

Observations of Microstructure Evolution and Damage of SAC305 Solder Alloys during Thermal and Mechanical Loadings

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Declaration of Originality

This thesis is submitted for the degree of Doctor of Philosophy. The research work described herein was carried out by myself in the period of October 2015 to December 2018, under the supervision of Dr T. Ben Britton and Dr Christopher M. Gourlay, the Department of Materials in Imperial College London.

I hereby declare that this thesis is the result of my own work and includes nothing which is the outcome of work done in collaboration except where specifically indicated in the text.

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December 2018

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Abstract

Creep of directionally solidified Sn-3Ag-0.5Cu wt.% dog-bone samples with a controlled $\langle 110 \rangle$ or $\langle 100 \rangle$ fibre texture is investigated under constant load tensile testing (stress level: 17 - 44 MPa) and at a range of temperatures (298 – 473 K). The tensile creep strain rate and the localised strain gradient are studied by two-dimensional optical digital image correlation. A transition of creep mechanisms from climb-controlled dislocation creep to lattice-limited diffusion creep is found beyond $\sim \frac{T}{T_M} = 0.7$ to 0.74 which changes depending on microstructure length-scale. The dominating creep mechanisms explored using electron backscatter diffraction, which enables the understanding of the heterogeneity in microstructural evolution at different strain levels, temperatures and strain rates. This heterogeneity is associated with grain boundaries and two distinct microstructural regions, i.e. the β -Sn in dendrites and eutectic containing Ag_3Sn and Cu_6Sn_5 . Formation of subgrains and recrystallisation are observed with increasing strain levels, and these correlate with local lattice rotation. Tensile deformation of grains with $[100]$ along the loading direction deform less than grains oriented with $[110]$ along the loading direction. For $[110]$ grains, the $(1\bar{1}0)[001]$ slip system activates. For $[100]$ grains, the $(110)[1\bar{1}1]/2$ and $(1\bar{1}0)[\bar{1}\bar{1}1]/2$ slip systems activate.

The heterogeneous evolution of microstructure for a single Cu/SAC305/Cu solder joint is investigated using in-situ thermal cycling. Local deformation due to thermal mismatch results in heterogeneous lattice rotation, localised towards the corners of the Cu metal substrate and decreases towards the centre of the solder joint. This deformation is induced due to the constraint from the coefficient of thermal expansion mismatch between the β -Sn, Cu_6Sn_5 and Cu at the interface. Formation of subgrains with continuous increase in misorientation is revealed during deformation, implying that the accumulation of plastic slip at the strain-localised regions play a significant role in grain recrystallisation and crack nucleation which likely leads to joint failure.

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Nomenclature

A: Area

BCT: Body centred tetragonal

BGA: Ball grid array

BSE: Backscatter electron

CTE: Coefficient of thermal expansion

DIC: Digital image correlation

DS: Directionally solidified

E: Young's modulus

EBSD: Electron backscatter diffraction

EDM: Electrical discharge machining

EDX: Energy dispersive X-ray

FIB: Focused ion beam

FSE: Forescatter electron

H: Hardness

HAGBs: High angle grain boundaries

HR-DIC: High spatial resolution digital image correlation

HR-EBSD: High angular resolution electron backscatter diffraction

IC: Integrated circuit

IMC: intermetallic compound

IPF: Inverse pole figure (EBSD)

L: Length

LD: Loading direction

LAGBs: Low angle grain boundaries

m: Schmid factor

Matlab: MATrix LABoatory

n: Stress exponent

ND: Normal direction

OPS: Colloidal silica

PCB: Printed circuit board

PECS: Polishing electron coating system

PLM: Polarised light microscopy
F: Force
PF: Pole figure (EBSD)
Q: Activation energy
R: ideal gas constant
RD: Rolling direction
ROI: Region of interest
RC: rolled and cut
SAC: Sn-Ag-Cu alloy
SDAS: Secondary dendrite arm spacing
SE: Secondary electron
SEM: Scanning electron microscope
STD: Standard deviation
t: time
T: Temperature
 T_M : Melting temperature
TD: Transverse direction
TEM: Transmission electron microscope
v: Growth velocity
 σ : Stress
 ϵ : Strain
 $\dot{\epsilon}$: Strain rate
 τ_{CRSS} : Critical resolved shear stress

Chapter 1 Introduction

In the transition to Pb-free electronics ^[1], Sn-3Ag-0.5Cu wt.% (SAC305) has become a widely-used commercial solder that can outperform Sn-37Pb joints in electronic interconnections ^[2] in electronic packaging, Integrated Circuit (IC) manufacturing and surface mount assembly of electronic components on printed circuit boards (PCB) within electronic devices (Figure 1.1).

However, the advantages of SAC305 over other compositions diminish as the severity of the thermal and mechanical cycle increases ^[2] and SAC305 often suffers from poor drop impact reliability ^[3, 4]. SAC305 is a Sn-based alloy, which contains more than 95% β -Sn phase. β -Sn has a body-centred tetragonal (BCT) structure and behaves anisotropically in thermophysical properties. For example, the coefficient of thermal expansion (CTE) is a factor of two times larger along the c-direction than along a=b and also c is about three times stiffer than a=b. Thus, SAC305 solder is particularly sensitive to thermal cycling and mechanical loading.

SAC305 is particularly sensitive to the creep deformation as it has a working temperature of 60-80% of its melting temperature (~ 490 K ^[5]), and creep occurs even at room temperature (298 K is $\frac{T}{T_M} \sim 0.6$).

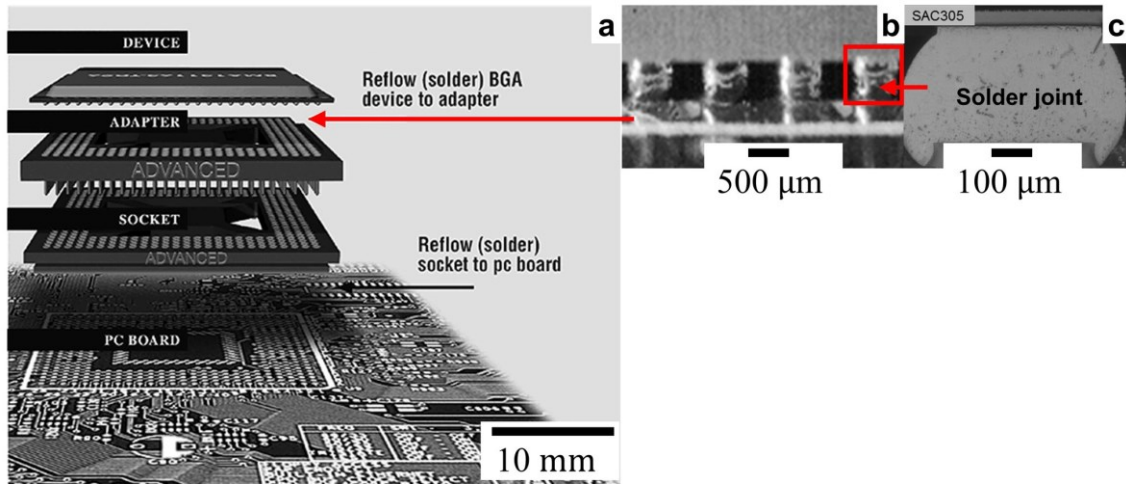


Figure 1.1. Electronic packaging with solder joint connection. (a) Ball-grid-arrays (BGA) solders in IC package assembly to PCB. (b) A layer of solder joints (c) Cross-section shows the microstructure of a solder joint ^[2].

In addition, SAC305 solder joints form a microstructure of β -Sn dendrites and β -Sn + IMC eutectic regions after solidification, which contains Ag_3Sn and Cu_6Sn_5 eutectic IMCs ^[6-8] (Figure 1.2a-1.2c). Each solder joint has different β -Sn crystal orientations and number of grains ^[7-11]. For SAC305, it normally has 1-3 β -Sn grains, which typically form single grain or cyclic twinned joints ^[12-15] (Figure 1.2d and 1.2e). Therefore, the role of IMCs in the eutectic, the behaviour of the two distinct microstructural regions (primary β -Sn dendrite and IMC-containing eutectic) and the crystal orientation of each solder sample need to be considered during the deformation process.

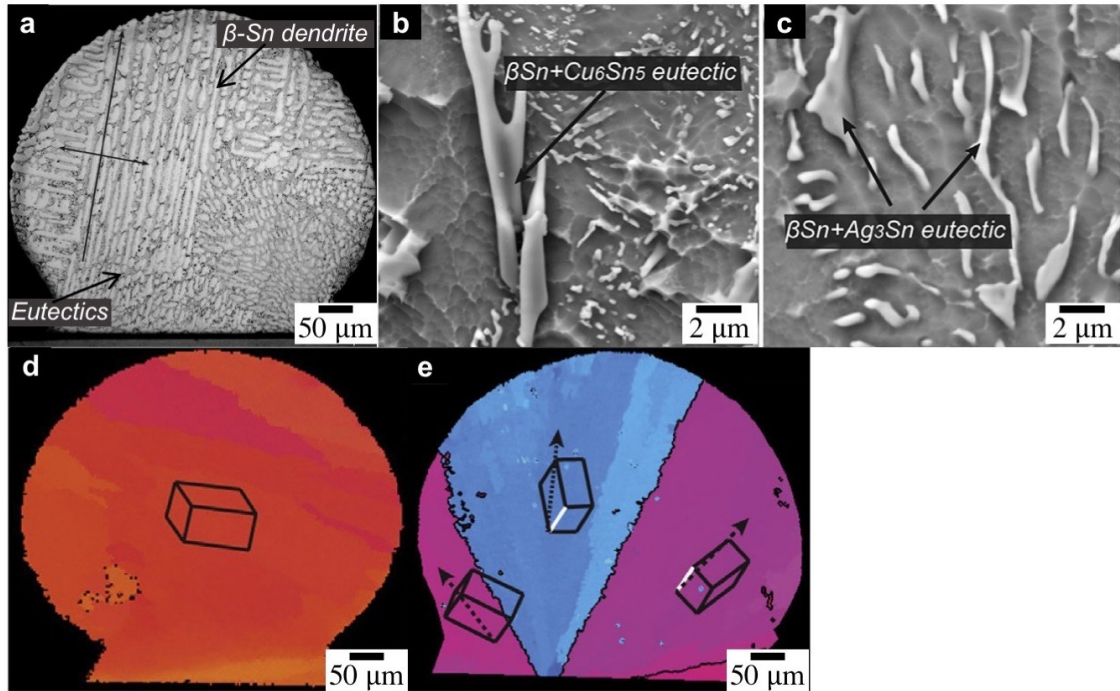


Figure 1.2. Micrographs of SAC305 solder joints modified from ref^[8]. (a) Optical image with β -Sn dendrite and eutectic region labelled, (b, c) the morphology of Cu_6Sn_5 (b), and Ag_3Sn (c). (d, e) EBSD maps of SAC305 solder joint, a single β -Sn grain joint (d), and a cyclic twinned β -Sn grain joint (e).

1.1 Aim and objectives

Based on the above introduction, there is a demand to better understand the behaviour of the SAC305 solder alloy, including the microstructural origin of deformation and failure to develop improved solder joints for high reliability applications.

The current project is focused on two main parts:

Part 1: The creep behaviour of the SAC305 solder. In this part, directional solidification (Bridgman growth) is applied to generate SAC305 bulk samples with reproducible β -Sn orientations and eutectic Ag_3Sn and Cu_6Sn_5 IMCs with reproducible size and spacing^[16, 17]. The creep behaviour of the controlled microstructures is investigated at a range of stresses and

Introduction

temperatures, and two different microstructural length-scales are studied at a constant stress at various temperatures.

Part 2: Thermal cycling of SAC305 solder joint. This part of the work is focused on the understanding for the microstructural deformation of ball grid array (BGA) scale SAC305 solder joints during thermal cycling. The localisation of plastic deformation is investigated with increasing number of thermal cycles and the effect of the microstructural features, such as the primary Cu_6Sn_5 IMC, eutectic Cu_6Sn_5 and Ag_3Sn and the interfacial layer of Cu_6Sn_5 on strain accumulation is studied.

The main questions to be addressed in the current project are the following:

1. Does heterogeneous deformation occur? Where does the heterogeneity occur?
2. Which microstructure is controlling the heterogeneity?
3. What is the influence of grain boundaries?
4. How does the microstructure evolve with increasing deformation?

1.2 Thesis structure

This thesis provides a detailed study of microstructural observations for SAC305 solder alloys under deformation from creep and thermal cycling.

After the introduction chapter, the related literature is reviewed in Chapter 2, which includes the fundamental properties and the deformation mechanisms of SAC305, together with the techniques used to characterise the microstructure. In the summary section, questions are addressed and answered later in the Results and Discussion in Chapter 4 - Chapter 9.

Introduction

Chapter 3 gives the methodology of sample fabrication and microstructural characterisation for creep tests. The samples are prepared by Bridgman growth, which creates samples with a few long columnar grains with strong directionality and reproducible crystallographic orientation of growth. The microstructure of the samples is characterised by scanning electron microscope (SEM) imaging and electron backscatter diffraction (EBSD), which analyses the phase and orientation information in detail.

Chapter 4 studies the effect of stress on creep behaviour of SAC305 solders at room temperature. The creep reliability for the SAC305 solders is investigated at a range of stress with the creep strain rate and the dominating creep mechanism is explored. EBSD is used to reveal the microstructural evolution, the effect of the two microstructural regions, and the role of intermetallic compounds (IMCs) during the creep deformation.

Chapter 5 investigates the effect of temperature on the creep behaviour of SAC305 solders at a constant stress. The dependency of temperature on creep reliability for the SAC305 solders is explored by exploring the creep strain rate and the dominating creep mechanism. The microstructural evolution and strain accumulation of the samples are determined by EBSD and two-dimensional digital image correlation (2D DIC) respectively, focusing on the transition between creep stages, i.e. the onset of secondary and tertiary stage creep. The effect of the presence of a grain boundary on heterogeneous deformation is also investigated.

Chapter 6 shows the effect of microstructure length-scale; i.e. the decrease in secondary dendrite arm spacing (SDAS), eutectic spacing and IMCs size on the creep behaviour and microstructural evolution of SAC305 solder at a constant stress and a range of temperatures.

Introduction

The influence on creep mechanism and strain accumulation are studied by utilising the same experimental set-up and analytical method as given in Chapter 5.

Chapter 7 solves the uncertain issues in the previous three chapters by carrying out in-situ creep test under a constant stress at room temperature. The dynamic evolution of the microstructure for the SAC305 solder is observed using foreshatter electron (FSE) imaging and EBSD during creep testing.

Chapter 8 explores the microstructural evolution of a single β -Sn grain Cu-SAC305-Cu BGA-like solder joint during in-situ thermal cycling. The heterogeneity in deformation and development of microstructure are observed by EBSD with increasing number of thermal cycles with a focus on the interfacial area.

Chapter 9 is an overall discussion from all results chapters, which overviews the plastic deformation mechanisms in SAC305 solders and solder joints.

Chapter 10 concludes the main results of this thesis and suggests potential future work.

1.3 Publications

Chapter 5 contains the works from the following published paper:

Gu, Tianhong, Christopher M. Gurlay, and T. Ben Britton. "Evaluating Creep Deformation in Controlled Microstructures of Sn-3Ag-0.5 Cu Solder." *Journal of Electronic Materials* (2018): 1-15.

Chapter 2 Literature review

2.1 Microstructure of SAC305 solder alloys

The SAC305 has a hypoeutectic composition and its microstructure consists of primary β -Sn dendrites and a eutectic region with Cu_6Sn_5 and Ag_3Sn embedded within the β -Sn matrix as shown in Figure 1.2a - 1.2c.

During the solidification of SAC305 (Figure 2.1), the primary IMCs, e.g. Cu_6Sn_5 and Ag_3Sn nucleate and grow in the course of solder cooling process for alloy addition (Ag and Cu) [18, 19]. With further cooling, the nucleation and growth of β -Sn dendrites takes place. In the final stage of solidification, the remaining liquid solidifies by eutectic reactions producing a Cu_6Sn_5 and Ag_3Sn IMC-containing eutectic between dendrite arms. The eutectic IMCs are much smaller than the primary IMC.

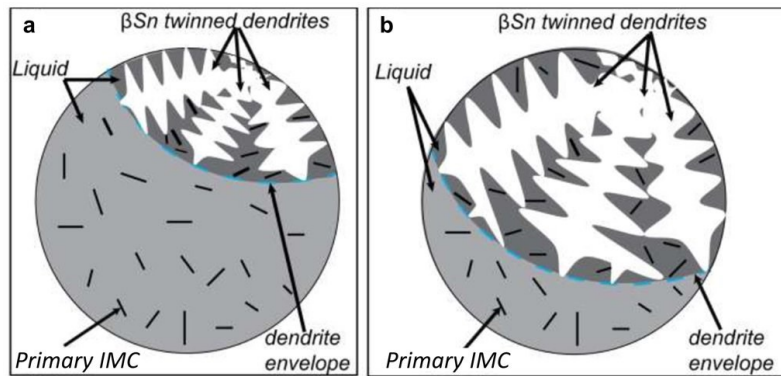


Figure 2.1. The schematic representation modified from ref^[20] showing the nucleation and growth of β -Sn in solder balls during solidification, (a) at early stage and (b) at later stage.

In the soldering of a joint, the liquid solder ball firstly reacts with the substrate (e.g. Cu) to form a layer of IMCs, e.g. Cu_6Sn_5 . The composition of the solder increases in Cu because of

Cu substrate dissolution. The β -Sn then nucleates near the IMC layer and spreads into the liquid volume to give the final microstructure of the solder joint ^[20, 21].

For β -Sn, the preferred dendrite orientation is $\langle 110 \rangle$ ^[22]. The lattice of the primary β -Sn dendrite is usually consistent with the β -Sn in the adjacent eutectic. Therefore, the grain orientation is determined by that of the initial dendrites at the nucleation stage.

2.1.1 Properties of β -Sn

β -Sn exhibits significant anisotropy in physical properties, such as mechanics and thermal expansion. The lattice parameters, coefficient of thermal expansion constants (CTE) and unit cell of β -Sn are shown in Table 2.1. The unit cell of β -Sn has its atoms positioned at (0 0 0), (0 0.5 0.25), (0.5 0.5 0.5), (0.5 0 0.75), producing four atoms per cell.

Lattice Parameters (nm) ^[23]			CTE ($\times 10^{-6} \text{ }^\circ\text{C}^{-1}$) ^[24]			Schematic diagram of β -Sn unit cell
a	b	c	a	b	c	
0.583	0.583	0.318	14.195	14.195	28.662	

Table 2.1. The lattice parameters, CTE and unit cell of β -Sn crystal at room temperature ^[23, 24].

Multiple studies in the literature have shown that SAC solders form a few Sn grains and normally have a single to triple grain structure ^[25-27]. Lehman et al. ^[28] observed single, cyclic twin (beach-ball) and interlaced twin structures in SAC solders, as shown in Figure 2.2, with composition such as Sn-3.0Ag-0.5Cu, Sn-3.9Ag-0.6Cu, Sn-3.0Ag-0.27Cu, and Sn-3.5Ag-0.5Cu, using polarised light microscopy (PLM).

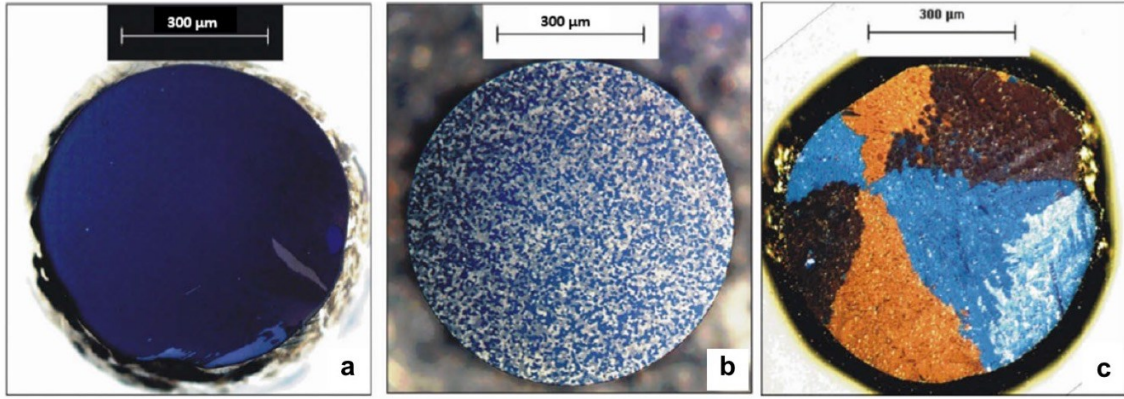


Figure 2.2. The Cross-polarised light micrographs of SAC solder balls with a near single β -Sn grain structure (a), interlaced twin grain structure (b), and cyclic twin grain structure (c) ^[18].

PLM has been used as a preliminary method to study the microstructures of SAC solders, which utilises the colour difference in individual crystal orientation, depending upon the birefringent properties of Sn (anisotropic material) because of the difference between two refractive indices to reveal the contrast of grains ^[29].

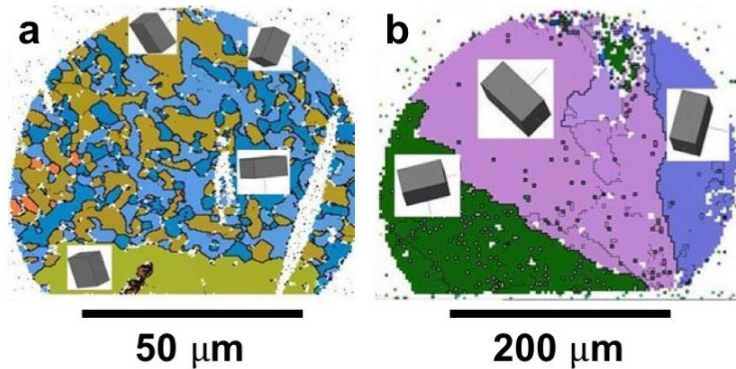


Figure 2.3. EBSD maps of SAC205 solder joint showing (a) an interlaced twin microstructure, (b) a cyclic twin grain microstructure ^[7].

The structure in Figure 2.3a and 2.3b shows six-fold twinned grains, which consist of three twin orientations on the $[100]$ axis by rotating 60° relative to each other ^[7]. These twins are nucleated and grown during the solidification. Lehman et al. ^[18] also reported that β -Sn has two twin systems, e.g. $\{101\}$ and $\{301\}$ as shown in Figure 2.4. The twinning angles are 57.2° and

62.8° for {101} and {301} planes respectively [18, 30]. Figure 2.3a shows an interlaced twinned solder ball, where all ‘grains’ have just three twinned orientations. Past work has shown that the interlaced twinned microstructure forms when there is a relatively large undercooling in solidification [7]. The cyclic twin solder ball in Figure 2.3b has the same three twinned orientations and forms at lower undercooling. In all cases in Figure 2.2 and Figure 2.3, there is only one nucleation event, which grows into a tri-crystal structure along the <110> directions through the entire solder ball [22].

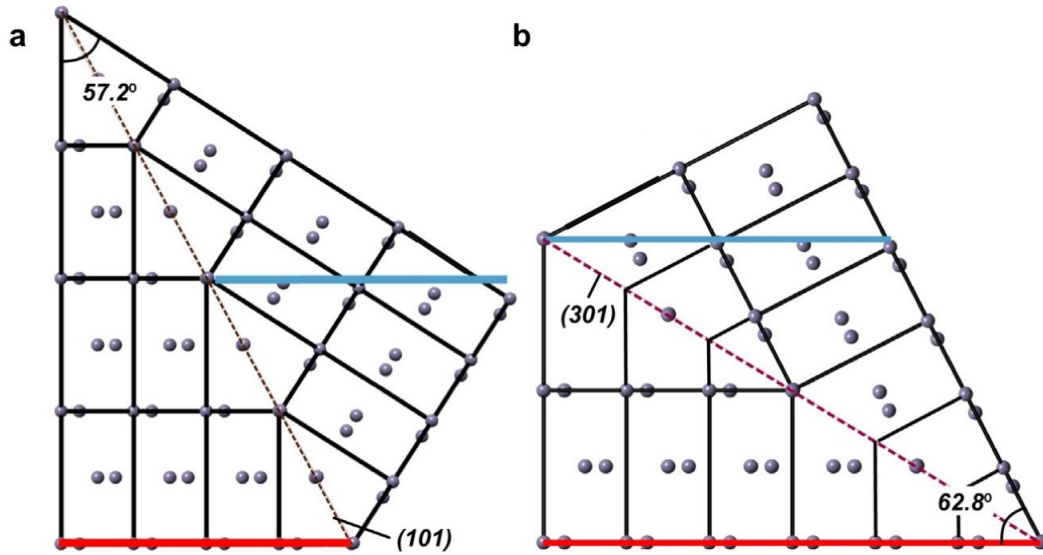


Figure 2.4. Projection view of β -Sn lattice on (010) plane showing (a) {101} and (b) {301} twin segments [30].

2.1.2 Role of intermetallic compounds

The IMCs form both within the solder matrix and at the interface of solder joint and affect the reliability of the solder alloys. The IMCs exhibit greater hardness and strength than β -Sn as given in Table 2.2. A reasonably fine IMC size and thin IMC layer can strengthen the solder bulk and joint and enhance both mechanical and thermal properties of it. However, a relatively

Literature review

large IMC size and thick IMC layer can increase the brittleness of the sample due to decreasing interfacial roughness ^[31, 32].

	β -Sn ^[33]		Cu ₆ Sn ₅ ^[34]		Ag ₃ Sn ^[35]
E (GPa)	26.3 ([100])	67.0 ([001])	155 ([0001])	133 ([11 $\bar{2}$ 0])	48.5
H (GPa)	0.01	0.04	8.2	6.7	1.1

Table 2.2. The Young's modulus (E) and hardness (H) values of the compositions in SAC305 e.g. β -Sn, Cu₆Sn₅ and Ag₃Sn determined by nanoindentation test ^[33-35]. The anisotropic properties Ag₃Sn is not considered in the according ref ^[35].

Ag₃Sn and Cu₆Sn₅ are the two IMCs formed in SAC305 solder during the soldering process (molten state) and grows continuously after solder solidification (solid state). The Ag₃Sn has an orthorhombic structure. The Cu₆Sn₅ has at least 4 polymorphs, namely, η , η' , η^6 and η^8 . The high temperature η -Cu₆Sn₅ forms a hexagonal unit cell ^[36] and all other polymorphs are superstructure ordered versions of η -Cu₆Sn₅.

It has been found that the Ag₃Sn particles in the solder matrix contribute to strengthen the sample by impeding dislocation movement ^[31, 37]. The Cu₆Sn₅ is reported to be much more brittle than the Ag₃Sn ^[35, 38].

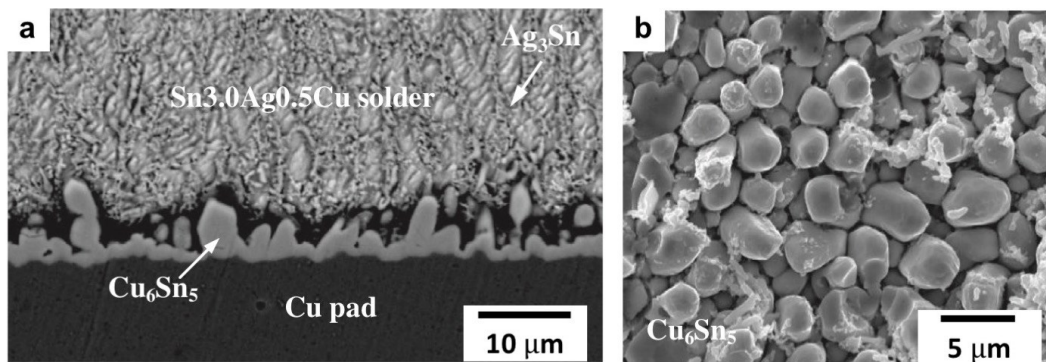


Figure 2.5. The SEM images of (a) SAC305/Cu interface, (b) morphology of Cu₆Sn₅ at interface ^[32].

In addition, Cu is the most widely used substrate for electronic soldering because of its good conductivity and good wetting of Sn (a substrate with high wettability of Sn can change the composition of the solder), so Cu_6Sn_5 constitutes an interfacial layer of IMC between the bulk β -Sn and Cu substrate (Figure 2.5). The Cu_6Sn_5 layer plays a crucial role in the composition, integrity and mechanical performance for SAC305/Cu solder joints, so it is vital to investigate the morphology and to understand damage effect of the interfacial IMC layer. The kinetics and microstructure of Cu_6Sn_5 layer growth have been intensively studied [31, 38-41] due to its importance on reliability at high shear rate. As this is not the focus of this thesis, Cu_6Sn_5 layer growth is not reviewed here.

2.2 Slip system and dislocation activities

Plastic deformation can occur via different slip system in the β -Sn matrix. Many literatures [42-53] have indicated observation of β -Sn slip systems, which are typically ranked in order of relative difficulty to activate. Table 2.3 illustrates a summary of the commonly observed 32 slip systems by Fujiwara et al. [52] and Zhou et al. [43] with their critical resolved shear stress (τ_{CRSS}) values calculated from by Zhou et al. [43], by considering of all three crystal orientations for single-crystal samples under uniaxial tensile test. Similar ranking was suggested by Matin et al. [47] but different exact values of τ_{CRSS} value were suggested. From Matin et al. [47] they select ‘consistent’ values of τ_{CRSS} for families, assuming a constant strain rate at 293 K and for family 1, 2, 9 and 10, these were extracted from experimental measurements (performed by others) from single-crystal experiments of pure β -Sn (99.99%) and family 3 - 5 are estimated values by correlating the slip systems with atomic line density (Peierls model based). Note that

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Matin et al. ^[47] also comment that τ_{CRSS} is a function of temperature, purity and dislocation density; and the temperature dependence implies that τ_{CRSS} may be strain rate sensitive.

Family		Slip system		τ_{CRSS} (MPa)	
1	{100}<001>	1	(100)[001]	1	1.9*
		2	(010)[001]		
2	{110}<001>	3	(110)[001]	1	1.3*
		4	($\bar{1}\bar{1}$ 0)[001]		
3	{100}<010>	5	(100)[010]	1.05	3.3*
		6	(010)[$\bar{1}$ 00]		
4	{110}< $\bar{1}\bar{1}$ >/2	7	(110)[$\bar{1}\bar{1}$]/2	1.1	2.3*
		8	(110)[$\bar{1}\bar{1}$]/2		
		9	($\bar{1}\bar{1}$ 0)[111]/2		
		10	($\bar{1}\bar{1}$ 0)[$\bar{1}\bar{1}$]/2		
5	{110}<0 $\bar{1}$ 0>	11	(110)[$\bar{1}\bar{1}$ 0]	1.2	-
		12	($\bar{1}\bar{1}$ 0)[110]		
6	{100}<011>	13	(010)[101]	1.25	3.8*
		14	(010)[$\bar{1}$ 0 $\bar{1}$]		
		15	(100)[011]		
		16	(100)[0 $\bar{1}\bar{1}$]		
7	{001}<100>	17	(001)[100]	1.3	-
		18	(001)[010]		
8	{001}<110>	19	(001)[110]	1.4	-
		20	(001)[$\bar{1}\bar{1}$ 0]		
9	{101}<10 $\bar{1}$ >	21	(101)[$\bar{1}$ 0 $\bar{1}$]	1.5	1.6*
		22	($\bar{1}$ 01)[101]		
		23	(011)[0 $\bar{1}\bar{1}$]		
		24	(0 $\bar{1}\bar{1}$)[011]		
10	{121}<10 $\bar{1}$ >	25	(121)[$\bar{1}$ 01]	1.5	1.7*
		26	($\bar{1}\bar{2}$ 1)[$\bar{1}$ 01]		
		27	($\bar{1}\bar{2}$ 1)[101]		
		28	($\bar{1}$ 21)[101]		
		29	(211)[0 $\bar{1}\bar{1}$]		
		30	($\bar{2}\bar{1}$ 1)[0 $\bar{1}\bar{1}$]		
		31	($\bar{2}\bar{1}$ 1)[011]		
		32	(2 $\bar{1}$ 1)[011]		

Table 2.3. The common Sn slip systems ranked by Fujiwara et al. ^[52] The values of critical resolved shear stress (τ_{CRSS}) are estimated from by Zamiri et al. ^[53] and the *values are from Matin et al. ^[47].

Generally for the crystal to obey Schmid's Law, the slip system with maximum Schmid factor (m) and minimum τ_{CRSS} will be activated first, where this critical value of the resolved shear stress required for plastic deformation for an individual slip system is called the τ_{CRSS} .

However, there is not only very limited number of works supporting the used τ_{CRSS} values, but also lack of systematic study on the τ_{CRSS} values for β -Sn.

Figure 2.6 shows a cylindrical single crystal sample under uniaxial tension, illustrating the relationship between the tensile stress applied to the crystal and the shear stress acting along the slip direction. The critical resolved shear stress is given by:

$$\tau_{CRSS} = \frac{F \cos \lambda}{A / \cos \phi} = \frac{F}{A} \cos \phi \cos \lambda \quad (2.1)$$

Where F is the applied force along tensile axis and equals to σA , $\frac{A}{\cos \phi}$ is the area of slip plane, ϕ is the angle between the tensile axis and the slip plane normal and λ is the angle between the tensile axis and the slip direction. The Schmid factor (m) is referred as

$$m = \cos \phi \times \cos \lambda \quad (2.2)$$

The slip system with minimum τ_{CRSS} and maximum m will be activated first ^[47]. And the weighted Schmid factor (m_W) of the sample can be calculated as

$$m_W = \frac{m}{\tau_{CRSS}} \quad (2.3)$$

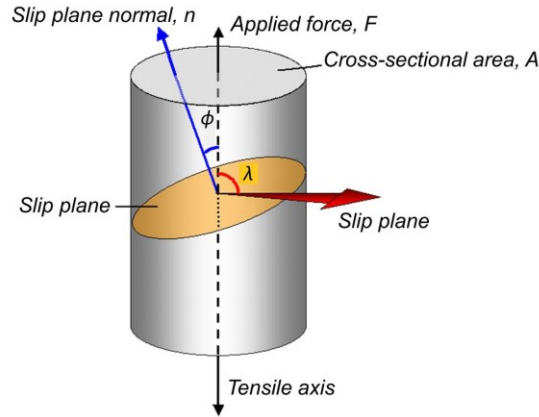


Figure 2.6. Schematic diagram modified from ref^[54] shows the Schmid's law.

According to the literature, the activity of slip systems for solder samples is typically identified using m and τ_{CRSS} values combined with slip trace observation. However, a large variation was obtained due to the influence of temperature history, composition, and orientation of the solder samples^[44, 47, 55].

Zhou et al.^[43], Bieler and Telang^[55], and Zuo et al.^[48] showed that the slip in the $[001]$ and $[111]$ directions on $\{100\}$ and $\{110\}$ type planes are the most active and are frequently observed for thermomechanical deformation, while activation in the slip of $[100]$ and $[101]$ directions were relatively hard^[43].

Bieler and Telang^[55] carried out a single shear lap test for 0.8 shear deformation at strain rate of 0.1 s^{-1} using an eutectic Sn-Ag/Cu solder to join two pieces of Cu half-dog-bone at 25°C . They concluded that the $(010)[101]$ slip system was activated and the slip systems in $\{101\}\langle 10\bar{1}\rangle$ and $\{121\}\langle 10\bar{1}\rangle$ families were very difficult to be activated for a sample with its $[001]$ direction near the loading direction.

Zuo et al.^[48] performed thermomechanical fatigue tests for SAC307 solder joints and discovered that the $(110)[1\bar{1}1]/2$ is the activated slip system. Meanwhile, the slip traces were

observed in the region with large Cu₆Sn₅, which resisted the development of low angle grain boundaries (LAGBs). In addition, it is also found that the τ_{CRSS} of the slip systems in #4 {110}<1 $\bar{1}$ 1>/2 family decreased exponentially with increasing temperature^[53] which means that high temperature was required to overcome certain energy barrier so as to initiate these slips.

However, Matin et al.^[46, 47] reported that the slip systems ($\bar{1}$ 10)[111]/2, ($\bar{1}$ 10)[001], (121)[$\bar{1}$ 01] and (121)[$\bar{1}$ 01] were activated for Sn-3.8Ag-0.7Cu bulk samples under mechanical^[47] and thermomechanical^[46] fatigue deformation. Slip traces and microcracks were observed predominantly in the eutectic regions. It is noted that the applied τ_{CRSS} values of slip family #9 and #10 by Matin et al.^[46, 47] were even lower than the highest ranking #1 {100}<001> slip family.

Furthermore, the activation of a slip system is largely dependent on the orientation of the particular solder joint^[26, 44, 51, 55]. Zhou et al.^[44] stated that the thermally cycled SAC305 solder joints had dominant red/orange orientations (c-axis is nearly parallel to chip-side interface) showing more slip activity than samples with green/blue orientations (c-axis is perpendicular to chip-side interface). They also reported that the recrystallisation generating new grains was caused by gradual lattice rotation, which was observed around Sn [110] axis and formed as a result of the easier activation of slip systems in the #2 {110}<001> family.

By carrying out uniaxial tensile tests, Kinoshita et al.^[51] and Dong et al.^[50] found that samples with orientation close to [001] had slip in the (101) plane activated easily for pure β -Sn and SAC305 respectively. For the [100] and [110] orientations, the (110) and (100) + (010) planes are less resistant to slip^[51].

2.3 Solder deformation during creep testing

Creep is time-dependent plasticity under a fixed stress at an elevated temperature, normally studied at a sufficiently high homologous temperature that a measurable amount of creep strain occurs within a reasonable time scale^[56].

2.3.1 General creep mechanisms

Solder samples susceptible to creep would be permanently deformed under stress below the yield point and have significantly reduced service lifetime. Therefore, creep properties are an important material selection parameter for structural design, especially in high temperature applications.

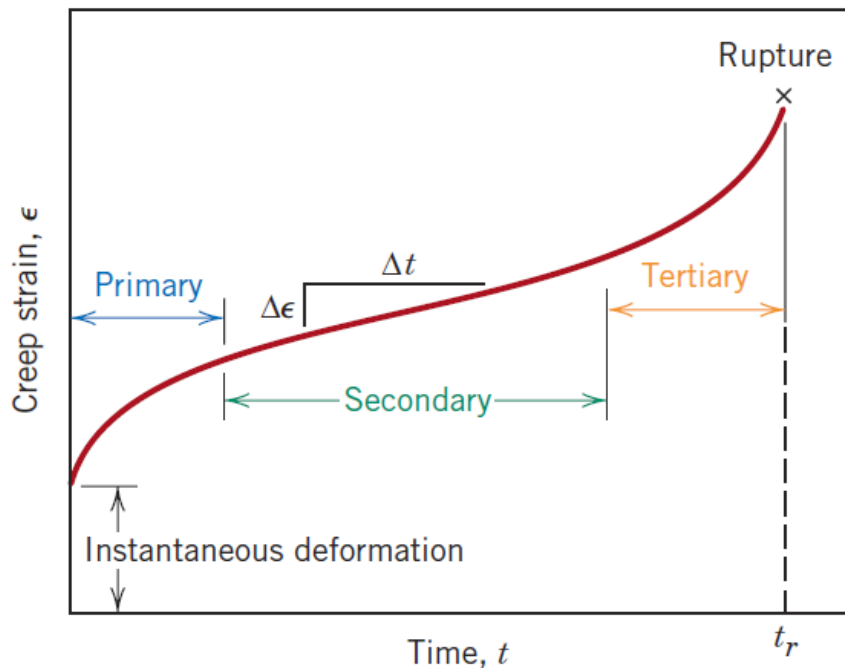


Figure 2.7. A typical creep curve^[56].

Figure 2.7 is a typical creep strain versus time curve showing three stages of creep, to which the slope at the steady stage of the curve is referred as the creep strain rate. In primary creep, the initial deformation takes place rapidly and the creep strain rate decreases because of strain

hardening, which occurs due to the dislocation multiplication during the plastic deformation. After that, the creep reaches secondary stage with constant creep strain rate, where the strain hardening and dynamic recovery compete with each other and allow dislocations to move through or around obstacles such as grain boundaries and IMCs, by cross slip, climb and thermally activated slip^[56]. Finally, in tertiary stage creep, the creep strain rate increases with significant microstructural damage accumulated. Effective reduction in cross-sectional area is often found due to necking and/or internal void formation^[37, 57-59].

The typically experimental stress-strain curves of reflowed SAC305 solders undergoing uniaxial tensile testing are illustrated in Figure 2.8 (Basit et al.^[60]). Figure 2.8a shows the data of four samples measured at different temperatures with constant strain rate and Figure 2.8b shows samples with constant temperature but different strain rate. It can be seen that the value of stress (flow stress) increases as the strain rate increases or temperature decreases. The strain rate determines the rate of dislocation generation and temperature determines the rate of dislocation removal.

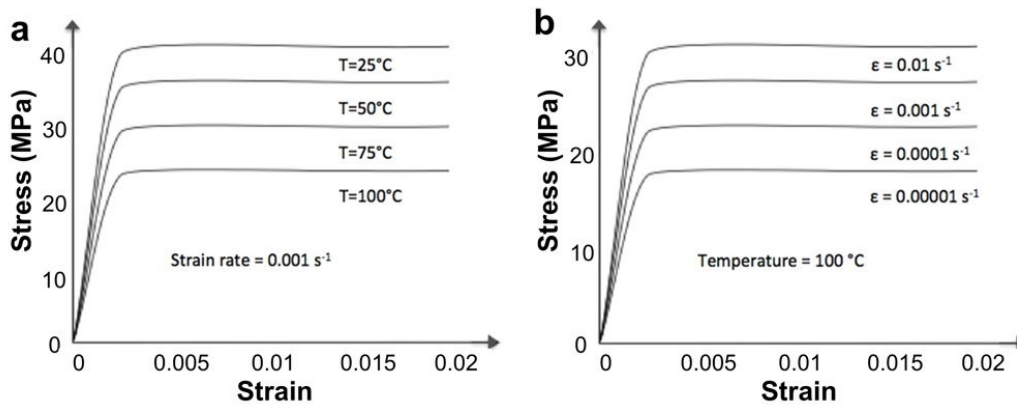


Figure 2.8. Reflowed stress-strain curves of SAC305 in uniaxial tension. a) with constant strain rate, b) with constant temperature replot from Basit et al.^[60].

Therefore, the understanding of the creep behaviour of SAC305 solder alloys was normally focused on three approaches, i.e. (1) loading condition ^[57, 61], (2) temperature range ^[62, 63] and (3) sample microstructure ^[64, 65].

The dominating creep mechanism is dependent on the stress and temperature, and is strain rate sensitive ^[56]. Figure 2.10 is a typical deformation map for Sn-37wt%Pb eutectic solder with the dominating creep mechanism shown for different regions of stress and temperature.

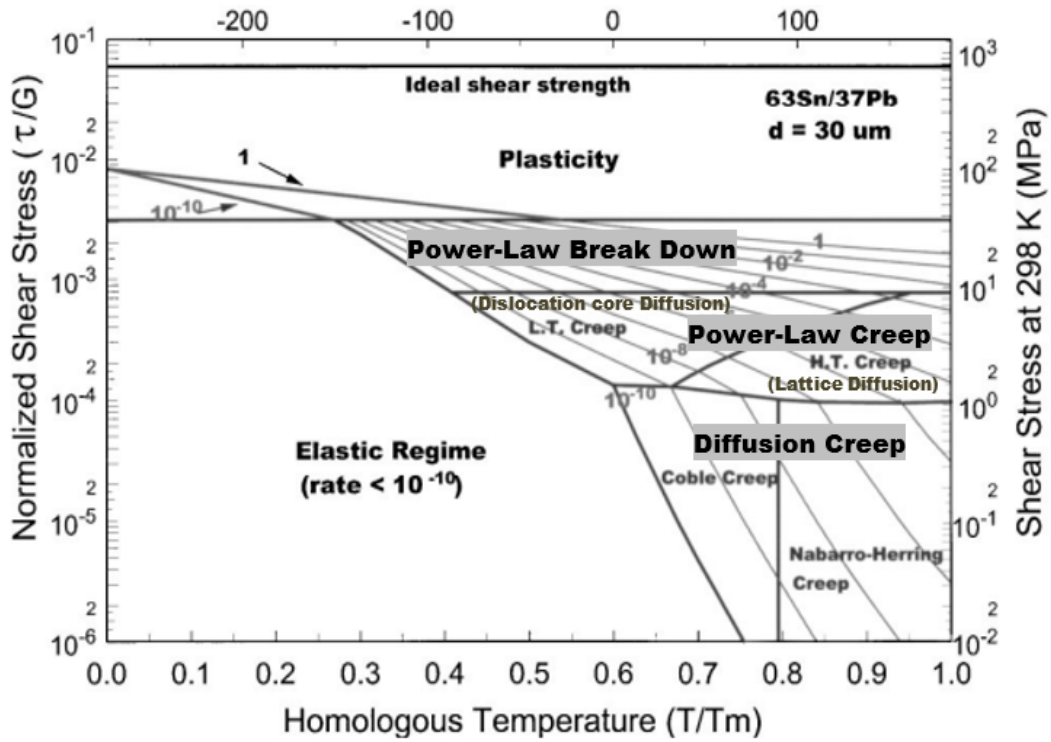


Figure 2.9. A typical deformation map for Sn-37wt%Pb with creep strain rates (s^{-1}) labelled on iso-lines, indicating the stress - temperature regimes with dominating deformation mechanisms, adapted from ref ^[66].

The stress exponent (n) and activation energy (Q) of creep can be used to distinguish creep mechanisms, i.e. between dislocation creep and diffusional creep, which can be determined from testing at variable stresses and temperatures in Sn-Ag-Cu, Sn-Ag and Sn-Cu solders in both bulk ^[57, 59, 62-65, 67-70] and joint form ^[37, 61-63, 71-80].

The creep strain rate, $\dot{\epsilon}$ is given as a function of temperature (T) and stress (σ).

$$\dot{\epsilon} = A\sigma^n \cdot \exp\left(-\frac{Q}{RT}\right) \quad (2.4)$$

Where R is the ideal gas constant of 8.314 J·Kmol⁻¹ and A is a constant.

$$\ln \dot{\epsilon} = \ln A + n \ln \sigma - \frac{Q}{RT} \quad (2.5)$$

By rearranging Equation (2.4), n and Q can be measured through the slope of $\ln(\dot{\epsilon})$ vs. $\ln(\sigma)$ and $\ln(\dot{\epsilon})$ vs. $1/T$ plot respectively.

The shape evolution of the sample can also be used to determine the difference between creep mechanisms. For samples, undergoing diffusion dominated creep, the elongation is fairly homogenous though the length-scale, while the dislocation dominated creep shows a more localised reduction in cross-section with a sharp neck at fracture^[56].

The disagreements in creep mechanism may be justified by differences in stress and temperature ranges, testing methods and sample preparation methods. By using transmission electron microscopy (TEM), Sidhu et al.^[62, 63] have concluded that, for several bulk and joint Sn-rich SAC alloys, climb-controlled dislocation creep is the dominating deformation mechanism and the deformation is controlled by subgrain formation from 25 to 130 °C. The value of n decreases with increasing temperature and the size of the subgrain is controlled by the IMC particles (Ag₃Sn and Cu₆Sn₅) in the bulk. Lin et al.^[57] suggested dislocation creep as the dominating mechanism from room temperature to 90 °C using tensile testing of bulk Sn-3.5Ag samples. The samples underwent ductile failure and the creep resistance decreased with increasing temperature, and increased with larger IMCs volume^[57]. The same mechanism was

also suggested by Ogawa et al. ^[61] with indentation testing from -20 to 160 °C, by Chen et al. ^{a[58]} with in-situ tensile testing between 25 and 100 °C and by Kariya et al. ^[59] with tensile testing of miniature SAC305 samples at 25 and 75 °C.

2.3.2 Change in creep mechanisms

2.3.2.1 Effect of temperature

The change in creep mechanism at higher temperature ($T \geq 0.7 T_M$) from dislocation to diffusion creep, i.e. a decrease in stress exponent (n) with increasing temperature has been suggested by prior research ^[37, 57-59, 61-65, 68, 74, 79]. Vianco et al. ^[64, 65] indicated clearly that a change in creep mechanism for bulk solder at high and low temperatures using solder Sn-3.9Ag-0.6Cu solder alloys. In the low temperature regime (-25 to 75 °C), dislocations act as obstacles to reduce diffusion path, which is perhaps the result of a recovery or recrystallisation assisted diffusion mechanism, while the high temperature regime (75 to 160 °C) predominantly occurred by lattice or bulk diffusion mechanisms. This change in mechanism is supported by Lee et al. ^[74] using Cu/Sn/Cu single-lap specimens, Mayer et al ^[81], who tested both pure Sn and Sn-Ag₃Sn eutectic with micropillar compression, and Mathew et al. ^[68] who showed good agreement between Sn-3.5Ag solder-bump array samples under shear loading and bulk samples through tensile testing between 20 to 150 °C, as well as Talebanpour et al. ^[79] using a cylinder indenter to carry out impression creep from -55 to 120 °C.

2.3.2.2 Effect of microstructure

Past work has shown that the creep behaviour of a given solder alloy is affected by the initial microstructure, which depends on the cooling rate during the solidification, i.e. the faster the cooling rate the smaller the dendrite arm spacing and eutectic IMC size and spacing ^[82-86].

Specifically, an increase in creep lifetime, secondary creep strain rate and ductility has been reported for samples cooled at a faster rate. Among them, Wu et al. ^[82] found that the creep life and creep strain rate of their tested Sn-3.5Ag dog-bone samples changed about 1.5 and 1-2 orders of magnitude respectively between fast and slow cooled samples. Less creep resistance and a reduction in ductility for Sn-Ag/Cu joints solidified at a slower rate was indicated by Yang et al. ^[83], who claimed it is caused by the brittle nature of the IMCs. Kim et al. ^[84] reported that, under tensile testing, dog-bone SAC305, Sn-3.5Ag-0.7Cu and Sn-3.9Ag-0.6Cu samples at faster cooling rate have a greater elongation and ductility with increasing size of Ag₃Sn. Moreover, Ochoa et al. ^[85] observed a slight decrease of the activation energy, Q from 40 to 35 kJ/mol by increasing the cooling rate from 0.08 to 0.5 °C/s for Sn-3.5Ag bulk solder.

There has been a large amount of work investigating the effect of aging, i.e. the coarsening of IMCs, on creep behaviour of solder alloys ^[64, 65, 87-91]. Overall, the creep strain rate increases with larger IMC size. Dutta et al. ^[87] studied Sn-3.5Ag and Sn-4Ag-0.4Cu and concluded that creep is controlled by dislocation climb and the rate is dependent on the eutectic spacing. For samples with larger IMCs, local dislocation climb takes place at the IMC and β -Sn interface. At constant stress and temperature, Talebanpour and Kumar ^[88] suggested that the creep strain rate of solder alloys is proportional to the IMC size and eutectic spacing. By comparing aged and rolled and cut 95.5Sn-3.9Ag-0.6Cu samples, Vianco et al. ^[64, 65] found a decrease in both stress exponent (n) and activation energy (Q) from aged to rolled and cut samples for temperatures in the range -25 – 160 °C. However, Basit et al. ^[91] and Talebanpour et al. ^[79] found no change in Q as the aging time and temperature increased for pure Sn, SAC105 and SAC305 solders tested between -25 and 125 °C.

2.3.2.3 In-situ tests

A limited amount of work has studied microstructural evolution of the solder during the in-situ creep deformation [77, 78, 92-96]. The main observations are an increase in surface roughness [92, 94], formation of shear bands [94, 95], growth of subgrains [77, 78, 93-96] and dynamic recrystallisation [94, 95] with continuous straining. Tian et al. [95] also reported that the creep deformation of each sample varies due to the different grain orientations (anisotropic property of β -Sn). Among them only Zhang et al. [77, 78, 96] has used in-situ EBSD to improve the understanding of dynamic deformation of Sn-Cu and Sn-Ag solder joints during creep tests. They found that at low strain rate of $1 \times 10^{-4} \text{ s}^{-1}$, a significant strain concentration is observed for Sn-Ag/Cu solder joint at the Sn-Cu₆Sn₅ interface with formation of shear bands, while a relatively uniform deformation is introduced with wave-like deformation bands formed for Sn-Ag/Cu solder joint. The grains deform showing lattice rotation, which depends on their orientation, leading to grain subdivision and grain boundary migration, however, for these tests recrystallisation is not observed [96].

However, the samples used in their works [77, 78, 96] have different orientations and number of grains, which will have a significant effect on the creep behaviour.

2.3.3 Failure mode

The solder interconnects are under ductile fracture after certain amount of plastic deformation, allowing the electronic parts to function [26]. As plastic deformation occurs, the stress and plastic strain concentrate within a region until fracture of the sample. Therefore, there is a large amount of energy absorbed before fracture. Generally, there are four steps of ductile fracture, namely, void formation, void coalescence (crack formation), crack propagation and fracture.

The propagation of a ductile crack involves substantially plastic flow and gives a rough fracture surface.

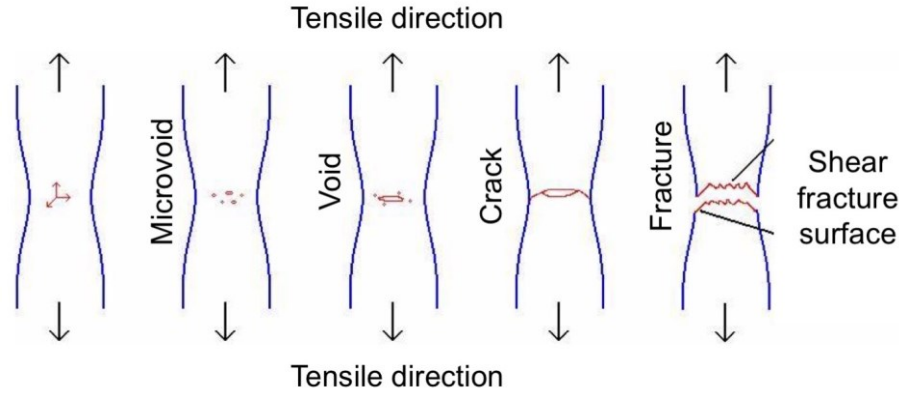


Figure 2.10. Mechanism of creep failure (a) necking, (b) void formation, (c) coalescence of voids to form crack, (d) crack propagation and fracture^[97].

The general ductile fracture mechanisms are shown in Figure 2.10 for solder in both bulk^[82, 98-100] and joint^[37, 75, 83, 101] form. Within a strain-localised region, necking initiates. Then microvoids develop inside the cross-section. As deformation continues, the microvoids enlarge and coalesce to form a long crack, which then propagates rapidly. The shear deformation finally proceeds at an angle of $\sim 45^\circ$ to the tensile axis to fracture of the sample^[97]. These were observed by Lin et al. with SAC305 and Sn-3.5Ag bulk samples, Xu et al.^[75] with SAC305 solder bulk, Kerr et al.^[101] using Sn-3.5 wt% Ag/Cu solder joints, and Mayer et al.^[81] using Sn-3.5Ag micropillar testing.

Choi et al.^[37] performed single shear lap tests for Sn-3.5Ag solder joints and suggested that intergranular fracture is usually observed as wedge-type cracks due to the void nucleation, which is correlated with the distribution of IMCs and grain boundaries. Transgranular fracture is associated with both necking and void nucleation and grows along the tensile direction, usually resulting in cup-cone failure, which was also reported by Ogawa et al.^[61] with

indentation testing on Sn-3.5Ag, Sn-0.7Cu and SAC305 solder bulk, and Kariya et al. ^[59] with tensile testing of miniature SAC305 samples with dimension of $0.5 \times 2 \text{ mm}^2$ for gauge cross-section.

2.4 Solder joint failure during thermal cycling

The anisotropic nature of β -Sn causes different mechanical responses for grains with different crystal orientations. The main concern reported from previous work relates to the microstructural evolution, i.e. polygonization and crack nucleation (Figure 2.11), which affects the reliability of solders ^[25, 26, 44, 102-104].

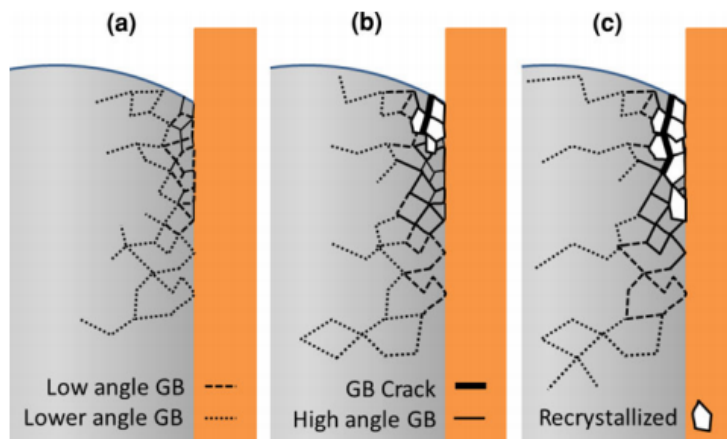


Figure 2.11. Schematic model for a single crystal solder joint at different stages of microstructural damage, (a) subgrain formation with high misorientations, (b) subgrain boundaries become mobile and initiation of recrystallisation, (c) Recrystallisation and crack formation with IMC coarsening ^[105].

2.4.1 Crack nucleation and propagation

The major failure mode of solder joints in thermal cycling is cracking after microstructure evolution in the bulk solder near the solder/substrate interface on the Si chip-side, which was observed in ref^[44, 102, 103, 106-109], as shown in Figure 2.12.

The crack propagates in the region where the mismatch of CTE is large and the induced thermal strain is high in comparison to the surroundings (larger misfit on Si chip side than PCB side of joint). The failure of the solder joints is caused by shear strain generated from anisotropic CTE between Si, solder and PCB in the normal direction of the joint^[110, 111] as shown in Figure 2.13.

