

Full Length Article

Microstructure and mechanical properties of curved AZ31 magnesium alloy profiles produced by differential velocity sideways extrusion

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Abstract

Lightweight curved profiles are widely utilised in the transportation industry considering the increasing need for improving aerodynamic efficiency, aesthetics and cutting emissions. In this paper, curved AZ31 Mg alloy profiles were manufactured in one operation by a novel process, differential velocity sideways extrusion (DVSE), in which two opposed rams were used. Effects of extrusion temperature and velocity (strain rate) on curvature, microstructure, and mechanical properties of the formed profiles were examined. Profile curvature was found to be more readily controlled by the velocity ratio of the bottom ram v_2 to the top ram v_1 , whereas extrusion temperature ($T = 250, 300, 350\text{ }^\circ\text{C}$) and extrusion velocity ($v_1 = 0.1, 1\text{ mm/s}$) slightly affect curvature for a given velocity ratio. A homogeneous microstructure with equiaxed grains ($\sim 4.5\text{ }\mu\text{m}$) resulted from dynamic recrystallisation (DRX), was observed after DVSE ($v_2/v_1 = 1/2$) at $300\text{ }^\circ\text{C}$ and $v_1 = 0.1\text{ mm/s}$, where the initial billet had an average grain size of $\sim 25\text{ }\mu\text{m}$. Increasing extrusion temperature leads to grain growth ($\sim 5\text{ }\mu\text{m}$) at $350\text{ }^\circ\text{C}$ and $v_1 = 0.1\text{ mm/s}$. DRX is incomplete at the relatively low temperature of $250\text{ }^\circ\text{C}$ ($v_1 = 0.1\text{ mm/s}$), and higher strain rate with $v_1 = 1\text{ mm/s}$ ($T = 300\text{ }^\circ\text{C}$), resulting in inhomogeneous bi-modal necklace pattern grains ranging in size around $1\text{--}25\text{ }\mu\text{m}$ for the former and $2\text{--}20\text{ }\mu\text{m}$ for the latter. Grain refinement is attributed to DRX during the severe plastic deformation (SPD) arising in DVSE, and initiates at the prior boundaries of coarse grains in a necklace-like manner. Compared with the billet, micro-hardness and ultimate tensile strength of the profiles have been enhanced, which is compatible with grain refinement. Also, an obvious increase in tensile ductility was found. However, yield strength slightly decreases except for the complete DRXed case ($300\text{ }^\circ\text{C}$, $v_1 = 0.1\text{ mm/s}$), where a slightly higher value was found, indicating strengthening by grain refinement is greater than softening caused by texture modification. The initial billet had a strong basal texture wherein the $\{0002\}$ basal plane is oriented parallel to the extrusion direction ('hard' orientation), while DVSE results in the profiles having weak basal textures and the $\{0002\}$ basal plane oriented $\sim 5\text{--}10^\circ$ to the extrusion direction (i.e. towards the orientation for easier slip). This significantly modified texture contributes to the softening of the profiles in the extrusion direction, in which tensile tests were performed, and the related elongation improvement.

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1. Introduction

Curved profiles utilising light metal alloys are widely used as constructional elements in various structures to realise aesthetic design as well as lightweight design. As reported

by Tekkaya and Chatti [1], improved aesthetics and aerodynamic properties can be achieved for lightweight structures constructed with curved profiles. As the lightest construction metal, magnesium alloy is of increasing interest for applications in structural construction [2,3]. Using curved magnesium alloy profiles in the frames of transportation vehicles such as automobile, railway train and aircraft bodies, has the potential to provide the ultimate in structural lightness.