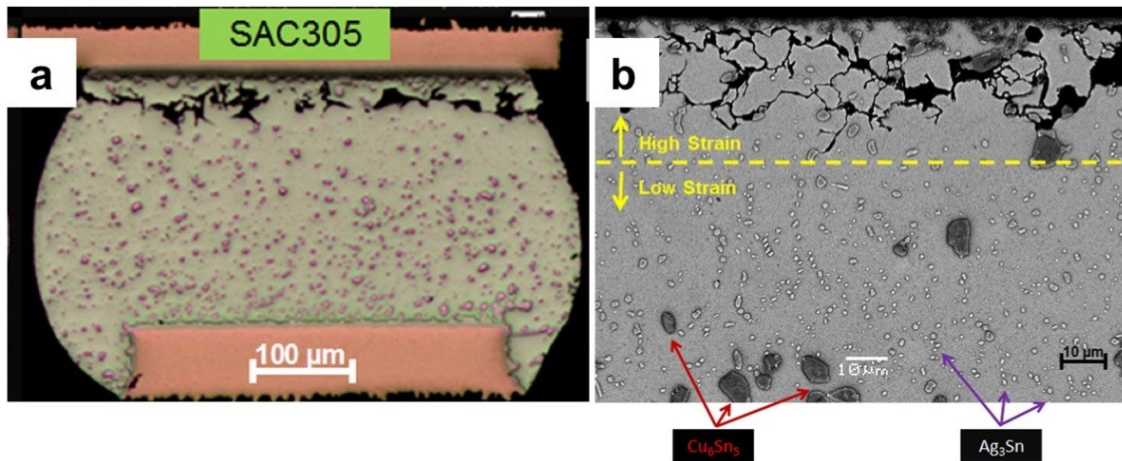


Figure 2.12. The micrographs of a failed SAC305 solder joint, (a) optical image with cracking near the interface, (b) EBSD map with recrystallisation shown around the crack^[2].

As the Cu_6Sn_5 layer thickens with increasing number of thermal cycles, failure can be induced near the solder/Cu interface because the Cu_6Sn_5 is extremely brittle and the different constituents have different CTE values. This varies the stress and strain build-up during thermomechanical loading near interface of the solder joint. George et al.^[112] studied the reliability of SAC305 and Sn3.5Ag solders during high-temperature cycling from -40 to 185°C with 15 minutes dwell time and $3.5^\circ\text{C}/\text{min.}$ ramp rate, and concluded that the failure

of solder occurred at the interface with maximum localised stress, where the Cu_6Sn_5 was observed and this was also caused by CTE mismatch between the three constituents.

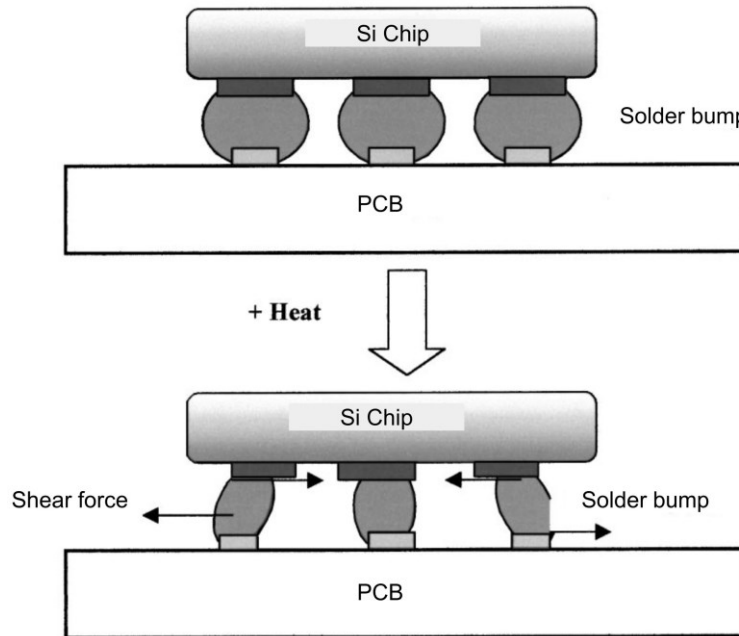


Figure 2.13. Solder joints subjected to shear strain during thermal cycling due to CTE mismatch between the Si chip, solder and PCB ^[111].

During thermal cycling, both intrinsic properties, i.e. the orientation difference between adjacent grains and defects in the solder matrix (flux voids and large primary IMCs) and extrinsic properties, i.e. the difference between constituents (solder / IMC layer / substrate) can induce the CTE mismatch, causing strain concentration at the interfacial area and localised plastic deformation ^[45]. The induced strains are mostly shear strains parallel to the interface plane of the solder joint ^[105]. Park et al. ^[25] reported that the elastic strain distribution in a solder joint was correlated with grain orientations, grain boundaries and primary IMCs under thermal cycling, because of the anisotropic behaviour of β -Sn. This resulted in plastic deformation and led to fatigue cracks along grain boundaries and near primary IMCs. The effect of the anisotropic CTE of β -Sn on thermomechanical fatigue of solder joints was also reported by Bieler et al. ^[26]. The grains with c-axis directed parallel to the substrate were damaged much

easier than when the a-axis and b-axis pointed parallel to the substrate during thermal cycling, which results in maximum CTE mismatch at solder joint interface. The CTE values of the commonly used constituents are shown in Table 2.4 and highly relate to orientation, which affects the performance of the solder joint. As the solder joint is thermally cycled, the CTE mismatch generates large thermal strain at the interface and results in damage in the solder joint near the substrate.

Axis	β -Sn ($^{\circ}\text{C}^{-1}$) [24]	η' -Cu ₆ Sn ₅ ($^{\circ}\text{C}^{-1}$) [24]	Cu ($^{\circ}\text{C}^{-1}$) [113]	Si [114]	PCB [115]
a	14.195	19.195	16.5	2.6	12-16
b	14.195	17.684	16.5	2.6	12-16
c	28.662	18.695	16.5	2.6	12-16

Table 2.4. The CTE values of the interface constituents. The values are determined using in-situ powder X-ray diffraction (PXRD) and calculated by fitting the PXRD data into corresponding crystal structure [24, 113-115]. A typical PCB material is a glass reinforced epoxy FR-4.

2.4.2 Recovery and recrystallisation processes

The heterogeneity of microstructure evolution has been extensively observed in the literature [26, 27, 40, 41, 102-104, 107, 116-119] for Pb-free solder joints during thermal cycling. When the strain level exceeds the limitation of elastic behaviour for the constituents, plastic deformation occurs and the stored energy in the crystal lattice is released by recovery, recrystallisation and grain growth [105].

In the situation for solder alloys (hot work), there could be sufficient thermal energy to activate all these processes even at room temperature. During deformation, dynamic recovery is reached, where the generation of new dislocations is balanced by the dislocation annihilation / rearrangement, i.e. the dislocation density is approximately constant. During recovery, the internal energy of the material (β -Sn) can be reduced by annihilation and rearrangement of

dislocations through climb and cross-slip that leads to the formation of sub-grain boundaries and LAGBs. The reduction of dislocation density occurs during the recovery process and the remaining dislocations pile-up to create LAGBs through rearrangement ^[54]. At elevated temperatures, the flow stress increases when the strain rate increases, and limits the ability of thermally activated mechanisms such as the unpinning dislocation structures and glide or thermally activated climb ^[60]. Therefore, it can be said that the dislocation density for dynamic recovery is strain rate and temperature dependent, where strain rate determines the rate of dislocation generation and temperature determines the rate of dislocation removal ^[40, 41, 103, 117].

During the recovery processes, the tested solder joints deformed by continuous orientation rotation, where the stored energy or the driving force can be released by forming subgrains with misorientation between 2 - 15° ^[44, 119, 120], as demonstrated in Figure 2.14. The surface roughness enhanced at the strain-localised region (Figure 2.14a) with formation of LAGBs, which is attributed to generation of dislocations and the operating slip systems can be analysed from the slip traces and the orientation of the solder sample.

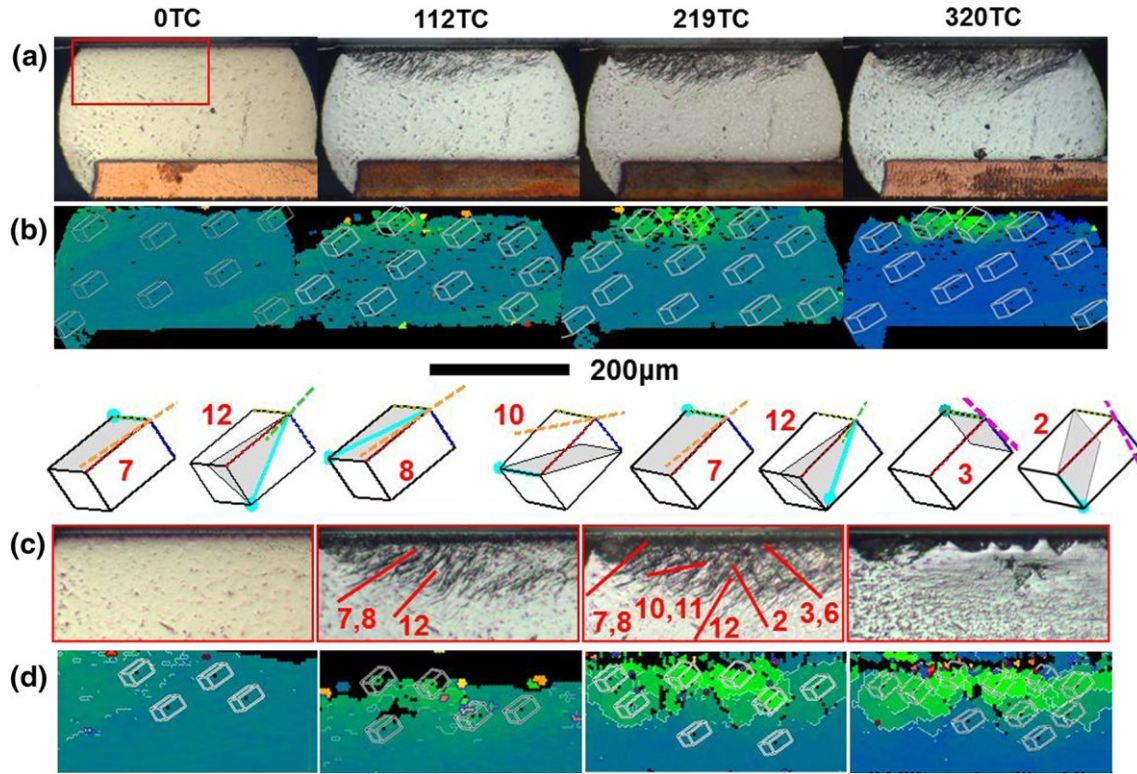


Figure 2.14. The micrographs of SAC305 solder joints at different number of thermal cycles, (a, b) the whole joint, (c, d) interfacial region at high magnification, (a, c) optical images, (b, d) EBSD maps. The calculated slip traces (red lines) are marked on (c) with the orientation unit cells given at high Schmid factors ^[44].

With increasing straining, the microstructure of the solder develops progressively and is often found to recrystallise, which is illustrated in Figure 2.14d. Continuous recrystallisation deforms the solder matrix by forming high angle grain boundaries (HAGBs) with enlarging misorientation (large green coloured recrystallised grains in Figure 2.14d). Recrystallisation can also take place discontinuously due to the presence of twin orientations and second phase particles (IMCs), which have higher dislocation densities than the surrounding grains and greater misorientation (rainbow-coloured small recrystallised grains in Figure 2.14d). This type of plastic deformation can result in grain boundary sliding and grain growth, providing an energy favourable site for cracking ^[26, 40, 41, 102, 107, 118].

In the work of Arfaei et al.^[107], the correlation of solder fatigue with both recrystallisation and crack growth were observed for SAC305 samples under two different thermal cycling profiles. In highly-strained regions, grain recrystallisation and crack propagation along the grain boundaries were observed. In addition, Bieler et al.^[103] studied how the anisotropic properties of β -Sn influence the microstructure and strain state of SAC305 solder joints with different thermal histories. The result demonstrated that the joint with higher strain stimulates recrystallisation faster because the recrystallised boundaries are high-energy boundaries and are easier for crack nucleation. Furthermore, Telang et al.^[108] reported continuous recrystallisation in a Sn-Ag joint during thermal cycling. Based on EBSD and 3D finite element modelling (FEM), newly recrystallised orientations grew to consume initial orientation(s) and the rate of recrystallisation was related to the elastic strain energy driving force. Moreover, the $\{110\}$ plane slip traces were generated with increasing amount of recrystallisation.

In addition, IMC coarsening has been reported by a considerable amount of works^[31, 35, 38, 39, 104, 106, 107]. The IMCs, namely, Ag_3Sn and Cu_6Sn_5 commonly formed in SAC305 solder joints act as nucleation sites for a new dislocation-free grains and can ease recrystallisation^[39]. The coarsening of IMCs often appeared in late stage of thermal cycling, meaning that it is a temperature and time dependent process^[31, 35, 38, 121]. In the recrystallisation region, less IMC particles and β -Sn dendrites were observed. According to Lee et al.^[104] the coarsening was related to crystal reorientation in highly-strained regions and corresponding to cumulatively discontinuous recrystallisation result from increasing strain energy, which causes grain boundary sliding and crack initiation.

2.5 Characterisation techniques

2.5.1 Electron backscatter diffraction (EBSD)

Since EBSD has been widely applied in studying Pb-free solder joints, an overview of this technique is given below in detail.

EBSD is an SEM based technique that allows the microstructural and crystallographic information of small volumes of crystalline material to be characterized using diffraction patterns. It is typically used in crystal orientation, grain texture, and crystal deformation analyses^[122, 123].

2.5.1.1 The set-up

Backscatter electrons (BSEs) are used as primary source for EBSD imaging. In order to achieve the best diffraction pattern and band contrast for EBSD imaging, the sample stage is tilted at a high angle of $\sim 70^\circ$ allowing the incident beam of electrons to focus on the sample surface at an angle of 20° . Electrons enter the sample, scatter, and some electrons may scatter with an escape angle that can cause electron diffraction. These diffracted electrons collide with the phosphor screen that is placed close to the sample to detect any electrons scattered between a $60\text{-}70^\circ$ capture angle. The EBSD detector set-up is depicted as schematic in Figure 2.15.

This collision causes the phosphor screen to fluoresce by forming the electron backscatter diffraction pattern (EBSP) and is captured by a sensitive charged couple device (CCD) camera. The quality of the diffraction pattern, i.e. the angular resolution of EBSP, can be improved by decreasing the capture angle, or increasing the screen distance, which leads to an increase in the signal-to-noise ratio of the image and decrease in the electron intensity of each pixel. Therefore, to measure the crystal orientation and identify a phase, a higher capture angle is

more suitable because the same number of pixels subtends to a larger angle ^[122, 123].

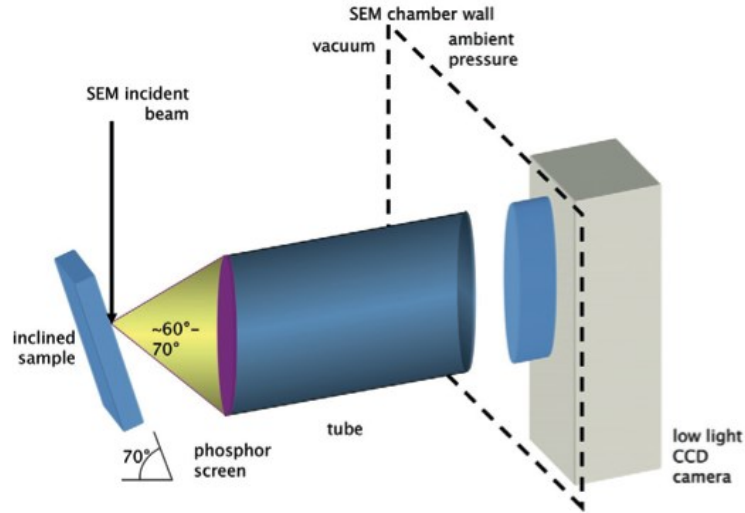


Figure 2.15. Schematic diagram of set-up for EBSD experiment ^[122].

2.5.1.2 Diffraction pattern

As the probe of electrons moves point by point across the sample, elastic collisions occur between the electrons and the sample. Electrons incident on the lattice planes of the sample diffract to form an EBSP. The incident angle of the electrons satisfies Bragg's Law.

$$n\lambda = 2d\sin\theta \quad (2.6)$$

where n is an integer, λ is the wavelength of the electrons, d is the spacing of the lattice plane, and θ is the incident angle of the electrons.

Bragg diffraction occurs when two coherent electron beams enter the lattice plane at angle θ and then are scattered by two atoms on different crystal planes with interplanar distance d . The two electron beams travel with a path difference of $2d\sin\theta$, so the two beams have same

wavelengths of different integer multiple and interfere each other constructively.

When the electron beam is ejected, it either hits or misses the Bragg angle of a lattice plane depending on the orientation of the crystal. Thus, the diffraction line (Kikuchi line) can be shown as dark or bright. As a set of electrons is inelastically scattered from the crystal, some of them are Bragg-scattered to give Kikuchi lines. During the crystal rotation, a different set of inelastically scattered electrons hits another lattice plane of the crystal [102, 123].

Therefore, the intensity of the EBSP is not uniform because of the constructive and destructive interference between the scattered beams and different crystal planes. The Kikuchi bands (diffraction bands) closer to the original incident beam have higher intensities and intensities decrease continuously with increasing angle from the original beam. Since the bands are reflected from different angles, the Bragg-scattered bands have different intensities to the background bands around them.

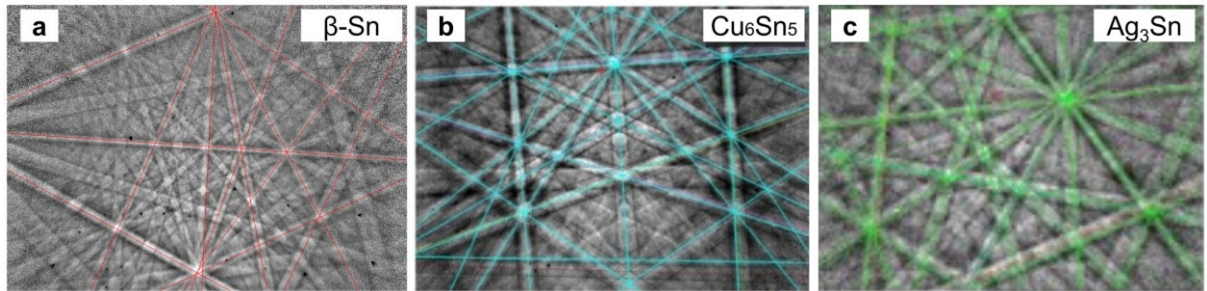


Figure 2.16. EBSPs of β -Sn, Cu_6Sn_5 and Ag_3Sn [20].

However, the Kikuchi bands are not straight lines but 3D cone shape, known as Kossel cones, as shown in Figure 2.17. They are observed when the cones intersect the plane of the detector and are shown as a set of pair nearly parallel lines. The cones of higher and lower intensities than their background are defined as ‘excess cone’ and ‘defect cone’ respectively [123, 124]. The two cones are separated by an angular distance 2θ with the plane of reflection in the middle.

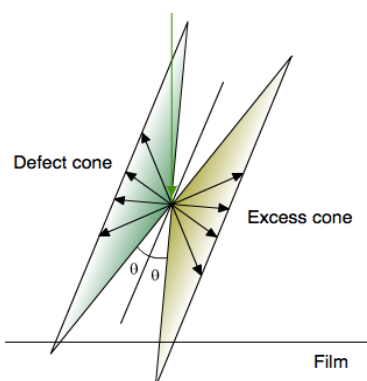


Figure 2.17. Schematic diagram of Kossel cones ^[124].

There is a number of factors able to affect the diffuseness and quality of the diffraction pattern. A diffuse or blurry pattern can be observed as a result of damage from poor sample preparation, contamination due to the inadequate storage, excessive deposit layer of coating, and/or defocusing of EBSD detector on the surface of sample. Furthermore, less crystalline perfection such as high density of point defects, dislocations, and lattice strains can introduce blur of diffraction pattern.

In practice, different parameters can be used to set-up EBSD scanning to achieve better pattern quality. For instance, a relatively large step size can be applied to measure the distribution of crystal orientations (overall texture) of grain. In the case of grain morphology, crack propagation and lattice damage, a smaller step size and longer exposure time are required for higher resolution EBSD ^[122, 125].

2.5.1.3 Indexing and measurement

To find out the crystal structure and lattice orientation from an EBSD, computer-assisted indexing and analysis can be performed rapidly in modern EBSD systems.

The Hough transform algorithm is commonly used to convert the Kikuchi pattern into Hough space in EBSD, i.e. transform the line information into points with knowledge of the

experimental geometry. With knowing the width and central position of some Kikuchi bands, it is possible to index the pattern without needing intensity measurements. The width of a Kikuchi band is correlated with the spacing of a crystal plane d and interplanar angle θ can be defined as

$$w \approx 2l\theta \approx \frac{nl\lambda}{d} \quad (2.7)$$

where l is the distance from the sample to the screen and n is an integer. Hence, planes with wider d -spacing have thinner Kikuchi bands and vice versa ^[122, 126].

The Hough transform takes a Cartesian straight line in form of $y = mx + C$, and transforms the Cartesian coordinates of diffraction pattern position (x, y) into polar coordinates (ρ, θ) in Hough space. The relation is given:

$$\rho = x\cos\theta + y\sin\theta \quad (2.8)$$

where ρ is the perpendicular distance from the origin to the line as $\rho = \sqrt{x^2 + y^2}$, θ is an angle between the line and the x -axis and the relationship between Cartesian and polar coordinates is shown as $(x, y) = (\rho\cos\theta, \rho\sin\theta)$. The polar equation can be rearranged into Cartesian form:

$$y = -x \frac{\cos\theta}{\sin\theta} + \frac{\rho}{\sin\theta} \quad (2.9)$$

Therefore, each point (x, y) in Cartesian space represents a unique sinusoid in Hough space, and on the other hand, every point in Hough space corresponds to an individual line in Cartesian

space. Meanwhile, the highest intensity occurs at the overlapping point of all sinusoid curves where each curve is produced by its corresponding point that exists in Cartesian space.

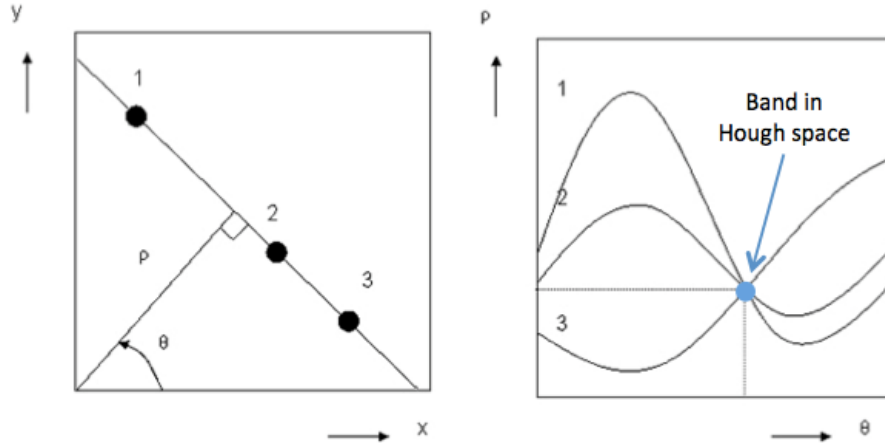


Figure 2.18. Hough transform transforms lines into points in Hough space^[127].

Hence, it is much easier to identify peak points in Hough space instead of locating lines and bands in the diffraction pattern as illustrated in Figure 2.18. In the case of a Kikuchi band, the Hough peaks are formed as ‘butterfly-shaped’ points^[128].

Once the peak intensity points on Hough transform are located, an iterative fitting algorithm is applied to determine the pattern centres. The approximate pattern centre coordinates and the corresponding crystal structure at interaction volume should be known. The EBSD is then indexed based on these approximate values and the theoretical band locations are compared to the detected ones using Hough transform.

Moreover, the interplanar spacing (width of the bands) and the interplanar angles (between the bands) can be calculated from only 3 - 4 families of $\{hkl\}$ crystal planes which are sufficient for most symmetrical crystals. Generally, only the most intense 5 -10 bands are indexed and the remaining bands are determined using symmetry operators of the crystal. These measured

values are then compared with the lookup tables with theoretical parameters obtained from a known crystal structure to index the crystal planes with Miller indices.

After indexing, the crystal orientation (absolute orientation accuracy: $\sim 2^\circ$ and 0.5° for misorientation ^[129]) of the grain can be easily described as Euler angles and rotation matrix.

The 3 Euler angles rotate about the standard basis axes of a coordinate system in 3D space and are denoted as $(\varphi_1, \varphi_2, \varphi_3)$ ^[130], when the rotation matrix performs the rotation from sample coordinate axes to crystal coordinate axes ^[131].

2.5.2 Digital image correlation (DIC)

The deformation at each step is recorded by a CCD camera and analysed by a computer program. A typical two-dimensional (2D) DIC setup is illustrated in Figure 2.19.

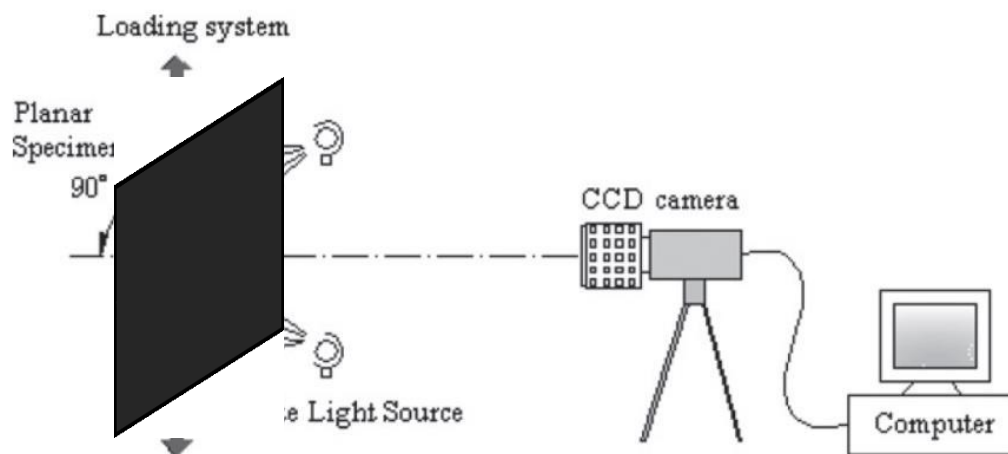


Figure 2.19. Typical set-up of optical image acquisition system for the 2D DIC.

2.5.2.1 Concept

Digital image correlation (DIC) is a non-contact optical measurement technique, which measures the surface deformation of a material by tracking the displacement changes in images.

This technique has been utilised broadly in mechanical tests to obtain the full-field strain map with increasing deformation ^[132].

In mapping the deformation, the region of interest (ROI) is divided into a number of subset windows, which make it easier to track the displacements within each image (rather than trying to track the motion of an individual pixel) ^[132]. The subsets are correlated by comparing image n with $n-1$, which measures the relative displacement change between the two successive images. Figure 2.20 shows the correlation between reference subset and deformed subset and their coordinates are (x_i, y_i) and (x'_i, y'_i) respectively. And the 2D deformation transformation can be described as

$$\begin{aligned} x' &= x + u + \frac{\partial u}{\partial x} \Delta x + \frac{\partial u}{\partial y} \Delta y \\ y' &= y + v + \frac{\partial v}{\partial x} \Delta x + \frac{\partial v}{\partial y} \Delta y \end{aligned} \quad (2.10)$$

Where u and v are the translations of displacement from the centre of the subset in x and y directions, Δx and Δy are the displacement from the centre to point (x, y) , and $\frac{\partial u}{\partial x}, \frac{\partial u}{\partial y}, \frac{\partial v}{\partial x}, \frac{\partial v}{\partial y}$ is the displacement gradients ^[132, 133].

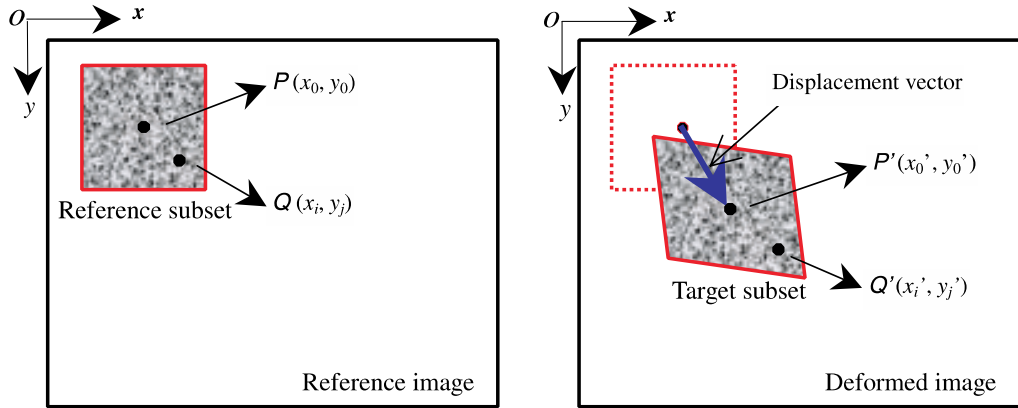


Figure 2.20. Schematic diagram shows a reference subset and a target subset after deformation within the same ROI ^[132].

2.5.2.2 DIC performance

The accuracy of the DIC measurement is also influenced by light resolution, alignment calibration, rigid body rotation, subset size, and speckle pattern.

A sufficient light source is needed during the image capture, which affects the image contrast. According to the scale of ROI, the 2D DIC can be carried out by optical microscope ^[134-136] or SEM ^[137, 138] to have a sensitive resolution.

The camera must be aligned parallel to the sample surface during the deformation because the magnification of the sample changes and leads to error in measurement of displacement.

The rigid body rotation causes excessive strain measurement and out-of-plane displacement as shown in Figure 2.21b, where some pixels have moved out of the reference subset (Figure 2.21a) and cause insufficient measurement ^[139]. Hung and Voloshin ^[140] reported that when the rigid body rotation is too large ($\geq 7^\circ$), displacement error is produced.

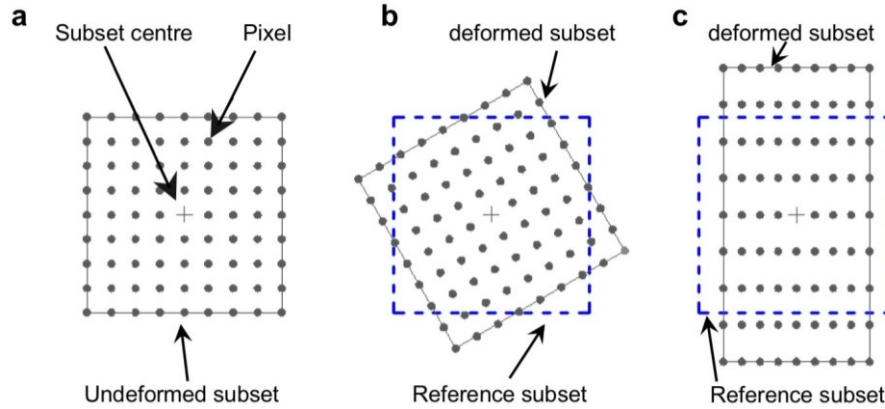


Figure 2.21. Schematic diagrams show (a) a reference subset, (b) a subset after rigid body rotation and (c) a subset after large deformation ^[132].

The subset determines the spatial resolution and precision of displacement calculated. Each subset must include at least 3 speckles. However, the minimum subset size cannot give a precise calculation and maximum subset size increases analytical time ^[141]. Therefore, there is a compromise between spatial resolution (subset size) and accuracy.

Speckle patterns are applied on the surface of the material to track the same pixels between successive images. The speckle patterns provide black and white contrast in intensity between different features with randomly homogenous distribution.

There are several methods to create speckle patterns, which must follow the surface during deformation and can be the natural or artificial patterns generated on the surface of the sample. The commonly natural patterns are the structural features of the sample, such as the morphology, phase and grain contrast, and can be revealed by etching and / or polishing. On the other hand, the artificial patterns are caused by depositing of layer of particles or producing defects. Moreover, tonner powder, airbrush and paint are often used for large scale sample (in mm), while in the case of sample in microscopic scale, atomic depositing and element etching (in nm) are usually used.

In order to image the deformation reliably, suitable subset and speckle sizes are required to provide enough number of features in a given subset of region, depending on the length-scale of ROI. For instance, Gioacchino et al. ^[138] used nano-scale gold layer as speckle patterns to map the plastic strain field of stainless steel, which underwent uniaxial tensile test with an effective pixel size of 0.036 $\mu\text{m}/\text{pixel}$. Wang et al. ^[135] observed strain distribution of the whole field (in mm) for a large-scale steel sheet during necking. The speckle patterns were made by powder spray with a ROI of 1 mm^2 . Vasco-olmo et al. ^[136] analysed the crack growth for pure grade 2 Ti under tension with a 13.5 $\mu\text{m}/\text{pixel}$ resolution obtained by airbrush. By using both high spatial resolution DIC (HR DIC) and high angular resolution EBSD (HR EBSD), the localisation of deformation was measured for a single-crystal Ni superalloy at room temperature by Jiang et al. ^[142]. The progressive development of slip systems was observed, showing as accumulation of plastic strain gradient.

2.6 Summary of literature review and what to investigate

In this chapter, a review has been carried out on the microstructure and performance of SAC305 Pb-free solder alloy for creep and thermal cycling deformation.

Since the EBSD and 2D DIC have been utilised in the current work and widely applied in studying Pb-free solders and in measuring deformation, an overview of the two techniques has been given in detail.

For the creep test, although the creep mechanisms have been studied by many researchers on the effect of various parameters, such as stress, temperature, grain size and IMC size, there is no systematic study on creep behaviour for SAC305 solder alloys with controlled microstructures (including crystallographic orientation). With controlled microstructure, more

Literature review

precise answers will be given for the effect of tested variables on deformation mechanisms and the determination of which slip systems are activated.

For thermal cycling tests, the literature has demonstrated the heterogeneity in plastic microstructure evolution for solder joints and the effect of anisotropic property of β -Sn and CTE mismatch at the solder / IMCs / substrate interface. However, there is limited in-situ EBSD evidence in support of these mechanisms. With in-situ thermal cycling and EBSD observation, the development of strain-localised region, lattice rotation and growth of subgrains and recrystallised grains will be understood.

Chapter 3 Sample fabrication and microstructure

3.1 Sample geometry and Bridgman growth

The alloy used throughout this thesis was Sn-3wt%Ag-0.5wt%Cu (SAC305), provided in ingot form by Nihon Superior Co., Ltd. The composition of this SAC305 batch was determined by X-ray fluorescence spectroscopy to be 2.92 wt%Ag and 0.586 wt%Cu with main impurities of 0.024 wt%Pb, 0.018 wt%Sb, 0.013 wt%Bi and minor impurities of Ni, Fe, Co, Zn each <0.006 wt%.

The first step of sample preparation was cold-rolling of a SAC305 bar to 1.5 mm thickness and cutting into a dog-bone shape using electrical discharge machining (EDM). A dog-bone shape was designed to confine creep deformation within narrow gauge region. Two geometries were used. For room temperature testing, the sample used had a 20 mm gauge length, 5mm width and 1.5 mm thickness (Figure 3.1a). For testing at elevated temperatures, the samples were cut into 10 (gauge length) $\times$ 2 $\times$ 1.5 mm size (Figure 3.1b), which can be perfectly fit into the Gatan Mtest 2000E tensile stage.

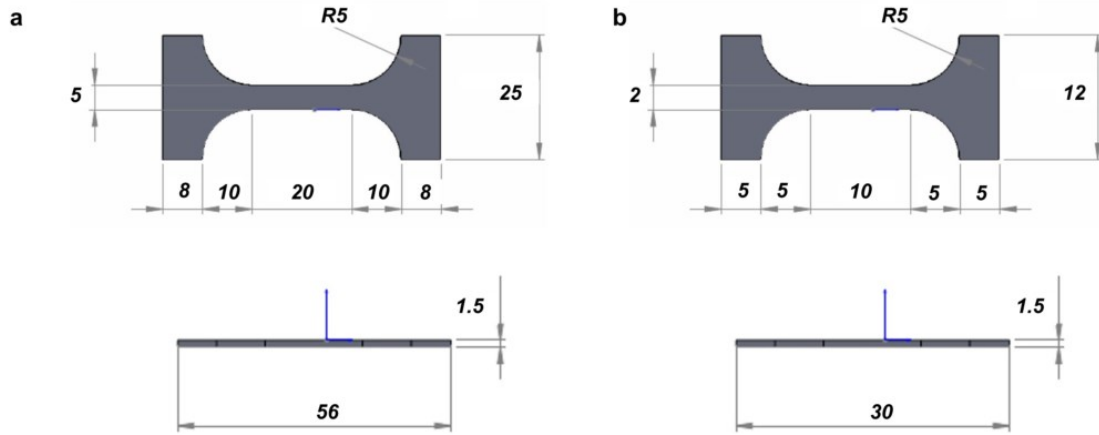


Figure 3.1. The dimensions of the SAC305 dog-bone sample used for creep testing (a) room temperature, (b) elevated temperature.

In the second step of sample preparation, controlled microstructures were grown in dog-bone samples by directional solidification (DS) using Bridgman growth, which provides control of the key parameters that determine microstructure formation during solidification: the thermal gradient, interface velocity and sample composition. This produced a directionally columnar microstructure and only 1-3 grains (a common misorientation angle with respect to ‘grain’ as identified with EBSD thresholding of grain boundaries at 5°) in the gauge length, which may enhance the thermomechanical performance of the material, especially creep resistance ^[143]. Note that in this thesis, I refer to a “grain” as a region of common orientation separated by a grain boundary. It may contain primary tin and eutectic. If the primary tin recrystallises, new grain boundaries may also be generated.

In this experiment, a Linkam TST 350 temperature controlled tensile testing stage was used as a temperature gradient furnace with a 350 °C hot zone and ~25 °C cold zone (Figure 3.2a), similar to that used in ref ^[144]. The Linkam stage has a nickel-plated heater with cooling block inside to operate at temperatures between -196 and 350 °C with 0.1 °C accuracy and the heating

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and cooling rates can vary from 0.01 to 30 °C/min. The position of the sample is fixed by a pair of stainless steel sample clamps at the jaws. The stage is able to open a distance from 0 to 77.00 ± 0.01 mm relative to the heater and apply pulling rates in the range 1-1000 $\mu\text{m/s}$.

The heater was set with a temperature of 350 °C and, once this temperature was reached, the sample was held for 20 minutes to ensure the temperature was stabilised, i.e. the sample and the heater reached thermal equilibrium. One of two pulling speeds was applied: 40 or 400 $\mu\text{m/s}$ but, because the two jaws were set to open in opposite directions, the actual solid-liquid (S-L) interfacial growth rate was half of the set pulling rate. The samples were pulled through a distance of 26 mm (Figure 3.2b), which was sufficient for the liquidus and solidus isotherms to grow along the entire gauge length (and is the maximum pulling distance for the stage). Once the maximum distance was reached, the heater was stopped and the sample was left in the pot to cool naturally in air. All samples in this study were fabricated under the same Bridgman growth conditions.

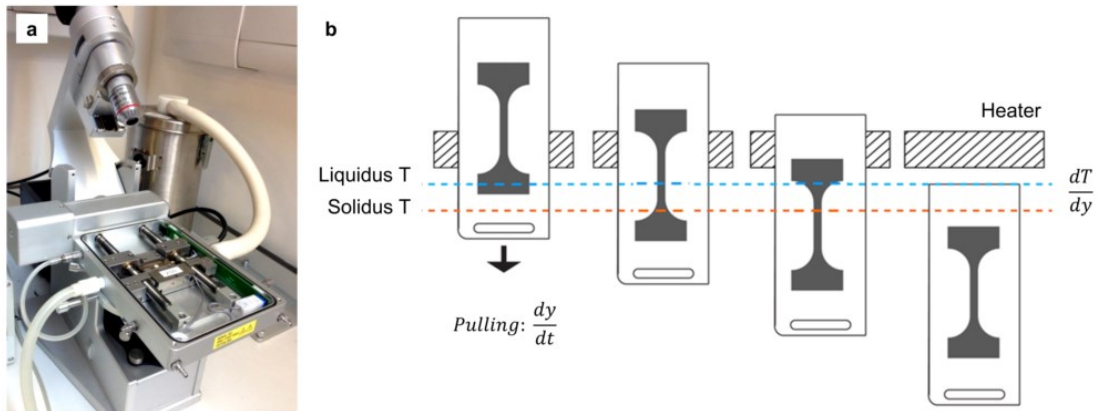


Figure 3.2. The Linkam TST350 Temperature control stage, (a) The overall view of Linkam stage, (b) The schematic of the sample from its initial position ($y=0$ mm) to its final position ($y=77$ mm) in distance. The orange dashed line shows the solidus line and the blue dashed line is the liquidus line.

The Linksys 32 software was used to record the temperature, position and force data, which are plotted in Figure 3.3 below. Figure 3.3a shows the set temperature and force versus time graph and Figure 3.3b gives the relationship between single jaw position and force with time for the 20 $\mu\text{m/s}$ sample. The constant growth region is highlighted as a green dashed line in Figure 3.3a and 3.3b, the measured force is $0.14 \pm 0.02 \text{ N}$ ($\mu \pm \sigma$) (max. force of the rig 20 N) and it can be seen that from Figure 3.3c this force is relatively stable within the growth distance $\sim 26 \text{ mm}$.

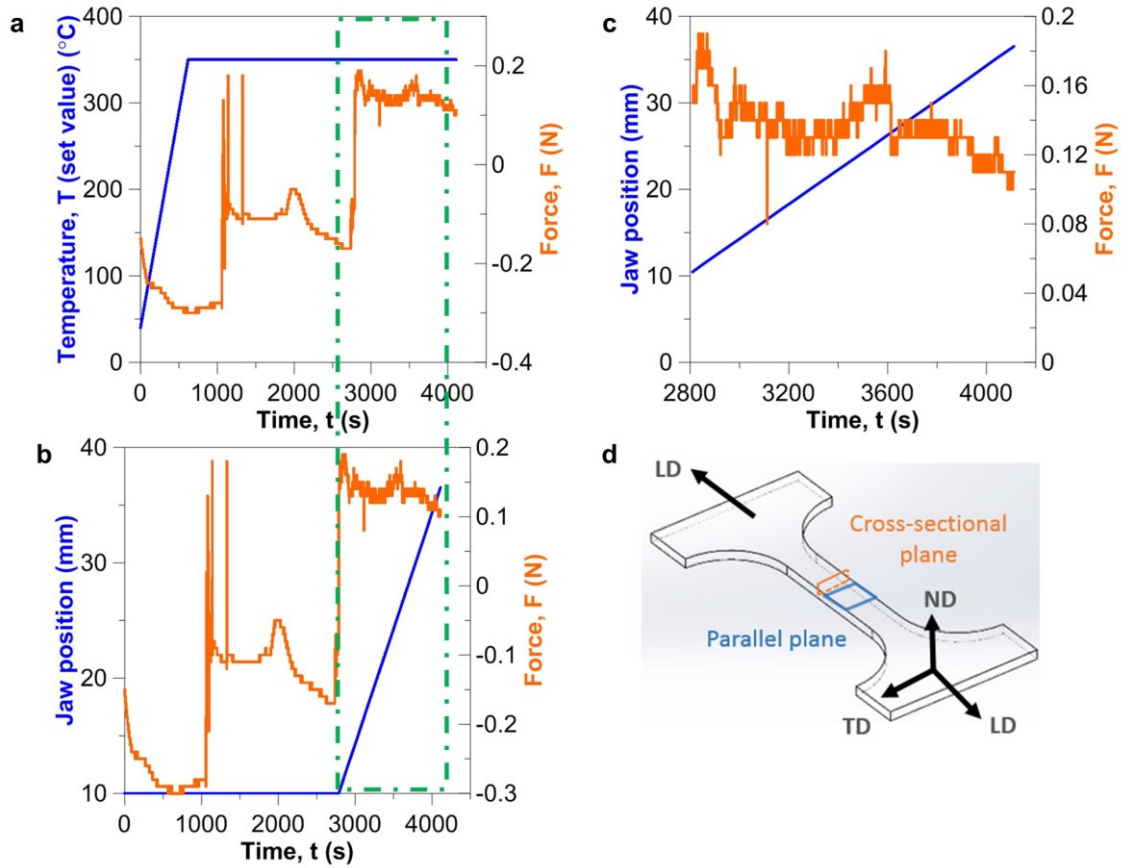


Figure 3.3. The plots drawn according to the data recorded by Linkam 32 software for the samples with a pulling speed of $v = 20 \mu\text{m/s}$. (a) Temperature and force vs. time graph. (b) The single jaw moving position and force vs. time graph, (c) The zoom-in graph of (b) on the constant growth region highlighted with green dashed lines in (a) and (b). (d) Schematic of the creep testing specimen

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showing the creep loading direction (LD) is the same as the DS pulling direction) in relation to the longitudinal plane and transverse direction (TD) and normal direction (ND).

The rolled and cut sample was fixed at one end and the single jaw was set to move at 20 or 200 $\mu\text{m/s}$ through the constant horizontal thermal gradient, $\frac{dT}{dy}$. These 2 growth rates (20 and 200 $\mu\text{m/s}$) were chosen to generate two reproducible microstructures, each containing β -Sn dendrites and β -Sn- Ag_3Sn - Cu_6Sn_5 eutectic at two significantly different microstructural length-scales. After growth, the microstructure of the sample was examined in both longitudinal and transverse section in relation to the pulling direction from the sample cross-section, as shown in Figure 3.3d.

During Bridgman growth, the shape of the sample is kept by placing it inside a designed mild steel sample holder and folded with a thin layer of polyimide sheet ($\sim 50 \mu\text{m}$), which gives a relatively good thermal conductivity between the sample and the heater. A piece of glass sheet ($\sim 5 \text{ mm}$) was placed on the top for achieving a flat sample surface. Before DS, the sample, the sample holder and the polyimide folder were cleaned with ethanol to avoid contamination.

3.2 Thermal gradient measurement

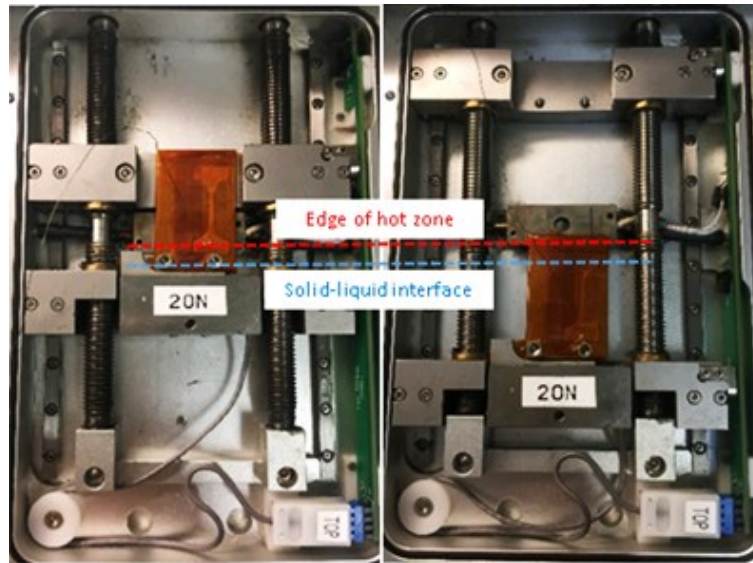


Figure 3.4. The image gives the initial sample position when pulling distance, $y=0$ mm and the final position of sample when $y=77$ mm shown in Linkam system. The red dashed line shows the edge of the heater and the blue dashed line shows the solid-liquid (S-L) interface of the sample.

In order to measure the thermal gradient ahead of the S-L interface, a 0.25 mm diameter K-type thermocouple wire was attached to the surface of the sample and located at the bottom edge of the heater (Figure 3.4), and temperature was recorded using a Pico-Log TC-08 recorder. The temperature (T) vs. pulling distance (y) data was plotted as shown in Figure 3.5.

The thermal profile is complex above 240 °C for the sample designed for room temperature creep testing (Figure 3.1a). The dip at high temperature (Figure 3.5a) is probably due to a viewing hole in the centre of the heater plate (Figure 3.4). However, the thermal field above 230 °C is fully liquid and is not expected to affect the solidification process (the liquidus temperature for SAC305 is 217 °C [5]). Below 230 °C, the thermal gradient is near linear with a ‘kink’ associated with the solidification front releasing latent heat and the difference in thermal conductivity between the solid and liquid phases (Figure 3.5b).

Figure 3.5c illustrates the temperature profile for the sample designed for elevated temperature creep testing (Figure 3.1b). It forms a relatively smooth curve during growth because the gauge of the sample is not being pulled through the hole of the heater (Figure 3.4). In the range 230-210 °C, the temperature gradient, $\frac{dT}{dy}$ at the β -Sn and eutectic S-L interface are found by fitting straight (red) trend lines to the temperature against jaw position curves (Figure 3.5b and 3.5d). The temperature gradient, $\frac{dT}{dy}$ is ~ 2.7 K/mm and 4.2 K/mm respectively for $v = 20$ and 200 $\mu\text{m/s}$ in the horizontal DS pulling direction (y). The position of the S-L interfacial line in Figure 3.4, was calculated from the thermal gradient, which was about 7 mm away from the edge of heater.

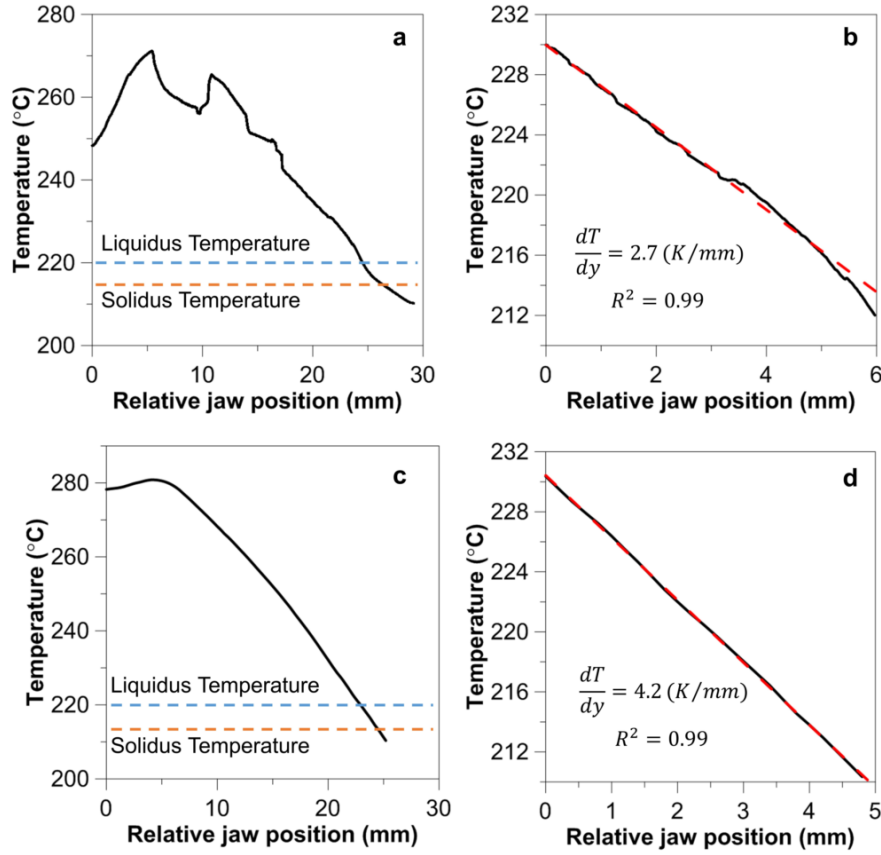


Figure 3.5. Temperature (T) vs. pulling distance (y) graphs for the DS samples. (a, b) at a pulling velocity of $v = 20 \mu\text{m/s}$, (c, d) at a pulling velocity of $v = 200 \mu\text{m/s}$. (a, c) The whole growth profile of the sample, (b, d) the profile during pulling, between 230-210 °C (217 °C is the ternary eutectic solidification temperature).

3.3 Sample preparation

To study the microstructure, the samples were mounted with cold mounting resin (Struers VersoCit acrylic resin). Grinding started with 800 grade paper and refined to 1200, 2000 and 4000 grades with water. After grinding, the sample was polished with a $0.05 \mu\text{m}$ colloidal silica (OPS) as a final step. Between each step, the samples were cleaned under running water and dried with an air gun and then briefly checked by optical microscopy to confirm the removal of large scratches from the previous stage. When a satisfactory optical image was obtained, the

sample was taken to the optical microscope and SEM for imaging and EBSD scanning. Since the cold mount is nonconductive, the top surface of mounted samples was coated with a layer of carbon powder (~ 3 nm), and the surrounding areas were covered with conductive silver paint to reduce charging effects in the SEM chamber.

A Quanta FEG 450 equipped with an x-ray energy dispersive X-ray (EDX) and a Bruker e-Flash^{HR} EBSD detector were employed to identify the elements included in the samples and determine the orientation, phase, and texture information of the sample. For EBSD, the SEM was operated at accelerating voltage of 20 kV, 5.5 spot size and approximate working distance of 15 - 20 mm. The EBSD detector was inserted to reach a distance of 10 – 15 mm towards the sample, which was tilted or pre-tilted using a special holder for 70° relative to the horizontal SEM chamber to obtain the best diffraction patterns. The data was analysed by Bruker ESPRIT 2.1 software during scanning with different step sizes and the EBSD maps were post-processed with “absorb surrounded zero solutions”.

The samples were properly prepared to a high-quality surface finish suitable for both DIC and EBSD. The sample was superglued on a hot-press Bakelite resin to achieve a $0.05\ \mu\text{m}$ colloidal silica mechanical polishing finish. Due to the fact that the β -Sn is much softer than the eutectic IMCs, slight roughness was generated between the β -Sn and IMCs during mechanical polishing. Therefore, the final specimen surface was polished with broad ion beam milling using at Gatan PECS II to firstly flatten the specimen surface and then introduced a very slight surface roughness which is suitable for DIC. The PECS II was operated in etching mode with dual modulation at a beam voltage of 3 keV and a tilt angle of 1° to flatten the IMCs, followed by 3 keV and 3° , in order to generate long strip speckle patterns on the surface, for 3 hours in total.

3.4 Microstructural Characterisation

3.4.1 Macro-etching

Before and after growth, the samples were firstly macro-etched using a 4.3% HCl + 8.7% FeCl₃ + 87% H₂O etchant for 30 seconds to reveal their grain structure. The grains with different orientations were etched at different rates due to their distinct susceptibility toward the etchant (Figure 3.6a, 3.6c and 3.6e), so the grains showed various brightness because of different reflectance under light illumination.

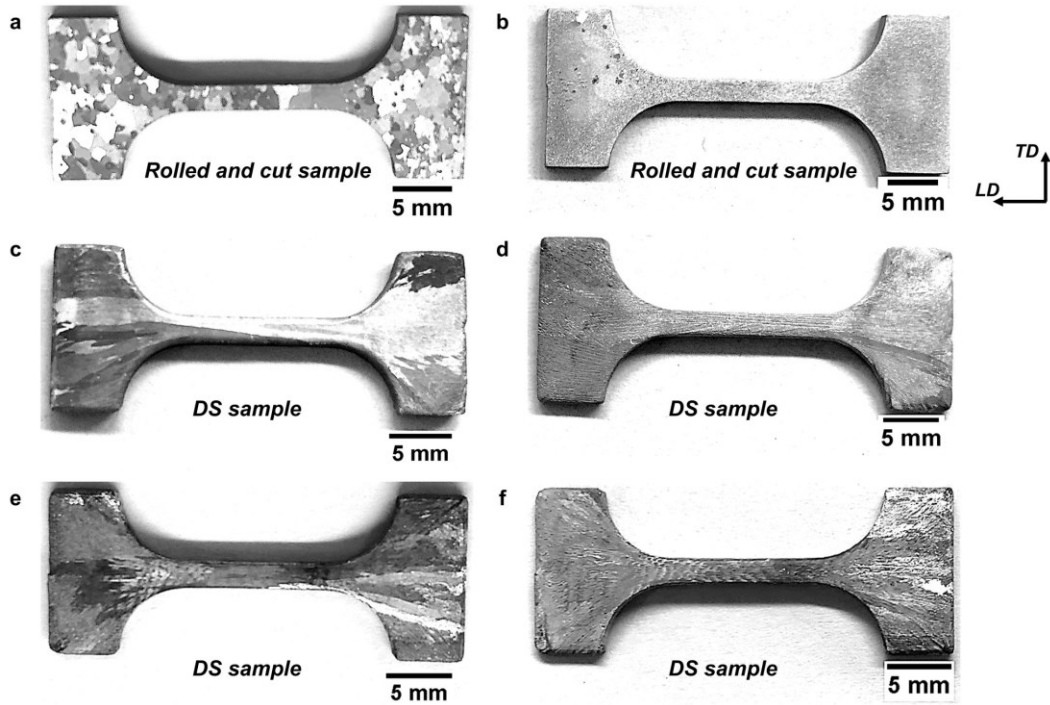


Figure 3.6. Macro-scale optical images of the dog-bone samples. (a, b) The rolled and cut samples, (c, d) the as-DS samples at $v=20 \mu\text{m/s}$, (e, f) the as-DS samples at $v=200 \mu\text{m/s}$. (a, c, e) the grain structures are revealed after grain etching, (b, d, f) the morphologies of the IMCs are shown after β -Sn etching.