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Curved profiles are currently mostly manufactured from semi-finished straight ones through a bending procedure and conventionally, forward extrusion of billet is used to produce virtually all straight profiles [4–7]. The widely used post-extrusion bending operations include press/ram bending, rotary draw bending, and three-roll bending. For magnesium alloy with a hexagonal close-packed (hcp) structure, its active slip systems at room temperature are inadequate as a result of its high critical stress for non-basal slip and twinning [8]. Thus the Von Mises criterion for sufficient plasticity where at least five independent slip systems are needed [9] cannot be fulfilled, resulting in a limited room temperature ductility and formability. Therefore for bending magnesium alloy profiles, heating of workpiece and tools is necessary. Wu et al. [10] examined the bending characteristics of circular magnesium tubes at elevated temperature in rotary draw bending, it was found that cross-sectional distortion and wall-thinning rate increase with increasing bending velocity and initial grain size. Hasegawa et al. [11] explored the press bending of square AZ31 tubes at room temperature and showed that axial tension helps to reduce wrinkling of the intrados. Although progress has been made to improve bending operations, issues such as springback, wrinkling and cross-sectional distortion which readily arise, are normally tackled by ad hoc approaches entailing cost and time. Stress-superposed bending process which superposes compression with bending has been developed to reduce the bending force and defects, as proposed by Chatti et al. [12,13]. Hermes et al. [14] later confirmed the favourable effect of stress superposition using torque. Some processes integrating extrusion and bending have been proposed to reduce the manufacturing procedures. Müller et al. [15,16] proposed a method using an external bending device (bending discs) pre-positioned at the extrusion die orifice. An external device can also be a guiding tool in-line controlled by a linear axes system, as reported by Selvaggio et al. [17]. Another approach is by extrusion die design. The orifice of the extrusion die designed by Shiraishi et al. [18–20] was inclined at a certain angle to extrude curved profiles. Alternatively, the extrusion ram was designed to have a step-like front end [21,22]. However, the above extrusion-bending integrated processes are mostly focused on aluminium alloy or steel and curvature variation of profiles cannot be conveniently achieved without utilising extra tooling. Moreover, the grains of the obtained curved extrudates are generally similar to those obtained by conventional forward extrusion.

It has been reported that grain refinement is able to improve mechanical properties of magnesium alloys, and high strength and ductility for magnesium alloys can be achieved through manipulating grain size and texture [8,23–27]. Severe plastic deformation (SPD) is extensively utilised to refine grains of metallic materials down to the microscale or even nanoscale regime. A large number of SPD strategies, including equal channel angular extrusion/pressing (ECAE/P) [28,29], high-pressure torsion [30], and accumulative roll bonding [31], have been proposed. ECAE/P has been proven to be one of the most effective techniques for refining grains

of metal alloys [32]. Kim et al. [33] outlined that through repetitive ECAE/P, both grain refinement and texture modification of magnesium alloy can be achieved, leading to the improvement of strength and ductility. However, several passes are needed for ECAE/P to achieve desirable grain refinement, especially for materials with initially a relatively large grain size, as pointed out by Figueiredo and Langdon [34–36]. Thus in the practical processing of metallic materials such as magnesium, aluminium alloys etc., ECAE/P can be costly and of low efficiency. Up to now, the commercialisation of SPD processed materials for components/products in industry has been focused on niche applications in which high material cost can be accepted, such as for aerospace structures [37], defence applications [38], and medical implants [39]. Moreover, the material for making these components is processed by SPD to refine grains before undergoing the shape forming process. The refined grains of the SPD processed material may detract from the subsequent shape forming process, especially in hot forming condition such as the extrusion, forging, rolling, bending, etc., and the scale up of SPD processed material needed for products in industry is not readily available. Kang et al. [40] successfully forged an impeller utilising ECAE/P processed magnesium alloy, the refined grains produced by ECAE/P were found to be affected by the forging condition.

The authors have developed a novel forming process called, differential velocity sideways extrusion (DVSE), where two opposed rams with independently controlled extrusion velocities can be used for direct manufacturing of curved profiles from billets in one operation [41]. Feasibility studies have been conducted using model material plasticine and profiles with adjustable curvature and undistorted cross-section, have been produced. It was also found that pronounced grain refinement and enhanced strength of AA1050 profiles can be achieved by DVSE at room temperature [42], and DVSE requires a low extrusion force and results in higher material strength of AA1050 profiles in comparison with forward extrusion for the same extrusion condition [43]. In this paper, effects of temperature and strain rate on curvature, grain structure, texture and mechanical properties of curved AZ31 magnesium alloy profiles fabricated by DVSE are presented for the first-time.

2. Experimental procedures

2.1. DVSE principle and experiments

The principle of DVSE has been elucidated in detail in [41], here a schematic of DVSE is given in Fig. 1a. An extrusion die orifice is situated perpendicular to the billet container, rams at each end of the container compress the billet simultaneously with different velocities, a velocity gradient is induced in the deforming region across the die orifice and thus a curved profile is extruded. The material used in this work was commercial AZ31 (Mg, 3% Al, 1% Zn, wt.%) magnesium alloy, which was received as extrusion rods with homogeneously distributed grains of average size $\sim 25 \mu\text{m}$. Cylindrical