For the room temperature creep samples, the 3D morphology of the IMCs was revealed in order to generate surface roughness (speckles patterns) of the sample surface for DIC during creep

testing (Figure 3.6b, 3.6d and 3.6f). The β -Sn was etched away using 3.5% 2-nitrophenol, 5% NaOH and 91.5% H₂O etchant at a temperature of about 70 °C with a hot plate. The heating was done by immersing the sample fully inside a beaker for 30 minutes of shallow etching. A glass cover was required to avoid large volume loss caused by vaporisation for long time etching. The sample was washed carefully with water and then dried naturally after etching.

Figure 3.6a and 3.6b give an example of a typically rolled and cut sample. After grain etching, the contrast between grains is revealed as bright and dark spots, which means that the rolled and cut sample has multiple and fine grains with numerous orientations (Figure 3.6a). The contrast between the β -Sn and the IMCs is enhanced after β -Sn etching and the IMCs are standing out but having no directionality (Figure 3.6b). The number of grains was significantly reduced after DS compared with the rolled and cut sample (Figure 3.6c). A few columnar grains have been generated through the length of the sample, where 2 and 3 main grains exist in the gauge region for $v = 20 \mu\text{m/s}$ (Figure 3.6c) and $v = 200 \mu\text{m/s}$ (Figure 3.6e) respectively. The morphology of the IMCs can be exposed clearly after selective etching of β -Sn and a significant increase in surface roughness of the samples can be observed (Figure 3.6d and 3.6f). The size and orientation of the grains were checked by optical microscopy and EBSD later.

3.4.2 Microstructure of rolled and cut sample

Figure 3.7 illustrates the large scale optical images, the secondary electron (SE) image as well as the EDX and EBSD maps of a typically rolled and cut sample from its polished and etched surfaces.

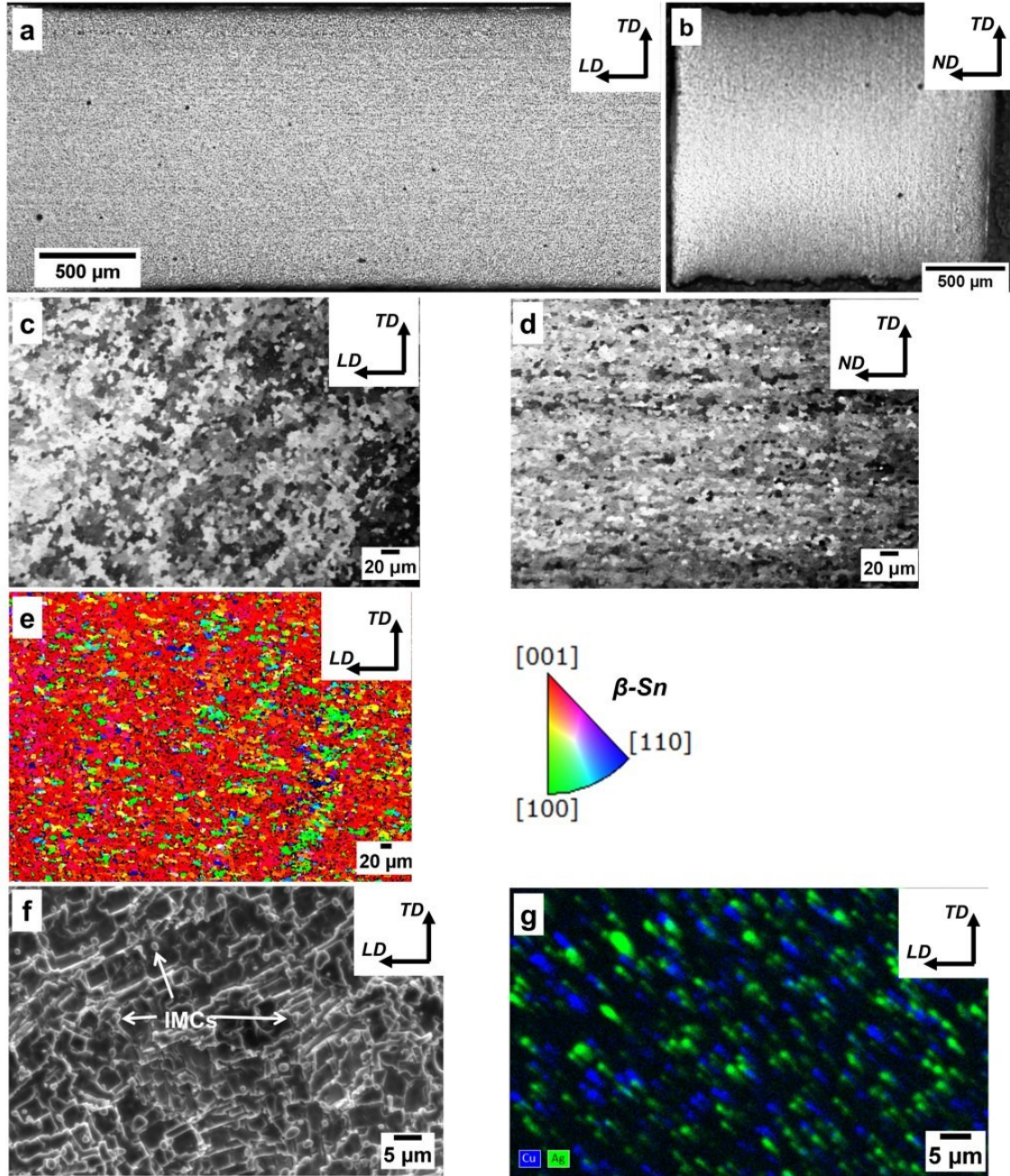


Figure 3.7. The microstructure of a typically rolled and cut sample. (a, b) The large scale optical image in (a) longitudinal direction (LD), (b) in transverse (ND) direction. (c, d) Higher magnification optical polarised light images show the grain contrast in (c) LD, (d) ND. (e) Crystal orientations represented with EBSD inverse pole figure (PF) colouring with respect to the loading direction (IPF-LD). (f) Higher magnification SEM secondary electron (SE) image after β -Sn etching with IMCs shown. (g) EDX map showing the distribution of the IMCs (blue: Cu_6Sn_5 and green: Ag_3Sn).

During the rolling process, the thickness of the SAC305 sheet was reduced significantly and introduced a high level of strain of ~170% to the sample, which is associated with a fine grain structure and stored deformation. It can be calculated from the reduction in thickness as

$$\varepsilon = \ln \frac{h_0}{h_f} \quad (3.1)$$

Where h_0 is the initial ingot thickness = 8.5 mm, and h_f is the final thickness after multiple passes = 1.5 mm.

The microstructure of the cross-section of the polished samples was firstly observed with an Olympus BX51-P Polarising Microscope. Both twinning and recrystallised grains occurred to accommodate the strains generated by rolling. No obvious microstructural features can be seen from the large scale optical image (Figure 3.7a and 3.7b). The grain boundaries can be distinguished using polarised light (Figure 3.7c and 3.7d) in both the LD and ND. The rolled and cut sample formed a large number of fine grains and the average grain size was approx. $15 (\pm 1) \mu\text{m}$ in both its longitudinal (Figure 3.7c) and transverse (Figure 3.7d) directions.

Figure 3.7e gives the EBSD IPF map in the LD, it formed a polycrystalline structure with main orientations close to [001] (Figure 3.7e), which means that the grains have same relationship between crystal lattice and sample coordinate systems. Figure 3.7f illustrates the β -Sn etched SE image of the sample with small round-shape IMCs ($\leq 1 \mu\text{m}$) uniformly embedded inside the β -Sn matrix. The two types of IMCs were not able to be distinguished from their shape. The SE image was obtained by a Zesis Auriga scanning electron microscope (SEM) in interpreting the IMCs morphology. No dendrite and eutectic morphology was observed from both the optical (Figure 3.7c 3.7d) and SE (Figure 3.7f) images.

The Figure 3.7g shows the EDX mapping of the sample, where the Ag_3Sn and the Cu_6Sn_5 are distinguished by the Cu and Ag content with blue and green colour identified. The two IMCs, i.e. Ag_3Sn and Cu_6Sn_5 are uniformly distributed within the sample matrix without forming IMC-free dendrite and IMCs-concentrated eutectic regions.

Therefore, the rolled and cut samples had a very different microstructure i.e. number of grains and grain size in its length-scale comparing to the real solder joints, which would make the testing result less effective to predict material behaviour for industrial application. This highlights the need to use a controlled solidification method (Bridgman growth) for generating microstructures that are relevant to solder joints for studies of creep mechanisms of the SAC305 solder in real applications.

3.4.3 Microstructure of directional solidified sample

The DS process produces a uniform microstructure of β -Sn dendrites with interdendritic eutectic regions, which contain Ag_3Sn and Cu_6Sn_5 IMCs (Figure 3.8). This process reproduces microstructures which are more similar to solder joints used in interconnection applications, i.e. similar number of grains (1-3) and a structure with β -Sn dendrites and IMCs in eutectic regions in its length-scale (compare with joints revealed in ref [7, 9, 11, 12]). As the direction and speed of growth were constrained, the dendrites were forced to grow in a well-aligned direction to the pulling direction with uniform dendrite arm spacing and reproducible eutectic spacing and IMC size. The microstructure of the β -Sn dendrites and the eutectic regions including IMCs, Ag_3Sn and Cu_6Sn_5 , was imaged using the optical microscope with a polariser within the cross-section of the polished samples using the same sample preparation method as in Section 3.4.2.

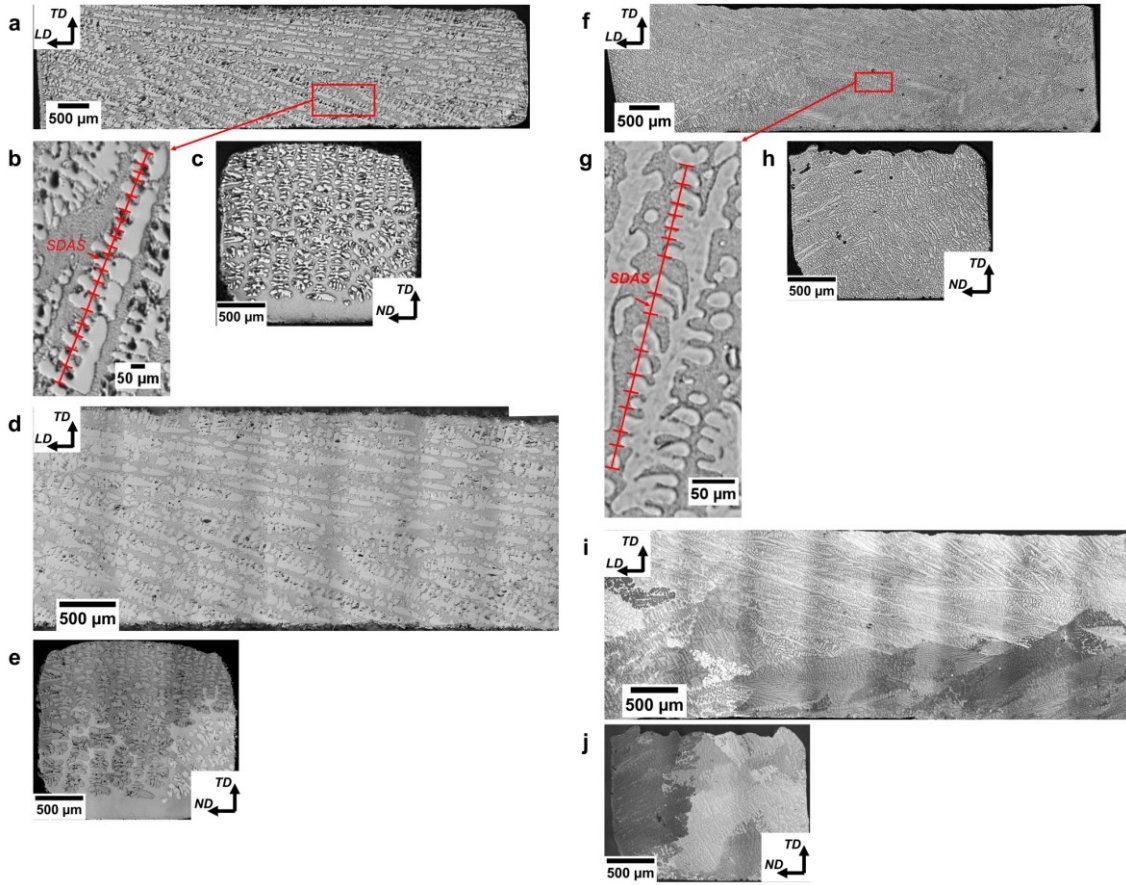


Figure 3.8. Optical images of polished DS samples, (a-e) at growth velocity of $20 \mu\text{m/s}$, (f-j) at growth velocity of $200 \mu\text{m/s}$. (a, d, f, i) in longitudinal direction, (c, e, h, j) in transverse direction. (d, e, i, j) with grain contrast shown by polarised light. The higher magnification images (b, g) show the dendrite arms with the secondary dendrite arm spacing (SDAS, λ_2) measured from 4 similar micrographs.

Figure 3.8a - 3.8e give examples of DS samples grown at $20 \mu\text{m/s}$. The β -Sn dendrites have a uniform secondary arm spacing (SDAS, λ_2) of $\sim 50 \pm 3 \mu\text{m}$, which was measured by drawing perpendicular straight lines through the secondary dendrite arms as shown in Figure 3.8b. By increasing the growth velocity to $200 \mu\text{m/s}$ (Figure 3.8f - 3.8j), the SDAS and IMC size are decreased greatly. The measured SDAS is $\sim 20 \pm 3 \mu\text{m}$ (Figure 3.8g). The SDAS is determined by

$$\lambda_2 = \frac{L}{N} \quad (3.2)$$

Where L is the length of the intersection line and N is the number of intersections of the line. The SDAS was calculated as a mean value by measuring 4 micrographs and the standard deviation (STD) of the value is given in Table 3.1 below.

Growth velocity, v (μm/s)	Length, L (μm)	Number, N	SDAS, $\lambda_2 = \frac{L}{N}$ (μm)		
				Average	STD
20	848	17	49.9	49.9	2.9
	683	13	52.5		
	857	19	45.1		
	883	17	51.9		
200	335	15	22.3	20.4	3.2
	226	12	18.8		
	96	6	16.0		
	340	14	24.3		

Table 3.1. Summary of SDAS (λ_2) of the DS samples.

The polarised light images in both longitudinal and transverse directions are shown in Figure 3.8d, 3.8e, 3.8i and 3.8j. The number of grains for the DS samples are significantly reduced. The 20μm/s has a single orientation in its longitudinal plane and 3 different orientations in its transverse plane, which is distinguishable by the contrast due to the interaction between the plane-polarised light and different Sn grains. The number of grains increases slightly as the growth velocity increases to 200 μm/s (Figure 3.8i and 3.8j) because it gives less time for the β-Sn to grow stably in the pulling direction.

To determine the microstructure and IMCs information of the sample, the Zesis Auriga SEM was utilised to obtain SE and BSE images for the DS sample at 20 and 200 μm/s growth rates. Figure 3.9 illustrates the micrographs in longitudinal directions (LD) for both samples with same scale bar.

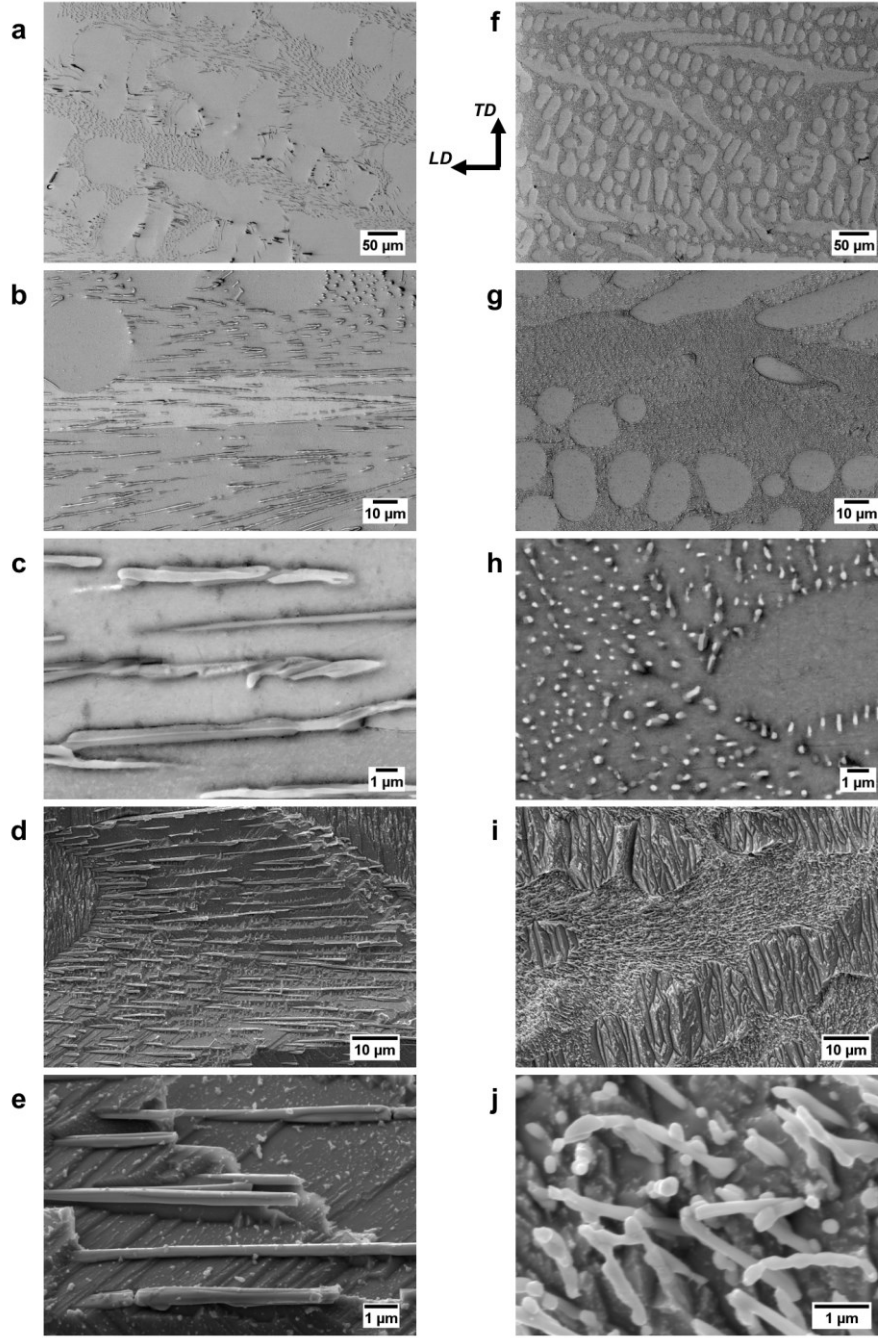


Figure 3.9. SEM images of the DS samples in longitudinal direction (LD), (a-e) at a growth velocity of 20 $\mu\text{m/s}$, (f-j) at a growth velocity of 200 $\mu\text{m/s}$, (a-c, f-h) show the BSE images after polishing with increasing magnification, (d, e, i, j) show the SE of the IMCs morphology after $\beta\text{-Sn}$ etching. (a, f) show the structure of $\beta\text{-Sn}$ dendrites, the higher magnification images (b, g) show the structure of the eutectic region, (c, h) show the morphology of IMCs.

Both the β -Sn dendrites and the IMCs are growing with strong directionality in the LD (growth direction). The dendrites are inclined approx. 15° to the pulling direction in longitudinal plane (Figure 3.9a and 3.9f).

With lower magnification (Figure 3.9a and 3.9f), the β -Sn dendrites appear as lighter grey region in the BSE micrographs. The ternary eutectic regions with IMCs of Ag_3Sn and Cu_6Sn_5 , formed between the dendrites, which correspond to the area involving black and dark grey features (Figure 3.9a and 3.9f). With increasing magnification (Figure 3.9b, 3.9c, 3.9g, 3.9h), the microstructure of the eutectic regions can be seen. The clear light grey colour regions are the eutectic β -Sn and the darker grey and black features are the Ag_3Sn and Cu_6Sn_5 IMCs. The IMCs can be distinguished due to their different atomic weight with respect to the backscatter electrons. The lightest Cu_6Sn_5 has the darkest black colour, while the heaviest Sn has the lightest grey colour (Figure 3.9c and 3.9h). EDX was used to determine the IMC compositions later in this section.

The effect of increasing growth rate is illustrated clearly in Figure 3.9. It can be seen that not only the size of dendrites and dendrite arm spacing (Figure 3.8) are decreased, but also the size of IMCs (Figure 3.9) are decreased by comparing the $200\text{ }\mu\text{m/s}$ sample with the $20\text{ }\mu\text{m/s}$ sample. Figure 3.9h shows the size of the IMCs are less than $1\text{ }\mu\text{m}$ in the longitudinal direction for the $200\text{ }\mu\text{m/s}$ sample, while the sample with $20\text{ }\mu\text{m/s}$ growth velocity has a IMCs size $\sim 10\text{ }\mu\text{m}$ in its longitudinal direction (Figure 3.9c and 3.9h).

The morphology of the IMCs is interpreted from the β -Sn etched samples using the SE image (Figure 3.9d, 3.8e, 3.8i, 3.8j). Large IMCs appeared in between β -Sn dendrite arms, as shown for the samples at both 20 and 200 $\mu\text{m/s}$ growth velocity. Figure 3.9d and 3.9i show that the eutectic regions have high surface roughness and the standing-out figures are IMCs. For the 20 $\mu\text{m/s}$ sample (Figure 3.9e), the IMCs are aligned well with the growth direction (LD) and the IMCs' spacing is uniform in length-scale. At higher growth rate of 200 $\mu\text{m/s}$ (Figure 3.9j), the IMCs are less match within the LD and have much smaller size by comparing to Figure 3.9e. The eutectic Ag_3Sn and Cu_6Sn_5 have a fully rod-like morphology for the 2 growth velocities.

Figure 3.10 illustrates the microstructure of the DS samples in the transverse direction. Similarly, the light grey regions are the β -Sn dendrites and the darker regions are the eutectic regions (Figure 3.10a and 3.10f) with IMCs growing out of the page (Figure 3.10c and 3.10h). The size of both β -Sn dendrites (Figure 3.10b and 3.10g) and IMCs (Figure 3.10d and 3.10i) decreases significantly with growth rate.

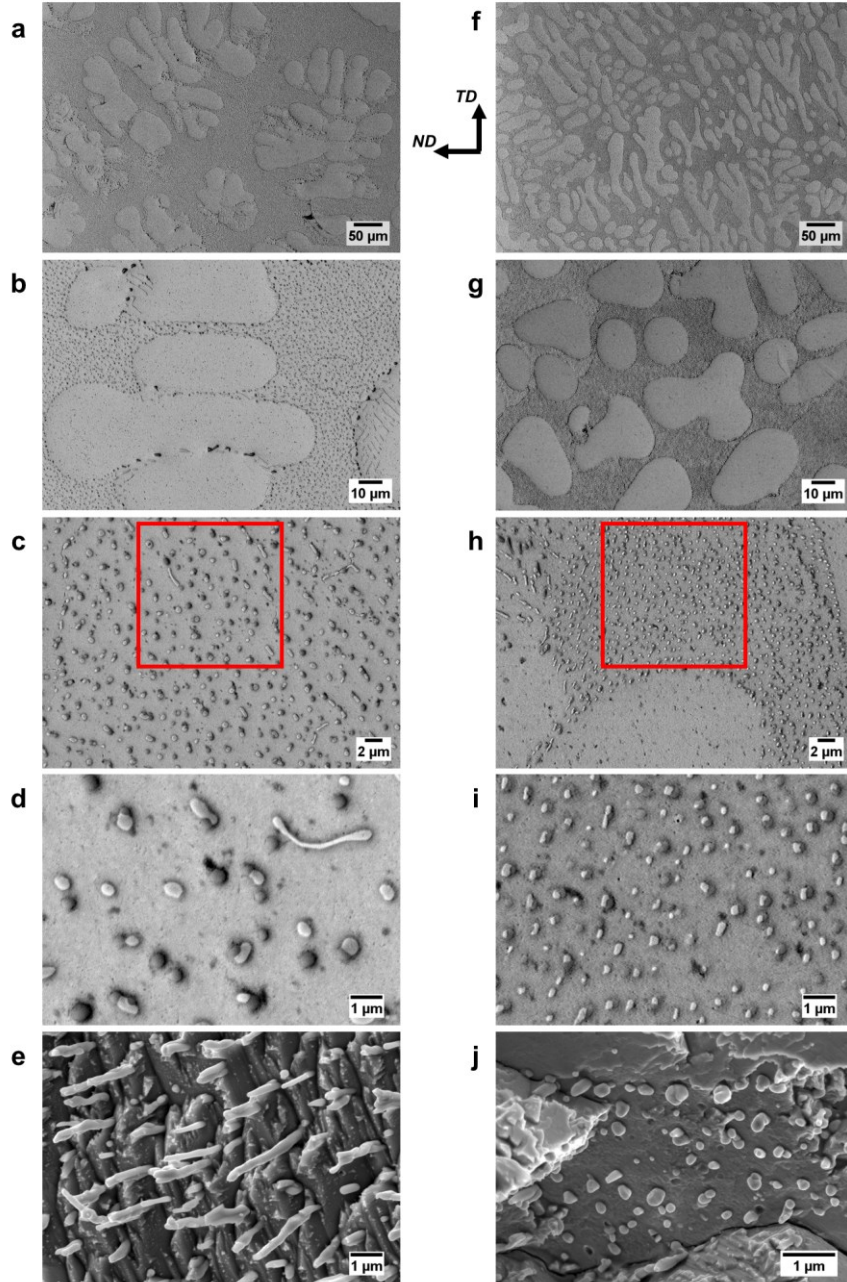


Figure 3.10. SEM images of the DS samples in transverse direction (ND), (a-e) at a growth velocity of 20 $\mu\text{m/s}$, (f-j) at a growth velocity of 200 $\mu\text{m/s}$, (a-d, f-i) show the BSE images after polishing with increasing magnification, (e, j) show the SE of the IMCs morphology after $\beta\text{-Sn}$ etching. The higher magnification images (b, g) show the structure of $\beta\text{-Sn}$ dendrites, (c, d, h, i) show the morphology of IMCs with the eutectic spacing (λ_e) measured from (c, h) within the red squared areas from 2 similar micrographs.

The spacing of rod eutectic morphology can be measured according to Flood and Hunt ^[145] as

$$0.5\lambda_e = S^r = 0.4 \left(\frac{A}{N} \right)^{0.5} \quad (3.3)$$

Where λ_e is the eutectic spacing in μm and $\frac{N}{A}$ is the number of IMC rods per unit area. A fixed area, A is selected and measured by Image J ^[146]. The number of particles, N are counted manually from Figure 3.10c and 3.10h. On average, the $\lambda_e \approx 1.18 \pm 0.02 \mu\text{m}$ and $\lambda_e \approx 0.68 \pm 0.02 \mu\text{m}$ for the 20 $\mu\text{m/s}$ and 200 $\mu\text{m/s}$ samples respectively. The Ag_3Sn (darker grey) and Cu_6Sn_5 (black) IMCs can be distinguished clearly by their grey-level in the BSE images (Figure 3.10d and 3.10i), however, a similar morphology is observed after β -Sn etching (Figure 3.10e and 3.10j).

Growth velocity, v ($\mu\text{m/s}$)	Area, A (μm^2)	Number, N		v ^{-0.5}	eutectic spacing, λ_e (μm)		
			Average			Average	STD
20	296	142	137	0.224	1.155	1.176	0.021
		132			1.198		
200	296	385	408	0.071	0.701	0.681	0.019
		431			0.663		

Table 3.2. Summary of eutectic spacing (λ_e) of the DS samples.

Therefore, both the SDAS (λ_2) and eutectic spacing (λ_e) decrease with the increasing growth velocity, whilst the morphology of the IMCs remains the same (rod-like) for the two tested velocities.

Figure 3.11 shows a typical DS sample at a growth velocity of 20 $\mu\text{m/s}$. It has two major orientations along its longitudinal direction (LD) (Figure 3.11a). The green-cyan orientation is close to the $\langle 100 \rangle$ and blue orientation is near $\langle 110 \rangle$ aligned along the longitudinal axis (i.e. tensile loading direction for which these are mechanically crept). These 2 orientations are

formed because $\langle 100 \rangle$ is the pulling direction and $\langle 110 \rangle$ is the favoured growth direction for β -Sn ^[147, 148]. It is noticed that there are 2 small orientations shown within the blue $\langle 110 \rangle$ orientation, e.g. the purple and yellow orientations (Figure 3.11a). The unit cell crystals show that the 3 orientations have a common $\langle 100 \rangle$ direction and a common (100) plane (twinning plane), which is normal to the [100] vector. Moreover, the 3 orientations (blue, purple and yellow in colour) are presented in the PF (Figure 3.11a) as 4 points, except for the green orientation. The circled point on the {100} PF shows a mixture of the three colours, which implies the three grains are cyclic twinned with approximately 60° to each other and a common (100) plane and $\langle 100 \rangle$ axis ^[12]. The green-cyan orientation (near $\langle 100 \rangle$), which is a single orientation and its orientation separated by 25° from the blue $\langle 110 \rangle$ orientation. Figure 3.11b gives the IPF map of the cross-section in the transverse direction (ND). The 2 distinct orientations are presented, as well in both the IPF map and the corresponding PF (Figure 3.11b). Figure 3.11c shows a FSE of a typical β -Sn dendrite and eutectic region with higher magnification. The FSE captures the image with the forward scattering electrons, which enables observations of the orientation, phase and topography through appropriate sample and detector positioning ^[149]. Figure 3.11c is taken with the detector at a ‘near’ position, where the detector is placed at approximate 13 mm towards the sample and $\sim 3.9^\circ$ (a low tilt angle) to the normal direction of the sample. These settings give optimised topography between the β -Sn and IMCs phases (the standing-out figures are the IMCs), but less information of orientation (the mapping region has single orientation indicated by EBSD). The IMCs Ag_3Sn and Cu_6Sn_5 are identified clearly in the eutectic region in EDS map in Figure 3.11d with green for Ag element and blue for Cu element respectively, while the dendrite region has no colour presented.

The phase information of all the 3 phases within a selected eutectic region is given in Figure 3.11e. The β -Sn dendrite shows a red colour, while the Ag_3Sn and Cu_6Sn_5 embedded are indexed clearly with green and blue colour respectively by EBSD. The 2 IMCs, namely Ag_3Sn and Cu_6Sn_5 are grown with strong directionality (Figure 3.11f and 3.12g). It looks like that the Ag_3Sn IMCs have orientation in between $[001]$ and $[100]$, which share a common $[010]$ axis (see annotation in the PF of Figure 3.11f). The $[001]$ of the Cu_6Sn_5 is nearly in parallel to the $[110]$ and the $[001]$ is rotating along the $[100]$ axis (see annotation in the PF of Figure 3.11g).

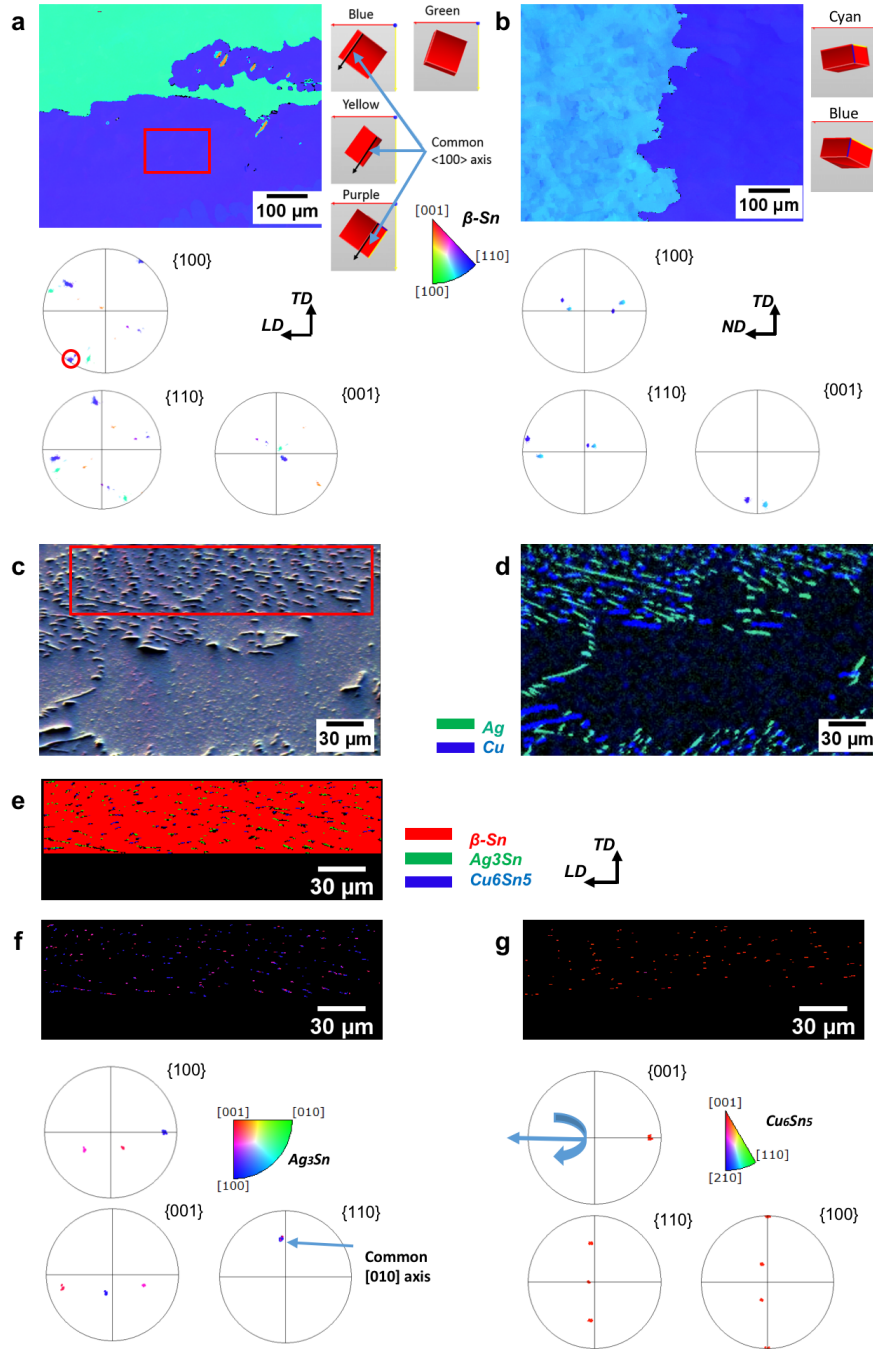


Figure 3.11. Crystal orientation and phase maps of a DS sample at $v = 20 \mu\text{m/s}$. (a, b) IPF maps of $\beta\text{-Sn}$ show 2 main orientations, the corresponding crystal unit cells and pole figures (PFs) are given, (a) in LD, (b) in ND. (c, d) High magnification map of the red squared region in (a) within the blue orientation, (c) Forescatter electron (FSE) image shows the topography between $\beta\text{-Sn}$ and IMCs, (d) EDS map with the Ag_3Sn and Cu_6Sn_5 identified. (e-g) EBSD maps of the red squared region in (c) at higher magnification within the eutectic region, (e) phase map with $\beta\text{-Sn}$, Ag_3Sn and Cu_6Sn_5 identified, (f, g) IPF-LD map of Ag_3Sn (f) and Cu_6Sn_5 (g) with texture given in the corresponding PF.

3.5 Summary of the Chapter

Controlled microstructures of bulk SAC305 solder alloy were generated by solidifying dog-bone samples by Bridgman growth which reproduced a similar number of β -Sn grains, β -Sn orientations, and length-scale of the eutectic Ag_3Sn and Cu_6Sn_5 in all samples prior to creep testing. The Bridgman grown samples enable consistent microstructures to be tested and promote understanding of fundamental mechanisms. This avoids addressing the stochastic nature of real solder joint microstructures, which have variations from joint to joint due to the differences in the nucleation undercooling of β -Sn and the random orientation of β -Sn at the point of nucleation.

The DS samples have key similarities to the microstructure of a real SAC305 solder joint, i.e. a similar number of tin grains (1-3), the similar microstructures with β -Sn dendrites, and fine IMCs in eutectic regions [7, 9, 11, 12]. The IMCs for the samples at a growth rate of $v = 20 \mu\text{m/s}$ are larger than found in a typical joint. A benefit of using larger IMCs is to enhance the localisation of strain during the creep deformation, so as to make it easier to observe the difference between the phases (the nature of heterogeneous deformation) during creep. The role of IMCs size on strain localisation can be explored during the creep deformation. The IMCs for the samples with a growth rate of $v = 200 \mu\text{m/s}$ are significantly smaller, i.e. nearly the same size as found in a typical joint. Hence, all the 3 phases need to be considered and to understand how these phases affect the creep reliability of SAC305 solder alloys.

Chapter 4 Effect of stress on creep behaviour of SAC305 solder

4.1 Introduction

In this chapter, the creep behaviour of the controlled microstructures produced by DS is investigated under a constant temperature ($\sim 25^\circ\text{C}$) at a range of stresses. The secondary creep strain rate, the dominating creep mechanism, and its stress dependence are explored. The microstructural evolution mechanisms of the sample are examined by EBSD. The role of IMCs in the eutectic within the bulk matrix is considered during creep testing to investigate their effect on microstructure evolution and creep reliability for the SAC305 solder alloys. These observations could be extended for understanding the mechanical loading of solder joints.

4.2 Experiment

The 10 kN Shimadzu AGS-X mechanical test machine with non-shift wedge type grips was used to carry out the room temperature creep tests with range of stresses under load control. The experimental parameters were set-up and recorded using TRAPEZIUM X data processing software and the creep strain curves were determined by the TRViewX non-contact digital video extensometer. Samples were tested from 150 to 350 N with a 50 N interval for stress level from 17 – 44 (± 2) MPa to fracture and tests were repeated for each loading for a more comparable result. The 250 N loading (~ 30 MPa) was repeated for 4 times with different initial orientations. The sampling interval was normally set to be 1 per second but was adjusted

accordingly due to the large variation of testing time. The uncertainty of the stress and temperature are obtained from the Figure 4.1. The temperature of the sample was monitored using the thermocouple Pico-Log TC-08 recorder together with the thermocouple wire, which was attached to the surface of the sample. The temperature uncertainty was ± 1 °C.

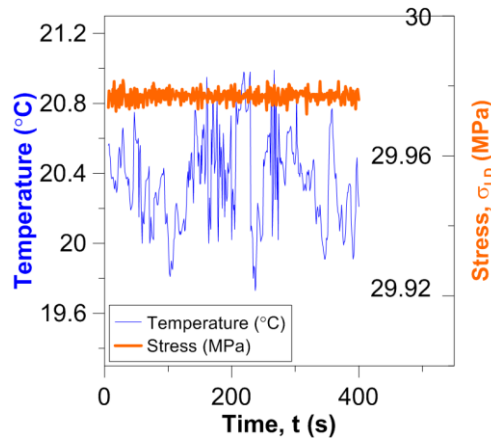


Figure 4.1. The uncertainty of the temperature and stress during creep testing for room temperature carried out by Shimadzu.

The DS samples with a growth velocity of $v = 20$ $\mu\text{m/s}$ were used to carry out the creep test at room temperature. The samples were shallowly etched before creep testing (see Chapter 3.4.1). Since the IMCs are uniformly distributed in within the tin matrix, where most of the deformation are accommodated. Thus, the effect of the etching on deformation damage can be neglectable. The fracture surface of the samples was studied in macroscopic scale by the stereomicroscope (Brunel Microscope) near crack tip in both longitudinal (LD) and transverse plane (ND) to determine the morphology of the fracture surface and indicate the difference in failure modes for different stresses after creep deformation. The cross-section was polished according to the method shown in Section 3.4.2. The microstructure of the fractured surfaces was revealed in LD using both optical microscopy and EBSD with a step size of 1 and 0.4 μm for the low and high magnification IPF maps respectively.

4.3 Results and Discussion

4.3.1 Creep strain curves

Figure 4.2 gives the creep curves for the fractured samples, which were tested at room temperature under constant load from 17 – 44 MPa (the stress is calculated using applied load divided by initial cross-sectional area measured). The creep strain against time curves (Figure 4.2a) show a dominating secondary creep region for all tested stresses and its gradient is the secondary creep rate, which is determined by plotting a linear fit through all of the regions (data is given in Table 4.1).

The creep strain curves from the primary stage to the early stage of secondary creep are shown in Figure 4.2b. The tested samples reached their secondary stage creep within the first 20 s of testing. Figure 4.2c gives the creep strain rate vs. strain curves for primary and secondary stage creep, which is plotted by differentiating the creep curves for all tested stresses. A linear relationship with a constant gradient close to 0 is obtained once secondary creep is achieved and the sample tested at higher stress has less increase in strain during the secondary stage creep (Figure 4.2c). The onset of secondary creep strain is approximately 2% (Figure 4.2c) and increases slightly with increasing stress (Figure 4.2d).

In the secondary creep region, the two competing processes of strain hardening and dynamic recovery are in balance and no localised creep damage is obtained. The strain rate of secondary stage creep increases and the creep lifetime decreases significantly with increasing stress (Figure 4.2a and Table 4.1). It is observed that for the tested samples, there is a substantial increase in creep strain with time for tertiary stage creep, which implies that the damage of solders accelerates at this stage.

Effect of stress on creep behaviour of SAC305 solder

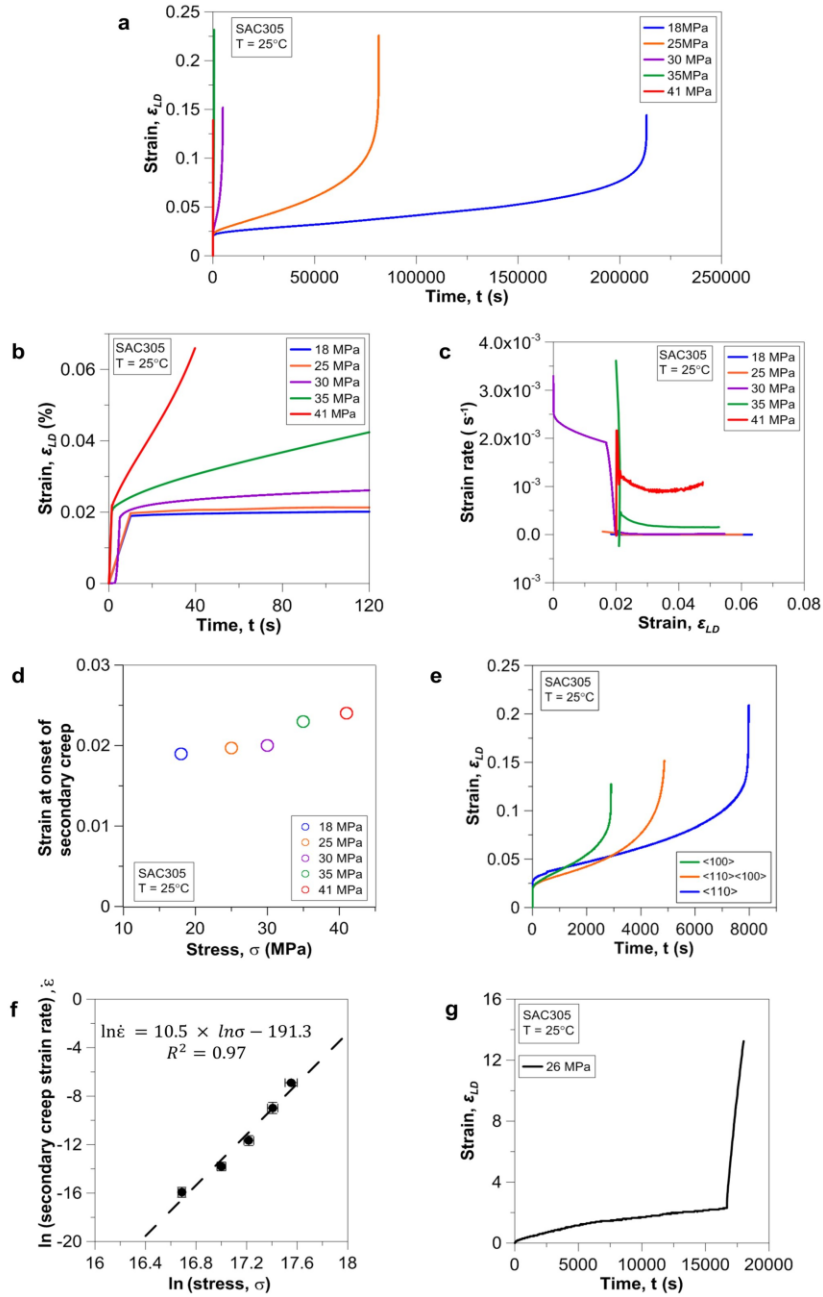


Figure 4.2. Constant temperature ($\sim 25^\circ\text{C}$) mechanical creep data from 18 to 42 MPa of SAC305 samples grown at $v = 20\ \mu\text{m/s}$. (a) Full creep curves until sample failure. (b) Creep curves at primary and early secondary stages of creep (first 120 s). (c) Creep strain rate vs. strain curves showing a gradient of 0 after the secondary stage creep is reached. (d) Variation of the onset of secondary creep strain with respect to stress. (e) Creep curves of repeated tests at constant stress of ~ 30 MPa with different initial orientations. (f) $\ln$ (secondary creep strain rate, $\dot{\epsilon}$) vs. $\ln$ (stress, σ) showing the value of stress exponent $n = 10.5$ and the error bars are drawn according to the standard deviation (STD) for both σ and secondary creep $\dot{\epsilon}$ in Table 4.1. (g) Creep curve of rolled and cut sample at ~ 26 MPa. (The symbols are included for completeness and represent orientations, as described in Table 4.2.)

Effect of stress on creep behaviour of SAC305 solder

The 250 N (~30 MPa) loading was repeated 3 times with different initial orientations and number of grains (Figure 4.2e). The lifetime almost doubles for the samples with an initial main <110> orientation compared the ones with a main <100> orientation as shown in Table 4.2. The lifetime of the bi-crystal and tri-crystal samples is between the <110> and <100> single crystal samples. It looks like the secondary strain rate varies with initial orientation and number of grains of the sample, but this variation with crystal orientation is relatively small compared with the effect of the applied stress.

Load (N)	Sample	σ ($\pm$ 0.1 MPa)	$\dot{\epsilon}$ (s ⁻¹)	ln σ			ln $\dot{\epsilon}$		
					Average	STD		Average	STD
150	DS2	18.2	8.0×10^{-8}	16.72	16.68	0.029	-16.34	-15.94	0.405
	DS7	17.1	1.8×10^{-7}	16.66			-15.53		
200	RC04	26.6	1.1×10^{-4}	17.10	17.10	-	-9.12	-9.12	-
	DS3	24.2	6.7×10^{-7}	17.00	17.00	0.035	-14.22	-13.80	0.357
	DS8	25.2	9.7×10^{-7}	17.04			-13.85		
	DS11	23.1	1.6×10^{-6}	16.96			-13.35		
250	DS4	29.2	5.2×10^{-6}	17.19	17.22	0.039	-12.16	-11.67	0.407
	DS9	31.5	1.6×10^{-5}	17.26			-11.04		
	DS12	30.9	8.7×10^{-6}	17.25			-11.65		
	DS14	28.6	7.4×10^{-6}	17.17			-11.81		
300	DS5	36.8	1.6×10^{-4}	17.42	17.41	0.041	-8.740	-8.973	0.456
	DS10	37.7	1.9×10^{-4}	17.45			-8.568		
	DS3	34.3	6.7×10^{-5}	17.35			-9.611		
350	DS6	40.0	1.1×10^{-3}	17.50	17.55	0.048	-6.812	-6.891	0.079
	DS13	44.0	9.4×10^{-4}	17.60			-6.970		

Table 4.1. Summary of the fractured samples tested under room temperature showing the information of load, stress (σ), secondary creep lifetime and creep strain rate ($\dot{\epsilon}$), as well as $\ln \sigma$ and $\ln \dot{\epsilon}$, to calculate the value of n . The rolled and cut sample (RC) is given as the highlighted data without repeating, so no value for STD.

The secondary creep curves can be analysed to extract the likely deformation mechanism based on Equation (2.4), it can be derived that under constant temperature, $\ln \dot{\epsilon}$ is proportional to $\ln \sigma$, and the gradient of the linear fit is the stress exponent n , which is used to determine the creep mechanism of the material. For each loading, the creep tests were repeated a few times, and therefore an average value and STD for each loading were obtained for both σ and $\dot{\epsilon}$. Table 4.1

gives the summarised data for all the tested samples, which is used to plot Figure 4.2f with error bars calculated by STD. Therefore, n is 10.5 for the DS SAC305, indicating the dominating creep mechanism is dislocation creep (see Section 2.3.2).

Figure 4.2g shows the creep curve of the rolled and cut sample at ~ 26 MPa. The sample has a much higher value of secondary creep strain rate and a significantly shorter lifetime comparing to the DS sample at the same stress (Table 4.1 and Table 4.2). This might be caused by the β -Sn grains of the rolled and cut sample, which are more refined and $[001]$ oriented ^[150, 151], since the grain boundaries provide short-cut paths for diffusion and therefore increases the probability of diffusion-assisted dislocation climb (see Section 2.3.2).

Load (N)	Sample	Average σ (± 0.1 MPa)	No. of grains in LD	Initial Orientation in LD	Secondary creep lifetime (s)
-	DS1	-	2	$\langle 100 \rangle, \langle 110 \rangle$	-
150	DS2	17.7	1	$\langle 100 \rangle$	130000
200	DS3	24.2	1	$\langle 100 \rangle$	50000
	RC04	26.6	Multiple	$[001]$	15000
250	DS4	30.1	1	$\langle 100 \rangle$	2000
	DS9		2	$\langle 100 \rangle, \langle 110 \rangle$	3500
	DS12		1	$\langle 100 \rangle, \langle 110 \rangle$	4500
	DS14		1	$\langle 110 \rangle$	6000
300	DS5	36.2	2	$\langle 100 \rangle, \langle 110 \rangle$	300
350	DS6	42.0	1	$\langle 100 \rangle$	30

Table 4.2. Summary of the fractured samples tested under room temperature with information of load, stress, number of grains, initial orientation and secondary creep lifetime shown. The rolled and cut sample (RC) is given as the highlighted data.

4.3.2 Creep fracture surface and failure mode

Figure 4.3 illustrates the stereomicroscope images of the creep fractured samples at fracture surface in both the longitudinal and transverse plane. Necking has been observed for all fracture samples indicating the DS samples are under ductile failure. It is observed that the fracture

surface changes as the applied stress increases and reduction of transverse surface area increases with increasing stress levels.

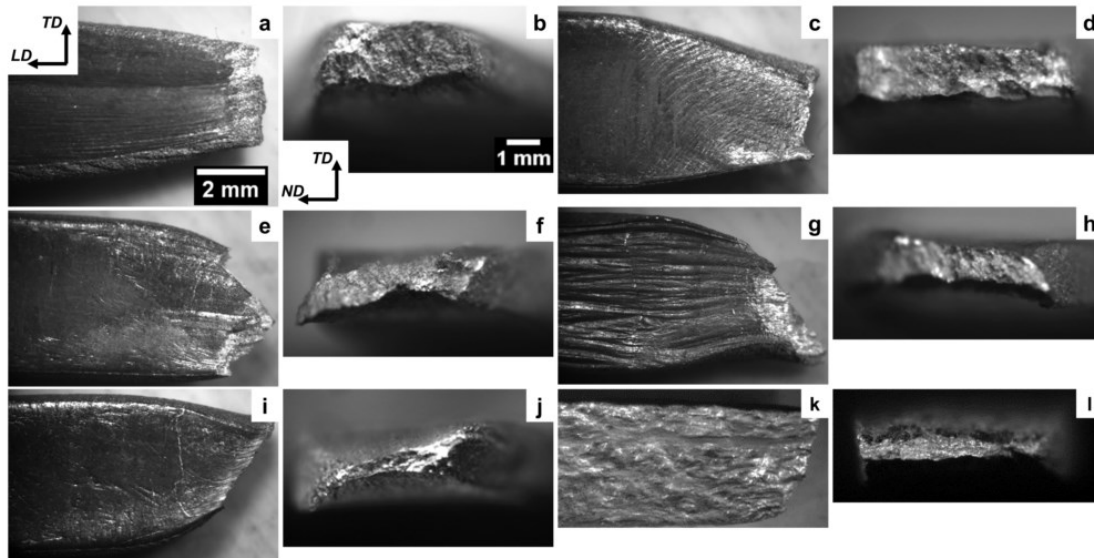


Figure 4.3. Stereo-micrographs of the fracture surfaces for (a-j) the DS samples, (k, l) the rolled and cut sample, tested at stress (a, b) ~18 MPa, (c, d) ~24 MPa, (e, f) ~30 MPa, (g, h) ~36 MPa, (i, j) ~42 MPa, (k, l) ~26 MPa, (a, c, e, g, i, k) in longitudinal plane (LD), (b, d, f, h, j, l) in transverse plane (ND).

The sample at ~18 MPa (Figure 4.3a and 4.3b) and ~24 MPa (Figure 4.3c and 4.3d) have a reduction in transverse area of ~ 20% and ~23% respectively with a near-flat fracture surface and large extension in length. The samples at stress level of ~36 MPa and ~42 MPa show a significant reduction in transverse area up to ~50% with an obvious shearing fracture surface at approx. 45° to loading direction, which represents localised necking. The sample at ~30 MPa shows a moderate reduction of ~38%. It forms a ragged surface with shear stress introduced. After the localised neck initiates, further deformation of the material concentrates in a localised region. The rough appearance of the transverse fracture surface implies the feature of cup-cone failure, of which the mechanism is shown in Figure 2.10 (See Section 2.3.3).

Long-strip deformation patterns appeared in the longitudinal plane for all DS samples (Figure 4.3a, 4.3c, 4.3e, 4.3g, and 4.3i), while the rolled and cut sample forms a block-shape deformation patterns at the macroscopic scale. The difference in the patterns is probably due to the microstructural difference, i.e. the DS samples have long strips of dendrites and long columnar grains in the longitudinal plane, and the rolled and cut sample, on the other hand, has small and square-shape grains.

At the macroscopic scale, elongating and thinning were observed during creep testing. Necking was observed for all samples after entering their tertiary creep regions. The micro-scale damages were supposed to occur, which will be referred to the section 4.3.3 in this chapter.

4.3.3 Microstructural analysis of creep fractured sample

Figure 4.4 gives an example of a typical creep fracture sample at ~18 MPa. The initial structure of the sample is distorted due to pulling. The sample was examined at 3 different positions, as labelled in Figure 4.4a.

The initial orientation of the sample can be expected from the IPF map at position 1. It is located in the non-necking region and has a mainly single orientation, which is between $[100]$ and $[110]$ in the loading direction with the crystal unit cell shown in Figure 4.4b. There are some small new orientations appeared near IMCs (indicated with red arrows) and slightly colour difference is observed between the dendrites and the eutectic regions. Figure 4.4c shows that the corresponding misorientation map (as indicated with a MO av. per grain map colour key 0 - 15°) has high spots in the eutectic regions around IMCs (labelled with red arrows), which is our hypothesis.

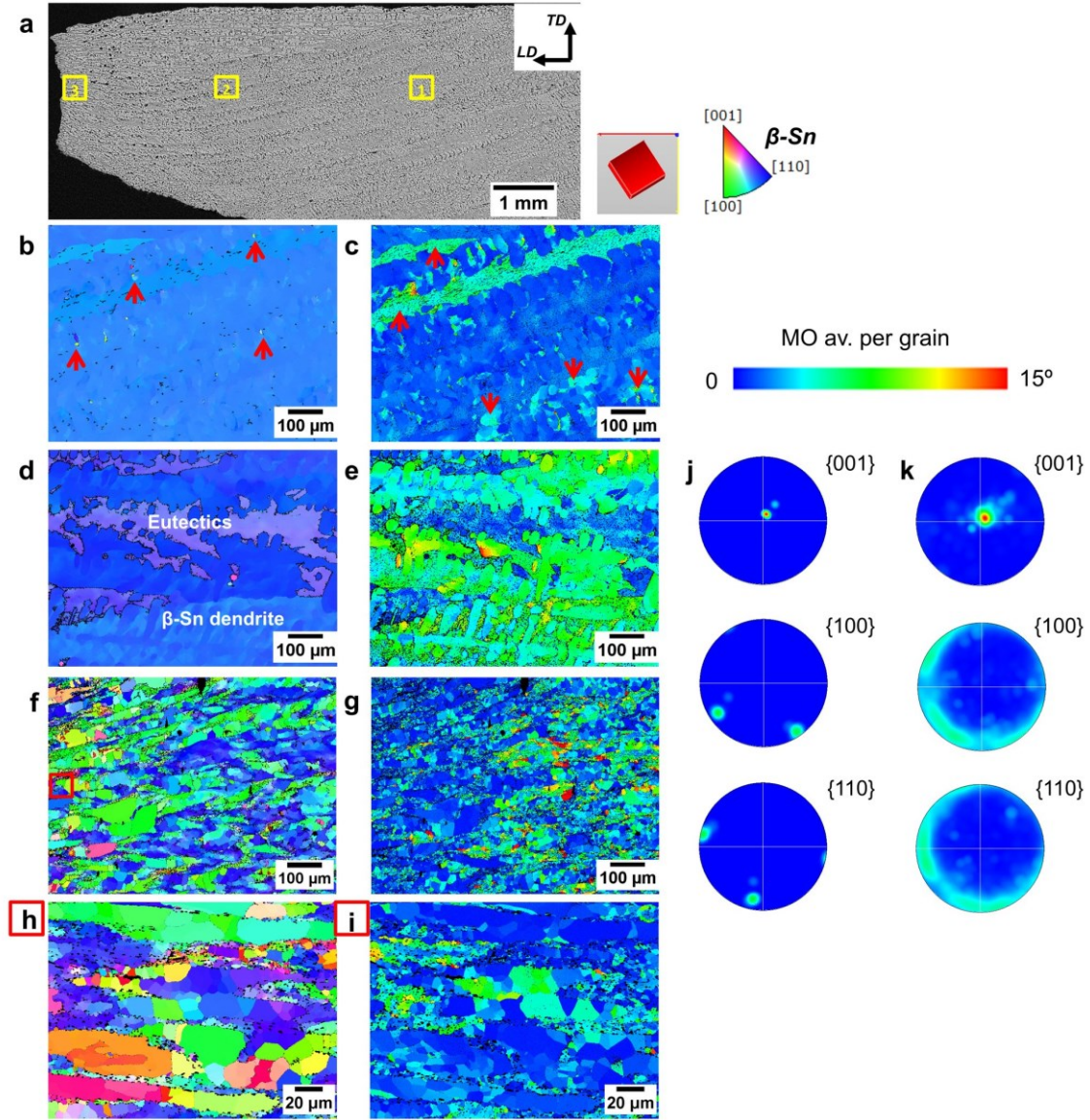


Figure 4.4. The creep fractured DS sample at ~ 18 MPa. (a) Large scale optical image at fracture surface in longitudinal plane. (b-i) EBSD maps in the yellow squared region in (a) (only the β -Sn has been indexed here), (b, c) at location 1 within the uniformly deformed region, (d, e) at location 2 in the necking region, (f, g) at location 3 near the fracture surface, (h, i) Higher magnification IPF-LD maps in the red squared region in (f). (b, d, f, h) IPF-LD maps show the evolution of crystal orientation with position. (c, e, g, f) MO av. maps show the variation of misorientation with position. (j, k) PFs of the sample at DS condition (j), at tertiary stage creep (k).