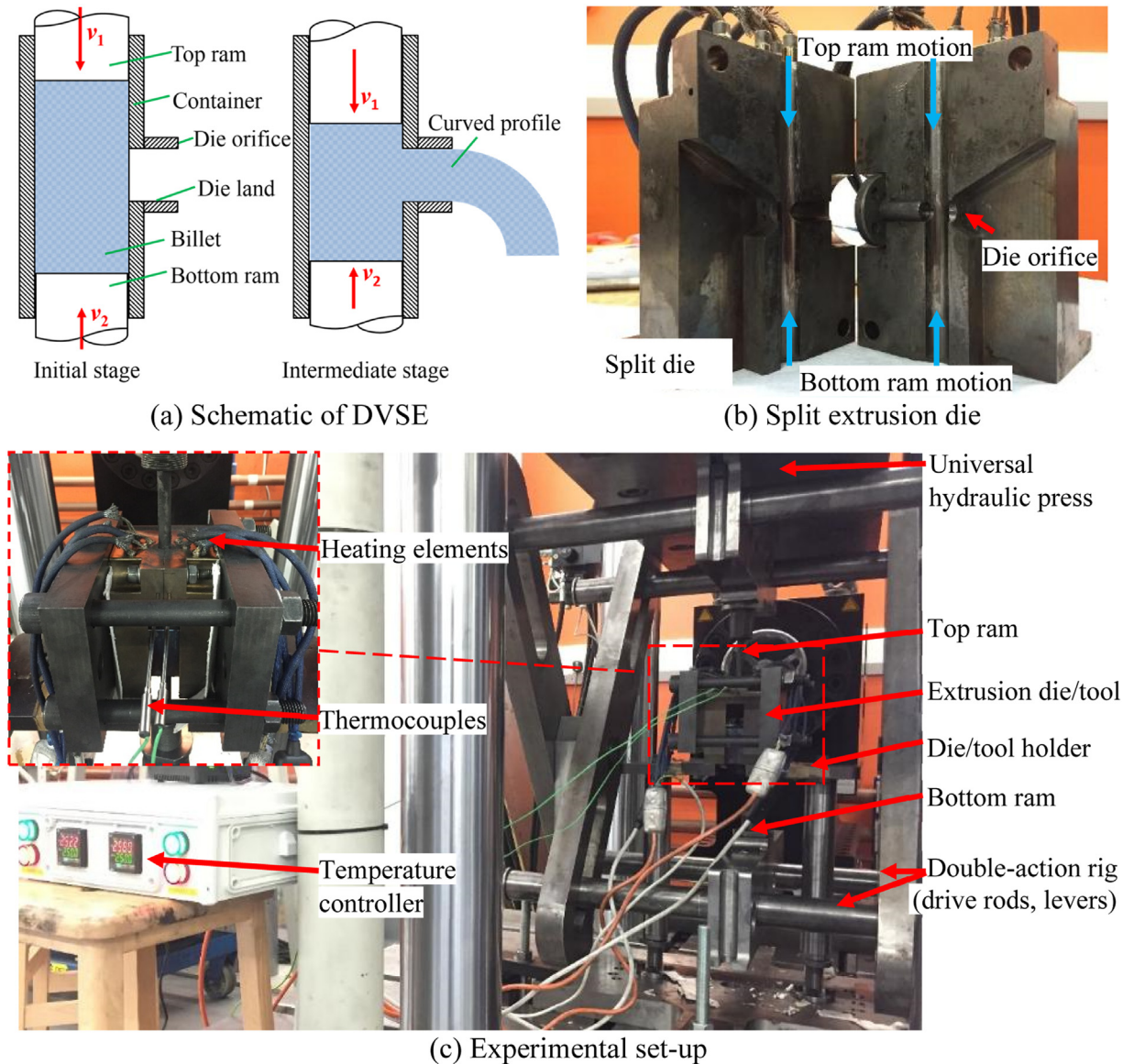


Fig. 1. Principle of the DVSE process, designed split extrusion die with heating elements, and experimental set-up with extrusion toolset mounted on an Instron press.

drical billets having a dimension of 11.5 mm diameter and 130 mm length were machined for the DVSE experiments, which were conducted using a split extrusion tool made of H13, as shown in Fig. 1b. The height of the container (vertical channel) was 150 mm and its diameter was 12.7 mm, very close to the 10 mm diameter of the die orifice (horizontal channel) in the side of the container. This enables a weak cross-section reduction effect by a nominal extrusion ratio of 1.61 and focus on the intense shearing after passing through the channel intersection region [42]. The die land was 2 mm in length. Fig. 1c shows the experimental set-up where the extrusion toolset was mounted on an Instron press. Graphite based Omega-35 grease was used as a lubricant and applied to both the billet and extrusion tool.