Figure 4.4d and 4.4e show the IPF-LD map near the necking line at position 2. The main orientation (Figure 4.4d) is rotating towards $[110]$ with a blue / purple colour. The eutectic

regions (purple) experience more lattice rotation than the dendrite (blue). In this region, the sample is much more constrained with high misorientation (Figure 4.4e).

At the fracture surface (Figure 4.4f and 4.4g at location 3 in Figure 4.4a), the dendrite arms are elongated and much thinner because of higher amount of straining experienced. The formation of rainbow colour recrystallisation can be observed in Figure 4.4f. The constrained misorientation (Figure 4.4g) has been released due to the generation of recrystallisation and fracture of the sample. Figure 4.4h and 4.4i present the higher magnification IPF map at the fracture tip. Clearly, the β -Sn in the eutectic regions has similar recrystallisation trends but with smaller grain size than the one in the dendrites. A fibre texture with a common [001] (the direction has the least amount of spreading among 3 directions) is observed for the sample and a spread in orientation is obtained by comparing to the PF at position 1 (Figure 4.4j) to the one at position 3 (Figure 4.4k).

The optical and EBSD maps in Figure 4.5 show the sample creep fractured at ~24 MPa at room temperature. The main orientation (green) is close to [100] which is given by the crystal unit cell in Figure 4.5b.

It can be seen from the IPF-LD at location 1 in Figure 4.5a (Figure 4.5b), that small red (close to [001]) new orientations form during the deformation, which are located in the dendrite at the boundary between dendrites and eutectic regions, where the higher misorientations are observed (Figure 4.5c), likely due to CTE mismatch between the 2 phases. The red orientation is formed due to the recrystallisation, i.e. the green and the red are separated with $\sim 63^\circ$ indicating the generation of deformation twin as given by the crystal unit cells in Figure 4.5b.

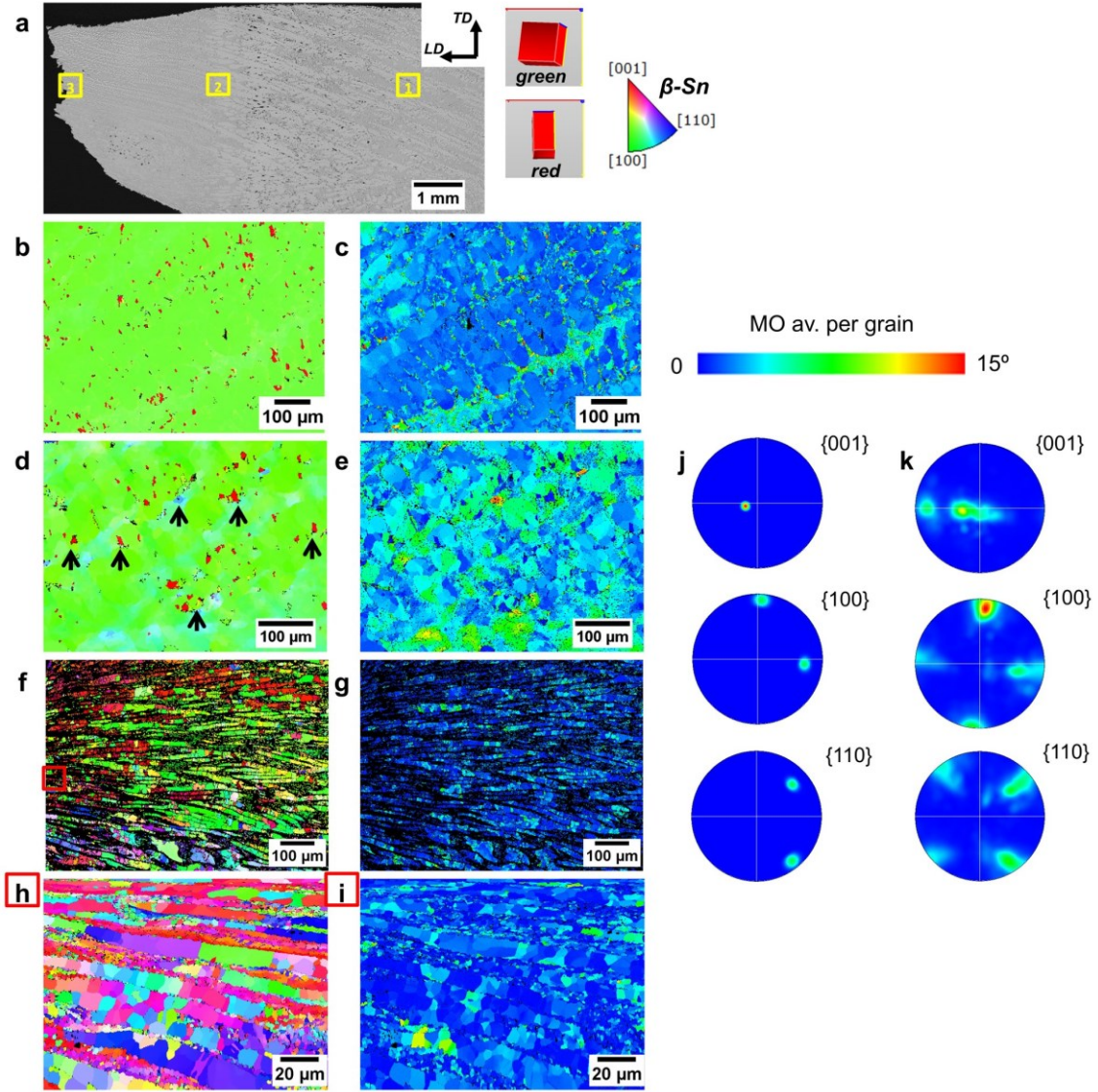


Figure 4.5. The creep fractured DS sample at ~24 MPa. (a) Large scale optical image at fracture surface in longitudinal plane. (b-i) EBSD maps in the yellow squared region in (a) (only the β -Sn has been indexed here), (b, c) at location 1 within the uniformly deformed region, (d, e) at location 2 in the necking region, (f, g) at location 3 near the fracture surface, (h, i) Higher magnification IPF-LD maps in the red squared region in (f). (b, d, f, h) IPF-LD maps show the evolution of crystal orientation with position. (c, e, g, i) MO av. maps show the variation of misorientation with position. (j, k) PFs of the sample at DS condition (j), at tertiary stage creep (k).

As moved to the necking region (Figure 4.5d and 4.5e located at location 2 in Figure 4.5a), more red orientations are developed and the third orientation (light blue) appears in the eutectic regions (indicated with black arrows), which might be caused by gradual grain rotation towards [110]. The deformation is running through the primary and giving massive subgrains, because

it is relatively easy for the grain boundaries to push through in the dendrite, where it is relatively difficult for these boundaries to run through the eutectic regions in which there are many other phases involved.

Both red and rainbow recrystallisation are developed at the fracture surface (Figure 4.5f and 4.5g at location 3 in Figure 4.5a). The red orientation can be due to the formation of a twin (perhaps tetragonal switch since the green and the red are having a common [100]) and leads to recrystallisation through the section (Figure 4.5f). The orientation spread is small and even in all directions (Figure 4.5j and 4.5k). The rainbow colour forms by β -Sn lattice rotation with progressive recovery followed by recrystallisation to change the orientation (Figure 4.5f), showing as spread of colours in the PF (Figure 4.5j and 4.5k). As same as the sample at ~ 18 MPa, the recrystallised grain in the eutectic regions have much smaller grain sizes and large spreading in orientations for the sample at ~ 24 MPa (Figure 4.5h). The misorientation has been decreased in both dendrites and eutectic regions at position 3 (Figure 4.5g and 4.5i).

The microstructure of the sample tested at ~ 30 MPa is presented in Figure 4.6. Although the IPF-LD map at location 1 in Figure 4.6a (Figure 4.6b) is almost single coloured, the formation of subgrains is observed in the misorientation map (Figure 4.6c) and they are localised within the eutectic regions in strong connection with the distribution of IMCs in the sample matrix (indicated with red arrows).

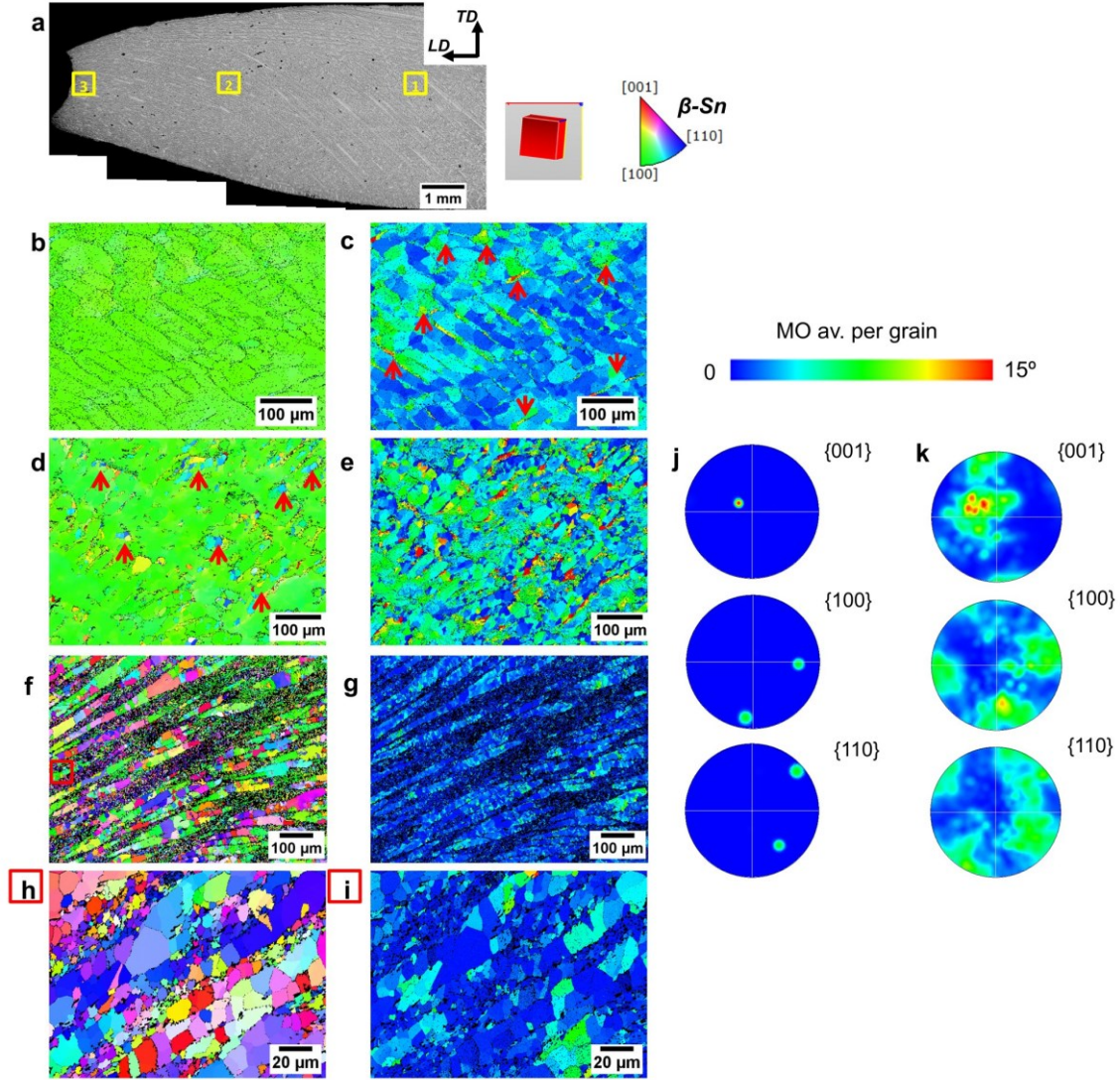


Figure 4.6. The creep fractured DS sample at ~ 30 MPa. (a) Large scale optical image at fracture surface in longitudinal plane. (b-i) EBSD maps in the yellow squared region in (a) (only the β -Sn has been indexed here), (b, c) at location 1 within the uniformly deformed region, (d, e) at location 2 in the necking region, (f, g) at location 3 near the fracture surface, (h, i) Higher magnification IPF-LD maps in the red squared region in (f). (b, d, f, h) IPF-LD maps show the evolution of crystal orientation with position. (c, e, g, i) MO av. maps show the variation of misorientation with position. (j, k) PFs of the sample at DS condition (j), at tertiary stage creep (k).

At the necking line (location 2 in Figure 4.6a), the misorientation increases significantly across the map (Figure 4.6e). The IPF-LD map (Figure 4.6d) shows a mainly single orientation, but new orientations are formed in between primary dendrites and eutectic regions (indicated with red arrows). Recrystallisation has been generated near the fracture surface (Figure 4.6f) in both

primary dendrite and eutectic regions (Figure 4.6h) with huge reduction in misorientation value (Figure 4.6g and 4.6i). Significant spread in orientation is obtained in all 3 directions on the corresponding PFs from position 1 to position 3 (Figure 4.6j and 4.6k).

Figure 4.7 shows a sample deformed at ~36 MPa and its initial orientation can be seen using IPF-LD map for position 1 in Figure 4.7a (Figure 4.7b). This sample consists of mainly a single (near [100]) orientation as shown by the unit cell next to the IPF-LD map. The local lattice misorientation starts to develop around the IMCs and the small new orientations are appearing near the IMCs. It looks like that the initial deformation is length-scale controlled by the IMCs.

Position 2 (Figure 4.7a) is located just before the necking line, the second orientation (cyan) appears in the eutectic regions (Figure 4.7d) and the misorientation becomes much more constrained, which implies that the initial orientation changes smoothly with a large number of subgrains formed (indicated with red arrows). Within the necking region, recrystallisation is much easier to carry through the primary dendrite, because the primary β -Sn is very soft material and relatively easier to achieve deformation by rotating. And it is expected that the recrystallisation would start along these boundaries at later stage of creep.

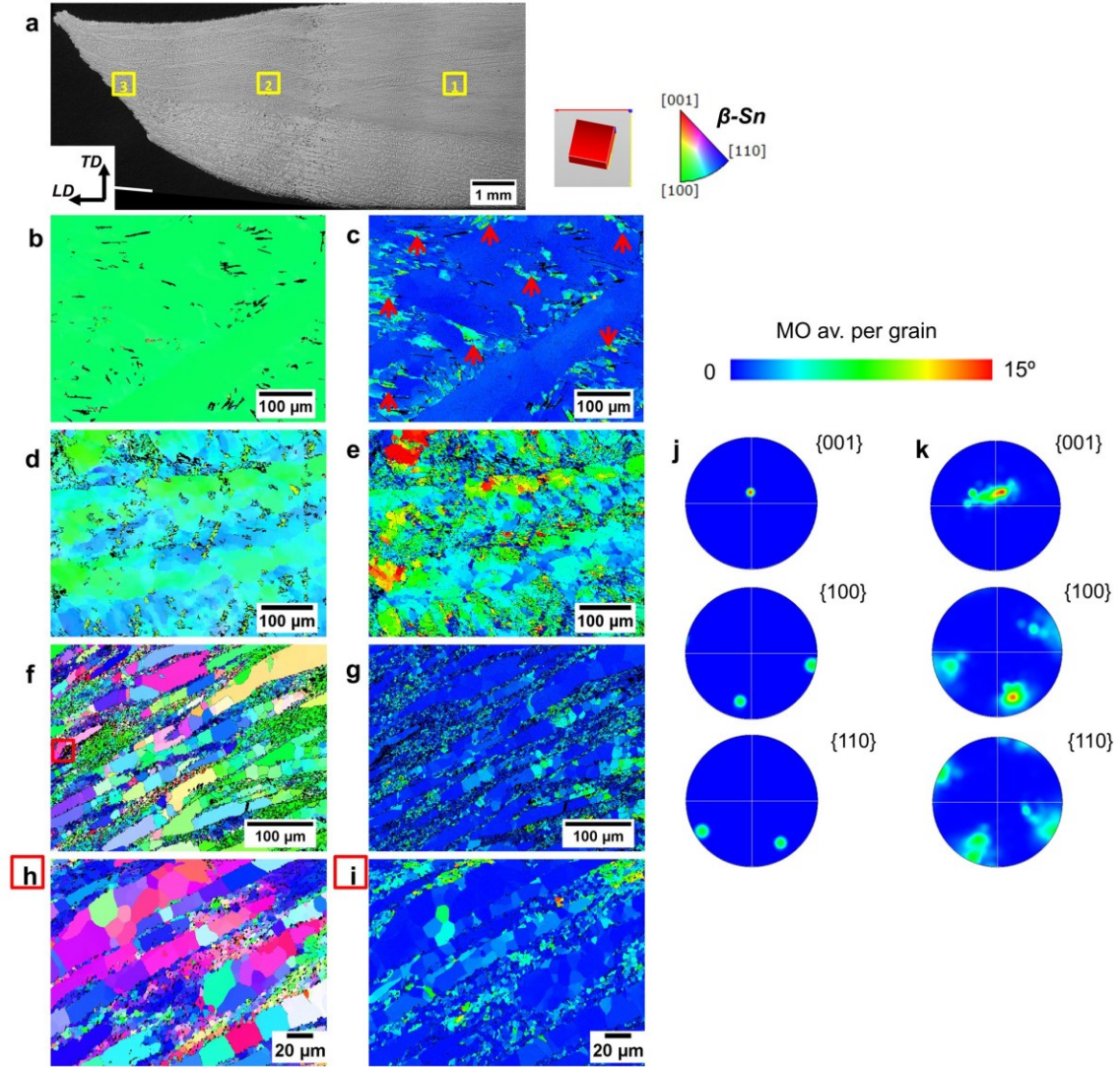


Figure 4.7. The creep fractured DS sample at ~ 36 MPa. (a) Large scale optical image at fracture surface in longitudinal plane. (b-i) EBSD maps in the yellow squared region in (a) (only the β -Sn has been indexed here), (b, c) at location 1 within the uniformly deformed region, (d, e) at location 2 in the necking region, (f, g) at location 3 near the fracture surface, (h, i) Higher magnification IPF-LD maps in the red squared region in (f). (b, d, f, h) IPF-LD maps show the evolution of crystal orientation with position. (c, e, g, i) MO av. maps show the variation of misorientation with position. (j, k) PFs of the sample at DS condition (j), at tertiary stage creep (k).

It can be seen clearly that a change from green to rainbow colour at the fracture surface (Figure 4.7f) and this indicates that stored energy has been released here (Figure 4.7g). Similar spreading in orientation in PFs is obtained between position 1 and 3 (Figure 4.7j and 4.7k) and this is similar to the samples tested at different stresses.

4.4 Summary of the Chapter

The creep performance of the large-scale DS SAC305 samples has been studied at room temperature under constant stress level in the range 17 – 44 MPa. The following conclusion can be drawn.

1. The DS samples contain two distinct microstructural regions, containing primary β -Sn dendrites and an eutectic consisting of β -Sn and fine scale IMCs. These deform differently during creep. An increase in misorientation is observed in regions close to the IMCs which highlights the role of strain gradients in these regions of high microstructure contrast. These regions dominate recrystallisation events, perhaps due to the role of stored energy which can act to initiate recrystallisation. Deformation within the eutectic and around the IMCs is highly localised, whereas the deformation within the dendrite is more spatially extensive. The difference in deformation patterning results in a change in the localisation of stored energy and therefore the likelihood of local recrystallisation. ‘Rainbow’ recrystallisation is promoted in the later stages of creep, where grains with a multiplicity of orientations are generated in the single crystal regions. The length-scale of this recrystallisation is related to the dendrite and IMCs spacing for each region.
2. The creep strain against time curves show a dominating secondary creep region for all stresses, where the creep strain rate accelerates steadily. Shorter creep lifetime and greater secondary creep strain rate are obtained at higher stresses. Table 4.3 summaries the results for all the testes at room temperature. The samples are under ductile failure and the reduction of cross-sectional area is increasing with stress.

Effect of stress on creep behaviour of SAC305 solder

Load (N)	Sample	Average σ (± 0.1 MPa)	Area (± 0.05 mm ²)	Reduction of Area	Failure mode	Microstructure
150	DS2	17.7	5.95	20%	Void coalescence Ductile	Small , Subgrain formation, Small recrystallised grain in primary and on eutectic interface
200	DS3	24.2	5.75	23%	Void coalescence + shear cup-cone failure at the bottom tip	Recrystallisation zones can be seen on the edge Red + rainbow colour
250	DS4	30.1	4.65	38%	Ragged, shear cup-cone failure	Recrystallisation (rainbow colour), segmentation of β – Sn, recrystallisation then continue to deform (orientation gradient involved)
300	DS5	36.2	3.75	50%	Shear cup-cone failure	Recrystallisation (rainbow colour), segmentation of β -Sn, recrystallisation before failed
350	DS6	42.0	3.25	57%	Shear, cup-cone failure	N/A

Table 4.3. Summary of the fractured samples tested at room temperature with information of area of reduction, failure mode, and microstructure after fracture.

3. The stress exponent, n is determined to be 10.5, which means dislocation creep is the dominating creep mechanism for DS SAC305 bulk samples.
4. The samples are under heterogeneously uniaxial deformation during creep testing. The deformation is starting with high misorientation development and leading to subgrain formation and further recrystallisation generation. Different recrystallisation mechanisms are observed between the β -Sn dendrite and eutectic regions. The eutectic regions are recrystallised at an earlier stage with much smaller grain size due to the incoherent boundaries between IMCs and β -Sn, which act as stress concentrators. The deformation then runs through the β -Sn dendrite by lattice rotation, when the grains are

rotating enough, there is enough energy to drive recrystallisation nuclei to segment the grain.

5. Overall, the DS samples tend to retain its initial microstructure, i.e. the same primary β -Sn dendrite regions and eutectic regions, even after failure. Therefore, the initial solidification microstructure of the SAC305 solder is highly relevant to its mechanical properties.

Chapter 5 Effect of temperature on creep behaviour of SAC305 solder

5.1 Introduction

In this chapter, the creep behaviour of controlled microstructures of the DS samples are investigated under constant load testing (~ 30 MPa) at a range of temperatures (20 to 200 °C). The secondary creep strain rate, the dominating creep mechanism, and its temperature dependence are explored. The microstructural evolution mechanisms of the sample are examined by EBSD and are mainly focused on the onset of secondary and tertiary stage creep. The strain field of the sample is measured by 2D DIC. The role of IMCs in the eutectic within the bulk matrix, the variation of crystal orientations and the number of grains, i.e. the presence of a grain boundary of each sample are considered during creep testing to investigate their effect on strain accumulation and microstructural evolution on creep reliability of the SAC305 solder alloys. These observations could be extended for understanding thermal loading of solder joints.

5.2 Experiment

The DS sample was fitted inside the 2 kN Gatan Mtest 2000E tensile stage for creep testing (Figure 5.1). Load controlled deformation was performed using closed loop control within the Microtest stage and the tests were carried out at a constant applied stress of 30 ± 2 MPa. Deformation was performed at a range of temperatures, including room temperature, 45, 60,

90 °C, 120, 150, 180 and 200 (± 0.4) °C. The tests were interrupted shortly after the onset of secondary stage creep (~ 120 s) and stopped at the end of tertiary stage creep. The sample was removed from the load frame and EBSD scans were performed in the SEM at these two stages. These interruptions of loading can cause statistic recovery and recrystallisation in the microstructure due to stress relaxation. The sample was then replaced within the loading frame to resume the creep test between the two EBSD scans. The EBSD maps were scanned with a step size of 6 μm and 0.4 μm for large scale and high magnification IPF maps respectively.

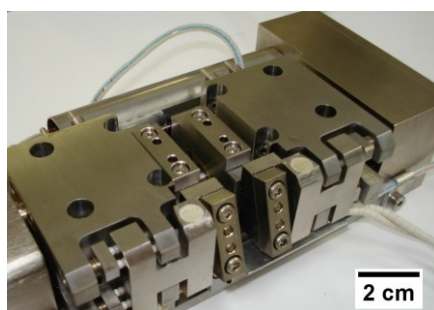


Figure 5.1. The Gatan Mtest 2000E tensile stage with a 2 kN load frame.

The stability of the stress and temperature are shown in Figure 5.2. The temperature of the sample was measured by attaching a thermocouple wire to the sample at the centre of its surface. The variation was determined as ± 2 MPa and ± 0.4 °C for the stress and temperature respectively.

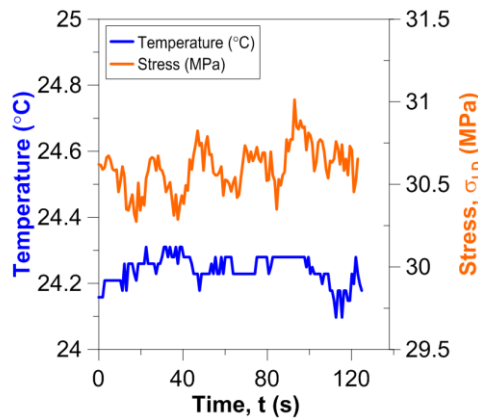


Figure 5.2. The variation of the temperature and stress during creep testing for room temperature carried out by Mtest 2000E.

The samples were prepared to a high-quality surface finish suitable for both DIC and EBSD. This was achieved using mechanical polishing with a 0.05 μm colloidal silica finish. Due to the fact that the $\beta\text{-Sn}$ is much softer than the eutectic IMCs, slight roughness was generated between the $\beta\text{-Sn}$ and IMCs during mechanical polishing. Therefore, the final specimen surface was polished with broad ion beam milling using at Gatan PECS II to firstly flatten the specimen surface and then introduced a very slight surface roughness which is suitable for DIC. The PECS II was operated in etching mode with dual modulation at a beam voltage of 3 keV and a tilt angle of 1° to flatten the IMCs, followed by 3 keV and 3° , in order to generate long strip speckle patterns on the surface, for 3 hours in total.

Creep strain curves were measured by 2D optical DIC. The curves were formed from fields of the normal strain along LD, extracted from the surface strain field plots by taking the average of each strain field map at each time-step. DIC was performed within DaVis with the following DIC settings: window size 65 pixels, overlap 16 pixels, field of view 10×2 mm, i.e. $\sim 860 \times 174$ pixels, with images captured at 14 bit depth, and an effective pixel size of $11.5 \mu\text{m}/\text{pixel}$. Each optical micrograph was captured with a QImaging Retiga QIClick™

camera attached to a Brunel Microzoom inspection microscope operating at a frame rate of 1 Hz.

5.3 Results and Discussion

5.3.1 Creep strain curves

The creep strain curves for primary creep and the early stages of secondary creep (the first 120 s) are given in Figure 5.3a for tests at 298 to 473 K. The creep strain rate vs. time and creep strain rate vs. strain curve for 298 to 473 K are given in Figure 5.3b and 5.3c respectively. These 2 figures are plotted by differentiating the creep curves from the primary to secondary stage creep (the data was smoothed before differentiation). Figure 5.3b shows the creep strain rate vs. time curves for the first 120 s of creep and the curves are fluctuated about their creep strain rate, which means all the tested samples reached their secondary stage creep with in the first 60 s of testing. From the creep strain rate vs. strain curves in Figure 5.3b, a linear relationship with a constant gradient close to 0 is presented once secondary creep is achieved, which allows the onset of secondary creep strain to be identified.

The strain at the onset of secondary creep decreases with the testing temperature (Figure 5.3d), especially between 298 K and 333 K. This might be caused by increasing dislocation climb at higher temperature (298 – 333 K), which enables recovery to more easily balance the work hardening. At even higher temperature (363 – 473 K), the deformation mechanism changes gradually with an inflection at ~343 K, as indicated in Figure 5.3e. This transformation could be a change from dislocation limited processes to diffusion limited deformation processes. Towards the melting temperature ($T_M = 490$ K) the onset of secondary creep strain rate tends

towards zero. It is anticipated that onset of secondary creep will occur at even greater strains at lower temperatures, as there is a total reduction in creep deformation at these lower temperatures due to the thermal activation of dislocation motion.

Figure 5.3e shows a plot of $\ln$ (secondary creep strain rate) vs. the reciprocal temperature. Analysis of the gradient of this data enables calculation of the activation energy, Q . The data can be represented by two different temperature ranges with separate linear fits. From room temperature to 333 K the Q is 41 kJ/mol, implying that a climb-controlled dislocation mechanism is the predominant mechanism in this low temperature regime. This value is within the range of 40 - 65 kJ/mol indicated from other works tested between room temperature to 393 K [59, 68, 80]. A much flatter slope occurs beyond 363 K with a Q value of 6 kJ/mol, suggesting a fast diffusion path might take place, probably due to the change into a lattice diffusion mechanism assisted by recovery and recrystallisation in the high temperature regime. This is also supported through observation of a decrease in the strain at onset of secondary creep at higher temperatures (Figure 5.3d), i.e. recovery process happens at lower strain level because of less dislocation formation and motion, but more diffusion occurs. This change in creep mechanism happens at $\frac{T}{T_M} \sim 0.7$, ~ 343 K, which is in agreement with some literature [64, 65, 74] that the change in mechanism from climb-controlled dislocation creep to lattice diffusion creep takes place at 70 - 80% of the melting temperature depending upon the initial microstructure of samples.

The creep curves from the onset of secondary creep until a creep strain of $\sim 10\%$ are shown in Figure 5.3f (obtained by 2D DIC). A shorter secondary creep time (and strain, Figure 5.3f) was

obtained with increasing temperature. The creep strain magnitude at failure is attributable to the temperature and also the initial crystal orientation and number of β -Sn grains in the sample.

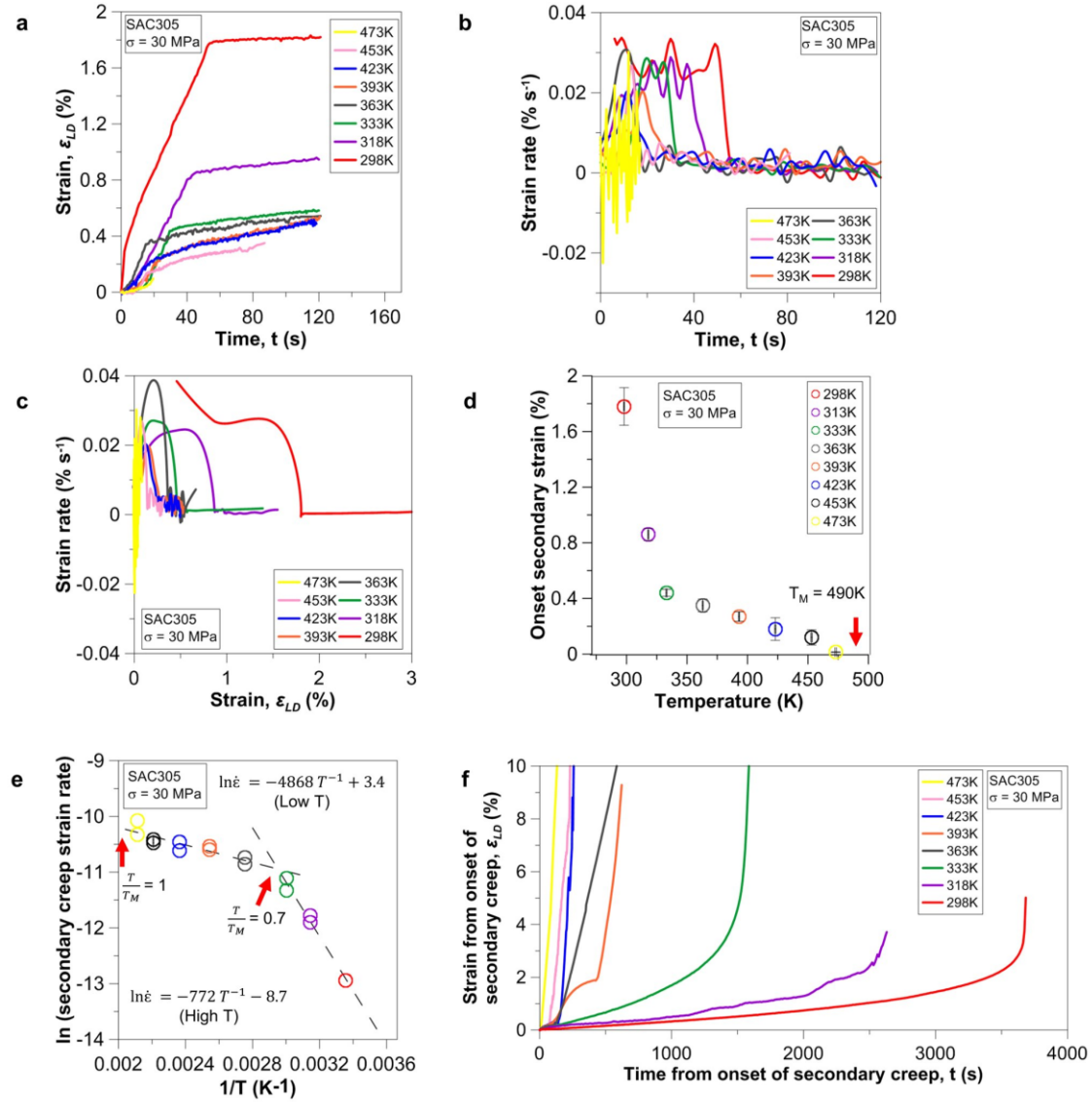


Figure 5.3. Fixed load (30 MPa) mechanical creep data from 298 to 473 K of SAC305 samples grown at $v=20\mu\text{m/s}$. (a) Creep curves from primary to early secondary stages creep. (b) Creep strain rate vs. time from primary to early secondary stages creep showing when the secondary stage creep is reached. (c) Creep strain rate vs. creep strain curves showing a gradient of 0 after the secondary stage creep is reached. (d) Variation of the onset of secondary creep with respect to temperature. (e) Creep model analysis of $\ln$ (secondary creep strain rate) vs. reciprocal temperature with two creep domains (high temperature and low temperature). (f) Creep curves from the onset of secondary to tertiary stage creep (maximum strain = 10%). (The symbols are included for completeness and represent orientations, as described in Table 5.1).

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Table 5.1 summarises the mechanical data from the creep tests together with microstructural information such as the orientation and number of β -Sn grains in each sample. The secondary creep strain rates increase with increasing temperature from the order of 10^{-4} to $10^{-3} \% s^{-1}$. The samples containing an initial near [110] crystal orientation achieve a relatively low creep strain at the end of testing, 6.8% and 5.5% for 298 and 318 K (low temperature regime: 298 – 333K), and 26.2% and 34.2% for 453 and 473 K (high temperature regime: 363 – 473 K), whilst the samples with main orientations near [100] achieve a higher creep strain 16.9% and 42.3% at fracture for the lower and higher temperature regime respectively. (The sample tested at 393 K was not fractured). In tension, a total creep strain before failure can be larger along [100] crystal direction, than [110]. The process of microstructural change is explored by EBSD in the next section.

T [K]	Strain at onset of secondary creep (%)	Secondary creep strain rate (s^{-1})	Failure creep strain (%)	Number of β -Sn grains	β -Sn Orientation measured by EBSD
298	1.78	3.79×10^{-6}	6.8	1	[110]
318	0.80	7.65×10^{-6}	5.5	3	[110] [110] [100]
	0.86	6.76×10^{-6}	9.8	2	[110] [110]
333	0.44	1.50×10^{-5}	13.6	3	[100] [100] [110]
	0.49	1.20×10^{-5}	7.2 (not fractured)	-	-
363	0.38	2.18×10^{-5}	10.7	3	[100] [110] [100]
	0.35	1.92×10^{-5}	16.9	1	[100]
393	0.30	2.51×10^{-5}	22.2	3	[100] [110] [100]
	0.27	2.65×10^{-5}	9.6 (not fractured)	2	[110] [100]
423	0.15	2.87×10^{-5}	42.1	3	[110] [110] [110]
	0.18	2.46×10^{-5}	25.2	-	-
453	0.09	2.82×10^{-5}	29.4	3	[110] [110] [110]
	0.12	2.92×10^{-5}	26.3	-	-
473	0.01	3.26×10^{-5}	29.8	2	[110] [110]
	0.01	4.21×10^{-5}	23.9 (not fractured)	-	-

Table 5.1. Summary of mechanical creep SAC305 samples grown at $v=20 \mu m/s$ under fixed load of ~ 30 MPa testing from 298 to 473 K showing the values of secondary creep strain rate, strain at onset of secondary creep, failure creep strain and grain orientation of the samples.

5.3.2 [110] single crystal sample at room temperature

5.3.2.1 Strain field and crystal orientation

A mainly single crystal orientation sample, which is close to [110] in the loading direction was deformed at room temperature, i.e. $\sim 298\text{ K}$ ($\frac{T}{T_M} \sim 0.60$). The crystal unit cells of the orientations are shown with the main orientation highlighted by a red square together with the IPF-LD map (Figure 5.4). Within this deformed gauge, the dendrite arms are bent and thinned, likely generated within tertiary stage creep, where the dendrite and eutectic regions are indicated with red and yellow arrows (Figure 5.4g). The variation in creep strain with respect to time and location of sample is illustrated in frequency histograms (Figure 5.4h and 5.14i) at 2 different locations 1 and 2 (as labelled in Figure 5.4b and 5.14f).

From the strain field (Figure 5.4 b and 5.4c), the sample deformed with a little heterogeneity from the primary to secondary stage creep, but, in general, there is fairly homogenous deformation. It reaches an average strain level of 1.8% at the onset of secondary stage creep for both Location 1 and 2 (Figure 5.4h).

With further straining (Figure 5.4 d - 5.4f), a bending neck formed gradually for this single [110] crystal sample at fracture, which reveals the gradual increase in heterogeneity during deformation. The deformation of the sample is ductile and relatively stable in this creep regime. From the IPF-LD map after fracture (Figure 5.4g), the gauge of the sample is elongated approx. 7% of its original length and the cross-sectional area is reduced approx. 18% through the gauge and 24% at the fracture surface. The fracture took place from the red squared region in Figure 5.4g.

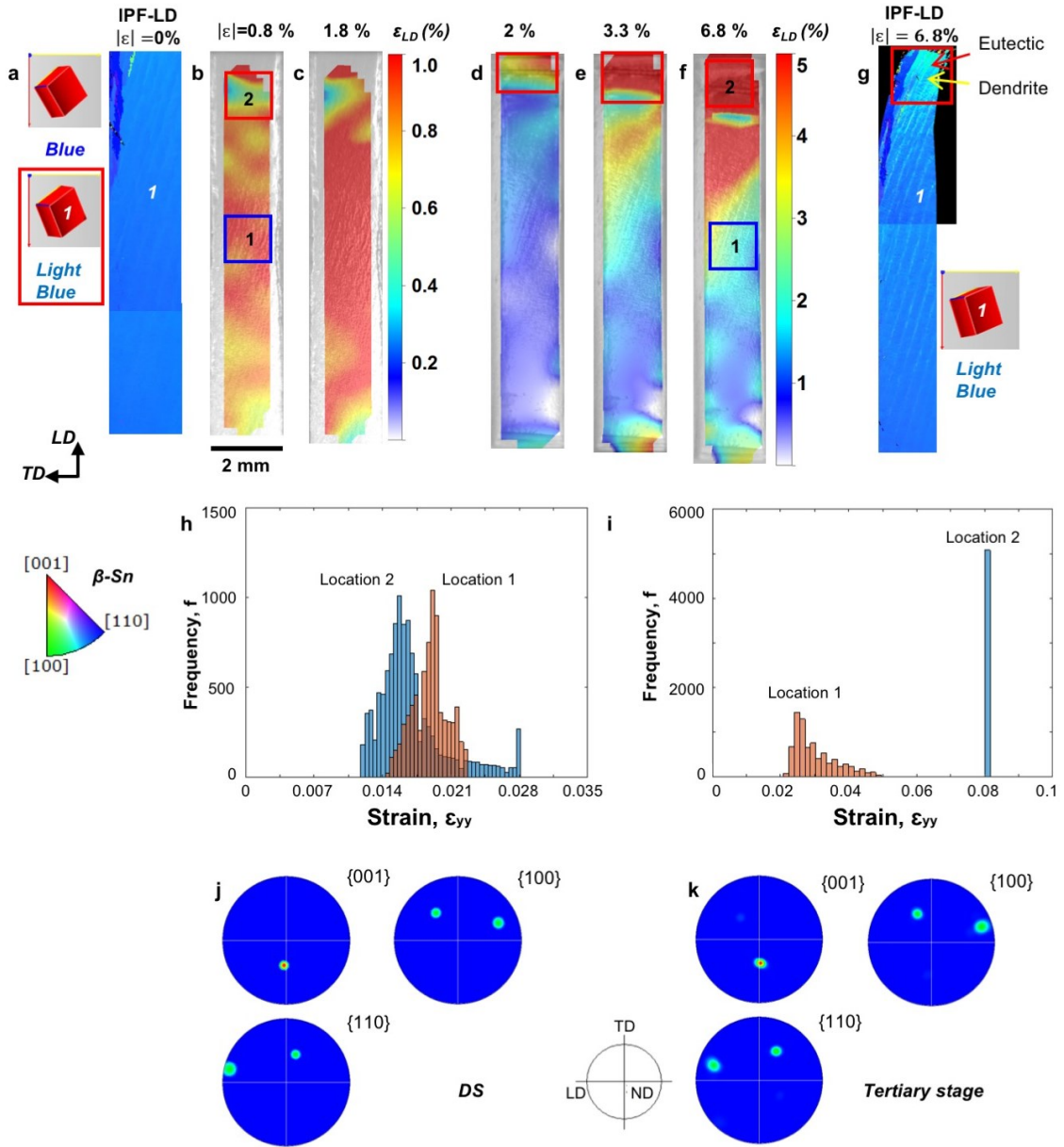


Figure 5.4. DIC figures and EBSD maps of a ‘coarse’ microstructure single grain sample tested at room temperature (298 K). (a) The IPF-LD map after DS with crystal orientations shown (the main orientation highlighted with red square). (b, c) DIC figures interrupted near onset of secondary stage creep at strain level (ϵ_{LD}) 0.8% and 1.8% respectively. (d-f) DIC figures stopped at tertiary stage creep (fracture) at strain level 2%, 3.3% and 6.8% respectively. (g) IPF-LD map at fracture with the crystal unit cell of the main orientation shown. (h, i) Frequency vs. local strain histogram at locations 1 and 2 shown at onset secondary stage creep (h), and at fracture (i). (j, k) PFs of the sample at DS condition (j), at tertiary stage creep (k).

The heterogeneity in strain development is increased as shown in Figure 5.13i (extracted from Figure 5.13f). Location 2 at the fracture surface generates 8% of strain, while Location 1 has approximately 3% of strain generated.

The second crystal orientation, green, is appearing in the eutectic regions during creep, which is indicated with red arrows in Figure 5.4g. The main orientation changed smoothly by rotating towards [100] (by comparing the unit cell 1 in Figure 5.4a and 5.4g). The corresponding crystal unit cell shows slight rotation for the main orientation (Figure 5.4g). A little spread in the PF is observed by comparing the as-DS condition (Figure 5.4h) with the one in tertiary stage creep (Figure 5.4i). Recrystallisation is only observed near the fracture surface and the detail of the microstructure is given in the next section (Figure 5.5 and Figure 5.6).

5.3.2.2 Microstructure of the sample within non-necking region

The microstructure of the single-crystal sample within the non-necking region is shown in Figure 5.5 for different stages of creep. It shows a clear single crystal orientation in the IPF-LD map (Figure 5.5 b and 5.5d) for both the DS condition and at the onset of secondary stage creep. The misorientation (as indicated with higher misorientation to average, MO av. with MO av. per grain map colour key 0 - 5°) starts to develop in the eutectic region around IMCs (indicated with red arrows in Figure 5.5e) at the onset of secondary stage creep, which is related to the incoherent boundaries between IMCs and β -Sn (a common misorientation angle with respect to 'grain' as identified with EBSD thresholding of grain boundaries at 5°). However, no special indication shows which IMCs, i.e. Ag_3Sn and Cu_6Sn_5 localises more strain.

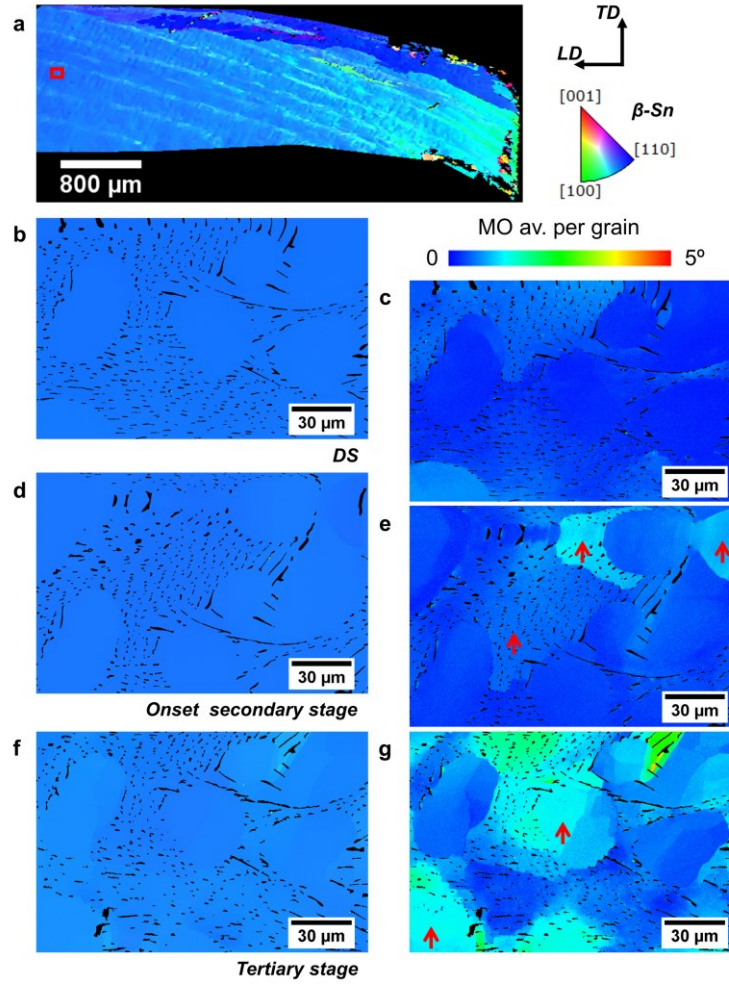


Figure 5.5. EBSD crystal orientation mapping showing evolution of microstructural heterogeneity highlighting evolution within the uniformly deformed region of the single crystal sample (only the β -Sn has been indexed here). (a) Large scale IPF-LD map at fracture. (b-g) Higher magnification IPF-LD maps of the red squared region in (a) within the non-necking region at different stages of creep, (b, c) at the DS condition, (d, e) at onset of secondary stage creep, and (f, g) at the tertiary stage creep. With increasing strain, there is slight change in the orientation in the IPF-LD map (b, d, f) but the MO av. maps show significant increase, especially in eutectic regions (the MO hot spots are indicated with red arrows) (c, e, g).

The misorientation in β -Sn dendrites has no obvious change from primary to secondary stage creep, while the eutectic regions show increased misorientation (Figure 5.5c and 5.5e). A significant increase in misorientation is observed at the tertiary stage creep and the high misorientation regions are mostly localised in the eutectic regions (Figure 5.5g). The crystal orientation (Figure 5.5f) is changed slightly by rotating towards the [100] direction and Figure

5.5g shows that misorientation starts to develop in the pure β -Sn regions and subgrains are formed.

5.3.2.3 Microstructure of the sample within the necking region

The role of the IMCs on heterogeneous creep deformation is illustrated within a necked region of the single crystal sample deformed at room temperature, as shown in Figure 5.6. The undeformed crystal orientation of this single crystal has an orientation close to [110] (i.e. a darker blue) which is observed within the dendrite regions. The intermetallic-containing eutectic regions show significant β -Sn lattice rotation towards the [100] crystal direction (in green / cyan), indicating that there is more creep deformation within these microstructural regions, and this results in a clear demarcation of the underlying microstructure within the IPF map in Figure 5.6a.

At the fracture surface, recrystallisation developed, which is very localised within this field (Figure 5.6a). The orientation field is also heavily modulated by the presence of the IMCs and the dendrite structure around them. The higher magnification FSE map within the bending region (Figure 5.6b), shows that the flow of β -Sn around the IMCs, which have popped out from the sample matrix at the highly-strained region, leads to elongation of the β -Sn dendrites and increases the surface roughness (Figure 5.6a). The main crystal orientation is moving close to [100] (Figure 5.6c). This implies that the initial crystal orientation is rotated gradually during creep to form the green-cyan orientation near [100] at the fracture surface (Figure 5.6e). The second orientation (yellow, which is indicated with red arrows) is appearing in the eutectic regions near IMCs (Figure 5.6c).

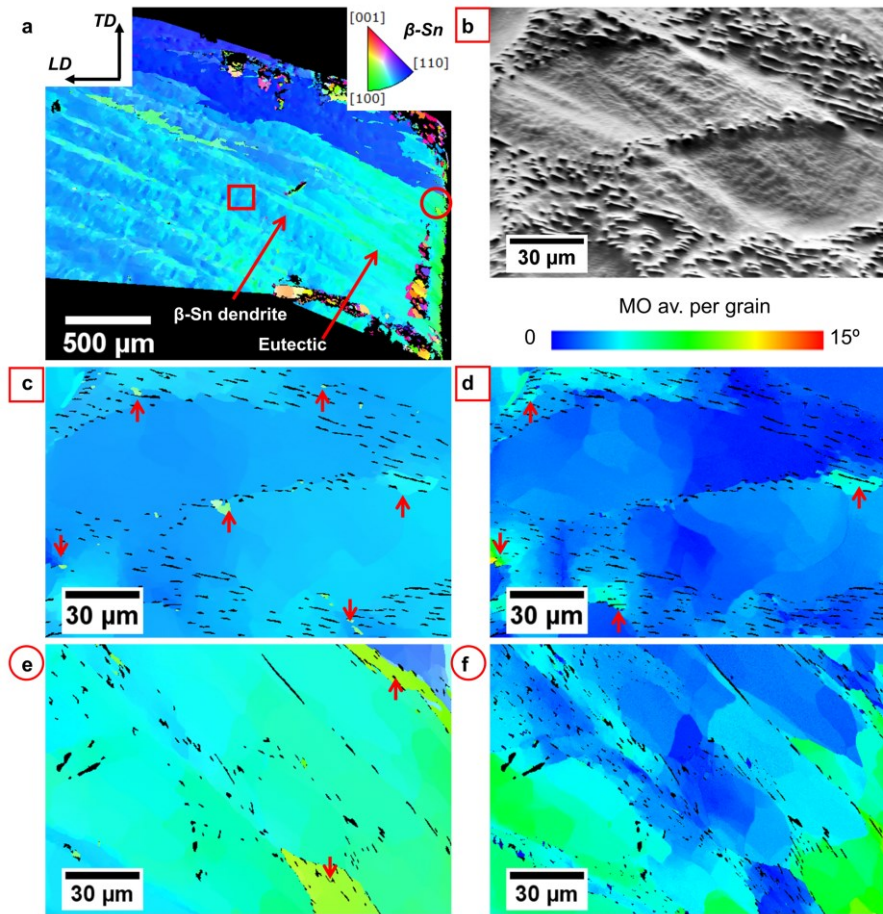


Figure 5.6. EBSD crystal orientation mapping showing evolution of microstructural heterogeneity highlighting evolution within the bending neck (only the β -Sn has been indexed here). (a) Large scale IPF-LD map at fracture. (b-f) Higher magnification maps of the same region in (a) within the bending region, (b-d) in the red squared region, (e, f) in the red circled region at the fracture surface. With increasing strain, there is significant increase in surface roughness in the forescatter electron image (FSE) (b), generation of recrystallised grain in the eutectic region (indicated with red arrows) (c, e), and increase in misorientation in the MO av. map (d, f).

Locations within the sample have high MO av., which increases greatly to 15°, as given in Figure 5.6d with MO av. per grain map colour key 0 - 15°. A high MO av. implies significant local constraint which results in an increase in crystal rotation ^[142]. Furthermore, the closer the distance to the fracture surface, the higher the misorientation of the sample (Figure 5.6d and 5.6f). The MO av. illustrated in Figure 5.6f has a much higher value at the fracture surface, indicating that there is a significantly stored energy within this region. Figure 5.6d and 5.6f also show strong subgrain formation in both the β -Sn dendrite and eutectic regions. The eutectic

β -Sn regions have more rotation relative to the β -Sn dendrites (Figure 5.6e) and a new crystal orientation with a component towards [001] (yellow) develops more in the eutectic regions.

5.3.3 [100] [100] [110] tri-crystal sample at 333 K

5.3.3.1 Strain field and crystal orientation

This section overviews a tri-crystal sample deformed at 333 K ($\frac{T}{T_M} \sim 0.68$). The large scale IPF-LD map shows the 3 crystal orientations which are near [100] and [110] in loading direction (Figure 5.7a). The DIC strain field shows that the sample generates a strong heterogeneity even from the primary to secondary stage creep (Figure 5.7b and 5.7c). The sample has a mean strain level of 0.44% at the onset of secondary stage creep (Figure 5.7b and 5.7c). And no preference is indicated regarding which grain localised more strain among the 3.

As the deformation continues (Figure 5.7d - 5.7f), most of the strain is accommodated at the red squared region, which fractured at the end of the test. A cup-cone fracture neck surface (w-type fracture surface) is observed. The length of the sample has been elongated over 13% and its cross-sectional area reduced 40% at the fracture surface and 6% outside the necking region, which shows a slight decrease of the ductility but more localisation of strain comparing to the single [110] sample (Figure 5.4).

The orientation of the 3 grains changes gradually by rotation. Figure 5.7a and 5.7g show that the orientations for cyan (1) cyan and green (2) green are rotated towards [110] and [001] to form a more cyan and green-yellow colour in the necked region respectively, while the orientation for purple (3) is not notably deformed. At the fracture surface (Figure 5.7g), the orientation for cyan (1) and green (2) have been developed more, while the purple (3)

orientation is thinning in width. This might be caused by strain induced grain boundary mobility. Figure 5.7h and 5.7i show that the orientations are rotated in $\{001\}$ PF and stayed the same in the $\{100\}$ PF. Therefore, it looks like the $[100]$ is the rotational axis. The detail of the microstructure evolution is given in the next section (Figure 5.8 and Figure 5.9).

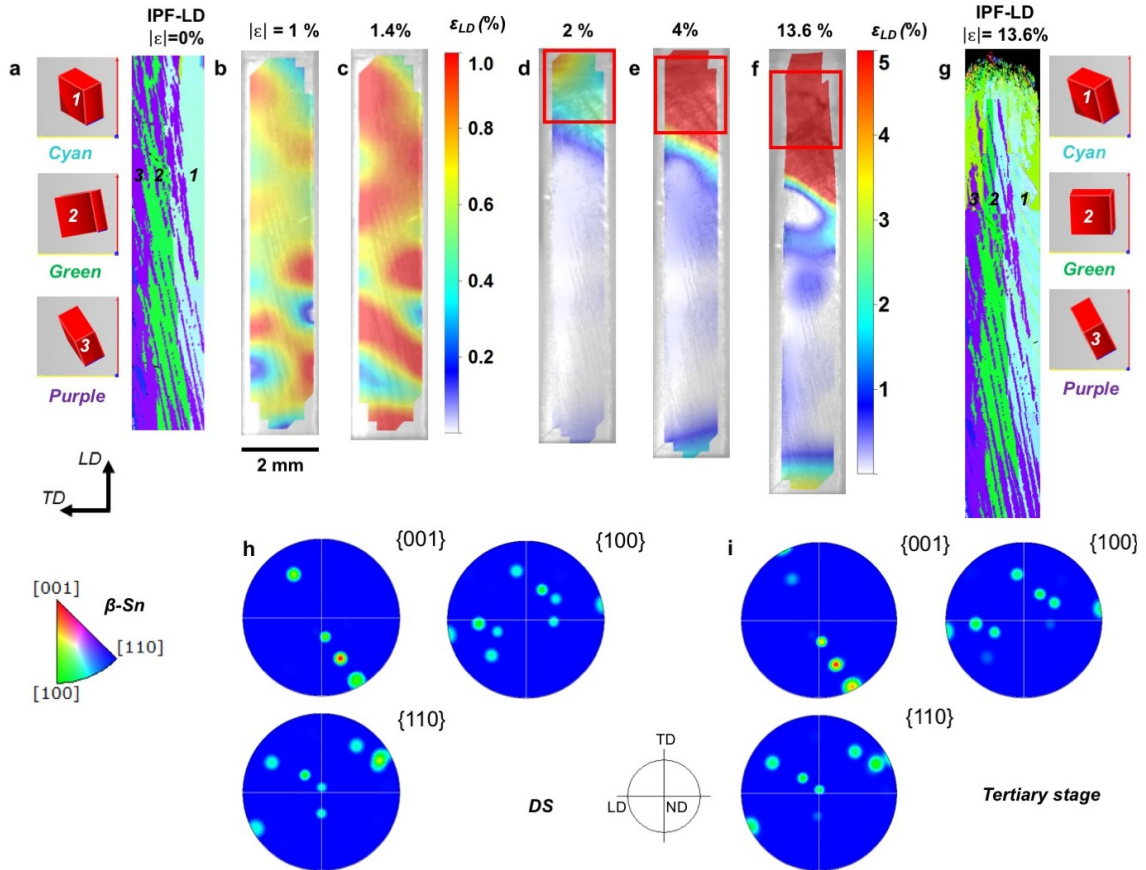


Figure 5.7. The DIC figures and EBSD maps of a 'coarse' microstructure tri-crystal sample crept at 333 K. (a) IPF-LD map after DS ($\epsilon_{LD} = 0\%$) with main crystal orientation shown. (b-c) DIC figures interrupted near onset of secondary stage creep at strain level (ϵ_{LD}) 1% and 1.4% respectively. (d-f) DIC figures at tertiary stage creep (fracture) at strain level 2%, 4% and 13.6% respectively. (g) IPF-LD map at fracture ($\epsilon_{LD} = 13.6\%$) with the main orientation shown. PFs of the sample at DS condition (h), at tertiary stage creep (i).