The test program is illustrated in Tables 1 and 2. The velocity of the top ram was set as $v_1 = 0.1, 1$ mm/s, respec-

Table 1
Test matrix for AZ31-effect of extrusion velocity ratio

Temperature T (°C)	Velocity v_1 (mm/s)	Velocity ratio v_2/v_1		
		1/3	1/2	2/3
300	0.1	✓	✓	✓

tively, by the Instron hydraulic press. The velocity ratio of the bottom ram to the top ram was set as $v_2/v_1 = 1/3, 1/2$ and $2/3$, respectively, by a specially designed double-action loading mechanism (Fig. 1c). Illustration of its kinematics can be seen in [41]. The dynamic recrystallisation (DRX) temperature for AZ31 Mg alloy is approximately between 260 °C and 320 °C [44,45], thus work-piece and tools were heated to a temperature between 250 °C and 350 °C to study DRX.

Table 2
Test matrix for AZ31-effects of temperature and strain rate

Velocity ratio v_2/v_1	Velocity v_1 (mm/s)	Temperature T (°C)		
		250	300	350
1/2	0.1	✓	✓	✓
	1		✓	

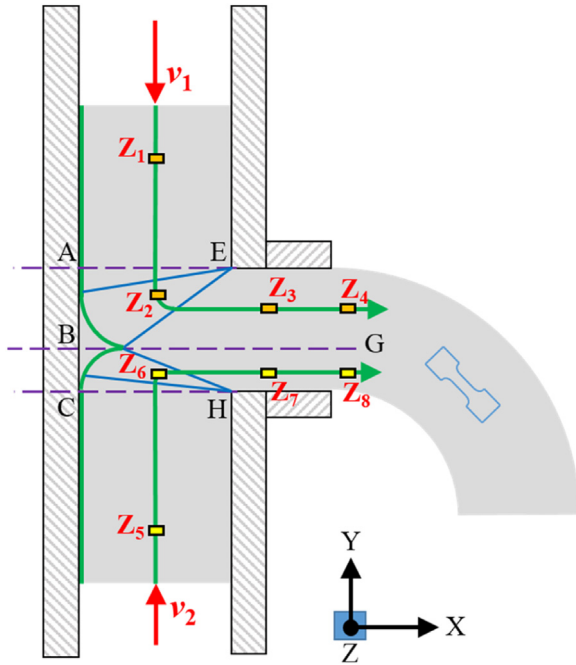


Fig. 2. Deformation mode and sampling locations for characterisation of microstructure and mechanical properties.

For each extrusion test, the billet was preheated to the extrusion temperature by furnace and transferred to the die heated to the same temperature, where it was held for 5 min before being extruded. The die was heated using eight cartridge heaters, four in each half of the die. A temperature controller was specially designed to control the cartridge heaters to heat the die to a prescribed temperature. A 3 mm diameter hole was drilled into each half die, and the end of the hole was ~5 mm away from the vertical channel wall. Two K-type thermocouples were inserted into these two holes to measure the real-time temperature of the die, which was fed back to the controller to control temperature. After DVSE, the curved profiles were quenched in water immediately.

2.2. Characterisation of microstructure and mechanical properties

Fig. 2 shows a 2D deformation mode considered on the symmetry plane XY of the die orifice and container. The billet is pressed with velocities v_1 and v_2 in the Y direction and flows out in the X direction. Material in the intersection region (ACHE) of the horizontal and vertical channels can be reasonably regarded as two segments divided by a line BG which is related with v_2/v_1 , and $AB/AC \sim 0.55$ when

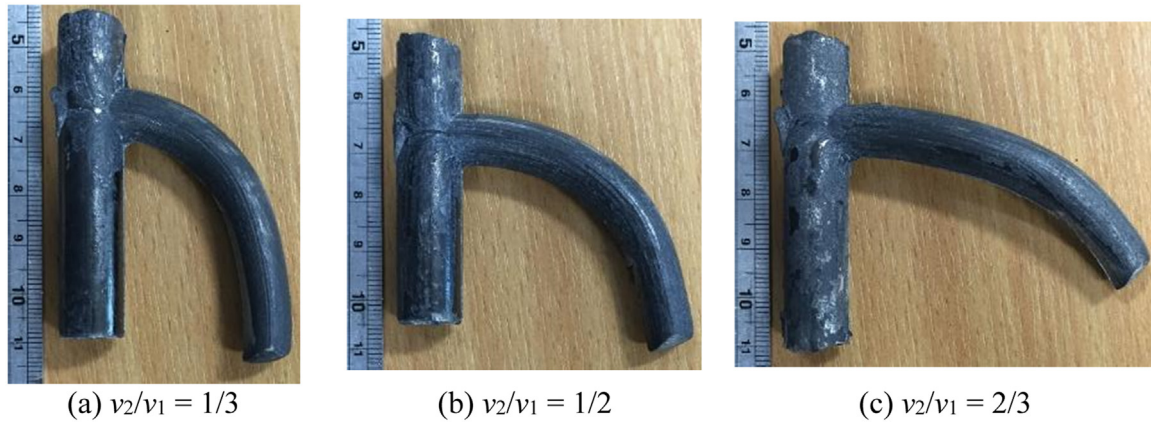
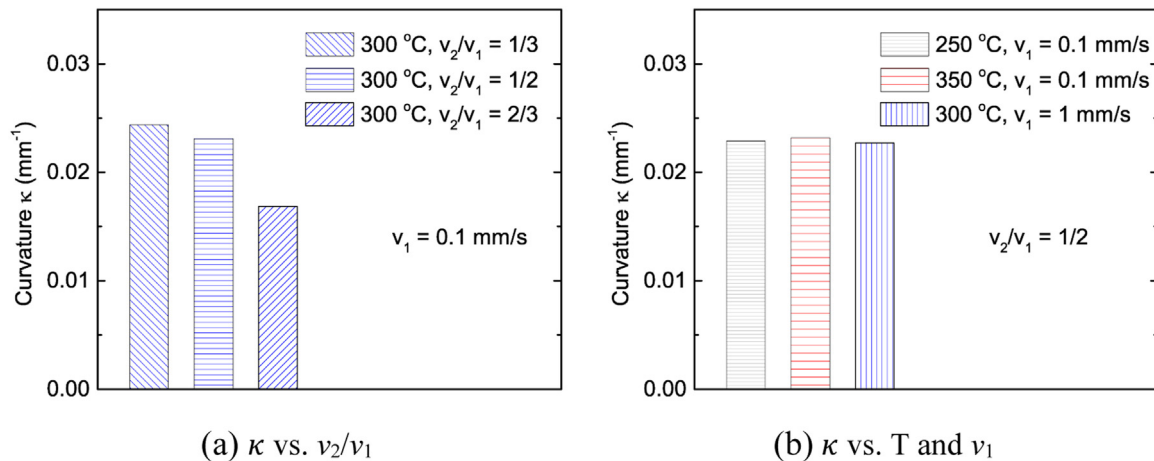
$v_2/v_1 = 1/2$ [46]. For the two segments in the region ACHE, when material passes through the intersection region of the entrance and exit channels, shear deformation occurs similarly to that in the channel angular extrusion, which mainly takes place in the zone (containing Z_2 or Z_6) near the ideal intersection plane ('EB' or 'HB') of the corresponding two channels [47,48]. Thus, on the container/orifice symmetry plane XY , locations Z_1 – Z_4 along the flow line in the middle of the upper part were chosen for microstructure characterisation, in which Z_1 is in the upsetting zone, Z_2 is in the shearing zone, Z_3 is in the forming zone (die bearing/land) and Z_4 is in the profile after forming. Similarly, locations Z_5 – Z_8 along the flow line in the middle of the lower part were chosen. Micro-hardness characterisation of the initial billet and zone Z_4 of the extruded profiles was undertaken on a Zwick Roell machine, the load and dwell time applied were 10 kgf (HV10) and 10 s, respectively. A Deben MICROTENSILE tensile stage was used to conduct tensile tests at room temperature, the loading speed was 0.2 mm/min. Specimens for tensile tests had gauge sections of 2 mm × 3 mm × 5 mm, which were obtained through electrical discharge machining of the billet and curved AZ31 profiles along their axes (Fig. 2).

Optical microscopy (OM) was conducted on a ZEISS optical microscope. Specimens for OM were polished with silicon carbide papers and etched for 25 s with an etchant composed of 1 ml acetic acid, 1 g oxalic acid powder, 1 ml nitric acid, and 150 ml distilled water. Electron backscatter diffraction (EBSD) characterisation was performed on a Hitachi S-3400N SEM equipped with a Bruker's QUANTAX EBSD analysis system; the operation voltage was 25 kV. Specimens for EBSD were polished using colloidal silica suspension (0.04 μm) and Metprep® Alpha cloth, then they were finished by argon ion polishing using the Gatan PECS II system.