5.3.3.2 Microstructure of the sample within non-necking region

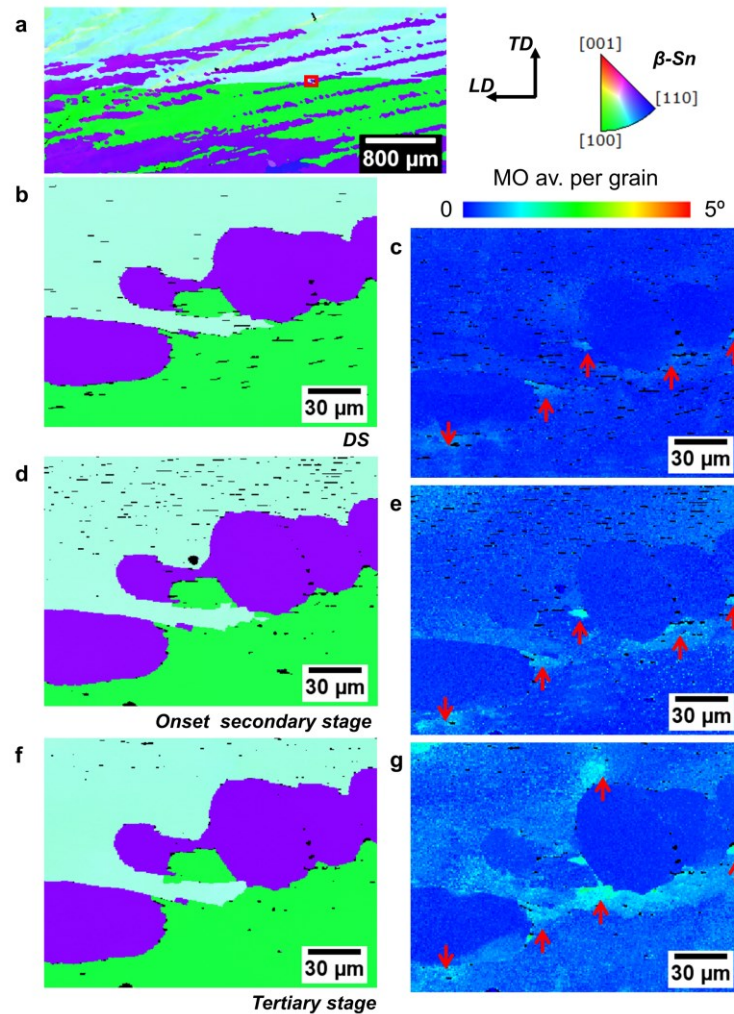


Figure 5.8. EBSD crystal orientation mapping showing evolution of microstructural heterogeneity highlighting evolution within the uniformly deformed region of the single crystal sample (only the β -Sn has been indexed here). (a) Large scale IPF-LD map at fracture. (b-g) Higher magnification IPF-LD maps of the red squared region in (a) within the non-necking region at different stages of creep, (b, c) at the DS condition, (d, e) at onset of secondary stage creep, and (f, g) at the tertiary stage creep. With increasing strain, there is slight change in the orientation in the IPF-LD map (b, d, f) but the MO av. maps show significant increase, especially in eutectic regions and near grain boundaries (the MO hot spots are indicated with red arrows) (c, e, g).

The high magnification IPF-LD maps (Figure 5.8b - 5.8g) are taken at the interface between the 3 grains at different stages of creep in the red-squared region within the non-necking region in Figure 5.8a. In the as-DS condition, the IPF-LD map shows 3 different colours (Figure 5.8b),

indicating the 3 orientations. The misorientation hot spots are presented in the as DS-ed condition in the MO av. map (Figure 5.8c) around IMCs near grain boundaries (indicated with red arrows), while the β -Sn dendrite is nearly subgrain free. The misorientation starts to develop within eutectic regions, especially at grain boundaries from primary to secondary stage creep (Figure 5.8e), but not very much in the regions of β -Sn dendrites. From the secondary to tertiary stage creep (Figure 5.8f and 5.8g), significant subgrain formation is developed in the eutectic regions and the length-scale is modulated by the spacing of the IMCs (Figure 5.8g). The subgrain structure starts to grow into the primary β -Sn dendrites and the length-scale is controlled by IMCs as well. However, no obvious orientation change has been observed in the IPF-LD maps (Figure 5.8b and 5.8f).

5.3.3.3 Microstructure of the sample within necking region

Within the necked region (Figure 5.9a), a larger volume of recrystallisation has been generated comparing to the single [110] crystal sample (Figure 5.4). The FSE image in the necked region highlighted by the red square (Figure 5.9b) shows the dendrite arms are strongly elongated and tilted towards LD and the increase in surface roughness is more notable than the single-crystal sample (Figure 5.4). This is likely due to the more recrystallisation volume and less homogenous deformation were generated in the process. Moreover, a few microvoids are formed as indicated with red arrows and they are located near the dendrite – eutectic interface. Figure 5.9c is the IPF-LD map of the red squared region, which shows a change in orientation to yellow / orange colour (close to [001]). The corresponding misorientation (Figure 5.9d) increases greatly across the map with a large number of subgrains formed in both eutectic regions and dendrites. The highest misorientation spots appear near the location of the

microvoids which are pointed out with white arrows in Figure 5.9d. Recrystallisation can be seen near the fracture surface (Figure 5.9e), the recrystallised grains in the eutectic regions have much smaller grain size than the ones in the β -Sn dendrites.

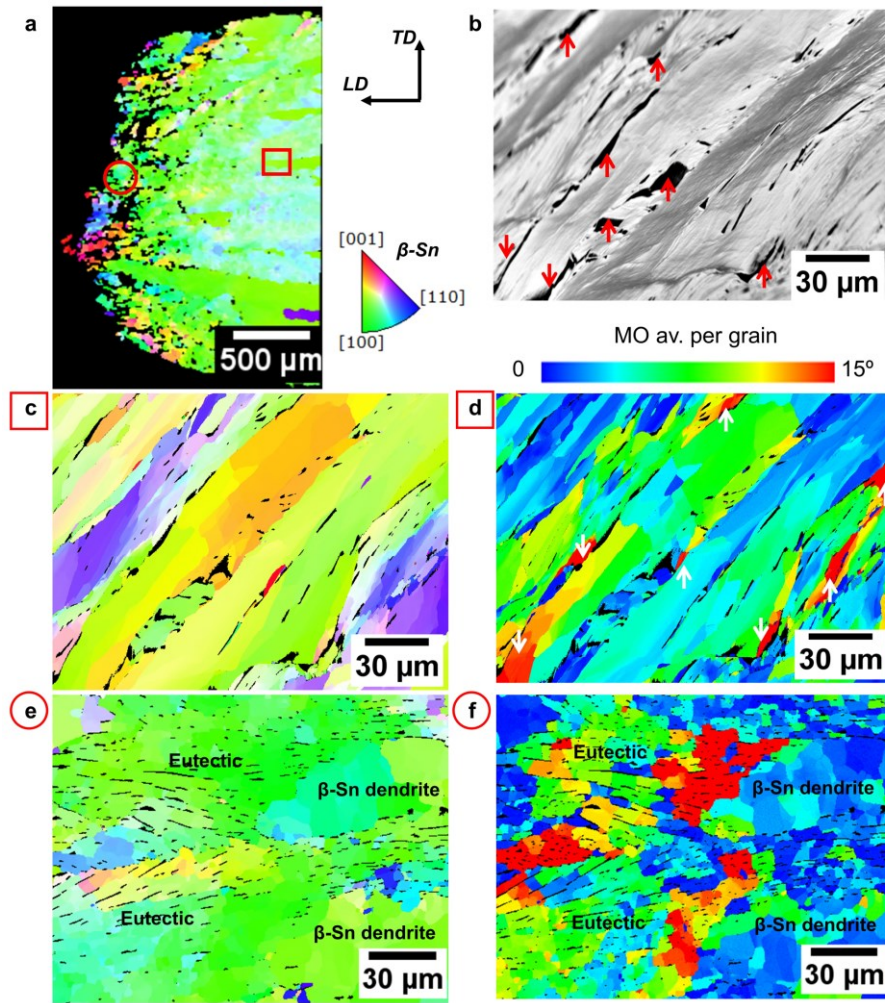


Figure 5.9. EBSD crystal orientation mapping showing evolution of microstructural heterogeneity highlighting evolution within the necking region (only the β -Sn has been indexed here). (a) Large scale IPF-LD map at fracture. (b-f) Higher magnification maps of the same region in (a) within the necking region, (b-d) in the red squared region, (e, f) in the red circled region at the fracture surface. With increasing strain, there is significant increase in surface roughness in the FSE (b), generation of recrystallised grain in the eutectic region (c, e), and increase in misorientation in the MO av. map (d, f). (e, f) are taken from the polished surface of the sample after fracture.

5.3.4 [100] single crystal sample at 363 K

5.3.4.1 Strain field and crystal orientation

A single grain crystal sample with main [100] orientation in the loading direction was deformed at 363 K ($\frac{T}{T_M} \sim 0.74$). Its main orientation is given by the crystal unit cell in Figure 5.10a.

Similar to the [110] single-crystal sample in Figure 5.4, this sample is deformed relatively homogenous from primary to secondary stage creep according to the strain field in Figure 5.10b and 5.10c. It reaches a lower strain level at the onset of secondary creep approx. 0.35%, which is the result of the higher temperature experienced comparing to the [110] single crystal sample.

As the testing continues, i.e. from secondary to tertiary stage creep, more strain is accommodated at the red-squared region in Figure 5.10d – 5.10f. The sample necked at the highly-strained region and fractured gradually within the necked region. During necking, there are both significant localisation of creep strain and surface roughening (Figure 5.10f) within the region. The elongation of the sample is approx. 17% in length and the cross-sectional area reduces approx. 55% at the fracture surface while only reducing ~3% for the rest of places. The IPF-LD map at fracture (Figure 5.10g) shows that the generation of recrystallisation and formation of new grain orientations are very localised at the fracture surface. The fracture happens from the top right side (Figure 5.10g) with more recrystallised grains in [110], which means that grain boundaries act as strain concentrators and lead to fracture of the sample. Significant amount of strain localisation takes place at the unstable neck, which indicates that this [100] single-crystal is deformed with less ductility than the tested [110] single crystal sample (Figure 5.4). The corresponding PFs (Figure 5.10h and 5.10i) show the orientation spread for all 3 directions presented. The {001} shows relatively small spreading among the 3

directions. Gradual rotation to $[110]$ is observed from the IPF-LD map (Figure 5.10g), likely with a $[001]$ rotational axis.

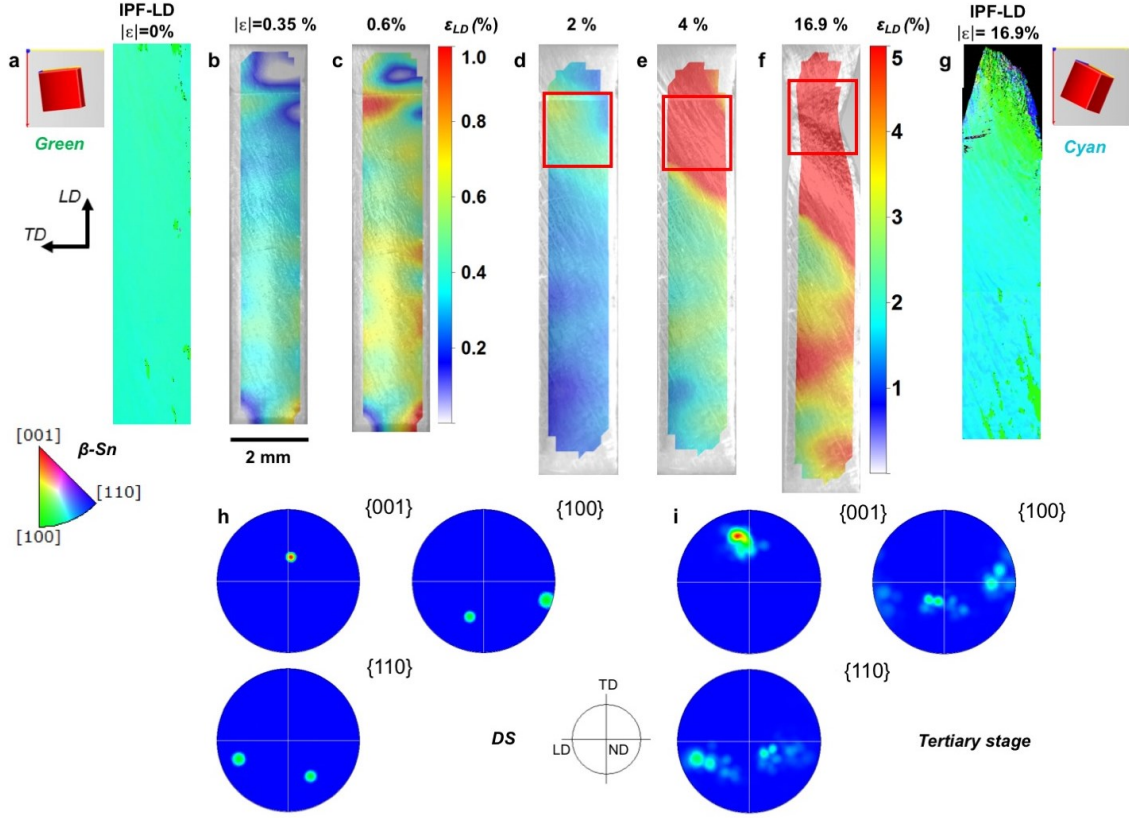


Figure 5.10. DIC figures and EBSD maps of a ‘coarse’ microstructure single-crystal sample crept at 363 K. (a) IPF-LD map after DS ($\epsilon_{LD} = 0\%$) with main crystal orientation shown. (b-c) DIC figures interrupted near onset of secondary stage creep at strain level (ϵ_{LD}) 0.35% and 0.6% respectively. (d-f) DIC figures at tertiary stage creep (fracture) at strain level 2%, 4% and 16.9% respectively. (g) IPF-LD map at fracture ($\epsilon_{LD} = 16.9\%$) with the main orientation shown. PFs of the sample at DS condition (h), at tertiary stage creep (i).

5.3.4.2 Microstructure of the sample within non-necking region

High magnification images are given at different stages of creep within the red-squared region in the non-necking region in Figure 5.11a. The same development of misorientation from the DS-ed to tertiary stage creep is observed the same as the samples tested at room temperature and 363 K (Figure 5.4 and Figure 5.7).

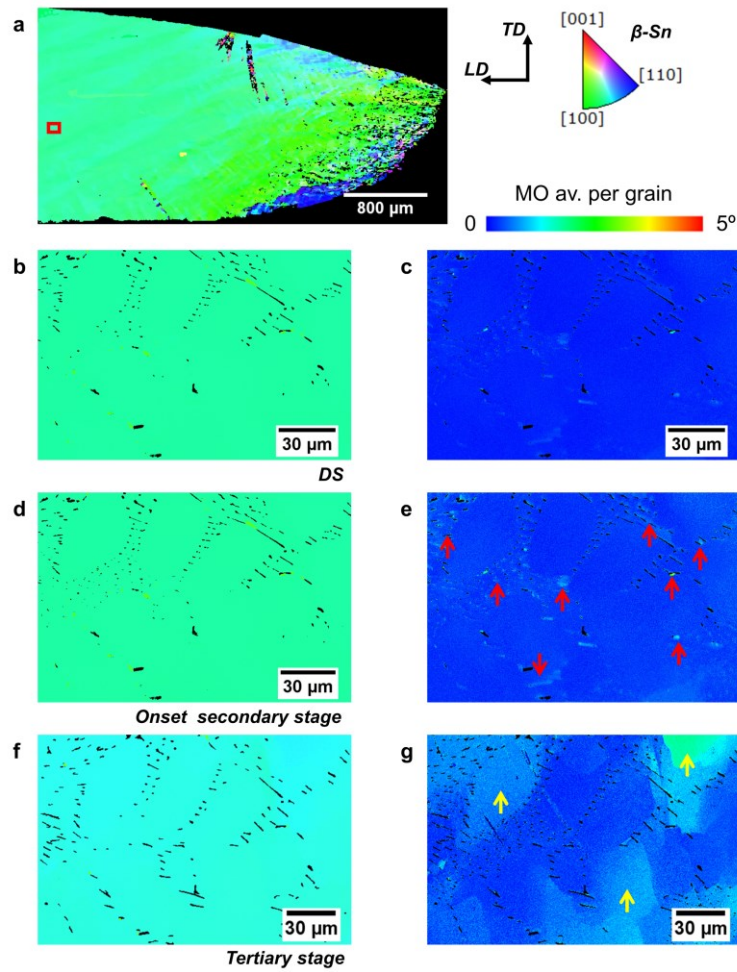


Figure 5.11. EBSD crystal orientation mapping showing evolution of microstructural heterogeneity highlighting evolution within the uniformly deformed region of the single crystal sample (only the β -Sn has been indexed here). (a) Large scale IPF-LD map at fracture. (b-g) Higher magnification IPF-LD maps of the red squared region in (a) within the non-necking region at different stages of creep, (b, c) at the DS condition, (d, e) at onset of secondary stage creep, and (f, g) at the tertiary stage creep. With increasing strain, the orientation changed slightly in the IPF-LD map (b, d, f), the misorientation increased significantly in the MO av. maps (c, e, g), firstly in eutectic regions (e) and then in the pure β -Sn dendrites (g) (the MO hot spots are indicated with arrows).

Initially, the misorientation is often localised in the eutectic region near IMCs at as-DS stage (Figure 5.11c) and at the onset of secondary stage creep (Figure 5.11e), where the misorientations are developed in the eutectic region near IMCs (indicated with red arrows). It looks like that the orientation changes smoothly by rotation towards [110] and the colour of the IPF-LD maps change visibly from green (Figure 5.11b and 5.11d) to cyan (Figure 5.11f).

5.3.4.3 Microstructure of the sample within necking region

The as-DS sample has a mainly single crystal orientation close to [100] (i.e. a green colour) which is observed within both the dendrite and eutectic regions. Slightly stronger deformation is observed in the IMC-containing eutectic regions, which shows more significant β -Sn lattice rotation towards the [110] crystal direction with a cyan colour (Figure 5.12a).

Within the necking region (Figure 5.12a), it shows a green / cyan colour in the IPF-LD map (Figure 5.12b), but the misorientation has increased greatly with a large amount of subgrain formed (Figure 5.12c). The size of the subgrains in the eutectic regions is much smaller than the ones in dendrites. The length-scale of the subgrains is controlled by the spacing of the IMCs.

At the fracture tip, there is a significant increase in surface roughness (Figure 5.12d) and decrease in misorientation (Figure 5.12f). Significant grain recrystallisation and tearing microvoids (labelled with annotation) are observed (Figure 5.12e). The small recrystallised grains, namely, red, pink and yellow orientations are formed by rotating from the parent green orientation and the microvoids appear in the eutectic regions (Figure 5.12e). The new grains have crystal orientation close to [110] (indicated with red arrows) and have a similar length-scale as the formed subgrain in the early stage (Figure 5.11f and 5.11g). More recrystallised grains are obtained from the fracture side (bottom side of Figure 5.12a) and they are much closer to [110] with a blue / purple colour. The constrained misorientation has been released due to the formation of the microvoids and fracture of the sample (Figure 5.12f).

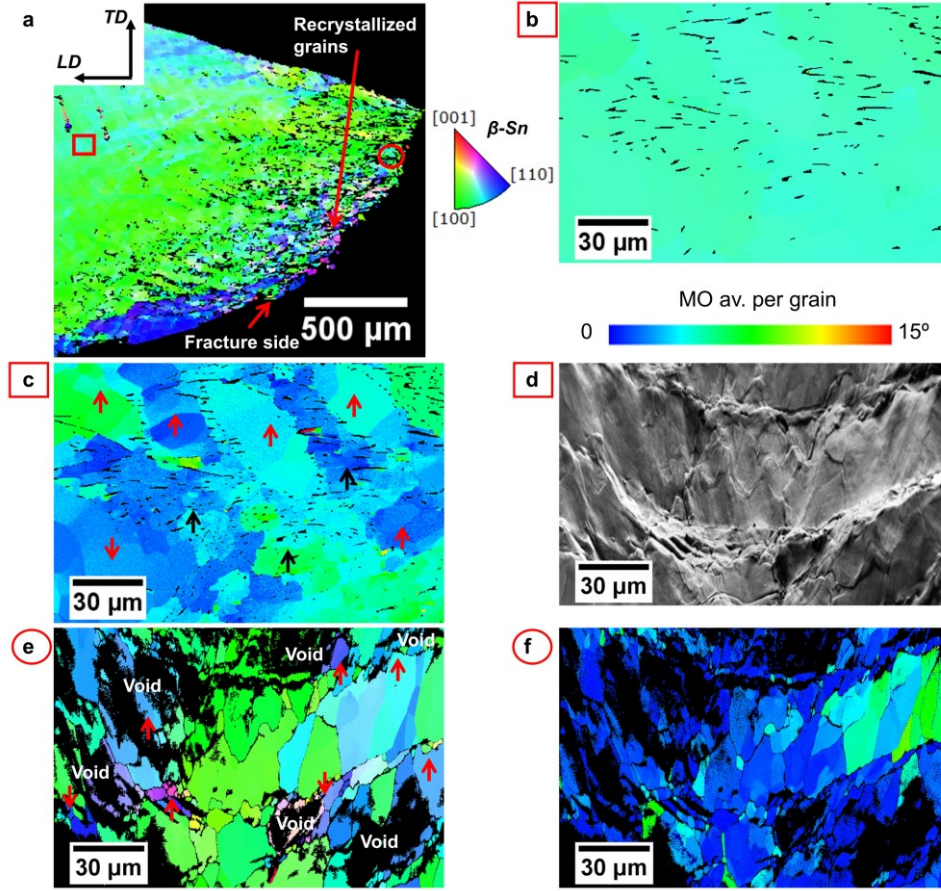


Figure 5.12. EBSD crystal orientation mapping showing evolution of microstructural heterogeneity highlighting evolution within the necking region (only the β -Sn has been indexed here). (a) Large scale IPF-LD map at fracture. (b-f) Higher magnification maps of the same region in (a) within the necking region, (b, c) in the red squared region, (d-f) in the red circled region at the fracture surface. With increasing strain, there is significant increase in surface roughness in the FSE (d), generation of subgrains (b) and recrystallised grains (e), and change in misorientation in the MO av. map (c, f).

5.3.5 [100] [110] bi-crystal sample at 393 K

5.3.5.1 Strain field and crystal orientation

The large scale IPF map in Figure 5.13a shows the initial crystal orientations and grain structure of the bi-crystal sample tested at 393 K ($\frac{T}{T_M} \sim 0.80$). There are two major crystal orientations and these two orientations are near [110] and [100] in the loading direction.

The DIC figures show that the bi-crystal sample has significant deformation heterogeneity in crystal length-scale (i.e. mm) during creep straining. The high local strain starts to develop within the red squared section in Figure 5.13b and most of the creep strain initiated at the interface between the blue [110] and the green [100] crystal orientations on the IPF-LD map in Figure 5.13a, and the [110] grain seems to accumulate more creep strain at this stress level. The sample has a mean strain of 0.54% at the onset of secondary stage creep (Figure 5.13b and 5.13c).

The heterogeneity in strain development is interpreted by a frequency histogram (Figure 5.13h), which illustrates the spatial variations in strain field between Location 1 and 2 near the onset of secondary creep at 120 s (extracted from Figure 5.13c). Location 2 generates 0.8% of strain and has a main blue orientation close to [110], while Location 1 has approx. 0.4% of strain with a main green crystal orientation near [100], which is half of the location 2.

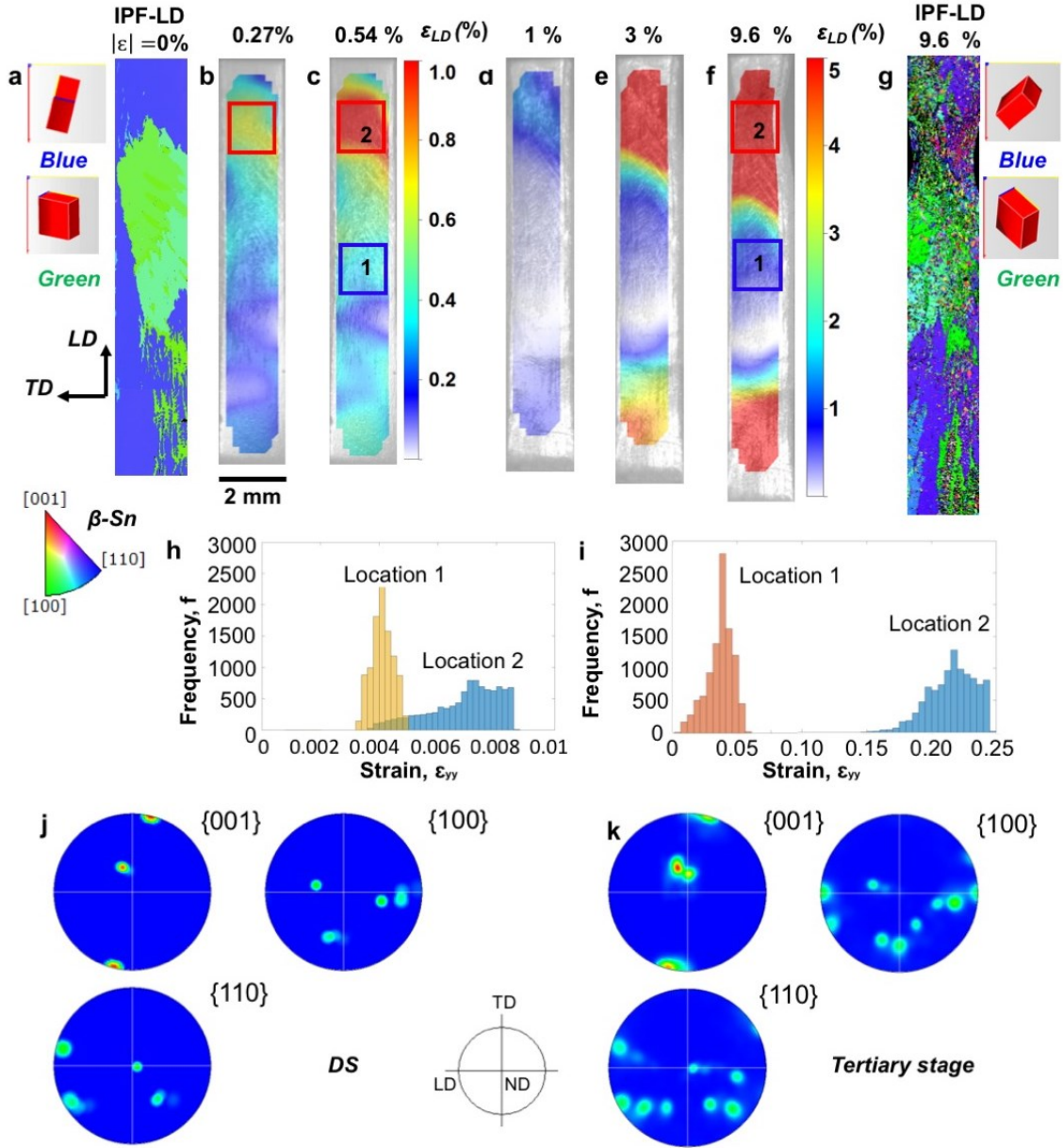


Figure 5.13. DIC figures and EBSD maps of a 'coarse' microstructure bi-crystal sample tested at 393 K. (a) IPF-LD map after DS ($\epsilon_{LD} = 0\%$) with crystal orientations shown. (b, c) DIC measurements after interruption near onset of secondary stage creep at strain (ϵ_{LD}) level 0.27% and 0.54% respectively. (d-f) DIC figures in tertiary stage creep at strain level 1%, 3% and 9.6% respectively. (g) IPF-LD map at tertiary stage creep ($\epsilon_{LD} = 9.6\%$) with the crystal unit cell of the main orientations shown. (h, i) Frequency vs. local strain histogram at locations 1 and 2 shown at onset secondary stage creep (h), and at tertiary stage creep (i). (j, k) PFs of the sample at DS condition (j), at tertiary stage creep (k).

As the testing continues, more strain is accommodated at the two ends (Figure 5.13d – 5.13f).

A rapid increase in creep strain rate is obtained when the sample forms a semi-stable neck

which localises the heterogeneous deformation and initiates near the grain boundary at Location 2 (Figure 5.13f). During necking, there is a significant localisation of creep strain (Figure 5.13f) and surface roughening. The IPF-LD map (Figure 5.13g) indicates that there is more significant grain fragmentation than the 2 single crystal samples (Figure 5.4 and Figure 5.10) and a new grain orientation is observed in this region (likely due to recrystallisation). Moreover, the sample is elongated approx. 10% in length, 33% reduction in cross-sectional area at the neck and 7% though the gauge (the sample was not fractured).

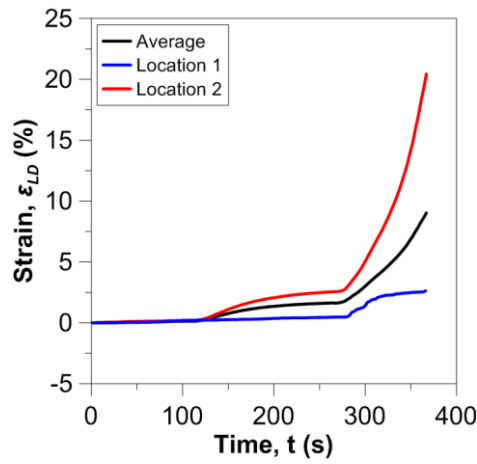


Figure 5.14. Creep curves from the onset of secondary to tertiary stage creep for the SAC305 sample tested at 393 K showing the heterogeneity in strain localisation through the length-scale (mm) of the sample. The Location 1 (blue line) and 2 (red line) are extracted from Figure 5.13 in the corresponding locations, which are compared to the average creep curve of the sample (black line).

Figure 5.13i gives the frequency histogram at Location 1 and 2 at the end of the creep test (extracted from Figure 5.13f). The localised creep strain at Location 2 is ~21% on average, while Location 1 only has ~ 2.6% strain, which means that the heterogeneity of deformation increases during creeping. It is believed that Location 1 with main orientation near [100] grain has not reached tertiary stage creep at the end of the test, as illustrated in Figure 5.14. The orientation spreading and formation of new orientations are observed by comparing the PFs in the as-DS condition (Figure 5.13j) with the one in tertiary stage creep (Figure 5.13k), which

indicates the gradual rotation of orientation and the generation of recrystallisation. The detail of the microstructure within the two grains are explored in the next section.

5.3.5.2 Microstructure of the sample within non-necking region

The microstructure of the bi-crystal sample within the non-necking region, i.e. location 1 in Figure 5.13c and 5.13f, is given at different stages of creep in Figure 5.15b – 5.15f, which come from the red squared region in Figure 5.15a. As shown in Figure 5.15a, there are 4 noticeable grain orientations in this location after DS. 2 small grains (labelled as 1 and 2) are embedded inside the main orientation, which is close to [100].

The subgrains are formed even in the DS condition as shown in Figure 5.15b and 5.15c. Their growth is preceded by the formation of significant misorientation and these are localised around IMCs and annotated with red arrows in Figure 5.15c. Regions of high misorientation area are also generated within the dendrites, which are annotated with yellow arrows in Figure 5.15c, likely due to CTE mismatch between the β -Sn dendrite and IMCs and the softness of β -Sn. Overall, the initial deformation is heavily length-scale modulated by the IMCs.

For deformation in the early stages of secondary creep, the MO av. decreases smoothly across the map as given in Figure 5.15e with red and yellow arrows labelled for eutectic and β -Sn dendrite regions respectively. The 2 small grains 1 and 2 are removed from the initial microstructure (Figure 5.15d), which might be caused by recovery processes where the material is annealing because deformation happens at $\frac{T}{T_M} \sim 0.80$ (T_M of SAC305 ~ 490 K) in this test.

At this high temperature ($\frac{T}{T_M} \sim 0.80$), the subgrain formation becomes stronger and faster, because there is greater amount of dislocation climb and grain boundary mobility. This might

be a result of initiation of lattice diffusion mechanism as well since exceeding $\frac{T}{T_M} \sim 0.70$, the creep mechanism changed in Figure 5.3d.

Figure 5.15f and 5.15g illustrate that the high misorientation regions developed more significantly at the tertiary stage creep with a creep strain level (average in location 1) approx. 2.5% (Figure 5.13f). These regions are introduced in the eutectic region as a result of the heterogeneity associated with the presence of IMCs with red arrows labelled. Qualitative observations indicate that there is correlation between the larger area region of high misorientation (Figure 5.15g) with the spatial region where the 2 small grains 1 and 2 were located initially. This region of high misorientation grows in area and extends the initial grain boundary, which is labelled with red lines. This implies that the compatibility requirements associated with the grain boundary is evident due to the localisation of plastic strain gradients (related to stored dislocations) and causing the formation of subgrains. Although recrystallisation has not been retriggered here yet, the start of the recrystallisation story can be seen visibly.

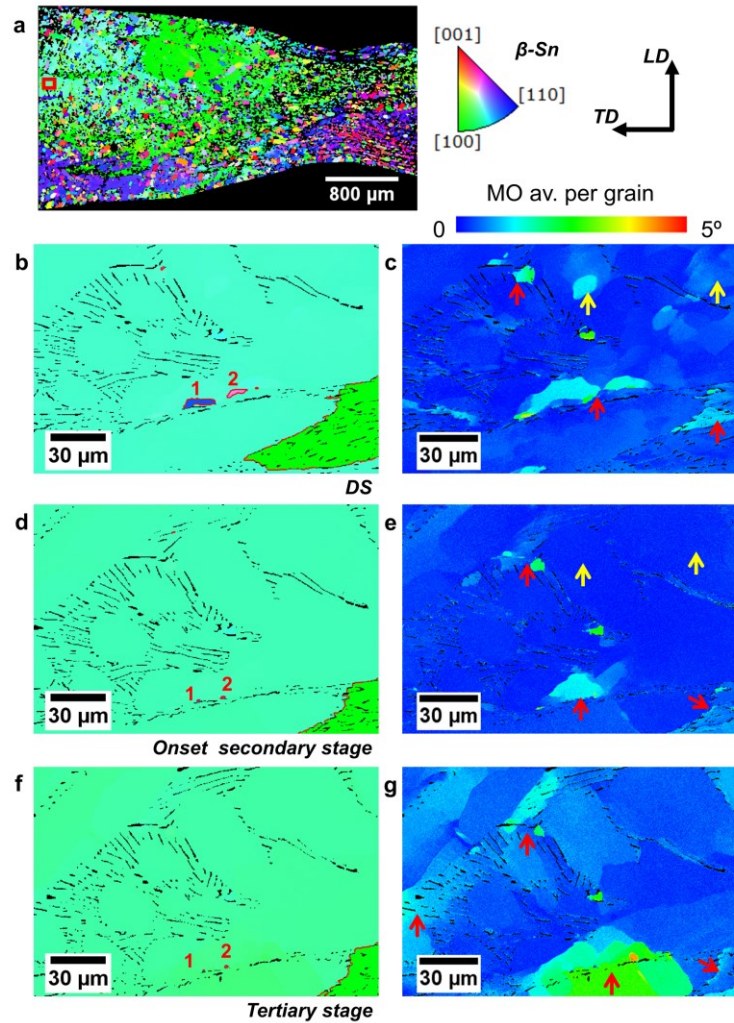


Figure 5.15. EBSD crystal orientation mapping showing evolution of microstructural heterogeneity highlighting evolution within the uniformly deformed region for the bi-crystal sample (only the β -Sn has been indexed here). (a) Large scale IPF-LD map at tertiary stage creep. (b-g) Higher magnification IPF-LD maps of the red squared region in (a) within the non-necking region at different stages of creep (the grain boundaries larger than 5° are labelled with red lines and the 2 embed small orientations are labelled with 1 and 2), (b, c) at the DS condition, (d, e) at the onset of secondary stage creep, and (f, g) at the tertiary stage creep. With increasing strain, there is a gradual orientation change in the IPF-LD map (b, d, f), while the MO av. shows significant change, especially in eutectic regions near IMCs and at grain boundary (c, e, g).

5.3.5.3 Microstructure of the sample within necking region

The heterogeneity in microstructural deformation of the sample is presented within the necking region with significant amount of recrystallisation in Figure 5.16a. The sample has been locally

strained to approx. 12% (as measured from DIC, in Figure 5.13f). The surface roughness of the sample (Figure 5.16b) has increased greatly in this region comparing to the non-necked region (Figure 5.15) and the soft β -Sn has flowed around the hard IMCs. Recrystallised grains generated (Figure 5.16c and 5.16e) have a similar length-scale as the formed subgrains in the early stage (Figure 5.15f and 5.15g). These recrystallised grains tend to have minimal internal misorientation (as expected from a recrystallisation process) and the misorientation maps (Figure 5.16c and 5.16e) show that some recrystallised grains are close in orientation.

The centre of the necking region (Figure 5.16e and 5.16f) has a greater amount of local deformation. Figure 5.16f shows that the constrained misorientation (Figure 5.16d) has been released due to the generation of recrystallisation. The MO av. is still relatively higher in the eutectic region around IMCs than around the β -Sn dendrites. It is observed from Figure 5.16e that the β -Sn within the eutectic region has similar ‘rainbow colour’ recrystallisation as the β -Sn within the dendrite, but the β -Sn in the eutectic has a much smaller recrystallised grain size.

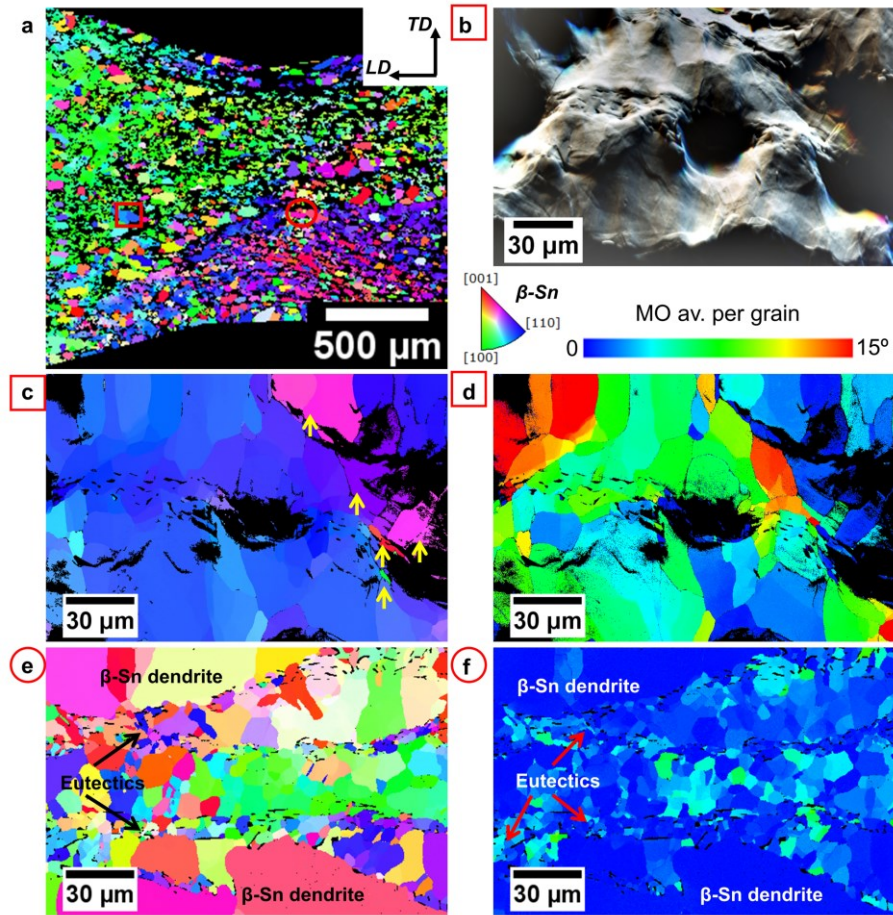


Figure 5.16. EBSD crystal orientation mapping showing evolution of microstructural heterogeneity highlighting evolution within the necking region (only the β -Sn has been indexed here). (a) Large scale IPF-LD map for the bi-crystal sample within the necking region at tertiary stage creep. (b-f) Higher magnification maps of the same region in (a) within the necking region, (b-d) in the red squared region, (e, f) in the red circled region. With increasing strain, there is significant increase in surface roughness in the foreshatter electron image (FSE) (b), increase in recrystallisation volume in both dendrite and eutectic regions (the eutectic β -Sn has much smaller grain size than in dendrite) (c, e), and decrease in misorientation in the MO av. map (d, f). (e, f) are taken from the polished surface of the sample after fracture.

5.3.6 [110] [110] [110] tri-crystal sample at 423 K

5.3.6.1 Strain field and crystal orientation

The as-DS IPF-LD map (Figure 5.17a) has 4 orientations in its length-scale, however, this sample at 423 K ($\frac{T}{T_M} \sim 0.86$) is considered as a tri-crystal sample because the green orientation has no effective deformation itself. The other 3 grains are deformed by rotating in the same way and they are close to the [110] orientation with orientation unit cell given in Figure 5.17a. The blue 1 and blue 2 grains are twinned (often called cyclic twinning in the Sn-based solder literature^[12-15]), the PF (Figure 5.17i) has a common [110] direction and the circled point shows a mixture of colours. The corresponding misorientation distribution histogram (Figure 5.17h) has a peak at approximately 60° indicating the presence of twinning. The boundary between the blue 2 and purple 3 grains is a random high-angle grain boundary.

The DIC figures (Figure 5.17b and 5.17c) show slight heterogeneity at the top half of the sample (within the red square) from primary to secondary stage creep, perhaps because of containing one more grain (the green grain), so the heterogeneity of strain field is related to the presence of the grain boundary. The strain at the onset of secondary stage creep decreases to 0.18% (Figure 5.17b), which is associated with increasing of temperature.

More strain is localised at the top end (highlighted with red square in Figure 5.17d – 5.17f) at tertiary stage creep. A slight bending neck is observed near the fracture surface (Figure 5.17g) and the orientation changes within the bending region. The length of the sample has been elongated more than 40% from its original length, reduced 23% and 11% in cross-sectional area. The spread in orientation is tiny for this sample (Figure 5.17i and 5.17j) and only slight rotation in orientation around [100] axis can be seen from the crystal unit cell of this sample

(Figure 5.17a and 5.17g). These might be caused by the presence of twin grain boundary, which is relatively more resistant to damage ^[152] than random high-angle grain boundary. The details of microstructure evolution are shown in Figure 5.18 and Figure 5.19 below.

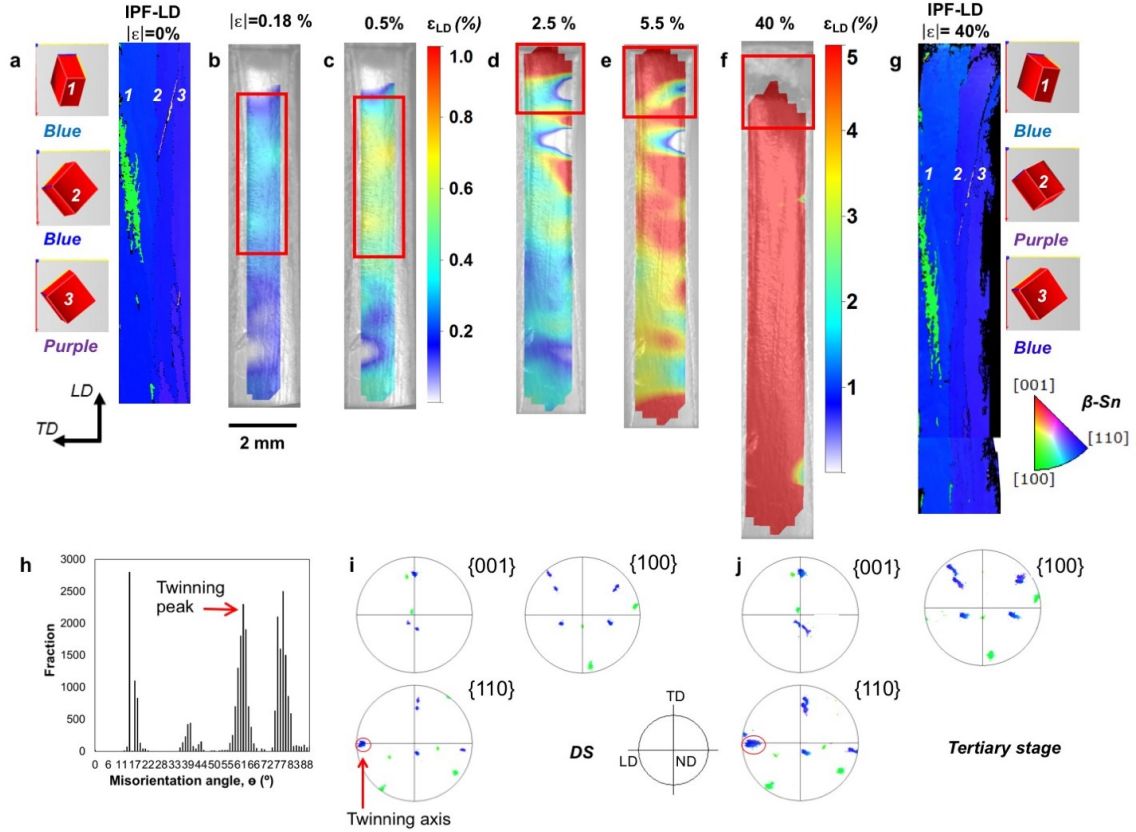


Figure 5.17. DIC figures and EBSD maps of a ‘coarse’ microstructure tri-crystal sample crept at 150 °C. (a) IPF-LD map after DS ($\epsilon_{LD} = 0\%$) with main crystal orientations shown. (b-c) DIC figures interrupted near onset of secondary stage creep at strain level (ϵ_{LD}) 0.18% and 0.5% respectively. (d-f) DIC figures at tertiary stage creep (fracture) at creep strain level 2.5%, 5.5% and 40% respectively. (g) IPF-LD map at fracture ($\epsilon_{LD} = 40\%$) with the main orientations shown. PFs of the sample at DS condition (h), at tertiary stage creep (i).

5.3.6.2 Microstructure of the [sample within non-necking region]

The red square highlighted region is located at the grain boundary between the 2 blue grains in Figure 5.18a, which are twinned approx. 60° with [110] axis. Figure 5.18b has relatively high

misorientation at as-DS condition, which is localised in the eutectic region at the grain boundary and near IMCs with respect to the region containing only one grain (Figure 5.5 and Figure 5.11).

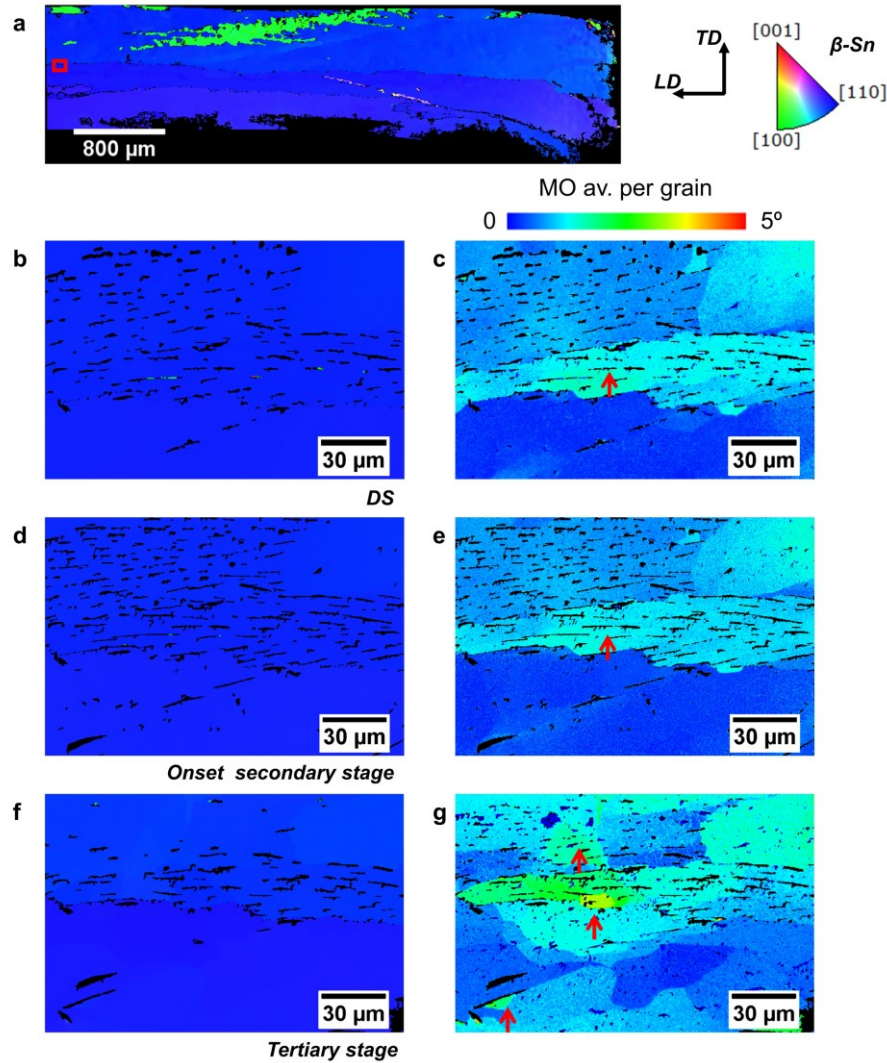


Figure 5.18. EBSD crystal orientation mapping showing evolution of microstructural heterogeneity highlighting evolution within the uniformly deformed region for the tri-crystal sample (only the β -Sn has been indexed here). (a) Large scale IPF-LD map at tertiary stage creep. (b-g) Higher magnification IPF-LD maps of the red squared region in (a) within the non-necking region at different stages of creep, (b, c) at the DS condition, (d, e) at the onset of secondary stage creep, and (f, g) at the tertiary stage creep. With increasing strain, the orientation changed slightly in the IPF-LD map (b, d, f), while misorientation changed significantly, especially in eutectic regions around IMCs and near the grain boundary (c, e, g).

The same decreasing in MO av. from the as-DS (Figure 5.18c) to the onset of secondary stage (Figure 5.18e) is observed as the 393 K sample (Figure 5.15) due to the recovery annealing at high temperature. Development of subgrains is observed from secondary to tertiary stage creep in both eutectic regions and β -Sn dendrites (Figure 5.18g). The highest misorientation spots are located at the grain boundary around IMCs. The two blue orientations are rotating during deformation and their colour become more distinguishable in the IPF-LD map at the tertiary stage creep (Figure 5.18f), i.e. the misorientation are deviating from the twin grain boundary during deformation. And again, coarsening of IMCs cannot be seen from the Figure 5.18b and 5.17g.

5.3.6.3 Microstructure of the sample within necking region

Figure 5.19a gives the IPF-LD map of the sample at fracture surface. Recrystallisation and subgrains have been generated and are localised around the fracture surface, which means the transition to the tertiary creep is very localised. Within the highly-strained region (Figure 5.19b), no obvious increase in misorientation (MO av. 0 -15°) is observed within the grain comparing to the region in the non-necking region (Figure 5.18g with MO av. 0 - 5°). Slightly misorientation hot spots appear around IMCs at the β -Sn dendrite and eutectic interface. The dendrite arms have been largely extended due to grain diffusion mechanism (Section 2.3.2.1).

The red circled region in Figure 5.19a shows the increasing of surface roughness (Figure 5.19d) and misorientation (Figure 5.19f) as the distance is becoming close to the fracture surface and significant rotation in orientation and development of subgrains are observed in the corresponding IPF map (Figure 5.19e). Small and new orientations (indicated with white

arrows) are generated within the eutectic region in the large purple grain (Figure 5.19e), which are formed by rotating from the parent grain near the IMCs, where deformation is localised.

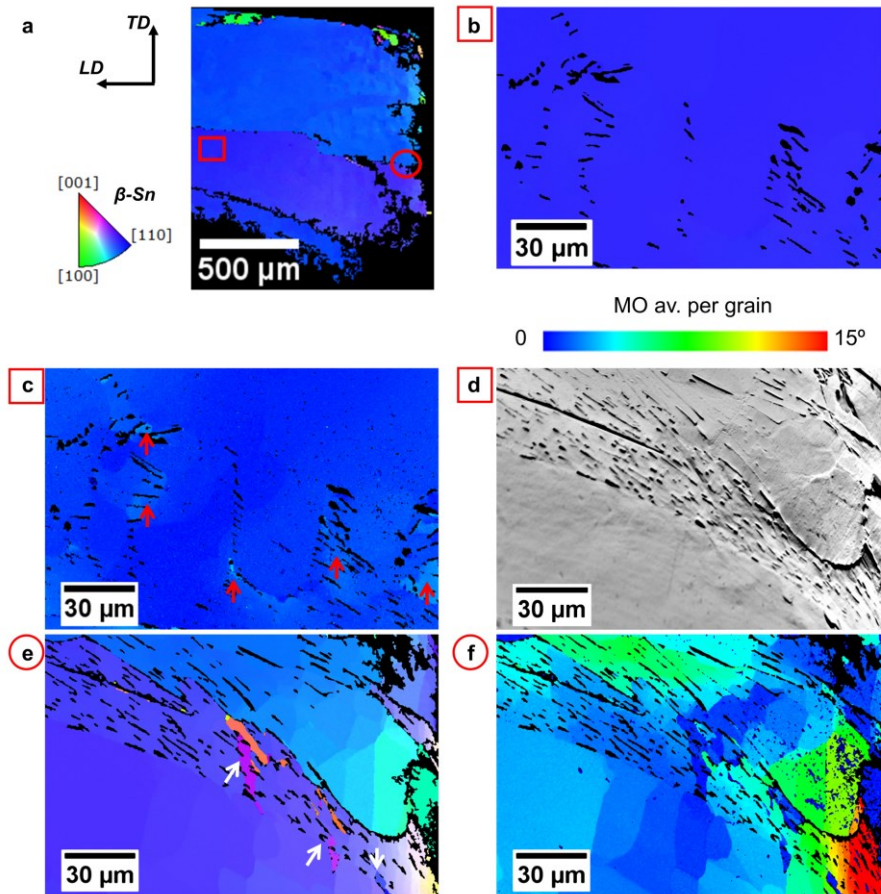


Figure 5.19. EBSD crystal orientation mapping showing evolution of microstructural heterogeneity highlighting evolution within the necking region (only the β -Sn has been indexed here). (a) Large scale IPF-LD map for the tri-crystal sample within the necking region at tertiary stage creep. (b-f) Higher magnification maps of the same region in (a) within the necking region, (b, c) in the red squared region, (d-f) in the red circled region. With increasing strain, the surface roughness increased is significantly at the fracture surface in the FSE (d), generation of subgrains (b) and recrystallised grains (e), and increase in misorientation in the MO av. map (c, f), especially in the eutectic regions near the grain boundary and at the fracture surface.

5.3.7 [110] [110] bi-crystal sample at 473 K

5.3.7.1 Strain field and crystal orientation

The bi-crystal sample tested at 473 K ($\frac{T}{T_M} \sim 0.97$) achieved a similar deformation process to the samples deformed at 423 K. The 2 crystal orientations shown in the IPF-LD map (Figure 5.20a) are close to [110].

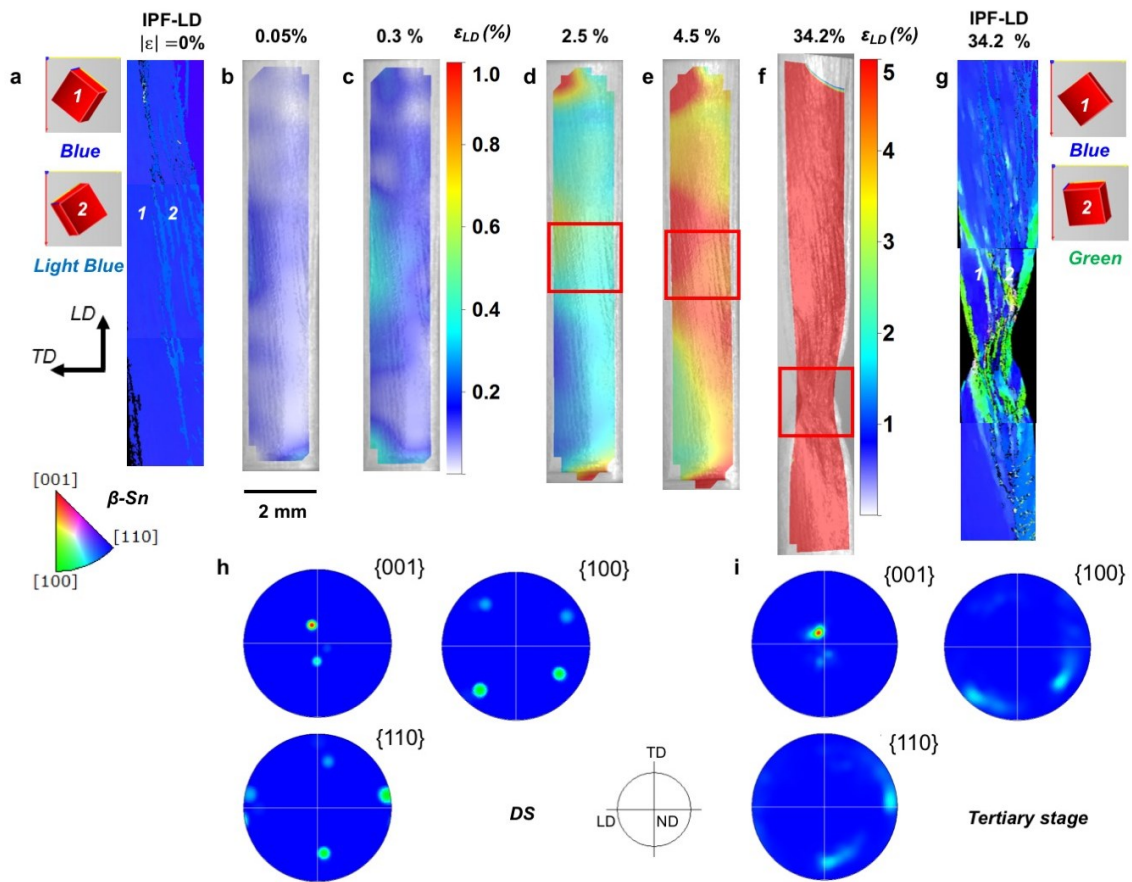


Figure 5.20. DIC figures and EBSD maps of a ‘coarse’ microstructure bi-crystal sample crept at 200 °C. (a) IPF-LD map after DS ($\epsilon_{LD} = 0\%$) with main crystal orientations shown. (b-c) DIC figures interrupted near the onset of secondary stage creep at strain level (ϵ_{LD}) 0.05% and 0.3% respectively. (d-f) DIC figures at tertiary stage creep at strain level 2.5%, 4.5% and 34.2% respectively. (g) IPF-LD map at tertiary stage creep ($\epsilon_{LD} = 34.2\%$) with the main orientation shown. The PFs of the sample at DS condition (h), at tertiary stage creep (i).

The strain level at onset secondary stage creep decreases to 0.05% indicating that the sample enters secondary stage creep straight way and has almost no dislocation pile-up during the deformation (Figure 5.20b).

As deformation continues (Figure 5.20d – 5.20f), more strain is accommodated at the red-squared region, where a stable neck is formed with broader size comparing to the sample tested at lower temperatures (Figure 5.4, Figure 5.7, Figure 5.10, Figure 5.13 and Figure 5.17). The length elongation of the sample is 34% and the cross-sectional reduction are 5% and 37% at the necking and non-necking regions respectively. At the tertiary stage creep (Figure 5.20g), the rotation in orientation from light blue 2 [110] to green [100] is observed. This is also illustrated as spread in the PFs (Figure 5.20h and 5.20i) and rotation in corresponding the crystal unit cells (Figure 5.20g).

5.3.7.2 Microstructure of the sample within non-necking region

The high magnification IPF-LD maps (Figure 5.21b – 5.21g) are taken at the red squared region in Figure 5.21a, which is located within the non-necking region at the interface between the 2 orientations. At as-DS condition (Figure 5.21b and 5.21c), relatively high misorientations appear in the eutectic regions and at the grain boundary. The MO decreases and becomes evenly distributed from primary to secondary stage because of the recovery promoted at higher temperature (Figure 5.21d and 5.21e). At tertiary stage (Figure 5.21f and 5.21g), misorientation starts to develop again at the grain boundary and subgrains are also formed here. With further deformations, it is noticed like that the grain boundary between the 2 orientations becomes rougher (Figure 5.21f), i.e. the 2 grains are permeating each other, which means the induction of grain boundary mobility causing by lattice diffusion mechanism.

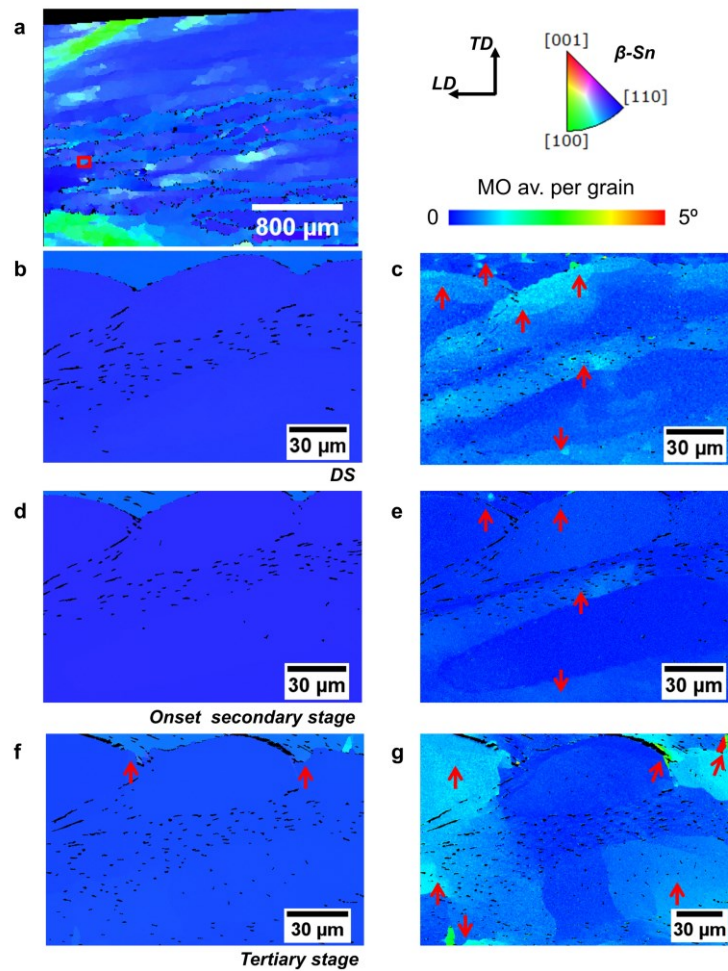


Figure 5.21. EBSD crystal orientation mapping showing evolution of microstructural heterogeneity highlighting evolution within the uniformly deformed region for the bi-crystal sample (only the β -Sn has been indexed here). (a) Large scale IPF-LD map at tertiary stage creep. (b-g) Higher magnification IPF-LD maps of the red squared region in (a) within the non-necking region at different stages of creep, (b, c) at the DS condition, (d, e) at the onset of secondary stage creep, and (f, g) at the tertiary stage creep. With increasing strain, the orientation changed slightly in the IPF-LD map (b, d, f), while misorientation changed significantly, especially in eutectic regions near the grain boundary (c, e, g).

5.3.7.3 Microstructure of the sample within necking region

Within the highly-strained region (necked region), significant elongation of β -Sn in both dendrite and eutectic regions is observed (Figure 5.22a and 5.22b). At the edge of the necked region (Figure 5.22c and 5.22d), there are very less increase in misorientation and subgrain

formation relative to the non-necking region (Figure 5.21g) but high misorientations are observed at the centre of the neck (Figure 5.22f). Similar to the 423 K sample, not many fine recrystallised grains have been generated within the necked region (Figure 5.22a), probably due to the short deforming time. Large volume of green orientation and new orientations in yellow and pink (close to $[001]$) are formed at the tertiary stage creep (pointed out with red arrows in Figure 5.22e) as a result of gradual lattice rotation showing evidence of recovery.

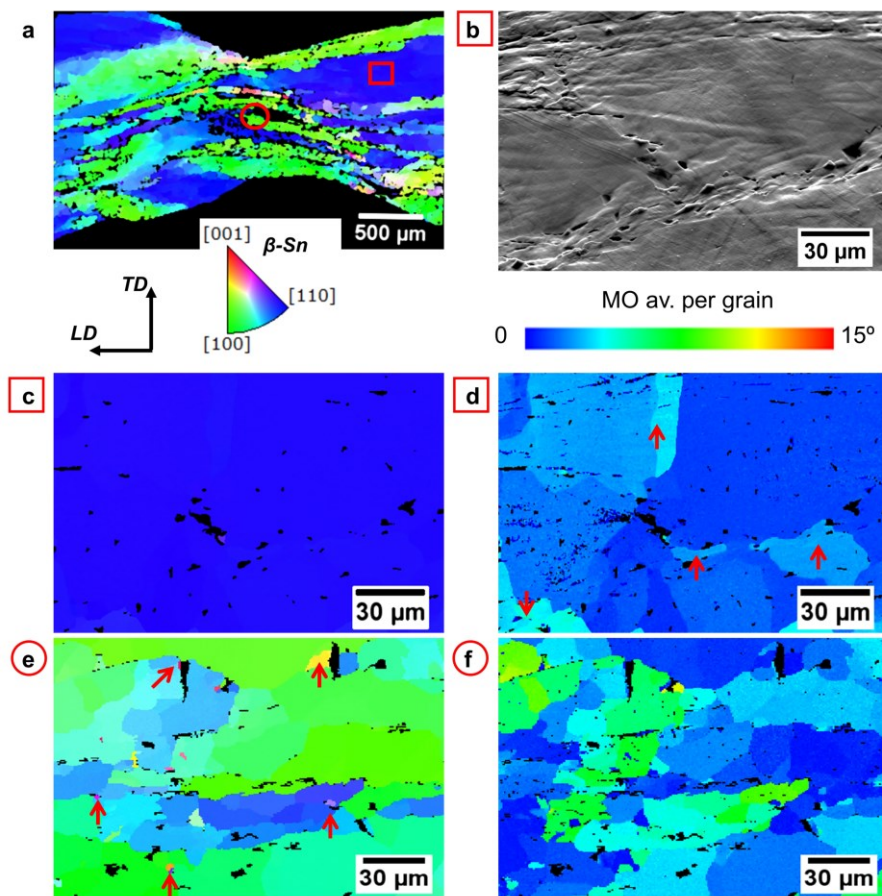


Figure 5.22. EBSD crystal orientation mapping showing evolution of microstructural heterogeneity highlighting evolution within the necking region (only the β -Sn has been indexed here). (a) Large scale IPF-LD map for the bi-crystal sample within the necking region at tertiary stage creep. (b-f) Higher magnification maps of the same region in (a) within the necking region, (b-d) in the red squared region, (e, f) in the red circled region. With increasing strain, there is significant increase in surface roughness in the FSE (b), growth in subgrains (c) and recrystallised grain (e) and increase in misorientation in the MO av. map (d, f) (indicated with red arrows). (e, f) are taken from the polished surface of the sample after fracture.

5.4 Summary of the Results

Table 5.2 summaries the orientation and creep result for all of the samples. The elongation of length increases with increasing temperature and the reduction of cross-sectional area is increased with temperature and is associated crystal orientation of the sample as well, i.e. the sample with a [100] orientation seems reduced more than one with an [110] orientation in cross-section.

Temperature (K)	Initial orientation	Final orientation	% elongation in length	% reduction in cross-sectional area	Secondary creep strain rate (s ⁻¹)
298	[110]	[110] [100]	7	24	$3.4 (\pm 0.4) \times 10^{-6}$
318	[110] [110] [100]	[110] [001] [100]	10	28	$7.2 (\pm 0.4) \times 10^{-6}$
333	[100] [100] [110]	[100] [110][001]	13	40	$1.4 (\pm 0.2) \times 10^{-5}$
363	[100]	[100] [110]	17	55	$2.1 (\pm 0.2) \times 10^{-5}$
393	[110] [100]	[100] [110] [001]	10 (not fractured)	33	$2.6 (\pm 0.1) \times 10^{-5}$
423	[110] [110] [110]	[110] [110] [110]	40	23	$2.8 (\pm 0.1) \times 10^{-5}$
453	[110] [110] [110]	[100] [110] [110]	35	31	$2.9 (\pm 0.1) \times 10^{-5}$
473	[110] [110]	[100] [110]	34 (not fractured)	35	$3.4 (\pm 0.1) \times 10^{-5}$

Table 5.2. The orientation, elongation and cross-sectional reduction of each sample. The error in strain rate is determined by standard deviation (STD) for averaging of 2 repeat measurements.

In summary at this point, it is believed that the deformation of the sample is initiated in the β -Sn within the eutectic region near IMCs and runs through the β -Sn dendrite as evidenced by in-grain lattice rotations, which corresponds to an increase in stored energy. This eventually leads to enough energy to drive recrystallisation nuclei to segment the initial grain, which occurs in tertiary stage creep.

Testing at higher temperatures shows that strain heterogeneity increases indicating that microstructure sensitivity between grains increases. This heterogeneous deformation is

observed, even during primary and secondary stage creep, and increases when transitioning from secondary to tertiary stage creep. At higher temperatures, thermally activated processes are enhanced. For deformation heterogeneity, temperature will influence hardening (i.e. dislocation forest generation and pile-up) and softening (i.e. recovery and removal of dislocation substructure), as well as the relative activity of slip systems (i.e. strain rate sensitivity). An increase in heterogeneity implies that the relative activation of these processes is dependent on crystal orientation, grain length scale, and the number of grains.

5.5 Summary of the Chapter

The creep behaviour and microstructural evolution of DS SAC305 solder alloy have been studied at a constant stress of 30 MPa at a range of elevated temperature. The following conclusion can be drawn from this work.

1. Shorter secondary creep lifetime and greater secondary creep strain rate are obtained at elevated temperature. The strain at the onset of secondary creep decreases with temperature, which is caused by less dislocation formation and motion, but more diffusion at higher temperatures, i.e. recovery takes place at lower creep strain level at higher temperature, because of less dislocation motion, but more diffusion.
2. A significant change in secondary creep strain rate is found beyond a homologous temperature $\sim \frac{T}{T_M} = 0.7$. This is associated with a change in creep mechanism from climb-controlled dislocation creep to lattice diffusion creep. This causes a decrease in the activation energy, Q , in the high temperature regime (363 – 473 K) relative to the

low temperature regime (298 – 333 K), the values of Q are 41 kJ/mol and 6 kJ/mol respectively.

3. Heterogeneous deformation is obtained during creep, even from the primary to secondary stage creep and this heterogeneity enhances from secondary to tertiary stage creep. All single, bi-, and tri-crystal samples are under ductile failure during creep. The ductility of the samples increases with temperature. The sensitivity in microstructure (number of grains and orientations) increases at more elevated temperatures. Bi-crystal and tri-crystal samples tend to deform with stronger heterogeneity than single-crystal samples. This deformation heterogeneity is associated with the presence of the grain boundary.
4. The dendritic primary β -Sn and eutectic β -Sn deform differently during creep, i.e. the length-scale of recrystallisation is much smaller in eutectic region than in the dendrite, consistent with the findings in Chapter 4. The deformation of samples is initiated in the eutectic regions near IMCs with high misorientation development and runs through the β -Sn dendrite by lattice rotation. When rotated enough, it leads to subgrain formation, and provides sufficient energy to generate recrystallisation which is localised near the ultimate fracture point of each specimen. This failure phenomenon is enhanced at elevated temperature due to the increase in dislocation climb and grain boundary mobility.

Chapter 6 Effect of microstructure on creep behaviour of SAC305 solder

6.1 Introduction

In this chapter, the effect of change in microstructure on the creep performance and microstructural evolution of DS SAC305 samples are studied. As mentioned in Chapter 3, the SAC305 samples in the current work were grown at two different growth rates, 20 and 200 $\mu\text{m/s}$, and a substantial difference in the microstructure, namely, secondary dendrite arm spacing, SDAS (λ_2), eutectic spacing (λ_e) and IMC (Cu_6Sn_5 and Ag_3Sn) size was obtained between the samples at the two growth rates. The samples used in this chapter are grown at a faster growth rate of $v = 200 \mu\text{m/s}$, which have a finer microstructure, i.e. SDAS, $\lambda_2 \approx 20 \pm 3 \mu\text{m}$ and eutectic spacing, $\lambda_e \approx 0.07 \pm 0.02 \mu\text{m}$, compared with the samples grown at $v = 20 \mu\text{m/s}$, which have relatively coarser microstructure with $\lambda_2 \approx 50 \pm 3 \mu\text{m}$ and $\lambda_e \approx 1.18 \pm 0.02 \mu\text{m}$. Therefore, the microstructure effects are explored by comparing the two microstructures during creep deformation.

6.2 Experiment

The samples used in the current chapter are grown at $v = 200 \mu\text{m/s}$ with a finer microstructure and prepared by the same method as given in Chapter 3. The samples are deformed at constant stress of $30 (\pm 2) \text{ MPa}$ between room temperature and $200 (\pm 0.4) ^\circ\text{C}$ as described in Chapter 5. The difference in the creep performance between the two different microstructures is

compared and two detailed EBSD scans were performed at 333 and 393 K to show the difference in microstructural evolution at low and high temperatures (the experimental procedure is as same as applied in Chapter 5).

6.3 Results and Discussion

6.3.1 Creep strain curves

Figure 6.1a shows the creep curves from primary to the early stage of secondary creep (the first 120s) for tests at 298 to 473 K (only the secondary creep regime is shown for the samples at 423, 453 and 473 K). All the samples reached their secondary stage creep within the first 120 s of deformation, as can be seen from the creep strain rate vs. time curves in Figure 6.1b and a gradient of 0 in the creep strain (%) vs. strain curves in Figure 6.1c. The creep curves were differentiated and smoothed to plot Figure 6.1b and 6.1c.

The strain at the onset of secondary creep decreases with increasing temperature, as determined from Figure 6.1c and shown in Figure 6.1d. The activation energy, Q can be calculated by analysing the gradient of Figure 6.1e. By applying two linear fits the square of the correlation coefficient, R^2 improves significantly from 0.87 (full temperature range) to 0.99 (low and high temperature regimes) (Table 6.1). The activation energy, Q was calculated from Figure 6.1e using (2.4) as 34 and 6 kJ/mol for low and high temperature regimes respectively.

	Q ($T_{\text{low}} = 298 - 333 \text{ K}$)	Q ($T_{\text{high}} = 363 - 473 \text{ K}$)	Q ($T_{\text{full}} = 298 - 473 \text{ K}$)
Q (kJ/mol)	34	6	20
R^2	0.99	0.99	0.87

Table 6.1. Comparison of activation energy, Q between low and high temperature regimes and full temperature range. R^2 is the square of the correlation coefficient of the corresponding liner fit.

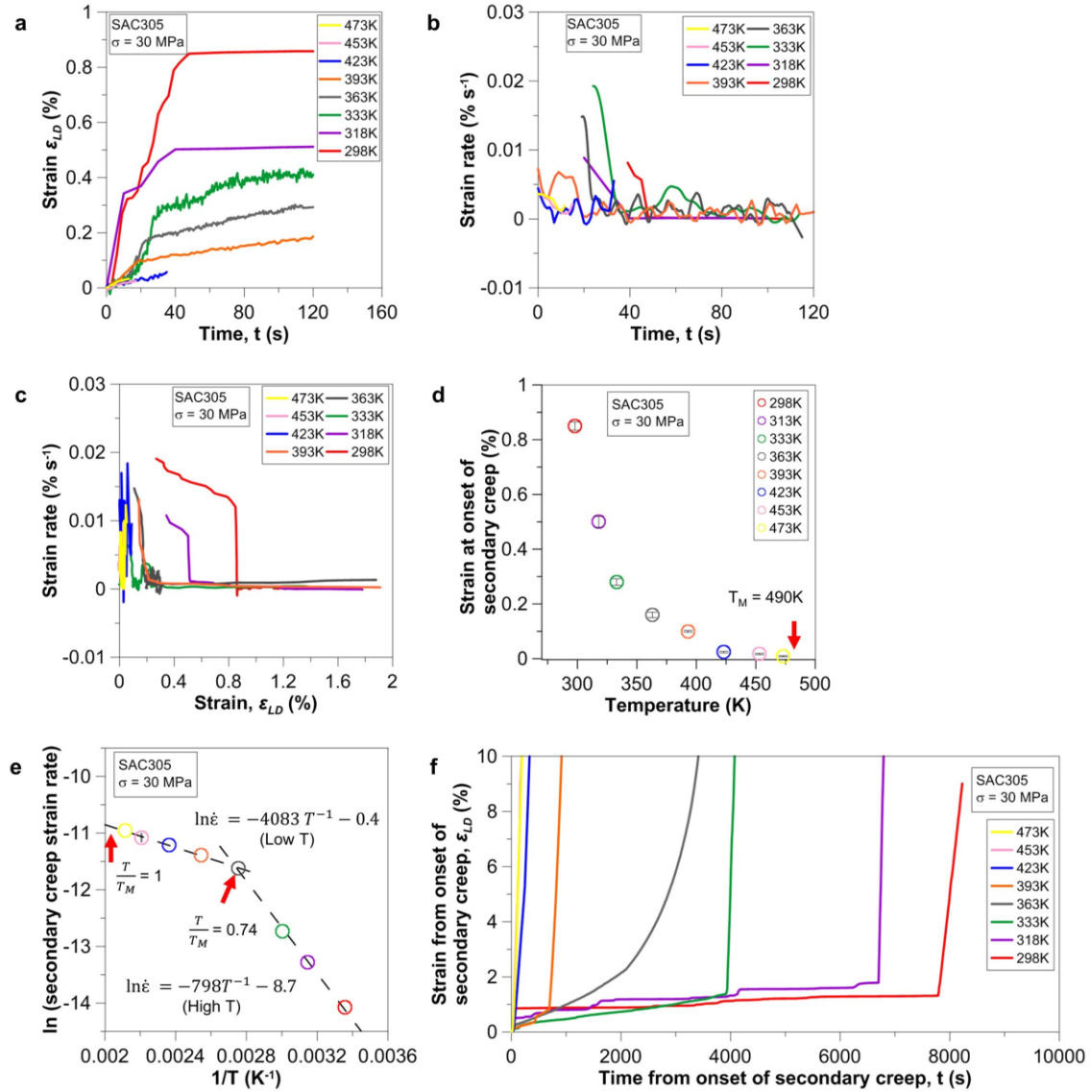


Figure 6.1. Fixed load (30 MPa) mechanical creep data from 298 to 473 K of SAC305 samples grown at $v=200\mu\text{m/s}$. (a) Creep curves from 298 to 473 K at primary and early secondary stages of creep. (b) Creep strain rate vs. time in the primary and early secondary stages of creep showing when secondary stage creep is reached. (c) Creep strain rate vs. creep strain curves showing a gradient of 0 after the secondary stage creep is reached. (d) Variation of the onset of secondary creep with respect to temperature. (e) Creep model analysis of $\ln(\text{secondary creep strain rate})$ vs. reciprocal temperature with two creep domains (high temperature and low temperature). (f) Creep curves from the onset of secondary to tertiary stage creep (maximum strain = 10%). (The symbols are included for completeness and represent orientations, as described in Table 2.3).

The transition between the low and high temperature regimes appears i.e. the recovery process happens after a shorter creep time for the sample tested at higher temperature and the creep mechanism changes from climb-controlled dislocation creep to lattice diffusion associated

processes. The measured activation energies and strain at onset of secondary creep indicate that at low temperatures climb-controlled dislocation mechanism operates, and at higher temperatures lattice-associated diffusion mechanism operates. The transition, when fitted with linear fits, implies that there is a change in mechanism at the homologous temperature $\sim \frac{T}{T_M} = 0.74 \pm 0.02$ (363 K). The same change in mechanism is described in Chapter 5. Linear fitting for these two regimes indicates that this transition is sharp, but in practice these linear fits could be viewed as asymptotic fits for the mechanisms with a transition regime of 353 – 373 K, which is estimated through error analysis. The creep curves from the onset of secondary creep until a creep strain of $\sim 10\%$ are shown in Figure 6.1f (obtained by 2D DIC). Generally, the secondary creep lifetime decreases with temperature.

T [K]	Strain at onset of secondary creep (%)	Secondary creep strain rate (s^{-1})	Failure creep strain (%)	Number of β -Sn grains	β -Sn Orientation measured by EBSD
298	0.85	7.71×10^{-7}	9.0	1	[110]
318	0.50	1.71×10^{-6}	26.2	2	[110] [100]
333	0.28	2.96×10^{-6}	38.5	2	[110] [110]
363	0.16	8.98×10^{-6}	22.7	3	[110][110][100] ^T
393	0.10	1.13×10^{-5}	25.8	3	[110] [110] [110]
423	0.03	1.35×10^{-5}	29.8	2	[110] [100]
453	0.02	1.54×10^{-5}	21.6	2	[110] [110] ^T
473	0.01	1.74×10^{-5}	21.2 (not fractured)	3	[110] [110] [100] ^T

Table 6.2. Summary of SAC305 mechanical creep samples grown at $v=200 \mu m/s$ under fixed load of ~ 30 MPa testing from 298 to 473K showing the values of secondary creep strain rate, strain at the onset of secondary creep, failure creep strain and grain orientation of the samples. The superscript T stands for the grain boundary in transverse to loading direction (the orientation at failure for all the samples are given in Figure 6.2).

Table 6.2 illustrates the creep data, the orientation and number of β -Sn grains of each sample given in Figure 6.1. The main orientations of each sample are shown on the corresponding IPF-LD plot under the EBSD IPF-LD map to compare the orientations that were tested during creep. The secondary creep strain rates increase from the order of 10^{-4} to $10^{-3} \% s^{-1}$ with

increasing temperature. The failure creep strain generally increases with increasing temperature, but it decreases when the number of grains increases and when grain boundaries are transverse to the loading direction.

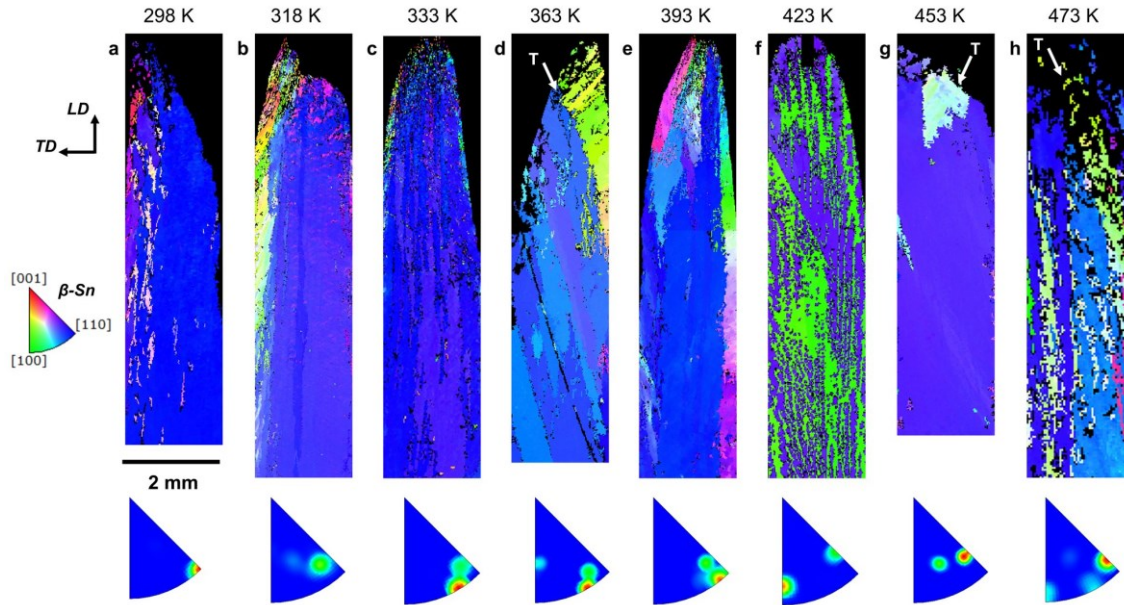


Figure 6.2. EBSD IPF-LD map of creep fractured SAC305 samples grown at $v = 200 \mu\text{m/s}$ from 298 to 473 K (a-h) with the main orientation plotted on the corresponding IPF-LD texture plot under each sample showing the number of grains and orientation information. The transverse grain boundaries are labelled.

The samples with a finer microstructure, i.e. smaller SDAS, eutectic spacing and IMC size have a longer creep lifetime (Figure 6.3a), smaller secondary creep strain rate (Figure 6.3b), and lower strain at the onset of secondary creep (Figure 6.3c) comparing with the samples with a coarser microstructure.

The activation energy, Q decreases to 34 kJ/mol in the low temperature regime and stays the same with a value of 6 kJ/mol in the high temperature regime (Figure 6.3d).

The change of creep mechanism happens at a homologous temperature of $\sim \frac{T}{T_M} = 0.74 \pm 0.02$ for the samples with a finer microstructure. This shifts to higher temperature of

$\sim \frac{T}{T_M} = 0.70 \pm 0.02$ for the samples with a coarser microstructure. The uncertainty was determined by drawing two linear fits of maximum and minimum slopes that satisfy the data. The difference in the transition temperature implies that the microstructural length-scale affects the creep mechanisms.

The decrease in the onset of secondary creep strain and Q at lower temperatures suggests that lower energy paths are available for dislocation to climb during creep. Therefore, it seems that the deformation is by obstacle-controlled dislocation motion which is dependent on the SDAS and IMC size. In the high temperature regime, there is less influence from changing the microstructure because creep deformation is mainly lattice diffusion limited which is based on the grain size.

Developing ideas from Chapter 5, the IMCs control heterogeneous deformation within the Sn-rich regions in these alloys. Varying the IMC length-scale in these two materials reveals that smaller IMCs reduce heterogeneity of deformation in the material, likely due to hardening of the deformation near the IMCs and associated length-scale effects. This limits strain localisation and the generation of sufficiently local stored energy to initiate recrystallisation.

The two IMCs Ag_3Sn and Cu_6Sn_5 are not differentiated because they have same morphology (Chapter 3) and are too small for EBSD to index at $v = 200 \text{ } \mu\text{m/s}$ with the used magnification, so the Ag_3Sn and Cu_6Sn_5 are referred to simply as IMCs in the remainder of this chapter.

The process of microstructural change is explored by EBSD in the next section.

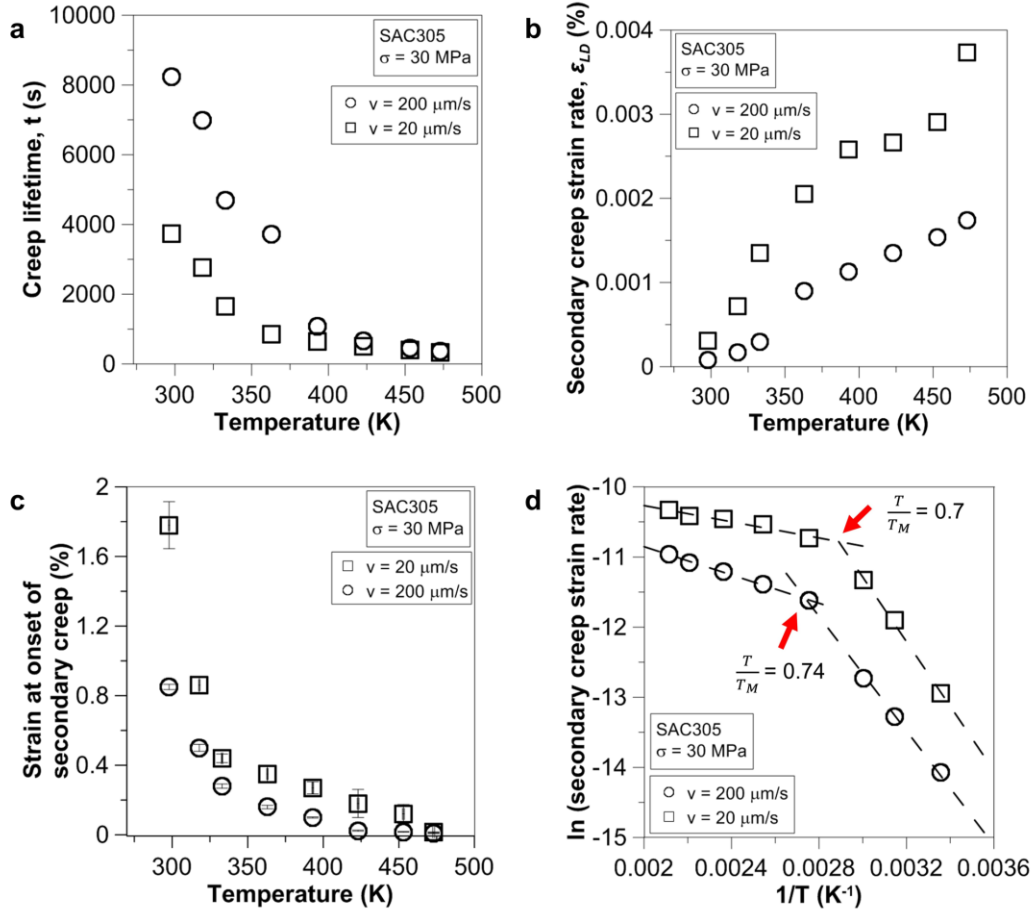


Figure 6.3. Comparison of the fixed load (30 MPa) mechanical creep data for the SAC305 samples from 298 to 473 K between the 2 growth rates of $v = 20$ and $200 \mu\text{m/s}$. (a) The variation of (a) creep lifetime, (b) the secondary creep strain rate, and (c) the onset of secondary creep strain with temperature data, showing the sample with a finer microstructure ($v = 200 \mu\text{m/s}$) has longer creep lifetime (a), smaller secondary creep strain rate (b), and lower onset secondary creep strain (c). (d) The $\ln(\text{secondary creep strain rate})$ vs. reciprocal temperature with 2 creep domains (high temperature and low temperature).

6.3.2 [110] [110] bi-crystal sample at 333 K

6.3.2.1 Strain field and crystal orientation

Heterogeneous creep deformation of a bi-crystal sample is shown in Figure 6.4 where the interface localises plastic strain significantly.

The sample deformed at 333 K ($\frac{T}{T_M} \sim 0.68$) has 2 main crystal orientations as shown in Figure 6.4a (the IPF-LD map after DS), which are close to [110]. A strong deformation heterogeneity is observed from the DIC strain field even at the very early stage of secondary creep (Figure 6.4b and 6.4c), but there is no clear link between the heterogeneity and the microstructure. The sample achieves a mean strain level of 0.28% at the onset of secondary stage creep (Figure 6.4b).

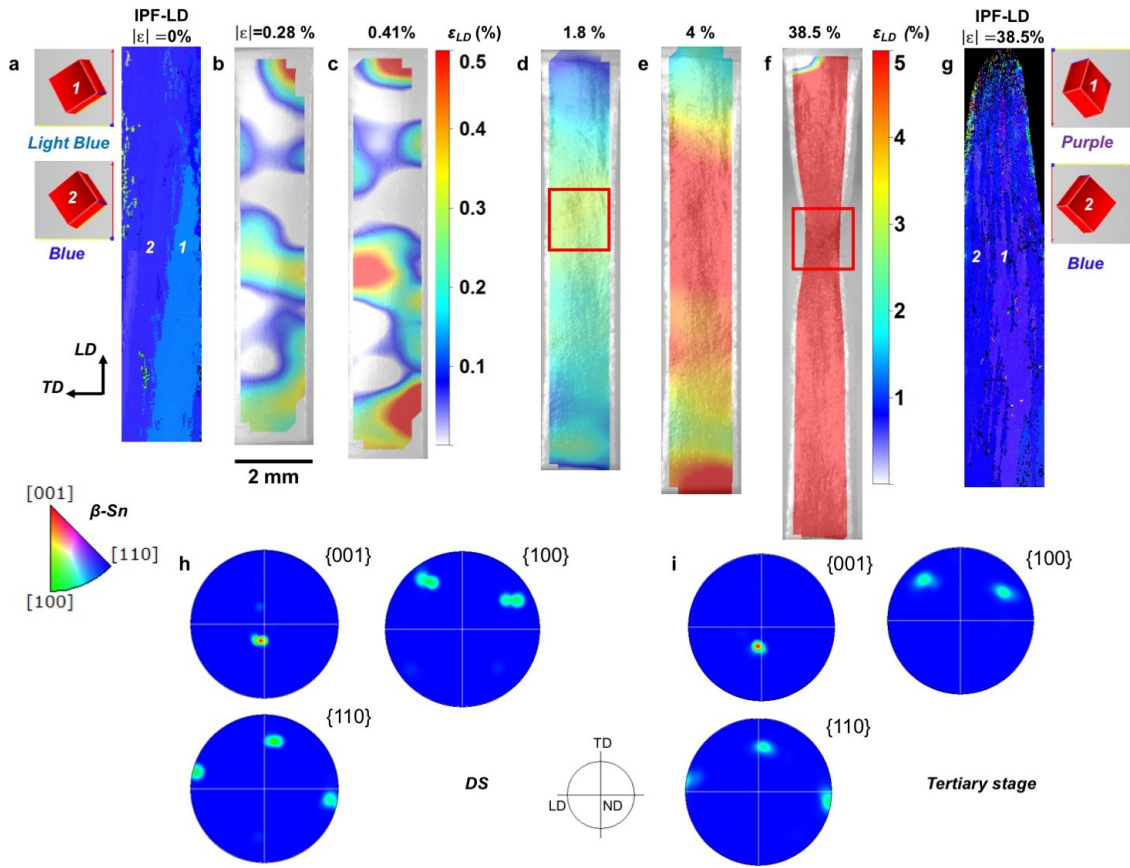


Figure 6.4. The DIC figures and EBSD maps of a ‘fine’ microstructure bi-crystal sample crept at 333 K. (a) IPF-LD map after DS ($\epsilon_{LD} = 0\%$) with main crystal orientation shown. (b-c) DIC figures interrupted near onset of secondary stage creep at strain level (ϵ_{LD}) 0.28% and 0.41% respectively. (d-f) DIC figures at tertiary stage creep (fracture) at strain level 1.8%, 4% and 38.5% respectively. (g) IPF-LD map at fracture ($\epsilon_{LD} = 38.5\%$) with the main orientation shown. PFs of the sample at DS condition (h), at tertiary stage creep (i).

In this bi-crystal sample, Figure 6.4d - 6.4f demonstrates that with increasing deformation, strain is accommodated near the grain boundary (highlighted by the red square in Figure 6.4d), and within the grain of light blue orientation (1) (i.e. close to [110]), which has a tilt grain boundary to the loading direction.

The sample necked and fractured at the highly-strained region during tertiary stage creep. The length of the sample has been elongated by over 35% and its cross-sectional area reduced by 38% at the fracture surface and 8% in the uniformly deformed region, which shows significant increase in ductility comparing to the tri-crystal sample with a coarser microstructure deformed in Chapter 5. The orientation of the [110] light blue (1) is rotated further towards [110] to form a purple colour, while the blue orientation (2) has no noticeable change in colour in Figure 6.4g. This might be because the orientation closer to [110] is relatively harder to deform.

At the fracture surface (Figure 6.4g), small green [100] orientations are observed, which are formed likely due to recrystallisation of the β -Sn. With increased strain, subgrain rotation occurs, leading to increase in stored energy and transition from subgrain boundaries to HAGBs at the highly-strained region. Figure 6.4a and 6.4g show that both orientations are rotated towards [110], the light blue (1) rotated more significantly by changing to purple in colour during the deformation. The PFs show that the orientations are much closer to [110] with slight spreading after deformation (Figure 6.4i) than in the as-DS condition (Figure 6.4h).

6.3.2.2 Microstructure of the sample within the non-necking region

The high magnification FSE and IPF-LD maps in Figure 6.5b – 6.5h were taken within the blue (2) orientation at different stages of creep in the red-squared region within the non-necking region in Figure 6.5a. The FSE image clearly indicates the dendrite and IMC eutectic regions

(Figure 6.5b). It is noticed that there are 3 small orientations shown within the blue $\langle 110 \rangle$ orientation, e.g. the purple, yellow and cyan orientations in the IPF-LD in the as-DS condition (Figure 6.5c). Initially, the misorientation hot spots are located within the eutectic regions and at the grain boundaries which are indicated with red arrows in the MO av. map (Figure 6.5d).

For the early stage of secondary creep (Figure 6.5e and 6.5f), local misorientation develops more in the eutectic regions and near grain boundaries and starts to grow in the β -Sn dendrites with subgrain formation (indicated with red and yellow arrows in Figure 6.5f respectively). The small purple orientation seems to grow larger in the corresponding IPF-LD map (Figure 6.5e).

In tertiary stage creep (Figure 6.5g and 6.5h), the orientation of the region changes slightly by rotation as noticed by the colour change (Figure 6.5g). The misorientation increases significantly across the map with development of subgrains in both dendrite and eutectic regions (Figure 6.5h), which is a result of the incoherent boundaries between IMCs and β -Sn and polygonization. New crystal orientation (cyan) is formed and located near the non-indexed features and in the eutectic regions, where more deformation is accommodated (Figure 6.5g). Non-indexed black features appear in the map, which are associated with increasing roughness (Figure 6.5g and 6.5h).

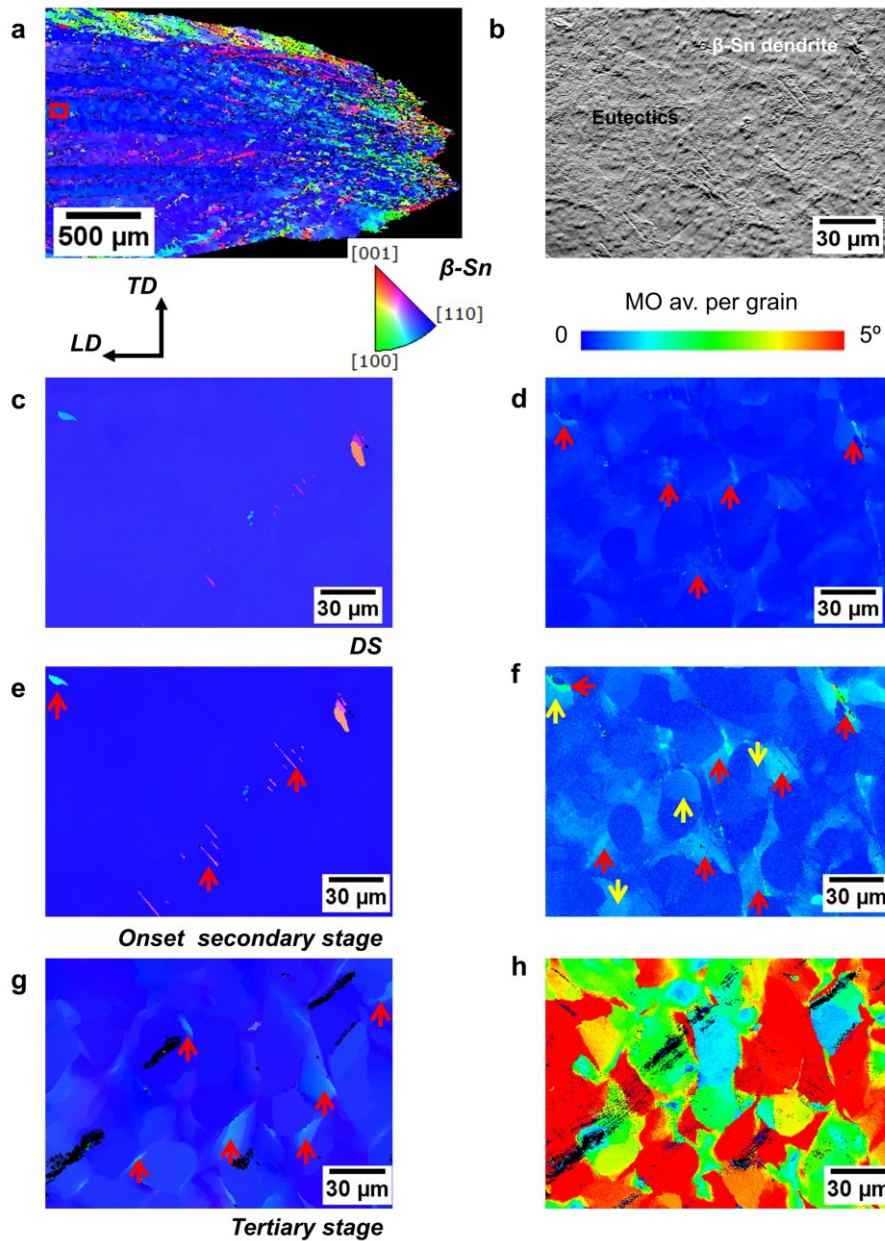


Figure 6.5. EBSD crystal orientation mapping showing evolution of microstructural heterogeneity highlighting evolution within the uniformly deformed region of the bi-crystal sample (only the β -Sn has been indexed here). (a) Large scale IPF-LD map after fracture. (b-h) Higher magnification maps of the red squared region in (a) within the non-necking region at different stages of creep. (b) FSE showing the morphology of the dendrite and eutectic regions at as-DS condition. (b, c, d) at as-DS condition, (e, f) at the onset of secondary stage creep, and (g, h) in tertiary stage creep. With increasing strain, the orientation in the IPF-LD map changes in the strain localised regions (c, e, g) but the MO av. maps show a significant increase (the MO hot spots in dendrite and eutectic regions are indicated with yellow and red arrows respectively) (d, f, h).

6.3.2.3 Microstructure of the sample within necking region

Within the necked region (Figure 5.9a) there are larger areas of recrystallised material. The FSE image within the necked region (the red squared region in Figure 5.9b) shows soft β -Sn flowed around the hard IMC phase. The relatively high surface roughness appears at the dendrite and eutectic interface, where recrystallised grains (Figure 5.9c) and regions of high misorientation are obtained (indicated with white arrows in Figure 5.9d). The recrystallised grains have green / cyan colour and close to $[100]$ orientation.

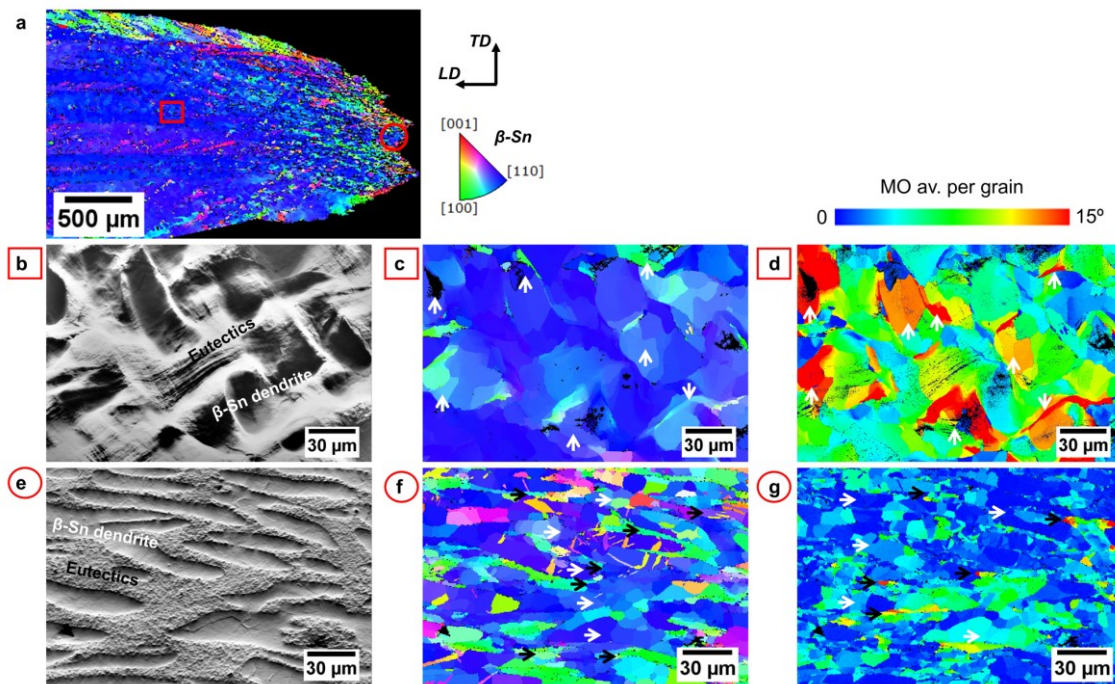


Figure 6.6. EBSD crystal orientation mapping showing evolution of microstructural heterogeneity highlighting evolution within the necking region (only the β -Sn has been indexed here). (a) Large scale IPF-LD map at fracture. (b-g) Higher magnification maps of the same region in (a) within the necking region, (b-d) in the red squared region, (e-g) in the red circled region at the fracture surface. With increasing strain, there is a significant increase in surface roughness in the FSE (b, e), growth in recrystallised grain in the eutectic region (c, f), and change in misorientation in the MO av. map (d, g). (e-g) are taken from the polished surface of the sample after fracture.

At the fracture surface (Figure 5.9e – 6.6g), the location of the eutectic and dendrites and the IMC length-scale are shown in the FSE image (Figure 5.9e). Fine recrystallisation has been

generated near the fracture surface (Figure 5.9f), the recrystallised grains in the eutectic regions (indicated with black arrows) have much smaller grain size than in the β -Sn dendrites (indicated with white arrows), which is consistent with the results in Chapter 4 and Chapter 5. A decrease in misorientation within each subgrain is observed, implying the stored energy has been released and this is likely due to the recrystallisation process.

6.3.3 [110] [110] [110] tri-crystal sample at 393 K

6.3.3.1 Strain field and crystal orientation

The sample tested at 393 K ($\frac{T}{T_M} \sim 0.80$) experienced heterogeneous deformation during creep (Figure 6.7). The as-DS sample has 3 main orientations labelled as 1, 2 and 3 in the as-DS IPF-LD map in Figure 6.7a.

The DIC figures Figure 6.7b and 6.7c) show heterogeneity in deformation even between the primary and early secondary stage creep. The strain at the onset of secondary stage creep in Figure 6.7b decreases to 0.1% (0.28% for the sample at 333 K in Figure 6.4b), which means the decrease of onset secondary creep strain is associated with increasing of temperature. The 3 main grains are deformed by rotating in the same direction towards the [100] orientation (Figure 6.7a and 6.7g).

As the deformation continues (Figure 6.7d- 6.7f), more strain is localised at the red squared region of the sample, where more grain boundaries are present Figure 6.7g). The sample forms a neck and fractures at the region (Figure 6.7f), so the grain boundary controls the heterogeneous deformation. The orientation changes within the necking region, the purple (2) and light blue (3) are changed to a green colour (orientation 4) close to [100]. The length of the

sample has been elongated approx. 36% from its original length, reduced 60% and 5% in cross-sectional area. The PFs show the significant spread in orientation and slight rotation in orientation around the [001] axis Figure 6.7h and 6.7i).

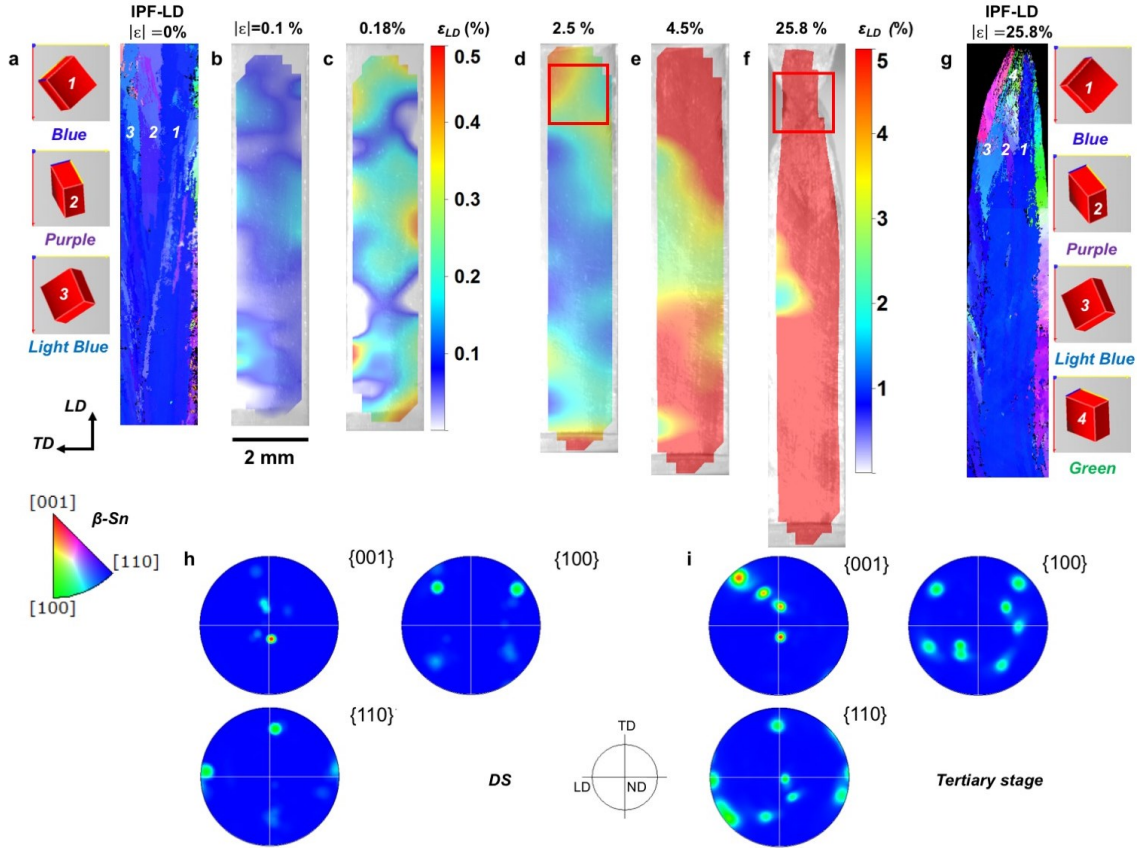


Figure 6.7. DIC figures and EBSD maps of a 'fine' microstructure tri-crystal sample crept at 393 K. (a) IPF-LD map after DS ($\epsilon_{LD} = 0\%$) with main crystal orientations shown. (b-c) DIC figures interrupted near onset of secondary stage creep at strain level (ϵ_{LD}) 0.1% and 0.41% respectively. (d-f) DIC figures at tertiary stage creep (fracture) at creep strain level 2.5%, 4.5% and 25.8% respectively. (g) IPF-LD map at fracture ($\epsilon_{LD} = 25.8\%$) with the main orientations shown. PFs of the sample at DS condition (h), at tertiary stage creep (i).

6.3.3.2 Microstructure of the sample within the non-necking region

The role of grain boundaries on heterogeneous creep deformation of the sample is presented within the uniformly deformed (non-necking) region is shown in Figure 6.8. The

microstructural evolution is observed from the high magnification maps (Figure 6.8b - 6.8h). The maps are taken within the red squared region, which are located at the grain boundary between the blue (1) and purple (2) orientations in Figure 6.8a. The dendrite and eutectic regions are distinguished in the FSE image (Figure 6.8b). Figure 6.8d has relatively high misorientations in the as-DS condition, which are localised in the eutectic region at the grain boundary and in the dendrite within the purple grain (indicated with red and yellow arrows respectively), which might be caused by the CTE mismatch during cooling at the end of solidification and the softness of Sn that will probably experience some deformation by changing in the orientation during handling (Figure 6.8c).

The misorientations decrease from the as-DS condition (Figure 6.8d) to the onset of secondary creep (Figure 6.8f) due to the recovery at high temperature (393 K). No obvious change in the orientation is observed in the IPF-LD map (Figure 6.8e).

Significant subgrains are developed during the tertiary stage creep in both the eutectic regions and β -Sn dendrites (Figure 6.8g and 6.8h). The highest misorientation spots (as indicated with the red arrows) are located at the grain boundaries and around IMCs, where dislocations concentrate (Figure 6.8h). The orientation changes slightly as shown by the colour difference at the grain boundary in Figure 6.8g).