3. Results and discussion

3.1. Effects of extrusion velocity ratio, temperature and strain rate on profile curvature

Photographs of profiles extruded at the same temperature (300 °C), top ram velocity v_1 (0.1 mm/s) and different velocity ratios ($v_2/v_1 = 1/3, 1/2, 2/3$) of the bottom ram v_2 to top ram v_1 are shown in Fig. 3. Fig. 4 illustrates profiles extruded at $v_2/v_1 = 1/2$ at three different temperatures (250, 300, 350 °C) and two different strain rates using velocity v_1 of 0.1 and 1 mm/s. It can be seen that extruded profiles are curved where no observable surface wrinkling or cross-sectional distortion were detected, though each extruded profile has a black surface due to the lubricant. Therefore, it is feasible for DVSE to form curved magnesium profiles directly from cylindrical billets. Since in DVSE the curvature is induced by differential material flow during extrusion instead of using subsequent bending operations on straight profiles, as is done in current processes, potential defects caused by these operations could be obviated. Fig. 3 shows that curvature can be regulated by adjusting the velocity ratio and lower velocity ratio results in greater profile curvature. Fig. 5 gives the estimated values of

Fig. 3. AZ31 profiles extruded at 300 °C, $v_1 = 0.1$ mm/s and different velocity ratios.Fig. 4. AZ31 profiles extruded at velocity ratio $v_2/v_1 = 1/2$ and different temperatures and strain rates.Fig. 5. Variation of curvature κ at different velocity ratios $v_2/v_1 = 1/3, 1/2, 2/3$, temperatures (250, 300, 350 °C) and strain rates (using velocity $v_1 = 0.1, 1$ mm/s).

curvature, which were obtained using the same procedure in previous work [41]. It shows that temperature and strain rate have a relatively small effect on the profile curvature, in comparison with velocity ratio. Profile curvature is mainly controlled by the velocity ratio. However, extrusion temperature and strain rate might affect the microstructure and mechanical properties of the profiles and this is discussed below.

3.2. Grain structure

3.2.1. Optical microscopy for grain evolution along flow lines

Optical microstructures along flow lines of the extruded shapes illustrated in Fig. 3b and Fig. 4, i.e. under the same velocity ratio $v_2/v_1 = 1/2$ but different temperatures and strain

rates, have been studied and the results are shown in Figs. 6–9. The average grain size of the initial billets was $\sim 25 \mu\text{m}$. Fig. 6a–h show the case of 250°C , $v_1 = 0.1 \text{ mm/s}$, $v_2/v_1 = 1/2$. Comparison of micrographs Z_1 to Z_4 indicate that the grains gradually split into a bi-modal distribution along the flow line, where fine grains of $\sim 1 \mu\text{m}$ and coarse grains of $\sim 20 \mu\text{m}$ exist. The formed profile has an average grain size of $\sim 3.5 \mu\text{m}$, as illustrated in Fig. 6d (Z_4). These measurements demonstrate that DVSE is effective in refining grains of AZ31 Mg alloy while forming curved profiles. The grain refinement initiates at the prior boundaries of coarse grains, as shown in Fig. 6b (Z_2), an inhomogeneous microstructure with bi-modal necklace pattern grains exists, indicating the occurrence of dynamic recrystallisation (DRX). The most remarkable grain refinement happens after passing the shearing zone (Z_1 to Z_3), where the number of dynamically recrystallised (DRXed) fine grains increases significantly. It is also noted that the coarse grains in Z_2 are elongated in Z_3 . As illustrated in Fig. 6c (Z_3), the elongation direction is obliquely upward, approximately in accordance with the direction of the ideal shear plane ‘EB’ shown in Fig. 2. These coarse grains in Z_3 are further elongated in the forming zone at the die bearing/land (Z_3 to Z_4) due to extrusion. Z_5 – Z_8 show similar grain evolution characteristics, thus for other temperatures and strain rates only the upper part is considered. It is seen that grain elongation direction of the lower part is obliquely downward (Fig. 6g (Z_7) and Fig. 6h (Z_8)), since the direction of the shear plane ‘HB’ shown in Fig. 2 is opposite to that of ‘EB’. Also, grains in the lower part (Z_6 – Z_8) have a slightly smaller size compared with those in the upper part (Z_2 – Z_4), especially for the coarse grains, although the difference is not obvious for the recrystallised fine grains. This is a result of the slightly greater effective strain induced in the lower part than that in the upper part for $v_2/v_1 = 1/2$ [46]. According to the previous work [41,46], DVSE could be reasonably considered to be two non-equal channel angular extrusion/pressing (N-ECAE/P) processes divided by a line BG shown in Fig. 2b (AB/AC ~ 0.55 for $v_2/v_1 = 1/2$), the upper part (corresponds to the faster top ram) of the extrudate has a lower effective extrusion ratio and less intensive shear deformation, resulting in a lower effective strain.