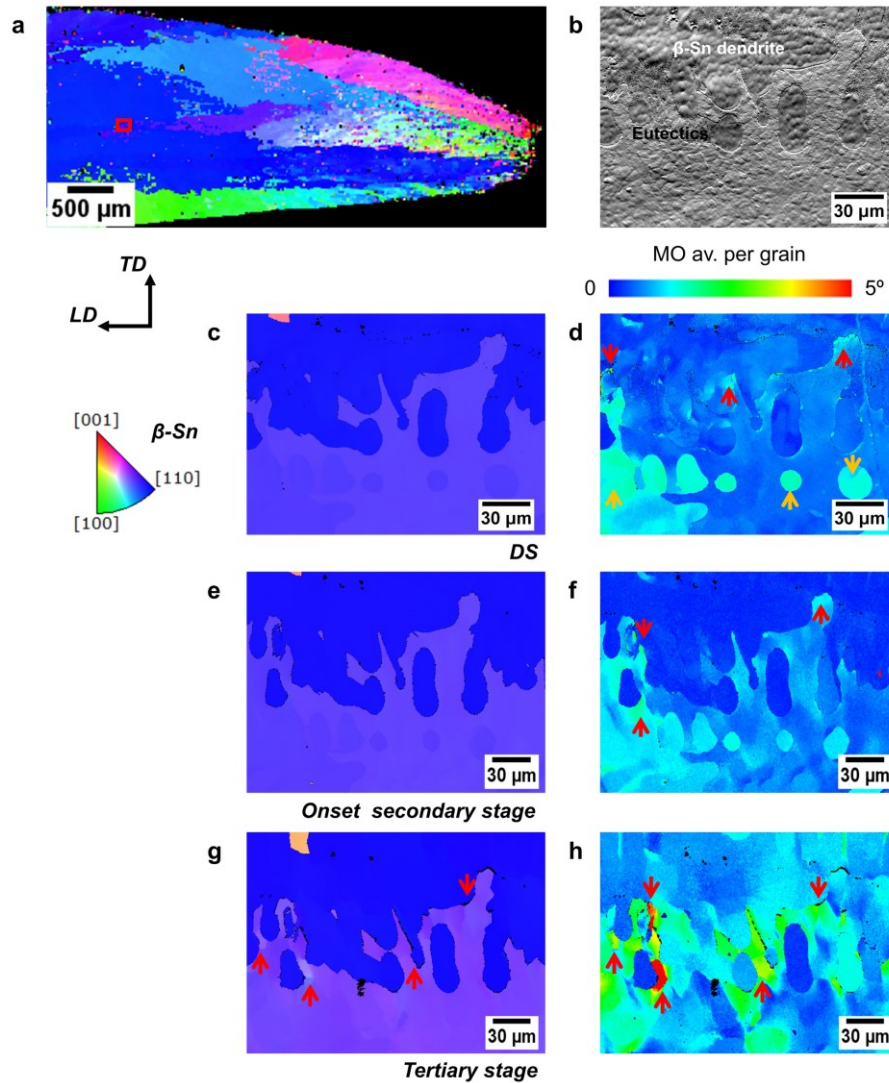


Figure 6.8. EBSD crystal orientation mapping showing evolution of microstructural heterogeneity highlighting evolution within the uniformly deformed region of the tri-crystal sample (only the β -Sn has been indexed here). (a) Large scale IPF-LD map after fracture. (b-h) Higher magnification maps of the red squared region in (a) within the non-necking region at different stages of creep. (b) FSE showing the morphology of the dendrite and eutectic regions at as-DS condition. (b, c, d) at as-DS condition, (e, f) at the onset of secondary stage creep, and (g, h) in tertiary stage creep. With increasing strain, the orientation in the IPF-LD map changes in the strain localised regions (c, e, g) but the MO av. maps show a significant change, especially near the grain boundary in eutectic regions and around IMCs (the MO hot spots in dendrite and eutectic regions are indicated with yellow and red arrows respectively) (d, f, h).

6.3.3.3 Microstructure of the sample within the necking region

The difference in microstructural deformation between the dendrite and eutectic regions is illustrated in Figure 6.9. The as-DS sample has 3 main crystal orientations, which are close to [110].

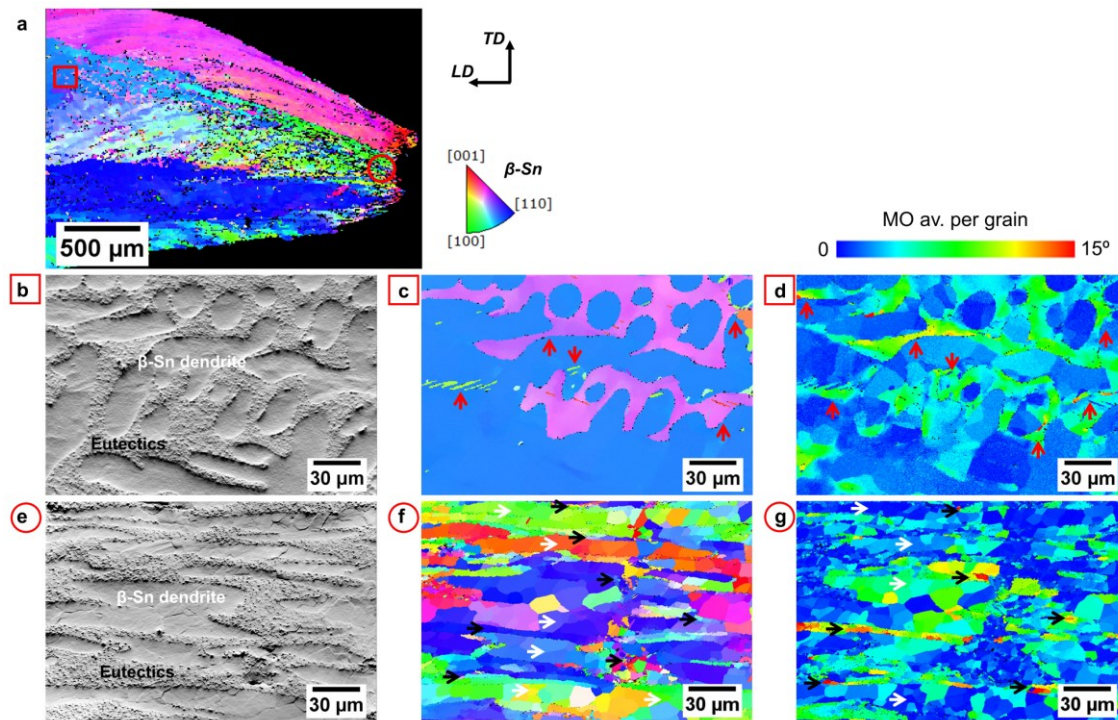


Figure 6.9. EBSD crystal orientation mapping showing evolution of microstructural heterogeneity highlighting evolution within the necking region (only the β -Sn has been indexed here). (a) Large scale IPF-LD map at fracture. (b-g) Higher magnification maps of the same region in (a) within the necking region, (b-d) in the red squared region, (e-g) in the red circled region at the fracture surface. With increasing strain, there is a significant increase in surface roughness in the FSE (b, e), growth in recrystallised grain in the eutectic region (c, f), and change in misorientation in the MO av. map (d, g). (e-g) are taken from the polished surface of the sample after fracture.

In the necking region, the dendrite (large smooth region) and the eutectic regions (containing standing-out features) are distinguishing clearly by near field FSE imaging (Figure 6.9b and 6.9e). Slightly stronger deformation is observed in the IMC-containing eutectic regions, which has new orientations near [001] (pink and yellow) formed in Figure 6.9c (as indicated by red arrows in Figure 6.9a). The MO av. map shows high misorientations are located in the eutectic

regions (as indicated with red arrows in Figure 6.9c), where a significant amount of subgrains formed in both dendrite and eutectic regions (Figure 6.9d). The length-scale of the subgrains is controlled by the dendrite arm spacing and the size of the IMCs.

At the fracture surface, there is a generation of rainbow-colour recrystallisation (Figure 6.9f) and a change in misorientation (Figure 6.9g). The constrained misorientation (Figure 6.9d) has been released mostly due to the generation of recrystallisation, but the MO av. is still relatively high in some of the eutectic region (the dendrite and eutectic regions are indicated with white and black arrows respectively). Again, the eutectic β -Sn are recrystallised with much smaller grain size than in the dendrite.

6.4 Summary of the Results

Temperature (K)	Initial orientation	Final orientation	% elongation in length	% reduction in cross-sectional area at fracture surface	% reduction in cross-sectional area at uniformly deformed region
298	[110]	[110] [100]	9	32	6
318	[110][100]	[110][100][001]	27	50	4
333	[110] [110]	[110][110][100]	35	38	8
363	[110][110][100]	[110][110][100]	23	56	5
393	[110][110][110]	[110][110][100]	36	60	5
423	[110][100]	[110] [100]	30	59	2
453	[110][110][100]	[100][110][100]	31	42	7
473	[110][110][100]	[110][110][100]	32	50	15

Table 6.3. The orientation, elongation and cross-sectional reduction of each sample.

The orientation and creep results for all the samples are summarised in Table 6.3. With involvement of temperature, the sensitivity to microstructure increases. Both the elongation in length and reduction in cross-sectional area increase with temperature and vary with orientation of β -Sn in the sample. The samples with main orientation near [110] have more elongation and less reduction in cross-sectional area.

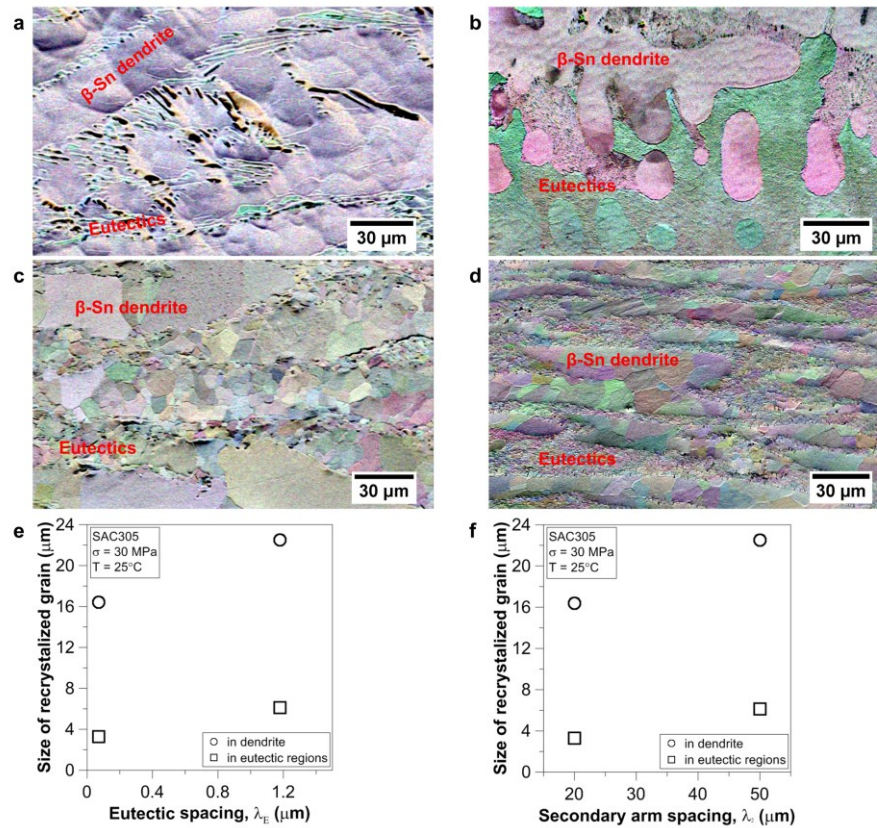


Figure 6.10. Comparison of the size for subgrain and recrystallised grains between the 2 microstructures from the samples at 393 K. (a-d) FSE images showing the morphology of the β -Sn dendrite and IMC together with the orientation contrast between sub-grains and recrystallised grains. The subgrains are shown in (a, b), the recrystallised grains are illustrated in (c, d) at tertiary stage within the uniformly deformed and the highly-strained regions of the sample respectively. (a-d) The subgrains and recrystallised grains are related to the initial microstructural length-scale of the sample (dendrite arm spacing, eutectic spacing and eutectic IMC size). The FSE images are taken at far position (~ 30 mm) between detector and sample. (e, f) The relationship between size of the recrystallised grains and eutectic spacing (e), and secondary arm spacing (f).

It is observed that the sub- and recrystallised β -Sn grains generated by the sample with finer microstructure (Figure 6.10b and 6.10d) are much smaller than in the samples with a coarser microstructure (Figure 6.10a and 6.10c). The generated subgrains and recrystallised grains are related to the microstructural length-scale of the initial sample (SDAS, eutectic spacing and eutectic IMC size). The relationship between the size of the recrystallised grains and SDAS and eutectic spacing are plotted in Figure 6.10e and 6.10f to quantify the change in size of recrystallised grains within dendrite and eutectic regions separately. These suggest that the size

of the recrystallised grains depends on the IMC size/spacing and dendrite arm spacing of the sample. Furthermore, the recrystallised grains in the eutectic regions have much smaller grain size than in the β -Sn dendrites for the two different microstructures.

6.5 Summary of the Chapter

The effect of change in microstructure length-scale, i.e. decrease in secondary arm spacing, eutectic spacing and IMC size, on the creep behaviour and microstructural evolution of DS SAC305 solder alloys have been studied at a constant stress of 30 MPa at a range of temperatures from 298 – 473 K. The following conclusions can be drawn from this work.

1. Heterogeneous deformation occurred during creep. The deformation mechanism for the samples with a finer microstructure (growth rate of $v = 200 \mu\text{m/s}$) is consistent with the results in Chapter 4 and Chapter 5, i.e. at the microscopic scale, the initial deformation of the sample starts in the β -Sn within the eutectic region near IMCs because dislocations often concentrate against the hard particles (IMCs) within a β -Sn grain (see Section 2.1.2). The soft phase, β -Sn, deforms by in-grain lattice rotation to form subgrains with high misorientation and leads to recrystallisation in tertiary stage creep. However, the size of the formed subgrains and recrystallised grains are significantly smaller than in samples grown at slower rate of $v = 20 \mu\text{m/s}$, which has a coarser microstructure (Chapter 5). Therefore, the initial microstructure of the sample, i.e. dendrite arm spacing, and eutectic spacing as well as the IMCs size controls the size of recrystallisation.
2. At the macroscopic scale, the deformation heterogeneity is associated with the presence of the β -Sn grain boundary. The misorientation starts to appear at the grain boundary,

which results in dislocation pile up to form subgrains leading to grain recrystallisation and crack nucleation at the grain boundary. The deformation (stress) is relatively easier to concentrate at the grain boundary transverse to the loading direction than one parallel to the loading direction. Furthermore, the misorientation increases with increasing deformation for samples tested at low temperature but, for samples tested at high temperatures, decreases first from the as-DS condition to secondary stage creep, then increases with further deformation. This phenomenon was the same for samples with the 2 different microstructures.

3. Longer creep lifetime, smaller secondary creep strain rate and lower onset of secondary creep strain are obtained by refining the microstructure of the sample at the same temperature. The activation energy, Q is 34 kJ/mol in the low temperature regime (298 – 333 K) and 6 kJ/mol in the high temperature regime (363 – 473 K). The change of creep mechanism from climb-controlled dislocation creep to lattice diffusion creep happens at homologous temperature of $\sim \frac{T}{T_M} = 0.74$. These changes imply that less energy is required for dislocations to climb during creep, which means that the deformation is dominated by obstacle-controlled dislocation motion, especially in the low temperature regime. In the high temperature regime, the creep deformation is mainly controlled by grain size (diffusion limited deformation), therefore less effect of IMC size and spacing was obtained.
4. The ductility of the samples decreased with coarsening the microstructure and the deformation became less localised (more deformation was introduced in the uniformly deformed region compared to the samples tested in Chapter 5).

Chapter 7 In-situ observation on creep behaviour of SAC305 solder

7.1 Introduction

Chapter 4, Chapter 5 and Chapter 6 demonstrate that the deformation process of DS SAC305 is heterogeneous and controlled by grain polygonization and recrystallisation, especially with respect to IMCs. The size of the recrystallised grains depends on the microstructure of the sample. Also, the two distinct microstructural regions, i.e. primary β -Sn dendrites and IMCs-containing eutectic β -Sn deform differently during creep. However, the following questions are remained to be fully addressed:

- (1) Does the recrystallisation occur before or after the creep failure?
- (2) When and where does exactly the recrystallisation take place?
- (3) More importantly, which microstructure is controlling the creep deformation and how does the strain accumulate in the 2 distinct microstructural regions?
- (4) How does the microstructure evolve once the sample reaches tertiary stage creep?
- (5) And for how long does the sample take from recrystallisation to crack initiation?

In this chapter, the above questions are answered by further observing the microstructural evolution on controlled microstructure of SAC305 solder using in-situ creep testing together with FSE and EBSD imaging.

7.2 Experiment

The creep test was performed with the 2 kN Gatan Mtest 2000E tensile stage (Figure 5.1 in Chapter 5) at room temperature (~ 298 K) and constant stress of 30 ± 2 MPa inside a Quanta FEI SEM. The test was carried out in tensile mode under load control until sample fracture without interruption. The microstructural deformation of the sample was observed in-situ using FSE and EBSD. Before the test, a small notch was made at the centre of the gauge behind the imaged area to ensure the sample fractures on top of the notch and the imaging was focused there.

Auto-FSE and auto-EBSD scans were set up in Bruker ESPRIT 2.2 software to track the microstructural evolution during the deformation. The far field FSE images were taken at a detector distance of ~ 30 mm with $1.2 \mu\text{m}$ step size and 1 minute capture time to optimise orientation based electron contrast. After the FSE imaging, the detector was moved automatically to the EBSD position (~ 10 mm) and the EBSD scan started directly as soon as the detector reached the set position. The EBSD maps were captured at $1.2 \mu\text{m}$ step size with 5×5 spatial binning (i.e. an ultimate EBSD step size of $6 \mu\text{m}$). This significantly reduces the mapping time to achieve the orientation of the sample. The duration of each auto-FSE and auto-EBSD pair was approx. 7 minutes. The as-DS microstructure and orientation of the gauge for the sample were scanned using a large-scale EBSD map before with step size of $12 \mu\text{m}$ inside the load frame before loading. After that, the creep test was performed and the auto-imaging was stopped until sample fracture. The orientation of the sample was obtained by a large-scale EBSD map after deformation (fracture).

7.3 Results and Discussion

7.3.1 Creep strain curves

Figure 7.1a shows the creep strain curves until a creep strain of ~36% (failure strain). Overall, the sample deformed with three stages corresponding to the difference in creep strain rate. The change in creep strain rate is illustrated by creep strain (%) vs. strain curves (Figure 7.1b). In the primary stage creep, the strain increases rapidly due to the continuous dislocation formation and rearrangement. When the deformation enters secondary stage, the creep strain rate decreases to a nearly constant value and it occupies most of the creep lifetime. With further deformation, the creep strain rate starts to increase again causing the failure of the sample. The strain at the onset of secondary and tertiary stage creep can be determined from Figure 7.1b, with a value of 1.9 and 16% respectively. The corresponding microstructural evolution at each stage of creep is explored in-situ in the next section.

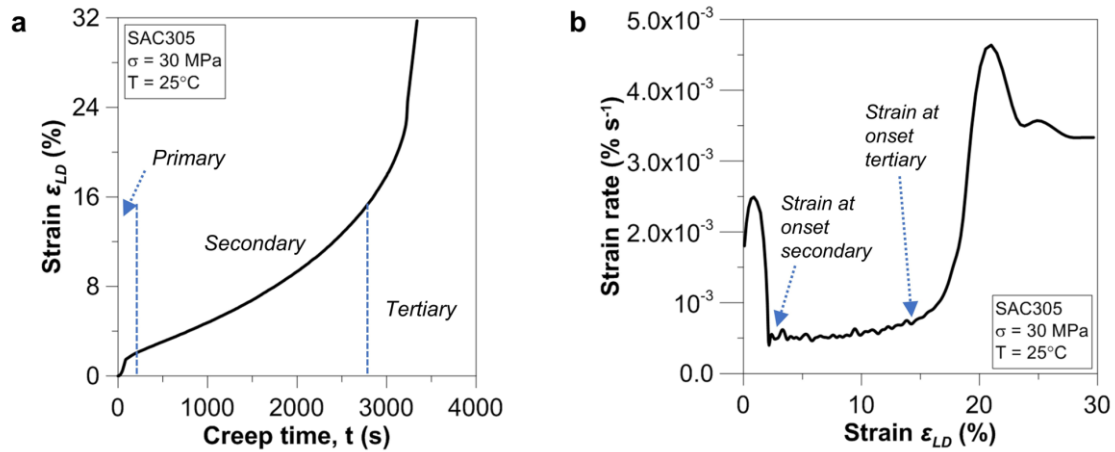


Figure 7.1. Fixed load (30 MPa) mechanical creep data of SAC305 samples grown at $v=20 \mu\text{m/s}$. (a) Creep curves from primary to tertiary stage creep with three stages labelled. (b) Creep strain rate vs. creep strain curves showing a gradient near 0 after the secondary stage creep is reached. The strain at onset secondary and tertiary stages are labelled.

7.3.2 Deformation of [100] [110] bi-crystal sample

The rotation of grain orientation together with continuous formation of subgrain and recrystallisation are observed during the in-situ creep deformation at room temperature. The sample forms two main orientations after DS, which are close to [100] and [110] as presented in large-scale IPF-LD map in (Figure 7.2a). The two microstructural regions (dendrite and eutectic regions) deformed differently and the initial heterogeneity in deformation is observed in the eutectic regions and near grain boundaries (Figure 7.2).

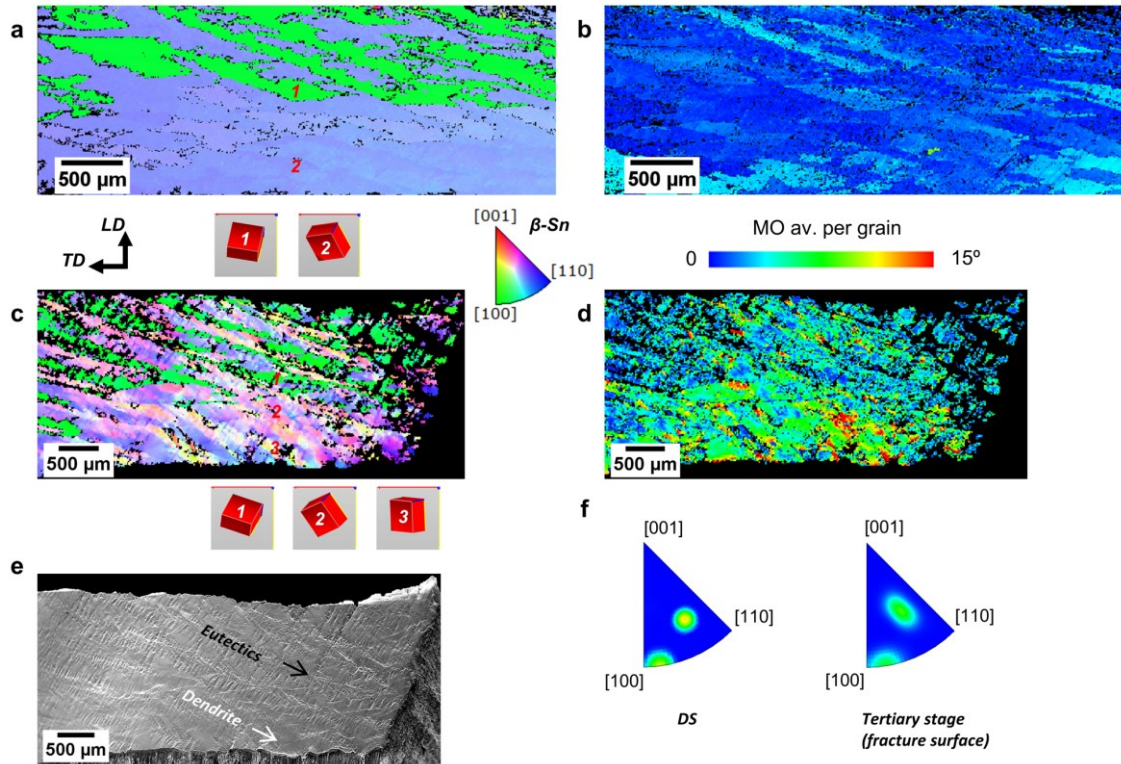


Figure 7.2. EBSD crystal orientation mapping showing microstructural evolution of a bi-crystal SAC305 sample (grown at $v=20 \mu\text{m/s}$) deformed under fixed load (30 MPa) and at room temperature (298 K). (a, b) Large scale maps after DS ($\epsilon_{LD} = 0\%$) with main crystal orientation shown. (c-e) Large scale maps at fracture ($\epsilon_{LD} = 32.3\%$) with the main orientation shown. With increasing strain, there are significant orientation change in the IPF-LD map (a, c) and increase in misorientation in the MO av. maps (b, d). The dendrite and eutectic regions are indicated in the SE image (e). The change in orientation of the sample are given in the IPFs at DS condition and tertiary stage creep (f).

The macroscopic deformation of the sample can be seen in Figure 7.2. Obvious colour difference is observed in the [110] purple (2) orientation before (Figure 7.2a) and after deformation (Figure 7.2c) suggesting the [110] grain experiences a faster rotation and spreads in orientation towards [110] and [001] continuously than the [100] grain illustrated by the IPF map in Figure 7.2f. The dendrite and eutectic regions can be distinguished clearly from the SE image with dendrite and eutectic region labelled in Figure 7.2e. New orientation yellow (3) is formed (Figure 7.2c) in the regions with high misorientation (Figure 7.2d), which is located in the eutectic regions (Figure 7.2e) and near the grain boundaries between the two orientations due to the heterogeneity in rotation between the two orientations (Figure 7.2c). The overall misorientation increases with deformation from the as-DS condition (Figure 7.2b) to the fracture of the sample (Figure 7.2d), which is associated with increasing roughness between the two microstructural regions (dendrite and eutectic region) and segmentation of the grain. The evolution of microstructural morphology and orientation of the bi-crystal sample is revealed by in-situ FSE and EBSD at different stage of creep in the sections below.

7.3.2.1 Microstructural evolution from primary to early secondary stage creep

The microstructural evolution of the sample is revealed between primary to early secondary stage creep in Figure 7.3. The deformation is not obvious and starts to develop at the interface between dendrite and eutectic region, as well as at the grain boundary, due to the phase and grain boundary heterogeneity.

The creep curve of the sample is given in Figure 7.3a. 8 groups of microstructural images are captured correlating to the three stages of creep.

Figure 7.3b – 7.3g show the microstructure of the sample at primary and early secondary stage creep with slight plastic deformation. The FSE image at as-DS condition shows a large contrast not only between grains and subgrains but also between the two microstructural regions, i.e. dendrite and eutectic regions, which is a result of the difference in topography between the two microstructural regions (Figure 7.3b). The as-DS sample has two main orientations, green (1) near [100] and purple (2) near [110] (Figure 7.3c). There are some regions with slightly higher misorientation, which is associated with the presence of subgrains (Figure 7.3d).

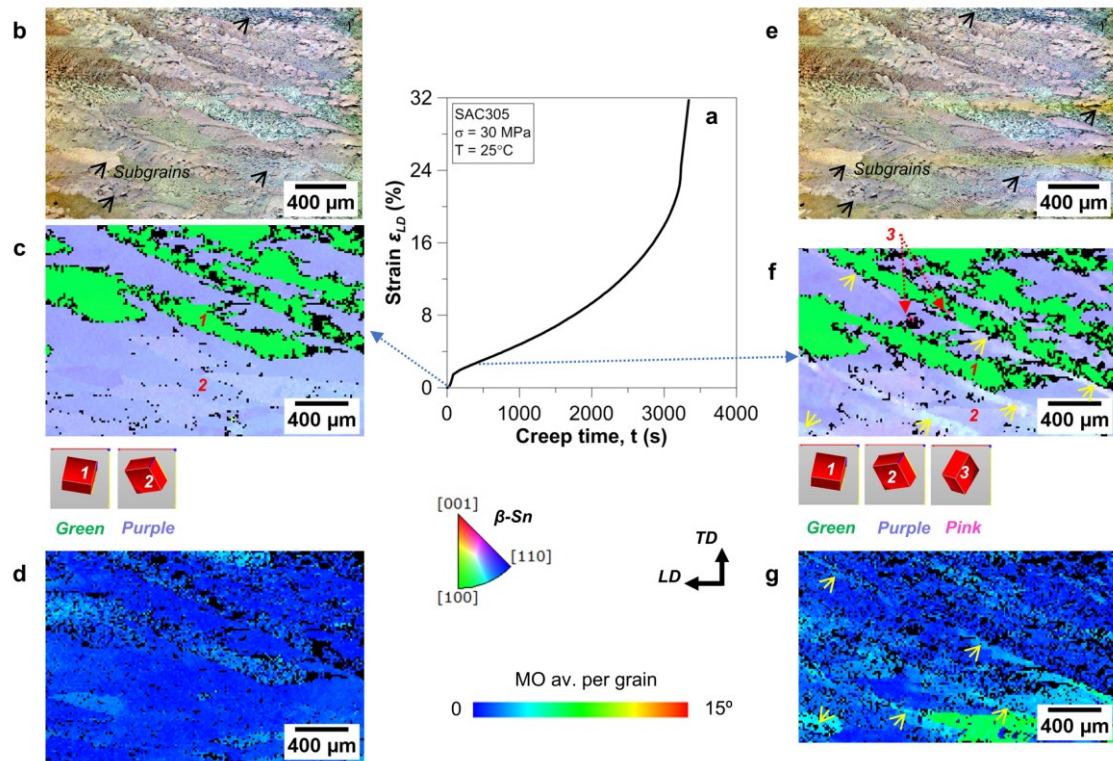


Figure 7.3. EBSD crystal orientation mapping showing microstructural evolution of a bi-crystal sample from primary to early secondary stage creep (only the β -Sn has been indexed here). (a) Creep curve from primary to tertiary stage creep with mapping position labelled. (b-g) EBSD maps at as-DS condition ($\epsilon_{LD} = 0\%$) (b-d), at early secondary stage ($\epsilon_{LD} = 3.9\%$) (e-g). The morphology of the dendrite and eutectic regions and contrast between the grains are shown in the FSE maps at as-DS condition (b) at early secondary stage creep (e) the subgrains are indicated with black arrows. With increasing strain, the orientation changes in the IPF-LD map (c, f) and misorientation increases in the MO av. maps (d, g) in the strain localised regions, especially near the grain boundary and at interface between dendrite and eutectic region (indicated with yellow arrows).

With increasing strain (Figure 7.3e – 7.3g), the small new orientation pink (3) appears at the grain boundary and it is also the location of the interface between the dendrite and eutectic region (Figure 7.3f). Slight colour change (light yellow) is observed in Figure 7.3f at the interface between dendrite and eutectic region due to the orientation rotation, leading to an increase in misorientation (indicated with yellow arrows in Figure 7.3f and 7.3g). The main orientations and the morphology of the microstructure in the scanned region show no change between the two stages of creep (Figure 7.3b and 7.3e).

7.3.2.2 Microstructural evolution during secondary stage creep

In secondary creep, the heterogeneity in deformation develops continuously (Figure 7.4). Significant increase in surface roughness, rotation in orientation and increase in misorientation are obtained especially in eutectic regions and at grain boundaries.

With continuous deformation (Figure 7.4b – 7.4m), the grain contrast in the FSE maps reduced significantly due to the increase in surface roughness (Figure 7.4b, 7.4e, 7.4h, 7.4k) which also causes less indexing in the EBSD map (Figure 7.4c, 7.4f, 7.4i, 7.4l). It is noticed that ‘flow’ lines appear gradually in the eutectic regions around IMC particles (indicated with black arrows in Figure 7.4e, 7.4h, 7.4k) and the ‘flow lines become more obvious with increasing strain. This means that the soft β -Sn phase flows around the hard IMC phase and forms several small bumps shown as black features.

In addition, massive new subgrain boundaries are formed (Figure 7.4c, 7.4f, 7.4i, 7.4l) during the secondary stage creep due to gradual increase in misorientation and the high misorientations (indicated with yellow arrows in Figure 7.4d, 7.4g, 7.4j, 7.4m) are located in the regions with high surface roughness. Due to the grain rotation, the colour difference becomes more obvious

along the grain boundary and at the interface between dendrite and eutectic region to form a new orientation yellow (4), which are annotated with yellow arrows in (Figure 7.4c, 7.4f, 7.4i, 7.4l). Meanwhile, small new orientations appear increasingly at the grain boundary with pink (3) and blue (5) colours.

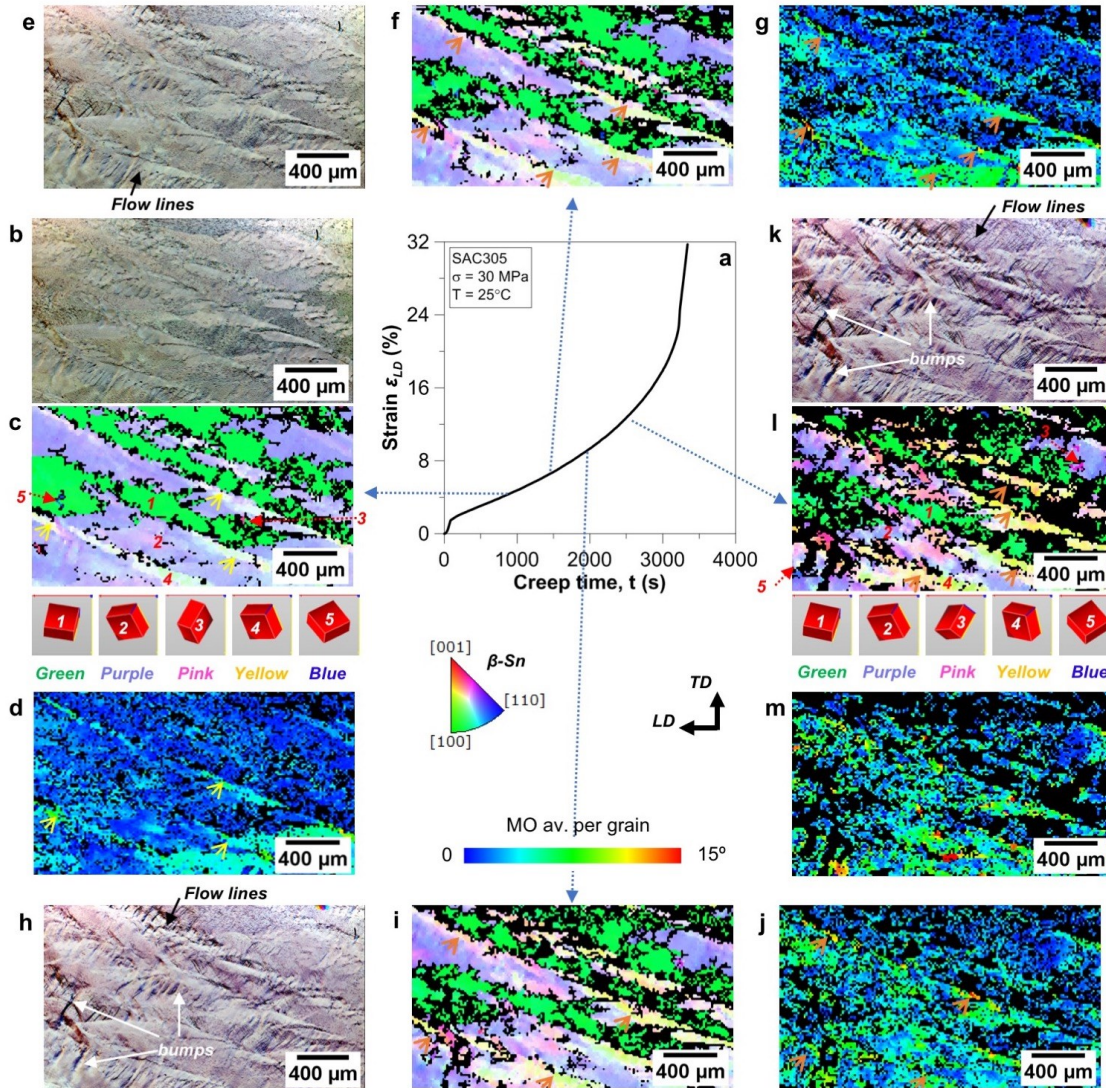


Figure 7.4. EBSD crystal orientation mapping showing microstructural evolution of a bi-crystal sample during secondary stage creep (only the β -Sn has been indexed here). (a) Creep curve from primary to tertiary stage creep with mapping position labelled. (b-m) EBSD maps during secondary stage creep, at strain $\epsilon_{LD} = 6.4\%$ (b-d), at strain $\epsilon_{LD} = 8.9\%$ (e-g), at strain $\epsilon_{LD} = 11.7\%$ (h-j), and at strain $\epsilon_{LD} = 15.0\%$ (k-m). With increasing strain, the surface roughness increases with formation of 'flow lines' in the FSE maps (b, e h, k), the orientation changes with continuous subgrain formation in the IPF-LD map (c, f, i, l) and misorientation increases in the MO av. maps (d, g, j, m) in the strain localised regions, especially in the eutectic region and at the grain boundaries (indicated with yellow arrows).

7.3.2.3 Microstructural evolution from secondary to tertiary stage creep

In tertiary creep, the creep strain rate increases leading to acceleration of the microstructural deformation and fracture of the sample. Serious plastic deformation occurs in the strain

localised regions with significant formation of subgrains and recrystallisation, which is correlated with the location of the bumped black features and ‘flow lines’ in the FSE maps (Figure 7.5b and 7.5e). Microcrack formation is observed early during the tertiary stage creep in the strain localised regions, where the black bumps are presented (Figure 7.4k). The microcrack then grows into a long crack and propagates through the sample in later tertiary stage creep (in Figure 7.5e).

The green and purple grains evolve differently during creep. The purple orientation (2) exhibits more colour change corresponding to much obvious rotation than the green orientation (1). The misorientation decreases here due to the continuous development of grain boundaries, which releases the constrained stored energy from pervious stages. Small recrystallised grains are observed at the grain boundaries with random orientations (indicated with red arrows in Figure 7.5c).

A region with high surface roughness (the highlighted region in Figure 7.5 e) is characterised by high magnification EBSD. The scanned map is located within the eutectic region, which has both Ag_3Sn and Cu_6Sn_5 phases identified in the green and blue colour respectively (Figure 7.5h) and has a green [100] orientation (Figure 7.5i). A large number of subgrains has been formed showing as high misorientation in the MO av. map (Figure 7.5j) and subgrain boundaries (dark lines) in the pattern quality map (Figure 7.5h). ‘Rainbow’ colour recrystallisation has been generated within the highly-bumped region (strain concentrated region) and the HAGBs are highlighted with red lines in Figure 7.5i. (A common misorientation angle with respect to ‘grain’ as identified) with EBSD thresholding of grain boundaries at 5°). The deformed microstructure has a wavy morphology with many ‘flow’ lines, which is attributed to the initial crystal orientation of the sample.

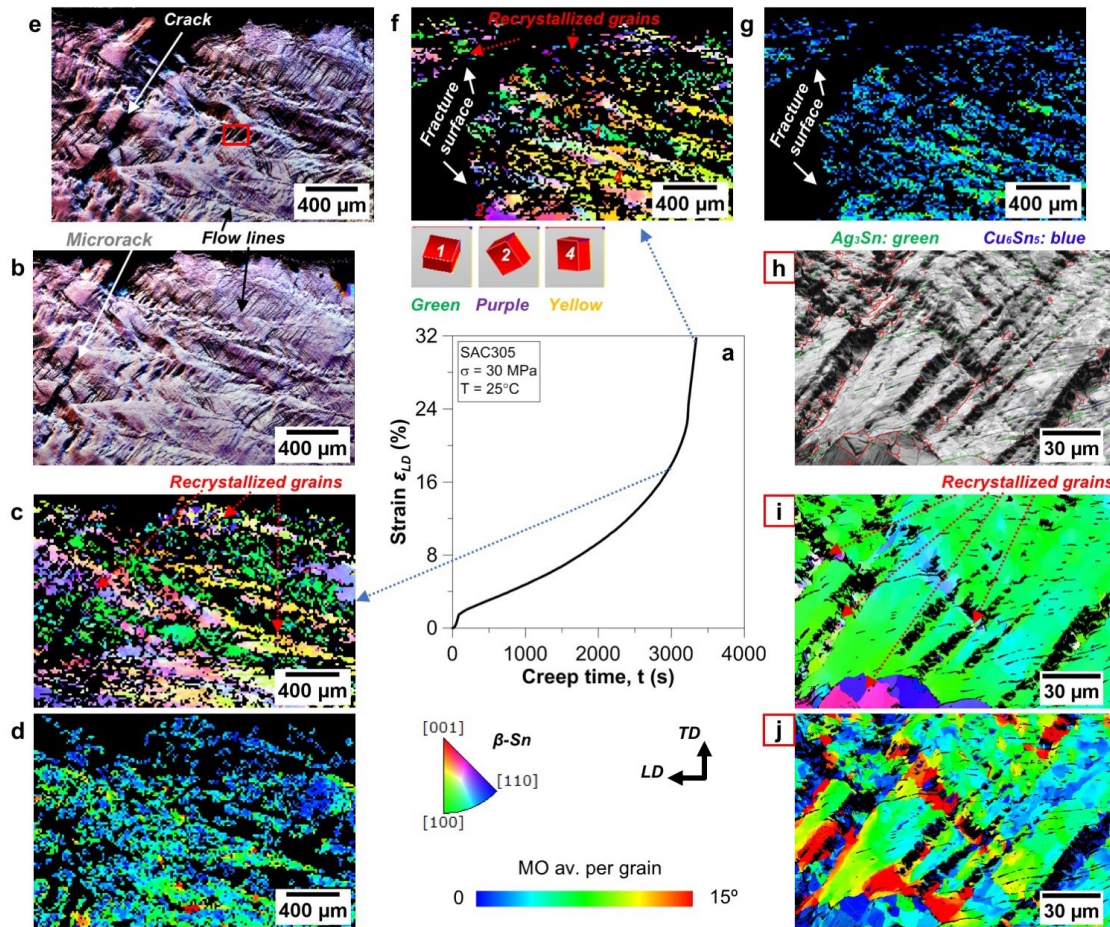


Figure 7.5. EBSD crystal orientation mapping showing microstructural evolution of a bi-crystal sample in tertiary stage creep (only the β -Sn has been indexed here). (a) Creep curve from primary to tertiary stage creep with mapping position labelled. (b-j) EBSD maps in tertiary stage creep, at strain $\epsilon_{LD} = 21.1\%$ (b-d), at strain $\epsilon_{LD} = 32.3\%$ (e-g). With increasing strain, the surface roughness increases with crack propagation along the 'flow lines' in the FSE maps (b, e), the orientation changes with continuous grain rotation in the IPF-LD map (c, f) and misorientation decreases in the MO av. maps (d, g) due to release of stored energy (d, g). (h-j) Higher magnification maps of the red squared region in (e), the high surface roughness is the eutectic region with Ag_3Sn (green) and Cu_6Sn_5 (blue) identified and high-angle grain boundaries highlighted (h), subgrain formation and recrystallisation are shown in MO av. map (j) and IPF-LD map (i) respectively.

7.4 Summary of the Chapter

The creep behaviour and microstructural evolution of DS SAC305 solder have been investigated through in-situ creep test under constant stress of 30 MPa and at room temperature

(~298 K). The following conclusions can be made, answering the addressed questions in the introduction.

1. The plastic deformation occurs during creep, which exhibits three stages including a strain hardening primary stage, a steady strain rate secondary stage and a strain accelerating tertiary stage.
2. The microstructural deformation evolved gradually by lattice rotation, which is influenced by the initial microstructural morphology and orientation of the sample. In the early stage of creep deformation, the grain with [110] orientation started rotating faster than the grain with [100] orientation. The misorientation increases to form subgrains at grain boundaries and IMC-containing eutectic regions due to the continuous dislocation formation and rearrangement. Small recrystallised grains are formed at the grain boundaries because of large heterogeneity in rotation between the orientations. During the steady stage creep, since recovery takes place, deformation accumulates constantly with increasing in surface roughness, formation of parallel ‘flow’ lines and the growth of subgrains (polygonization) in the strain localised regions. In the late stage of creep, the microstructural deformation accelerates in the region with high roughness to form significant subgrain and recrystallisation, leading to crack formation and fracture of the sample quickly.

Chapter 8 Thermal cycling of Cu/SAC305/Cu solder joints

8.1 Introduction

When electronics fail in service the cause of it is often thermomechanical failure of the solder joints. There is a strong demand in the oil and gas, automotive and aerospace industry to understand the mechanisms leading to thermomechanical fatigue failure, to be able to predict the joint microstructure that is most resistant to thermal cycling, and to develop ways of generating the optimum microstructure through alloy design and processing.

This chapter will be focused on investigating the role of strain localisation within a BGA SAC305 solder joints generated from CTE between the solder bulk and substrate, and microstructure evolution of the joint during thermal cycling. In order to reveal the microstructure and understand the fundamental deformation mechanisms of the SAC305 solder joint, an in-situ heating stage (Gatan Murano 525) is used together with EBSD inside the SEM chamber during thermal cycling. The microstructure development of the solder joint will be monitored during different number of thermal cycles.

Therefore, the following questions are addressed:

1. When does the microstructural deformation process of solder joint start?
2. Does the deformation increase with number of cycles?
3. How do the β -Sn grains (re)orientate?
4. Where does the strain accumulate?

5. What kind of microstructural deformation (recovery, recrystallisation, IMC coarsening and cracking) occurs?

8.2 Sample geometry and fabrication

In this project, a sandwich geometry solder joint (Cu-SAC305-Cu) was fabricated for the in-situ thermal cycling with SEM analysis, as illustrated in Figure 8.1. The sample has a dimension about $8 \times 8 \times 1.5 \text{ mm}^3$ and contains a full array of 5×5 solder balls with ball pitch size around 1 mm and pad size 0.5 mm (Figure 8.1). The solder balls were $\sim 500 \mu\text{m}$ in diameter, which connect two pieces of Cu substrates ($\sim 500 \mu\text{m}$ in thickness).

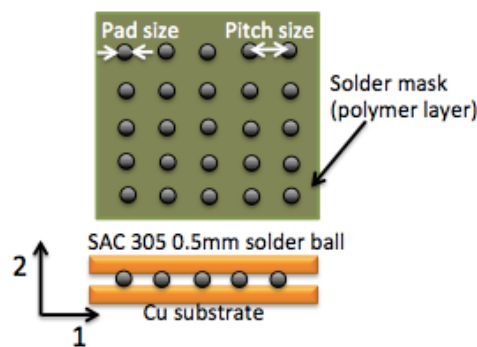


Figure 8.1. Schematic diagram of the Cu/SAC305/Cu solder joint geometry.

The solder balls were made from commercial SAC305 solder ingots, which were rolled into foils with thickness of $\sim 2.5 \text{ mm}$, then punched into thin disks. After that, the solder disks were melted on a hot plate at $\sim 250^\circ\text{C}$ and formed into near-spheres due to the surface tension ^[9].

The Cu substrates used were prepared by optical lithography with the following steps: (a) Cleaning – vibration cleaning was applied to eliminate contamination for the Cu substrates with acetone. (b) Pre-lamination – the substrates were baked in a hot laminator ($\sim 60^\circ\text{C}$) at the hottest position for 10 minutes and cooled under a dry environment for dehydration. (c) Masking – the solder mask and substrate were laminated together using laminating rollers. (d)

Lamination – the substrates were pressed through the laminator for 4 minutes and cooled under cold water to stabilise the mask layer and achieve good adhesion. (e) Exposure – the solder stop layer (semi-transparent paper with dots matrix) was placed on the substrates and was exposed to UV light for approximately 45 s with four 15-watt UV bulbs to project a mask pattern onto the substrates and then laid down in a dark place for 1 hour. (f) Development – an aqueous developer (1 L water and 10 g developer) at 45 °C was employed to develop the substrate by rubbing the substrates with a soft acrylic brush to profile the substrate.

After that, the solder balls were soldered to the Cu substrates with a mildly activated resin flux (RM-5) on a hot plate at a temperature of ~250 °C and then cooled to room temperature in air. The solder joint was then reflowed through a reflow oven (LFR400HTX TORNADO Surface Mount Technology, UK). The reflow profile measured in this experiment is given in Figure 8.2, which is undertaken under similar conditions as commercial BGA joints.

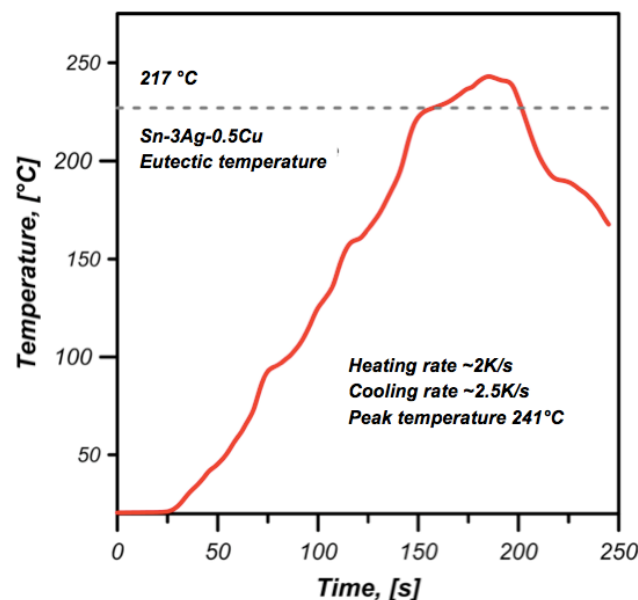


Figure 8.2. The applied reflow profile for solder joints.

All the samples were cleaned in an ultrasonic bath with ethanol to remove any flux residues and were then emerged in acetic acid for 20 minutes to remove the solder mask layer.

8.3 Experiment

8.3.1 Sample preparation

Since epoxy is not suitable for in-situ heat treatment experiments, an alternative polishing method was applied without mounting the joint inside epoxy. A Gatan Disk Grinder 623 was used to reveal the microstructure of the joint using the same recipe of polishing procedure as in Chapter 3. The joint was attached with superglue onto a specially made SEM sample stub, which can be held by the disk grinder, as shown in Figure 8.3. With the disk grinder, it is possible to control the polishing distance in ± 5 micrometres. When a satisfactory cross-sectional surface was obtained by optical imaging, the joint was detached from the sample stub by immersing inside acetone to dissolve all the superglue. The joint was then ion-beam milled using a PECSII at 3, 1 and 0.5 keV with a tilt angle of 3 and 1° respectively to flatten and improve the quality of the cross-sectional surface of the solder joint, required for a high resolution EBSD scan.

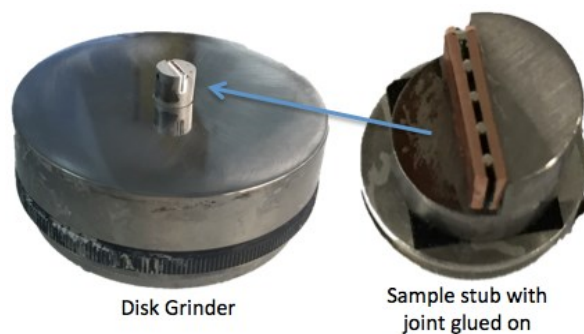


Figure 8.3. Picture showing the solder joint mounting on the disk grinder with a special SEM sample stub.

Afterwards, the joint was in-situ thermal cycled with EBSD scans inside A Quanta SEM.

8.3.2 Thermal cycling

In-situ thermal cycling was carried out with a Gatan Murano heating stage 525 inside the Quanta FEI SEM. A pre-tilted ($\sim 70^\circ$) heating module was used for EBSD imaging. The Murano stage contains a heater and thermocouple pins within its hotplate carriage assembly as shown in Figure 8.4a. Figure 8.4b shows the geometry of the sample mounting on the hotplate with EBSD shield on, which maximises the collection efficiency of EBSD patterns and reduces background noise from the heating and cooling process ^[153]. The joint was attached to the hotplate by using high temperature carbon paste.

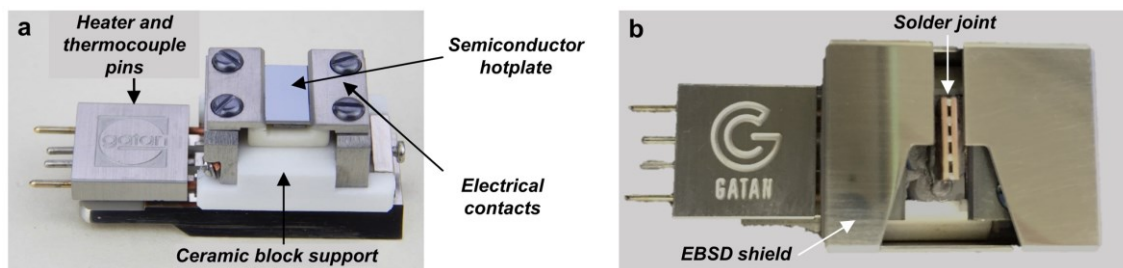


Figure 8.4. (a) Gatan Murano 525 hotplate carriage assembly modified from ref ^[153], (b) solder joint sample mounted on (a) with EBSD shield on.

The solder joint was thermally cycled from 20 to 150 °C and the dwell time was 10 minutes at both the lowest and highest temperature extremes to simulate conventional conditions. The heating rate was ~ 12.5 °C/min. and the cooling rate is ~ 4.5 °C /min as shown in Figure 8.5. The thermal profile is chosen according to the IPC9701 ^[154], which is a standard of “Performance Test Methods and Qualification Requirements for Solder”. The curve gives the measured temperature profile by the thermocouple inside the carriage assembly. An EBSD scan of the whole solder joint was performed every 12 cycles at room temperature with a step size of $1.8 \mu\text{m}$ and the solder / substrate interface was scanned with a step size of $0.2 \mu\text{m}$ (method described in Chapter 3). Due to limited equipment availability, between every 12 cycles (one overnight SEM section about 12 hours) the joint was detached from the hot plate

and put inside a vacuum desiccator to avoid oxidation.

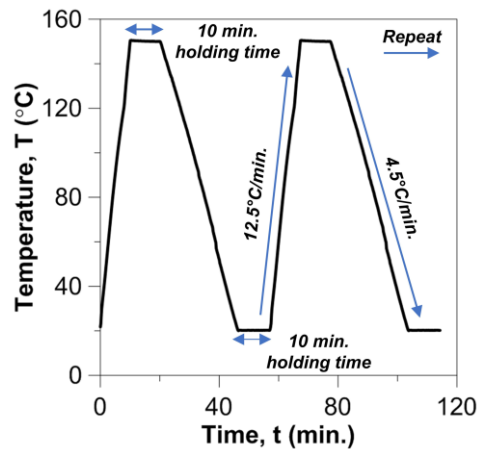


Figure 8.5. Thermal cycling temperature profile with dwell time, heating and cooling rate labelled on the curve.

8.4 Results and Discussion

8.4.1 Microstructure of as-soldered SAC305 joint

The as-soldered solder joint (Figure 8.6a) has a structure of β -Sn dendrite and IMC-containing eutectic region after solidification, which contains Ag_3Sn and Cu_6Sn_5 . The joint forms a reaction layer of Cu_6Sn_5 at the solder-substrate interface and large primary Cu_6Sn_5 in the solder matrix.

In soldering, the liquid solder ball reacts with the Cu substrate to form a layer of Cu_6Sn_5 at the solder / substrate interface (Figure 8.6b), where the Cu content increases because of Cu substrate dissolution into the solder matrix from the substrate. The primary Cu_6Sn_5 forms during the solder cooling process (Figure 8.6c) due to Cu dissolution into the alloy ^[18, 19].

During solidification, the β -Sn nucleates and grows into a dendritic morphology (Figure 8.6b), which occurs at the substrate and spreads into the liquid volume. The β -Sn and IMCs (Cu_6Sn_5

and Ag_3Sn) eutectic then grows in between dendrite arms to give the final microstructure of the solder joint as shown in Figure 8.6b. Figure 8.6d shows an area of etched eutectic in which the Cu_6Sn_5 and Ag_3Sn show a rod-like and a needle-like morphology respectively.

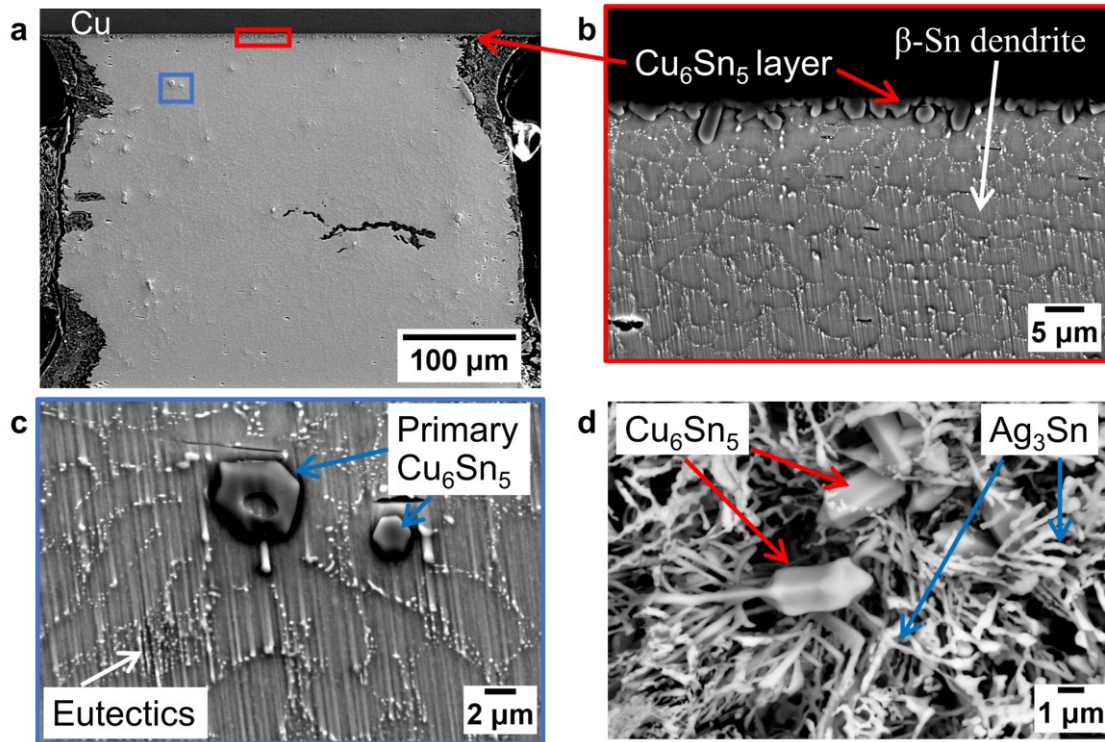


Figure 8.6. SEM images of an as-soldered solder joint. (a) SE image of the whole joint. (b,c) Higher magnification BSE image of (a) focusing on the solder / substrate interface (b), the microstructure within the solder matrix (c). (d) SE image of the etched eutectic regions showing the morphology of Ag_3Sn and Cu_6Sn_5 .

Figure 8.7a-8.7h show a typical single $\beta\text{-Sn}$ grain joint and a cyclic twinned joint with 3 orientations. The crystal orientation unit cells are superimposed on the IPFX map.