At 300°C , $v_1 = 0.1 \text{ mm/s}$, $v_2/v_1 = 1/2$, as shown in Fig. 7a, the grains in the upsetting zone Z_1 show similar characteristics as those in Fig. 6a. However, the grains in the shearing zone Z_2 (Fig. 7b) and forming zone Z_3 (Fig. 7c) are more uniform than those in Fig. 6b–c, though the latter has smaller fine grains due to the lower forming temperature. After forming, a typical microstructure with complete DRX is seen in the profile zone Z_4 (Fig. 7d). The grains are equiaxed and uniformly distributed, having an average size of $\sim 4.5 \mu\text{m}$.

Fig. 8 shows the microstructure of zone Z_4 of curved profiles ($v_2/v_1 = 1/2$) formed at a higher temperature (350°C) and strain rate (using velocity $v_1 = 1 \text{ mm/s}$), compared with the case of 300°C , $v_1 = 0.1 \text{ mm/s}$, $v_2/v_1 = 1/2$. At $v_1 = 0.1 \text{ mm/s}$, $v_2/v_1 = 1/2$, when increasing the temperature to 350°C , as illustrated in Fig. 8a, a slight increase in the size of

the DRXed fine grains ($\sim 5 \mu\text{m}$) is evident, compared with grains in Fig. 7d. This indicates that grain growth takes place at a higher temperature of 350°C .

At 300°C , $v_2/v_1 = 1/2$, when increasing the strain rate by increasing the extrusion velocity v_1 to 1 mm/s , bi-modal grains (~ 2 – $20 \mu\text{m}$) are formed, as shown in Fig. 8b. This could be the result of incomplete DRX due to the short extrusion time and time for dynamic recrystallisation. On the other hand, increased strain rate may lead to temperature increase which might cause grain growth. EBSD was conducted and results are shown in Section 3.3 to examine the subgrains and gain a better understanding of this case.

3.2.2. EBSD characterisation of grain evolution along flow lines

Fig. 9a–e show the EBSD inverse pole figure (IPF) maps of the initial billet and along the flow line of the extruded shape at 250°C , $v_1 = 0.1 \text{ mm/s}$, $v_2/v_1 = 1/2$. The white lines indicate low-angle boundaries (LABs) with misorientation $2^\circ \leq \theta < 15^\circ$, while the black lines indicate high-angle boundaries (HABs) with misorientation $\theta \geq 15^\circ$. Here, plane XY coincides the flow plane XY shown in Fig. 2. The LABs in the initial billet, upsetting zone (Z_1), shearing zone (Z_2), forming zone (Z_3) and profile zone (Z_4) are calculated to occupy 5.19%, 9.47%, 34.54%, 15.98%, and 13.97%, respectively, and their grain sizes to be 24.95, 19.98, 4.13, 3.61 and $3.47 \mu\text{m}$, respectively. During the deformation, a large number of sub-structural boundaries (LABs) are formed in the initial coarse grains due to the intense shearing (Fig. 9c (Z_2)). After passing through the shearing zone (Z_2), some of these sub-structural boundaries evolve into HABs and tiny recrystallised grains are generated at the boundaries, several long strip grains are also formed, as shown in Fig. 9d–e. This confirms the results from optical microscopy (OM) that dynamic recrystallisation (DRX) is the grain refinement mechanism for magnesium alloy AZ31 in the DVSE process. This grain refinement mechanism is different from that in fcc materials such as Al alloys, because the grain refinement in Mg-based alloys requires the activation of non-basal slip systems [49], which is favoured through the development of stress concentrations along the boundaries [50]. Hence the grain refinement for Mg-based alloys tends to initiate at the coarse grain boundaries in a necklace-like manner.