The single grain joint (Figure 8.7a) has only one peak shown on the misorientation histogram of $\beta\text{-Sn}$ at low angles (Figure 8.7c), while two peaks are observed in the $\beta\text{-Sn}$ misorientation histogram in Figure 8.7g, for the cyclic twinned joint (Figure 8.7e). A peak at approx. 60° indicates the presence of twinning (Figure 8.7g). This matches with the statement of previous literature, the twins usually grow at 57.2° and 62.8° on (101) and (301) plane ^[18].

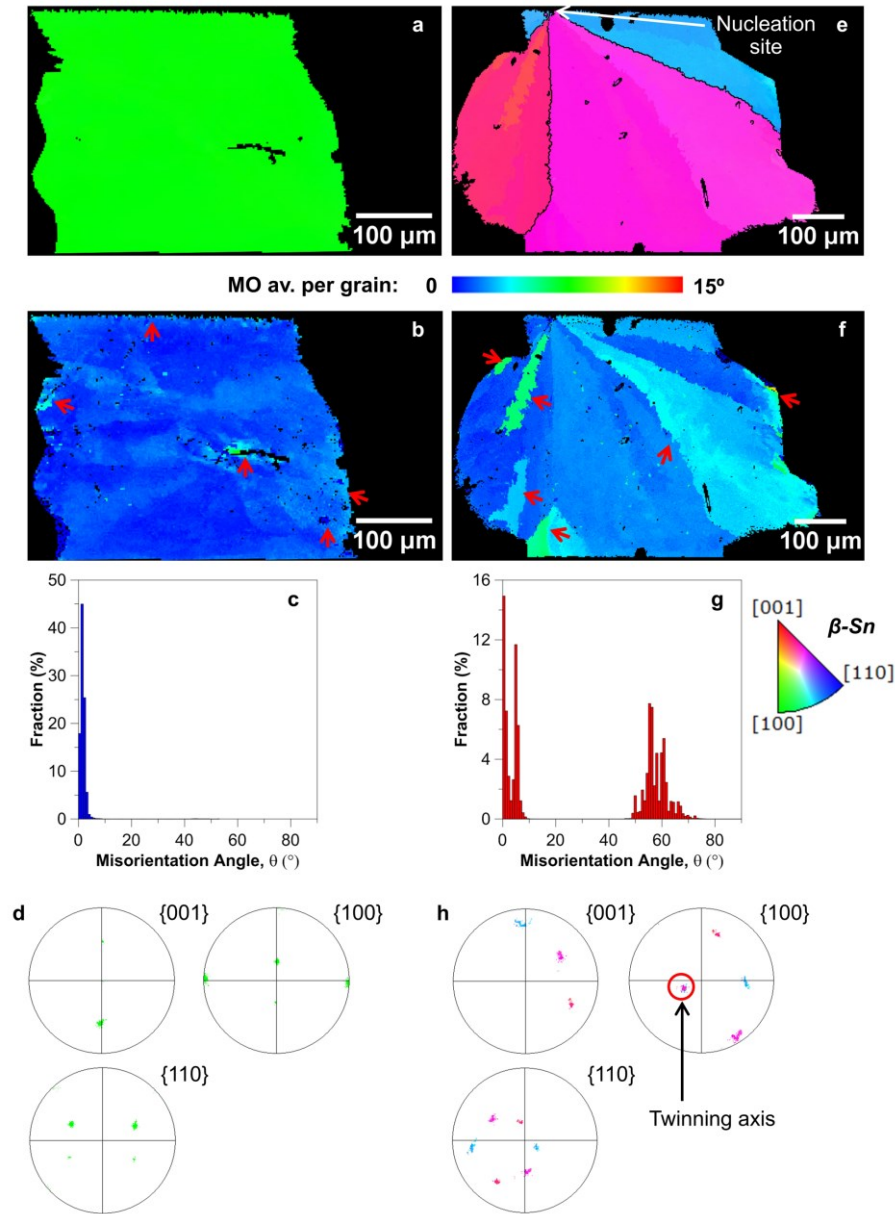


Figure 8.7. EBSD maps of the as-soldered SAC305 solder joint (only the β -Sn has been indexed here). (a-d) a single β -Sn grain joint, (e-h) a cyclic twinned β -Sn grain joint. (a, e) IPFX map with orientation crystal unit cells superimposed on the map. (b, f) MO av. maps show the distribution of misorientations within the joint, the MO hot spots are indicated with red arrows (MO av. per grain map colour key 0-15°). (c, g) Misorientation histogram of β -Sn. (d, h) PFs of the joint.

It is noticed that the nucleation site (Figure 8.7e), where the 3 orientations meet, is on / near the Cu_6Sn_5 layer for the cyclic twinned joint indicating that the 3 grains are 6-fold twinning with approx. 60° separation from each other and only a single nucleation event happened during solidification process (consistent with past studies on the nucleation of β -Sn in SAC305/Cu

BGA joints ^[13]). The 3 grains have a common [100] direction and a common (100) plane (twinning plane), which is normal to the [100] vector as shown in the PF (Figure 8.7h).

The corresponding misorientation of the two joints are illustrated in the MO av. maps (Figure 8.7b and 8.7f). Subgrain boundaries are formed for both joints. For the single grain joint (Figure 8.7b), relatively high misorientations are observed around a shrinkage pore/tear, near large primary Cu₆Sn₅ and at the edge of the joint. For the cyclic twinned joint (Figure 8.7f), a few subgrains are formed indicated with red arrows and the high misorientation also appears at the twin grain boundaries.

8.4.2 Microstructural evolution of SAC305 solder joint

In the early stage of deformation under thermal cycling (Figure 8.8), continuous rotation in orientation and the formation of subgrains are observed with increasing number of cycles for a single β -Sn grain joint. The plastic deformation is localised heterogeneously within the joint due to the CTE mismatch in the interfacial region.

The IPF maps in Figure 8.8 show that the joint forms a single β -Sn grain and has its z-axis points nearly in [001] direction, which is the shortest axis and has the largest CTE value. Therefore, the joint is expected to deform relatively significantly during thermal cycling.

As the joint is thermally cycled, a slight change in colour of the IPFX and IPFY maps at the corners of the joint from green to cyan for 36 (Figure 8.8d and 8.8e) and 60 thermal cycles (Figure 8.8g and 8.8h) comparing to the maps at 12 cycles (Figure 8.8a and 8.8b), indicating the orientation, is rotated gradually, as the number of cycles increases at the corners of the joint. This is caused by the CTE mismatch between the three constituents (β -Sn, Cu₆Sn₅ and Cu), which generates largely thermal strain at the interface and results in microstructural evolution

in the solder matrix (the CTE values are given in Table 2.1 in Section 2.1.1). After 84 thermal cycles, the accumulated plastic deformation causes noticeable reduction in indexing at the corners of the joint with no EBSD pattern detected.

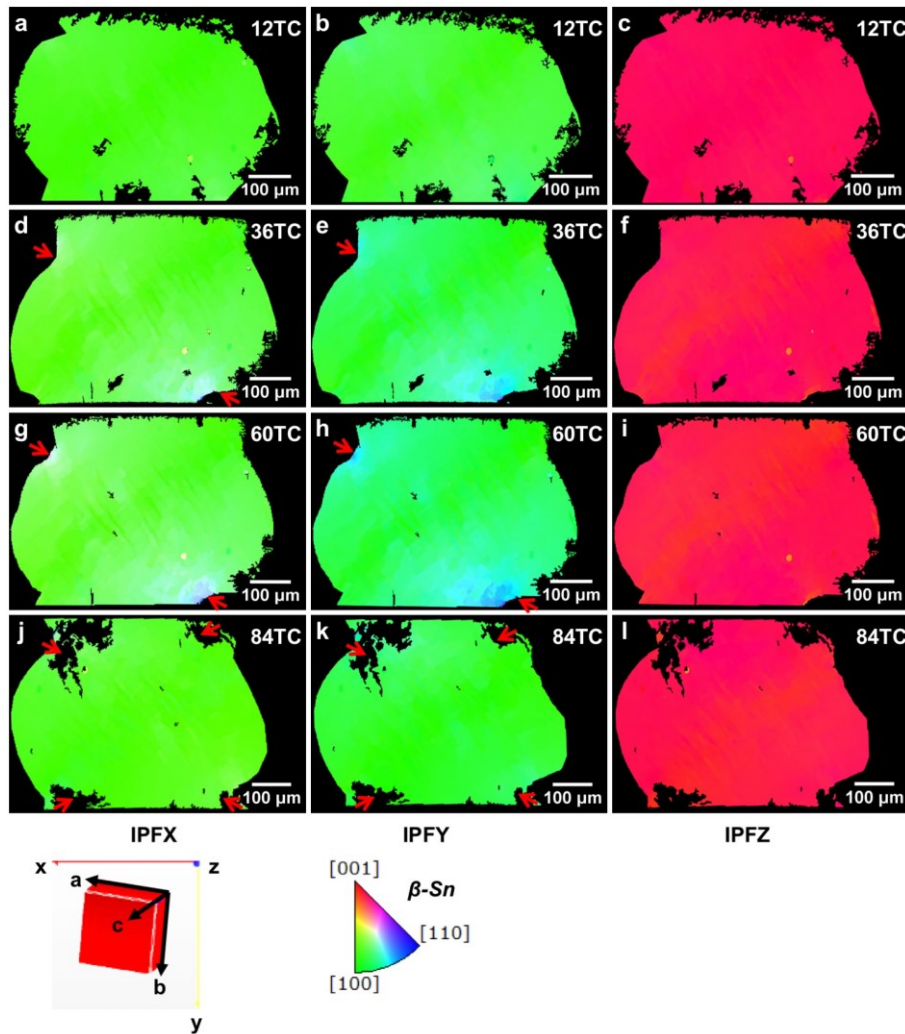


Figure 8.8. EBSD crystal orientation mapping showing heterogeneity in microstructural evolution of a single β -Sn grain SAC305 solder joint at different number of thermal cycles, (a-c) after 12 cycles, (d-f) after 36 cycles, (g-i) after 60 cycles and (j-l) after 84 cycles. (a, d, g, j) IPFX maps, (b, e, h, k) IPFY maps, (c, f, i, l) IPFZ maps (only the β -Sn has been indexed here).

The MO av. maps in Figure 8.9 give the in-grain misorientation at each position of the joint relative to the mean within the joint and form a clear cross band on each map. The highest misorientation appears at the corners with a MO av. colour key 0 - 15°. Each point labelled on

the maps (Figure 8.9a – 8.9d) has its corresponding orientation crystal unit cell shown and slight rotation in orientation is observed from the centre to the corner of the joint. As moving up through the cross band, i.e. from the centre to the corner of the joint (location 2 and 3), the orientation rotates counter-clockwise increasingly, while the orientation rotates clockwise as moving down the cross band (location 4 and 5).

The orientation rotation angle ($\Delta\theta$) vs. position (x) curves (Figure 8.9e) is drawn according to the red dashed line in each MO av. map, which shows the rotation of the orientation increases as the distance is becoming farther from the centre of the joint and the curve has larger gradient at higher thermal cycle. The sketched diagram of orientation unit cells (Figure 8.9f) illustrates the orientation of the β -Sn rotates around the [001] rotational axis with thermal cycles and the direction of rotation is indicated with black arrows.

After 84 cycles (Figure 8.9d), the non-indexing positions are located at the corners, where relatively higher misorientation were observed at previous cycles (Figure 8.9a – 8.9c). Generally, the misorientation increases with increasing thermal cycles, which is shown as increase in width and shift of low angle peaks to larger angles in the misorientation histogram at higher number of cycles (Figure 8.9e). When the solder is deformed by thermal cycling, the lattice misorientation within the solder joint is increased continuously by orientation rotation resulting from the crystal slip along a single rotational axis (Figure 8.9f). This orientation rotation indicates higher straining and more highly stored energy in the region, which causes formation of significant subgrain boundaries and is vital for generation of HAGBs (recrystallisation) and crack nucleation at later stages of thermal cycling ^[40, 41].

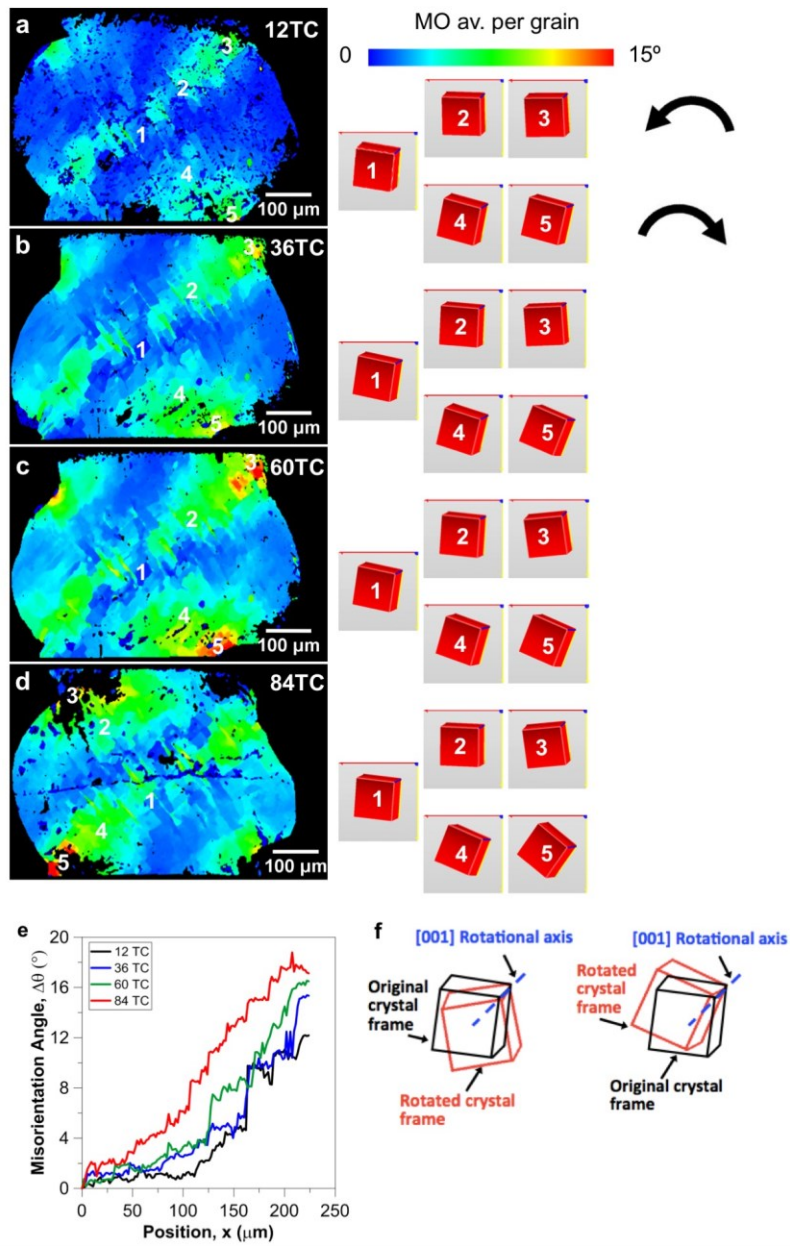


Figure 8.9. EBSD MO av. map showing misorientation evolution of a single β -Sn grain SAC305 solder joint at different number of thermal cycles, (a) after 12 cycles, (b) after 36 cycles, (c) after 60 cycles and (d) after 84 cycles. (e) Orientation rotation angle vs. position curves, the curve is drawn according to the red dashed line on (a-d). (f) Sketched diagrams showing the crystal rotation behaviour, the black crystal frame is the initial state of the grain and the red crystal frame is the rotated state of the grain (only the β -Sn has been indexed here).

8.4.3 Reaction zone of SAC305 solder joint

The microstructure of the β -Sn / Cu_6Sn_5 / Cu reaction zone interface is shown in the Figure 8.10 at 12 thermal cycles. In this strain-concentrated region, subgrain formation is observed in the β -Sn.

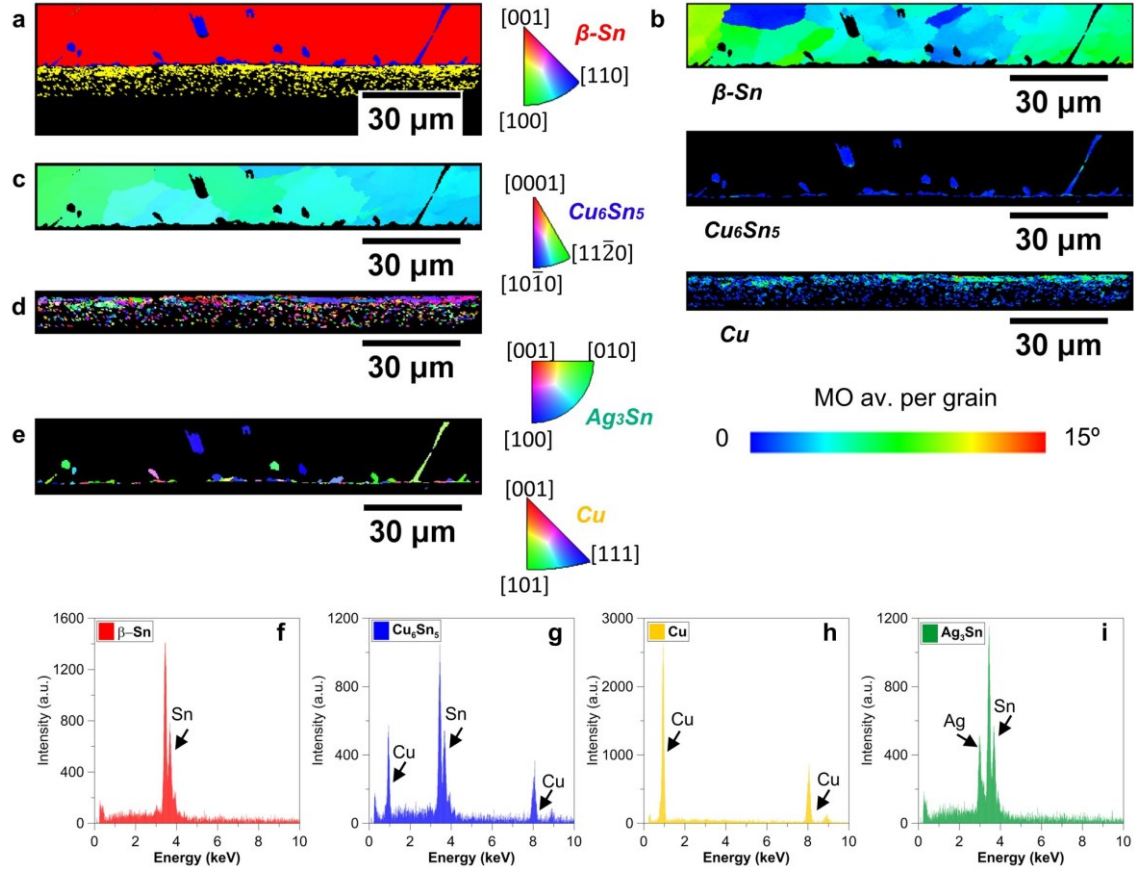


Figure 8.10. EBSD crystal orientation mapping showing the microstructure of the single β -Sn grain SAC305 solder joint at β -Sn / Cu_6Sn_5 / Cu interface after 12 thermal cycles. (a) Phase map (red = β -Sn, blue = Cu_6Sn_5 , green = Ag_3Sn , yellow = Cu). (b) MO av. map for β -Sn, Cu_6Sn_5 and Cu respectively. (c-e) IPFX maps of β -Sn (c), Cu_6Sn_5 (d) and Cu (e). (f-i) EDS spectrum at selected region in β -Sn matrix (f), at Cu_6Sn_5 layer (g), in the Cu substrate (h), and at Ag_3Sn particle (i).

The four phases: β -Sn, Cu_6Sn_5 , Ag_3Sn and Cu are identified clearly in the phase map (Figure 8.10a), with red, blue, green and yellow colour respectively. Only a few Ag_3Sn phase are indexed in the region because of its fine size (in nanometre scale). A layer of Cu_6Sn_5 and Cu_6Sn_5 in the bulk solder can be seen in Figure 8.10a, which are approx. 1 – 5 μm in size. At

the interface, the high misorientation is concentrated in the β -Sn phase (Figure 8.10c – 8.10e show that significant subgrains are formed in the β -Sn, and Cu_6Sn_5 and Cu have random orientations. The EDS spectrums (Figure 8.10f – 8.10i) indicate the elements present in the corresponding selected regions.

8.5 Summary of the Chapter

The microstructural evolution of a single β -Sn grain SAC305 solder joint with its z-axis in the [001] has been studied during the early stages of in-situ thermal cycling. The following conclusion can be drawn.

1. The orientation of the β -Sn changes gradually by in-grain rotation around the [001] axis with increasing plastic deformation during thermal cycling. The misorientation starts to localise at the corners of the joint to form subgrains by recovery processes where the highest CTE mismatch appears at the β -Sn / Cu_6Sn_5 / Cu interface.
2. The rotation in crystal orientation is heterogeneous, which increases from the centre to the corner of the joint. This implies accumulation of plastic deformation at corners of the joint and results in a highly-stored energy in these regions (likely due to the constraint imposed by the CTE mismatch), which plays a very important role in grain polygonization, recrystallisation, and ultimately crack formation and joint failure due to thermal cycling.

Chapter 9 Discussion

9.1 Plastic deformation of solder alloys

When the strain level exceeds the limitation of elastic behaviour, plastic deformation occurs due to gradual increase in stored energy in the solder matrix mainly in form of dislocations. During deformation, the stored energy in crystal lattice changes by deformation phenomena, namely, polygonization and recrystallisation leading to crack nucleation and propagation at the strain-concentrated regions ^[105].

The obtained stress exponent, n for directionally solidified SAC305 sample is 10.5, in the range of dislocation creep (see Chapter 4). Generally, the plastic deformation is caused by deformation in crystal lattice that creates dislocations and dislocation motions through the solder matrix. For solder samples undergoing constant deformation, it is noticed that the solders often experience a constant stress during the plastic flow, i.e. there is no or very little work hardening. Since the solders are being tested at hot working temperatures ($T/T_M \geq 0.6$), there is a sufficient thermal energy allowing dislocations to move out of their slip plane by mechanisms such as dislocation climb and cross-slip, which is the dominant deformation mechanism for solders ^[54].

9.1.1 Slip system and dislocation activities

For a crystal complying with Schmid's Law, the relative activity of the slip systems can be determined according to their critical resolved shear stress (τ_{CRSS}) and Schmid factor (m) ^[47].

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The slip system with maximum weighted Schmid factor (m_w) will be activated first (given in Equation (2.3) in Chapter 2.2).

Family	Slip system		τ_{CRSS} (MPa)	m [110]	m_w [110]	m [100]	m_w [100]
1	1	(100)[001]	1	0.388	0.388	0.051	0.051
	2	(010)[001]	1	0.163	0.163	0.012	0.012
2	3	(110)[001]	1	0.159	0.159	0.044	0.044
	4	($\bar{1}\bar{1}0$)[001]	1	0.391	0.391	0.027	0.027
3	5	(100)[010]	1.05	0.276	0.263	0.228	0.218
	6	(010)[100]	1.05	0.276	0.263	0.228	0.218
4	7	(110)[$\bar{1}11$]/2	1.1	0.301	0.280	0.397	0.361
	8	(110)[$\bar{1}\bar{1}1$]/2	1.1	0.194	0.176	0.429	0.390
	9	($\bar{1}10$)[111]/2	1.1	0.390	0.356	0.404	0.367
	10	($\bar{1}\bar{1}0$)[$\bar{1}\bar{1}1$]/2	1.1	0.110	0.100	0.423	0.385
5	11	(110)[$\bar{1}\bar{1}0$]	1.2	0.269	0.224	0.443	0.369
	12	($\bar{1}\bar{1}0$)[110]	1.2	0.269	0.224	0.443	0.369
6	13	(010)[101]	1.25	0.320	0.256	0.195	0.156
	14	(010)[$\bar{1}0\bar{1}$]	1.25	0.164	0.131	0.206	0.165
	15	(100)[011]	1.25	0.056	0.045	0.176	0.141
	16	(100)[$0\bar{1}\bar{1}$]	1.25	0.428	0.342	0.225	0.180
7	17	(001)[100]	1.3	0.388	0.298	0.051	0.039
	18	(001)[010]	1.3	0.164	0.126	0.012	0.009
8	19	(001)[$\bar{1}10$]	1.4	0.159	0.113	0.044	0.032
	20	(001)[$\bar{1}\bar{1}0$]	1.4	0.390	0.278	0.027	0.019
9	21	(101)[$\bar{1}0\bar{1}$]	1.5	0.388	0.259	0.368	0.245
	22	($\bar{1}01$)[101]	1.5	0.031	0.021	0.422	0.282
	23	(011)[$0\bar{1}1$]	1.5	0.136	0.091	0.016	0.010
	24	($0\bar{1}1$)[011]	1.5	0.040	0.027	0.029	0.019
10	25	(121)[$\bar{1}01$]	1.5	0.167	0.111	0.408	0.272
	26	($\bar{1}21$)[$\bar{1}01$]	1.5	0.393	0.262	0.123	0.082
	27	($\bar{1}21$)[101]	1.5	0.199	0.133	0.170	0.113
	28	($\bar{1}21$)[$\bar{1}01$]	1.5	0.244	0.163	0.440	0.293
	29	(211)[$0\bar{1}1$]	1.5	0.394	0.263	0.166	0.111
	30	($\bar{2}11$)[$0\bar{1}1$]	1.5	0.198	0.132	0.144	0.096
	31	($\bar{2}\bar{1}1$)[011]	1.5	0.010	0.006	0.143	0.095
	32	($2\bar{1}1$)[011]	1.5	0.068	0.045	0.101	0.067

Table 9.1. The common Sn slip systems ^[52] with critical resolved shear stress (τ_{CRSS}) ^[43], Schmid factor (m) and the weighted Schmid factor (m_w) calculated from loading along the [110] and [100] orientations of the bi-crystal sample in Chapter 7. Highlighting has been used to show Figure 9.1, Figure 9.2 and Figure 9.3.

The 32 slip systems are ranked according to their likelihood of activity based upon the τ_{CRSS} values ^[43]. The τ_{CRSS} values in Table 9.1 are based on single-crystal experiments of pure tin

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(β -Sn) with consideration of [100], [010] and [001] orientations for loading in all 3 directions^[43]. Later on in this section, the slip systems are re-ranked according to their weighted Schmid factor (m_w) and angular deviation ($\Delta\theta$) for [100] and [110] orientations in loading direction only.

The values of m are calculated using Matlab with Equation (2.1) given in Chapter 2.2. m_w and $\Delta\theta$ between the calculated and experimental slip traces of the [110] (Figure 9.1d and 9.1e) and [100] (Figure 9.2d and 9.2e and Figure 9.3d) orientations are ranked from the easiest to hardest for the bi-crystal sample performed under in-situ tensile test in Chapter 7. This provides a good illustration because the tested samples in the current works have either [110] or [100] crystal directions parallel to the loading direction.

The analysis of activated slip systems for the [110] orientation is shown in Figure 9.1. The calculated and experimental slip traces are illustrated in Figure 9.1a with pink and blue dotted lines for all 32 calculated and the experimental slip systems respectively. The experimental slip trace is measured from the FSE image (Figure 9.1b). Figure 9.1c represents the orientation unit cell with the highest weighted Schmid factor (m_w) for the [110] orientation. The slip plane (cyan shadowed planes), slip direction (green arrowed lines), calculated slip trace (pink dotted lines) and experimental slip trace (blue dotted lines) are labelled on the unit cell.

The most active slip system for the [110] orientation within the sample is #4 ($1\bar{1}0$)[001] according to its highest value of weighted Schmid factor, $m_w = 0.391$. The experimental slip trace (blue dotted line in Figure 9.1a) is approx. 1.6° from the calculated slip trace of #4 ($1\bar{1}0$)[001] the slip system (Figure 9.1a), which is the smallest angular deviation measured in the histogram in Figure 9.1e. Meanwhile, the # 9 ($\bar{1}10$)[111]/2, # 10 ($1\bar{1}0$)[$\bar{1}\bar{1}1$]/2 and # 12

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$(1\bar{1}0)[\bar{1}\bar{1}1]/2$ slip systems have the smallest angular deviations but their relatively lower weighted Schmid factor implies that they are unlikely to be active.

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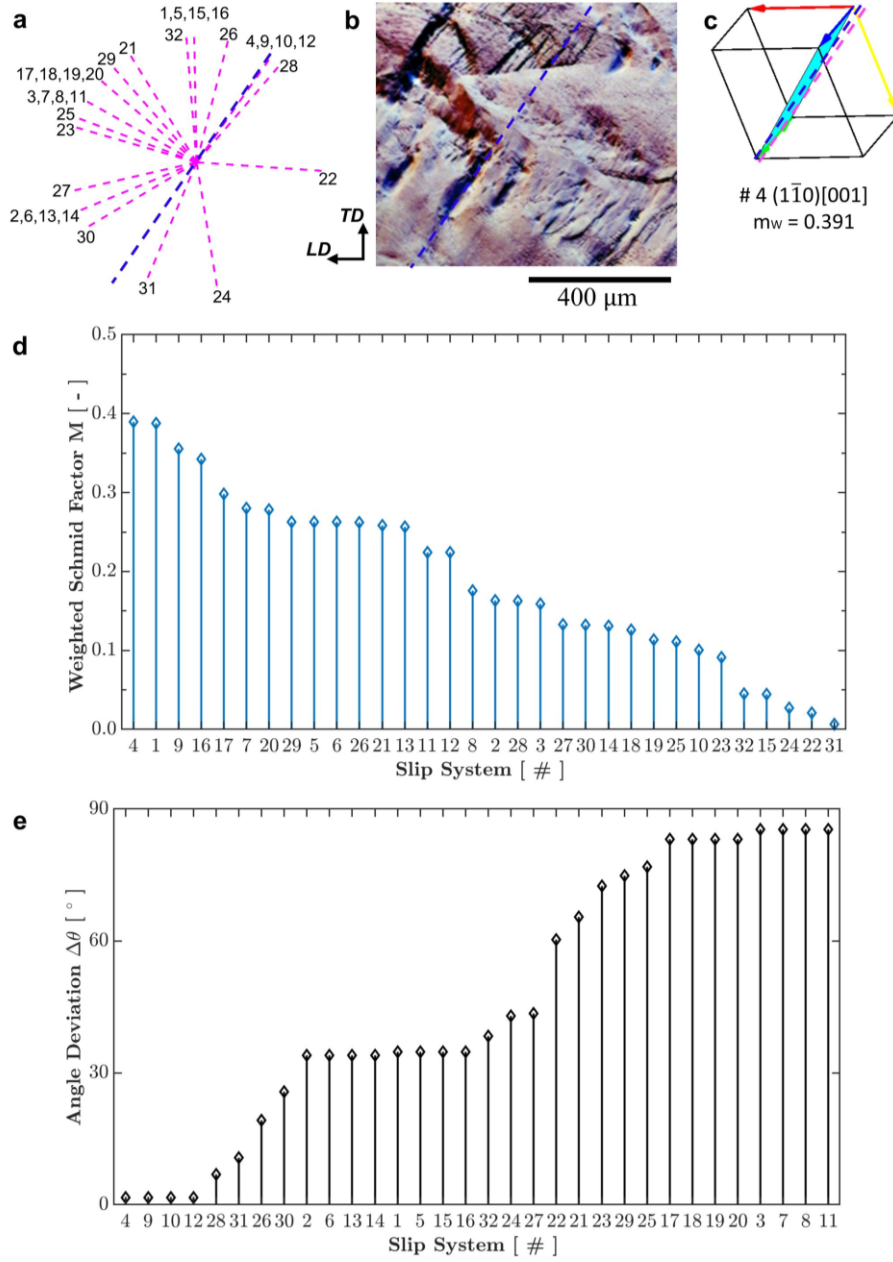


Figure 9.1. (a) The slip plane traces for the $[110]$ crystal orientation. (b) FSE image with experimental slip trace labelled. (c) The activated slip system shown on orientation unit cell with the highest weighted Schmid factor, m_w . The red, blue yellow and blue arrowed lines are the x, y, z crystal axes. The green arrowed lines are the slip directions. The cyan shadowed planes represent the slip planes. The dotted lines indicate the slip plane traces, the calculated slip traces are in pink and experimental slip traces illustrate in blue colour. (d) The weighted Schmid factor (m_w) distribution vs. the slip system (#). (e) The deviation angle ($\Delta\theta$) between experiential and calculated slip traces for the 32 slip systems (#). (d,e) The slip systems are ranked in terms of activation from the easiest to the hardest.

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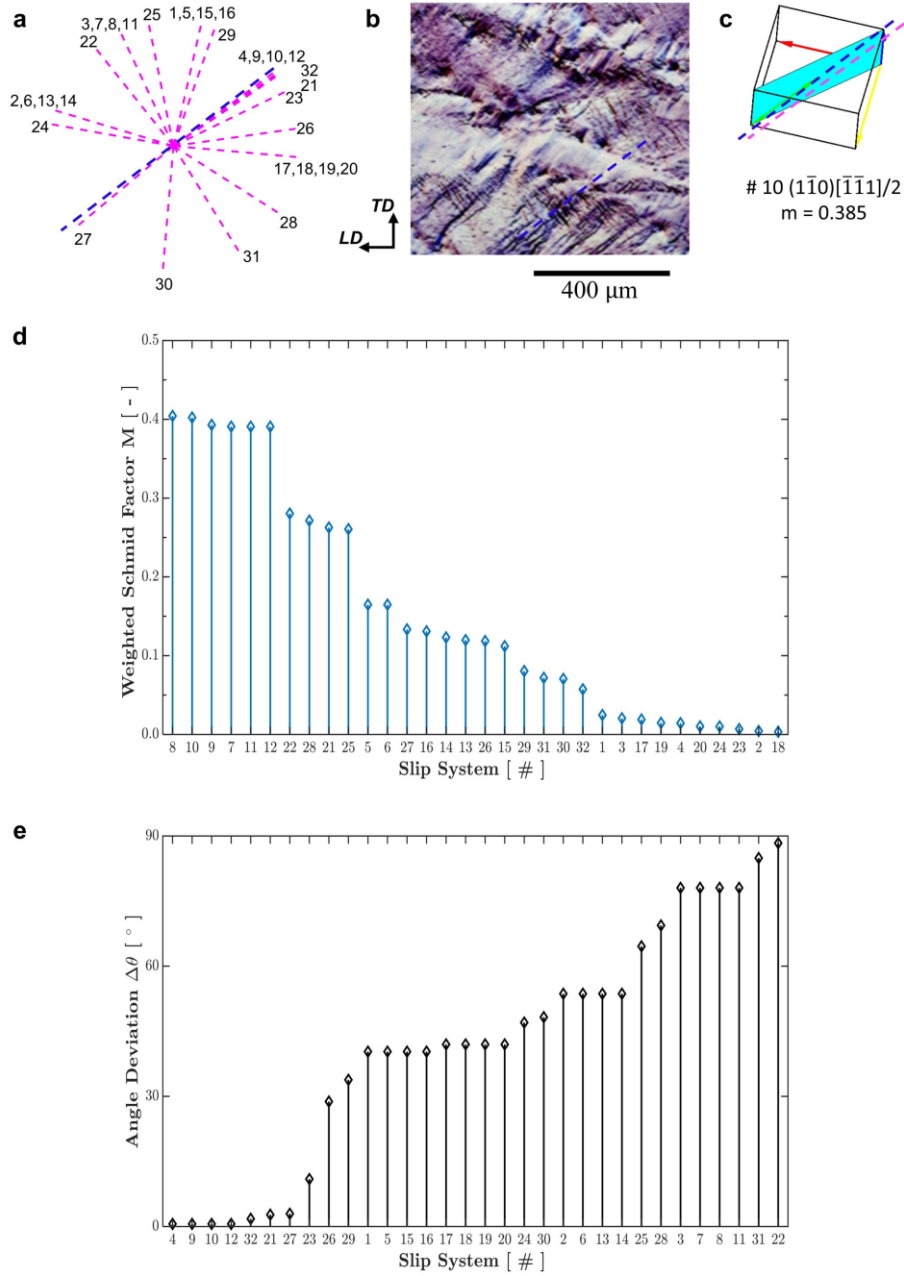


Figure 9.2. (a) The slip plane trace 1 for the [100] crystal orientation. (b) FSE image with experimental slip trace 1 labelled. (c) The activated slip system shown on orientation unit cells with the highest weighted Schmid factor, m_w for slip trace 1. The red, blue yellow and blue arrowed lines are the x, y, z crystal axes. The green arrowed lines are the slip directions. The cyan shadowed planes represent the slip planes. The dotted lines indicate the slip plane traces, the calculated slip traces are in pink and experimental slip traces illustrate in blue colour. (d) The weighted Schmid factor (m_w) distribution vs. the slip system (#). (e) The deviation angle ($\Delta\theta$) between experiential trace 1 and calculated slip traces for the 32 slip systems (#). (d,e) The slip systems are ranked in terms of activation from the easiest to the hardest.

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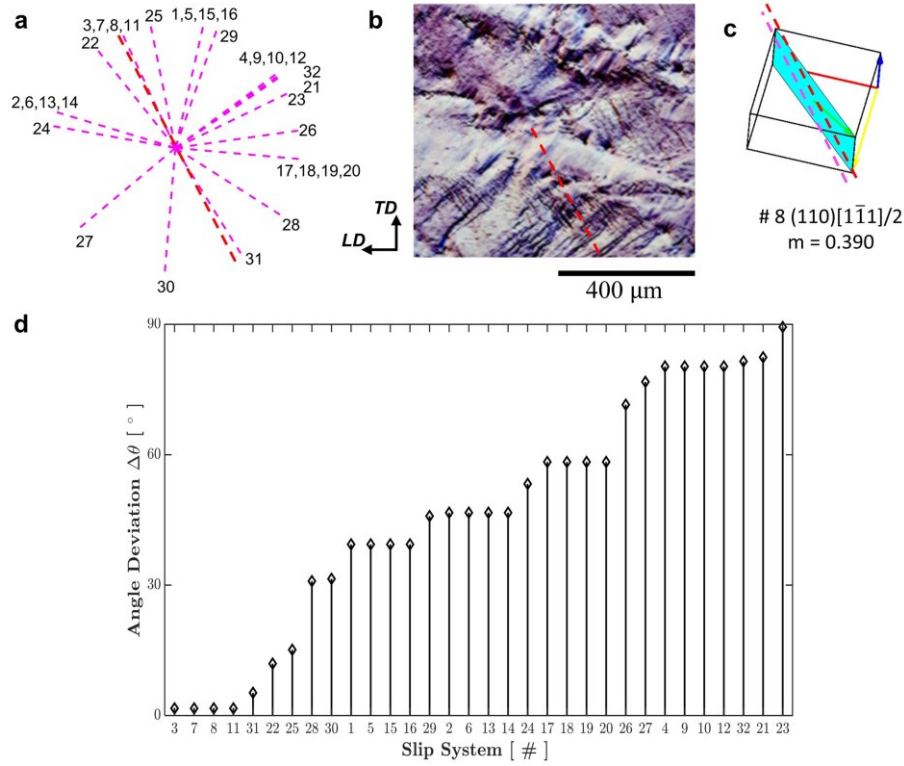


Figure 9.3. (a) The slip plane trace 2 for the $[100]$ crystal orientation. (b) FSE image with experimental slip trace 2 labelled. (c) The activated slip system shown on orientation unit cells with the highest 2 weighted Schmid factor, m_w for slip trace 2. The red, blue yellow and blue arrowed lines are the x, y, z crystal axes. The green arrowed lines are the slip directions. The cyan shadowed planes represent the slip planes. The dotted lines indicate the slip plane traces, the calculated slip traces are in pink and experimental slip traces illustrate in blue colour. (d) The deviation angle ($\Delta\theta$) between experimental trace 2 and calculated slip traces for the 32 slip systems (#), the slip systems are ranked in terms of activation from the easiest to the hardest.

Figure 9.2a Figure 9.3a shows the calculated slip systems (pink dotted lines) of the $[100]$ orientation with two lines match with the experimental trace lines (blue and red dotted lines). In addition, two different slip traces are observed and labelled with blue and red dotted lines in the FSE image of in Figure 9.2b and Figure 9.3b, which are corresponding to two activated slip systems.

By considering the weighted Schmid factor (m_w), the highest two for the $[100]$ orientations are shown in the orientation unit cells diagrams (Figure 9.2c and Figure 9.3c). The # 10 $(1\bar{1}0)[\bar{1}\bar{1}1]/2$ (Figure 9.2c) and # 8 $(110)[1\bar{1}1]/2$ (Figure 9.3c) are determined to be the most

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active slip system for the $[100]$ orientation, since the two experimental slip traces are in line with the calculated slip traces within 1.6 (Figure 9.2e) and 0.6° (Figure 9.3d) of deviation respectively. Although the # 3 $(110)[001]$, # 4 $(1\bar{1}0)[001]$, # 7 $(110)[\bar{1}11]/2$ and # 9 $(\bar{1}10)[111]/2$, # 11 $(110)[1\bar{1}0]$ and # 12 $(1\bar{1}0)[\bar{1}\bar{1}1]/2$ slip systems also have the smallest angular deviations, they are unlikely to be active because they have relatively lower weighted Schmid factor (Figure 9.2d) and the # 11 $(110)[1\bar{1}0]$ and # 12 $(1\bar{1}0)[\bar{1}\bar{1}1]/2$ slip systems are less favourable because of longer slip distance and greater value of effective yield stress than the slip systems for #8 $(110)[1\bar{1}1]/2$ and # 10 $(1\bar{1}0)[\bar{1}\bar{1}1]/2$.

Nevertheless, although the relative activity of slip systems for β -Sn can be identified using the weighted Schmid factor, there are exceptions for some slip systems due to the influence of testing temperature, purity of Sn, orientation and initial strain state of the sample^[44, 47, 55].

9.2 Recovery and recrystallisation mechanisms of solder alloys

When the strain is being applied, the microstructure evolution of the solders can be explained by the concept of dynamic recovery and recrystallisation. This process involves continuous generation of grains with new orientations. These grains are of low stored energy. They are generated from regions of high stored energy within a deformed solder matrix.

Dynamic recovery is demonstrated in Chapter 4 - Chapter 8, where the highly polygonised subgrains have been formed in regions which correlate previously with regions with high crystal misorientation. These subgrains are formed from continuous annihilations and rearrangement of dislocations via dislocation climb and cross-slip (as tin is so hot that thermally activated rearrangement processes are easier to activate). The remaining dislocations are piled up to form lower energy subgrain boundaries, with a misorientation from the initial crystal

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orientation. This entire process is the dynamic recovery, where the low angle subgrain boundaries contain stored energy and the density and misorientation of these boundaries correlates with the total stored energy within the substructure.

Dynamic recrystallisation is found as the deformation continues, where the stored energy can be further reduced by recrystallisation as the dislocation structures are absorbed by new grains and the interfaces between these grains and the previous material are HAGBs. The dislocations are removed by nucleation and growth of new grains that eliminates the driving force for further recrystallisation. New grains with high misorientation angle relative to the adjacent matrix can be called recrystallised grains ^[155] and they release the internally stored energy by forming HAGBs and increasing the grain boundary area as the new recrystallised grains consume the dislocated matrix. The nucleation of recrystallisation is controlled by stored energy, often in the form of dislocation density such that the nucleation rate and driving force for nucleation of recrystallisation are proportional to the dislocation density ^[40, 41, 103, 117]. The nucleation of recrystallisation is commonly initiated at grain boundaries and large IMCs, which are energetically favourable sites for nucleation ^[103]. In the primary dendrite tin, dislocations can accumulate (especially when two slip systems interact) and form structure, as evidenced with lattice misorientation. In the EBSD maps, these interactions are often observed difference in lattice rotation, and they are typically observed in regions with defects or second phase particles (IMCs) due to the deformation heterogeneity associated with their relative slip strength ^[156].

9.2.1 Role of grain boundaries

As we know, dynamic recrystallisation can be proceed by grain boundary migration (thermal activated process), which forms ‘strain-free’ new grains by consuming the driving force in the deformed structure.

In the presence of grain boundaries (bi- and tri-crystal sample in Chapter 4 - Chapter 8), discontinuous recrystallisation can take place initially, because the grain boundaries act as a barrier to concentrate dislocations and cause local evolution of high misorientation, providing a necessary site for recrystallisation nucleation.

The grain boundaries presented in Chapter 4 - Chapter 8 can be characterised in 3 groups, namely, LAGBs and HAGBs, special and random, as well as tilt and parallel boundaries. The migration of LAGBs is related to the dislocation motion, especially with respect to dislocation climb. By absorbing the LAGBs, the local migration of HAGBs may take place. This migration can be biased by the applied strain. For example, the deformation is relatively easier to concentrate at a tilt grain boundary than a parallel grain boundary to the loading direction because the motion of dislocations is easier to be blocked and pinned to form a high dislocation density region and evolve local orientation gradient ^[155]. It is also found that a twin boundary migrates faster than a random boundary because of its lower energy configuration and gives less localisation of deformation (Chapter 5 and Chapter 6). With increasing deformation, recrystallisation segments the initial grain and reduces the migration rate ^[155].

9.2.2 Role of intermetallic compounds

Due to additions of alloying elements, i.e. Ag and Cu, Cu_6Sn_5 and Ag_3Sn are formed in the SAC305 solder volume, which have 3.5% and are distributed with controlled spacing in the eutectic.

As illustrated in Chapter 4 - Chapter 8, recrystallisation is relatively easy to form in the IMC-containing eutectic regions with a finer size than in the eutectic-free dendritic regions. This is tempered with the balance of length-scale based strengthening, where the back stress from dislocation structures associated with the IMC interfaces will lead to higher stresses to activate and glide dislocations in the material. However, in comparison to many material systems where slip may be constrained to an individual slip plane, the relatively high temperature of the β -Sn here may reduce the potency of length-scale based hardening as the impact of strong pile-ups or tangles can be reduced through cross slip and climb of dislocations (either near the sources or near the IMCs).

Large deformation heterogeneities appear near IMC particles during deformation, which create regions of high local dislocation density and large orientation gradient, observed as high misorientations. These heterogeneities provide good nucleation sites for recrystallisation by reducing the stored energy and accelerate the evolution of microstructure and texture. This is so called particle stimulated nucleation (PSN) ^[155].

When the primary β -Sn (in dendrite) and eutectic β -Sn share the same orientation, the incoherent high-angle boundaries between the IMCs and β -Sn act as stress concentrators because the Cu_6Sn_5 and Ag_3Sn particles act as obstacles to dislocation movement. In regions

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near Cu_6Sn_5 and Ag_3Sn , the local constraint results in high stored dislocation content, corresponding to formation of LAGBs.

The size and amount of recrystallised grains play an important role in determining the performance of the initial microstructure and properties of the solder sample. By comparing the results in Chapter 5 and Chapter 6, the effect of initial microstructures on the formed recrystallised grains is shown. A less heterogeneity in deformation is achieved for the samples with finer microstructure, i.e. finer size and spacing of IMCs, implying that the plastic deformation is dominated by obstacle-controlled dislocation motion at the microscopic scale. As the size and spacing of IMCs increases, the coherency across the sample matrix is heavily decreased due to more pinning, which would prevent or delay recrystallisation and decay the overall thermal and mechanical properties of solder samples ^[155].

Furthermore, for the solder joint in Chapter 8, the Cu_6Sn_5 constitutes an interfacial layer of IMC between bulk $\beta\text{-Sn}$ and Cu substrate. The deformation of the solder joint induced at corner of solder matrix during thermal cycling where the Cu_6Sn_5 is formed. This is caused by the CTE mismatch between the constituents and relatively high brittleness of the Cu_6Sn_5 ^[39], which systematically varies the stress and strain build-up during thermomechanical loading near the interface of the solder joint ^[112].

The effect of recrystallisation on the development of crystal orientation is also revealed during deformation in Chapter 4 - Chapter 8. Within a single grain, the recrystallisation texture of the orientation spreads broadly from its initial orientation and it is associated to one of the PSN mechanisms of randomising texture ^[155].

9.2.3 Effect on microstructure

The microstructural texture develops during recrystallisation. Since recrystallisation is a process of nucleation and growth, the microstructure changes by forming new orientations and spreading the initial orientation as observed in Chapter 5 - Chapter 8. Due to the influence of large volume fraction, IMCs pin and prevent a strong deformation texture. So, the deformed orientations have no dramatic change in texture and relatively less deviation from their initial orientations.

Large crystal rotations appear near the fracture surface and within the heavily necked regions for all samples in Chapter 4 - Chapter 7, indicating that the amount of evolution in texture is proportional to the strain induced of the solder sample.

Moreover, different grains rotate differently in order to achieve new orientations with low stored energy grains, i.e. a preferred [110] grain tends to rotate towards [100] direction with one slip system activated and a preferred [100] grain tends to rotate in both [110] and [001] with activation of 2 slip systems. The driving force for the rotation varies with the crystal orientation and is also obtained by in-situ observing the heterogeneity in strain localisation for the [110] and [100] orientations for the bi-crystal solder sample (Chapter 7).

9.2.4 Effect on crack imitation and propagation

Ductile fracture takes place after a certain amount of plastic deformation is accumulated within a material. As plastic deformation occurs, the plastic strain region commonly work-hardens, the stress and strain increase in this region until the material fractures. Therefore, there is a large amount of energy absorbed before fracture. Generally, there are 4 steps of ductile fracture, namely, void formation, void coalescence (crack formation), crack propagation and failure^[157].

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The propagation of a ductile crack involves substantial plastic flow and gives a rough fracture surface.

The failure of solder is observed as propagation of crack and polygonization in the bulk. The crack propagates progressively at the region where the mismatch of CTE is large and the induced strain is high in comparison to the surroundings, i.e. grain boundaries and IMC-containing eutectic regions. During the deformation, the plastic deformation is localised to form parallel shear bands ('flow' lines) with high surface roughness. These 'flow' lines are suggested to be slip traces and corresponded to the initial orientation of the grain ^[77].

Referring to the EBSD maps in Chapter 4 - Chapter 8, the cracking of solder happens simultaneously with the evolution of microstructure in the strain-localised regions as the consequence of deformation heterogeneity. There is a strong correlation between recrystallisation and crack propagation, i.e. the highly-angled recrystallised grain boundaries assist the crack propagating across the solders matrix. The high-angle and high-energy boundaries produced by recrystallisation can facilitate grain boundary sliding and void formation at boundaries, which provide a favourable energy path for cracking ^[26, 40, 41, 44, 108, 118]. In the case of solder joints, both intrinsic (difference between grains and microstructure) and extrinsic (difference between constituents) can induce the CTE mismatch, which causes strain accumulation and localisation of plastic deformation, leading to crack propagation through the region ^[105].

Chapter 10 Conclusion and future work

I have explored the microstructural origin of deformation and fundamental deformation mechanisms of SAC305 solder alloys at both the large-scale bulk and solder joint scale.

10.1 Creep test

The creep test has been performed for SAC305 bulk samples. In order to avoid the stochastic microstructures of real solder joint, which is caused by the differences in the nucleation undercooling of β -Sn, a controlled microstructure of the bulk samples was successfully fabricated by Bridgman growth reproducibly. The produced microstructure (DS microstructure) had key similarities to the real SAC305 solder joints [7, 9, 11, 12], which has 1-3 β -Sn grains, a strong directionality with [100] or [110] orientations and similar length-scale of primary β -Sn dendrite arm spacing and the eutectic Ag_3Sn and Cu_6Sn_5 . The microstructure of the samples was controlled by varying the growth rate. A faster growth rate created a relatively finer microstructure with smaller β -Sn dendrite arm spacing, eutectic spacing and Ag_3Sn and Cu_6Sn_5 size and vice versa. Following this success on microstructure fabrication, the creep behaviour of the DS bulk samples was evaluated with minimising the uncertainty from microstructure.

It has been found that the DS bulk SAC305 deformed with three stages during creep testing, including a strain hardening primary stage, a steady strain rate secondary stage and a strain accelerating tertiary stage.

It has been discovered that the failure mode of the DS SAC305 samples changed from Mode I (open) to Mode II (shear) with increasing stress at a constant temperature (homologous

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temperature $\sim \frac{T}{T_M} = 0.6$) and the dominating creep mechanism was determined as climb-controlled dislocation creep with the value of stress exponent, $n = 10.5$.

Analysis of the creep curves suggests that the creep performance of the DS SAC305 solder alloys can be improved by reducing the applied stress, decreasing the temperature and refining the microstructure and indicated as a longer creep lifetime, especially for longer duration of secondary stage, and lower creep strain rate.

Calculation of the activation energy, Q has been interpreted for the transition of creep mechanisms depending on the initial microstructure of the sample. It has been suggested that the creep mechanisms changed from climb-controlled dislocation creep to lattice-limited diffusion creep beyond homologous temperature $\sim \frac{T}{T_M} = 0.7$ to 0.74 , which was represented as a decrease in onset of secondary creep strain as well. These behaviours have implied that the creep deformation was obstacle-controlled due to less energy required for dislocation motion below the transition temperature, while the deformation was controlled by grain size for diffusion-limited creep at higher temperatures, where the value of Q kept the same for different initial microstructures. Meanwhile, it also has been obtained that the greater ductility and shorter secondary stage creep time at high temperature.

In terms of microstructural deformation, the heterogeneity evolution has been characterised during creep and illustrated the stress, temperature and microstructure dependence on creep reliability. At the macroscopic scale, the heterogeneity was associated with the presence of a grain boundary; while at the microscopic scale, the heterogeneity was caused by the presence of two distinct microstructural regions, i.e. primary β -Sn dendrites and IMC-containing eutectic β -Sn regions. These phenomena have been explained by heterogeneous rotation in orientation and shown as polygonization (formation of subgrains and recrystallisation) with increasing of

misorientation continuously, which was caused by the incoherent boundaries between IMCs and β -Sn, as well as between different grain orientations acting as deformation concentrators. It is discovered that the grain with a $[100]$ orientation experienced less microstructural deformation and ductility than the grain with a $[110]$ orientation.

Localised recrystallisation has been generated within the strain-concentrated regions during deformation. The difference in deformation patterning can result in a change in the localisation of stored energy and also the development of local ‘rainbow’ recrystallisation. The length-scale of subgrains and recrystallised grains were related to microstructure (dendrite and IMCs spacing) of the sample, which retained its initial structure after failure.

Furthermore, the operation of slip systems has been determined by correlating with the developed of slip traces during the microstructural evolution. $(1\bar{1}0)[001]$ slip system was observed to be activated for a preferred orientation of $[110]$. On the other hand, two slip systems $(110)[\bar{1}\bar{1}1]/2$ and $(1\bar{1}0)[\bar{1}\bar{1}1]/2$ have been activated for a preferred orientation of $[100]$. With increasing deformation, more slip traces were formed and became more prominent by increasing the local surface roughness, which initiated crack and fractured the sample.

10.2 Thermal cycling

In-situ thermal cycling has been performed to investigate the heterogeneous evolution of microstructure for a single Cu/SAC305/Cu BGA solder joint with its z-axis in the $[001]$ direction, which experienced the largest CTE mismatch among the three directions of β -Sn at the early stage of deformation.

It has been revealed that during thermal cycling, the heterogeneous deformation was associated with both the constraint by the CTE mismatch between the three constituents at the interface and the anisotropy of β -Sn. The localisation of plastic deformation at the corners of the β -Sn / Cu₆Sn₅ / Cu interface resulted in heterogeneous rotation in orientation (the greater the distance from the centre of the joint the greater the rotation), which is a consequence of the increase in misorientation and formation of subgrains by recovery processes. This work provides new understanding on the early stages of microstructural deformation, which is the precursor to the more widely studied phenomena of recrystallisation and crack propagation in thermal cycling [26, 40, 41, 44, 108, 118].

10.3 Future work

For future work, in order to improve the understanding of the fundamental deformation mechanisms and evaluate the microstructure evolution of SAC305 solder alloys and joints, the following experiments should be planned:

- Low temperature creep test (below room temperature) will be required to compare the creep performance between low and high temperatures in order to observe whether the same creep mechanisms would be followed or not and explore how the microstructure evolves at low temperature.
- Creep test on samples with main [001] orientation, since the [001] direction is the stiffest for β -Sn and has the largest CTE values, it should be interesting to find out how the sample with a [001] orientation in the loading direction will behave under creep tests and will compare its creep performance to the samples with [100] or [110]

orientations. However, the difficulty presented in fabricating [001] orientation for β -Sn, is not the favourable growth direction.

- Further observation will be required to continue the in-situ thermal cycling experiment on evolution of microstructure in shape, morphology and orientation using SE, FSE and EBSD imaging. The position and shape of the joint should be expected to change during heating and cooling processes due to the induced shear stress ^[96]. Therefore, four FIB markers will be made at the corners of the solder joint on the Cu substrate to track the position and shape of the joint. The quantity of orientation rotation will be measured by attaching a small piece of Si wafer onto the joint, which enables the relative rotation angle between the β -Sn and Si determined from the EBSP at each step of cycling.
- It is known that the failure of solder joint often occurs in the highly-strained regions in the bulk β -Sn near the β -Sn / Cu_6Sn_5 / Cu interface where the largest CTE mismatch is present. Therefore, it is well worth determining the accumulation of strain and illustrating the heterogeneous distribution of strain for the joint with increasing number of thermal cycles. Therefore, the strain measurements will be carried out by HR-EBSD, which has been used efficiently in previous work ^[142, 158-160] to quantify the residual strain (stress) levels within the solder joint at different stages of thermal cycling and to find out the answers to the following questions:
 1. How does the strain level in a solder joint change with number of cycles? Does the strain level increase with number of cycles?
 2. At what strain level does microstructural deformation (increase in misorientation, formation of subgrains, generation of recrystallisation and coarsening of IMCs) initiate?

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3. At what strain level does the crack initiate and propagate in solder joints? Does cracking happen in the region with the highest strain level within the joint?
4. What is the effect of grain boundary on strain accumulation?
5. How does the primary IMCs and IMC layer affect the strain accumulation?

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