3.3. Effects of temperature and strain rate on grain structure

To further investigate effects of temperature and strain rate on subgrains and grain boundaries, which cannot be revealed in optical microscopy, EBSD IPF maps of the zone Z_4 of curved profiles formed at $v_2/v_1 = 1/2$ and different temperatures (300 , 350°C) and strain rates (using velocity $v_1 = 0.1$, 1 mm/s) are shown in Fig. 10a–c. Fig. 11 shows the corresponding distribution of grain size at each case. Quantitatively, the value of the average grain size has been calculated to be 3.47, 4.43, 5.08 and $6.70 \mu\text{m}$, respectively, for the cases: (i) 250°C , 0.1 mm/s , (ii) 300°C , 0.1 mm/s , (iii) 350°C , 0.1 mm/s ,

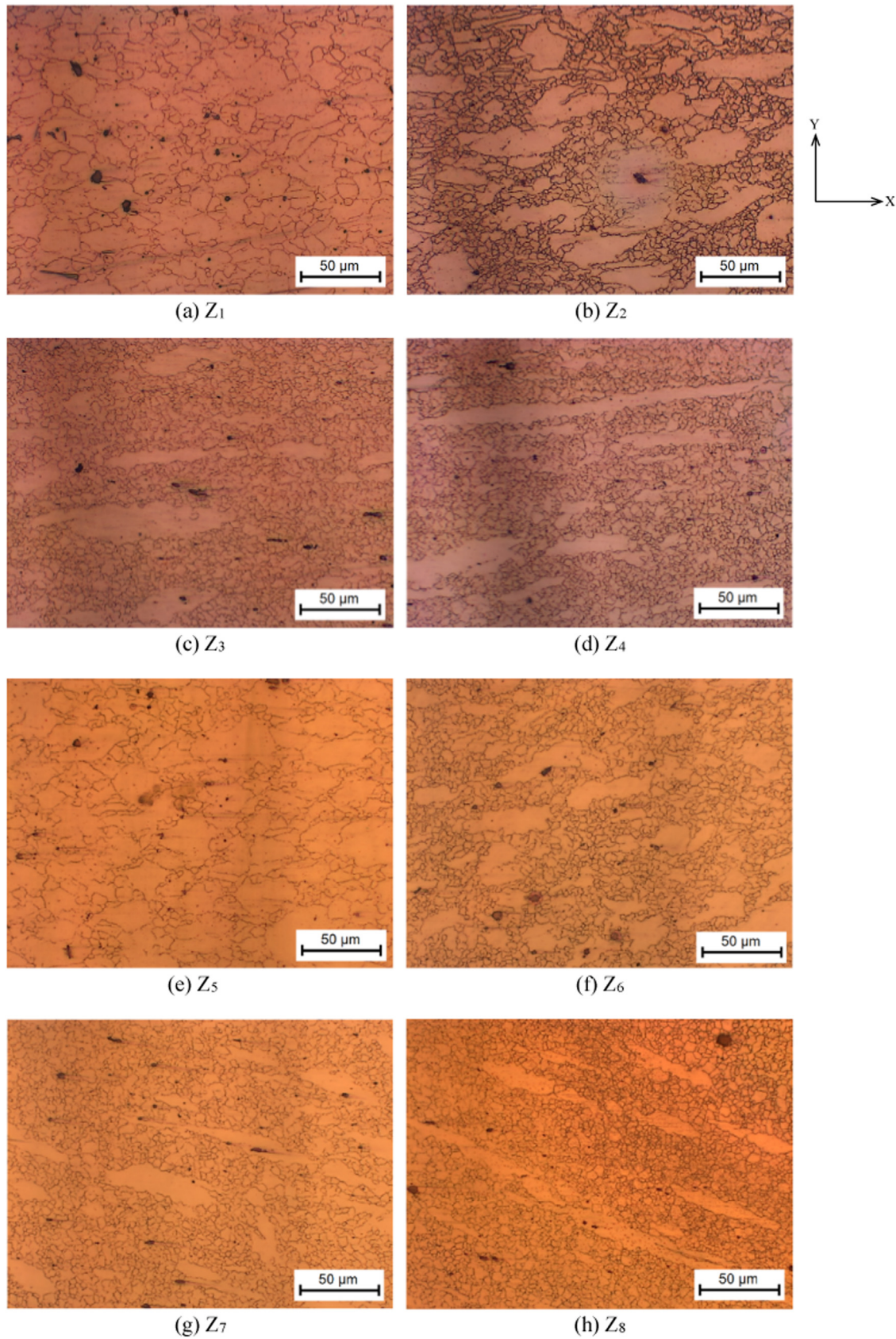


Fig. 6. Microstructure evolution along the flow line on the symmetry plane XY at 250 °C and 0.1 mm/s.

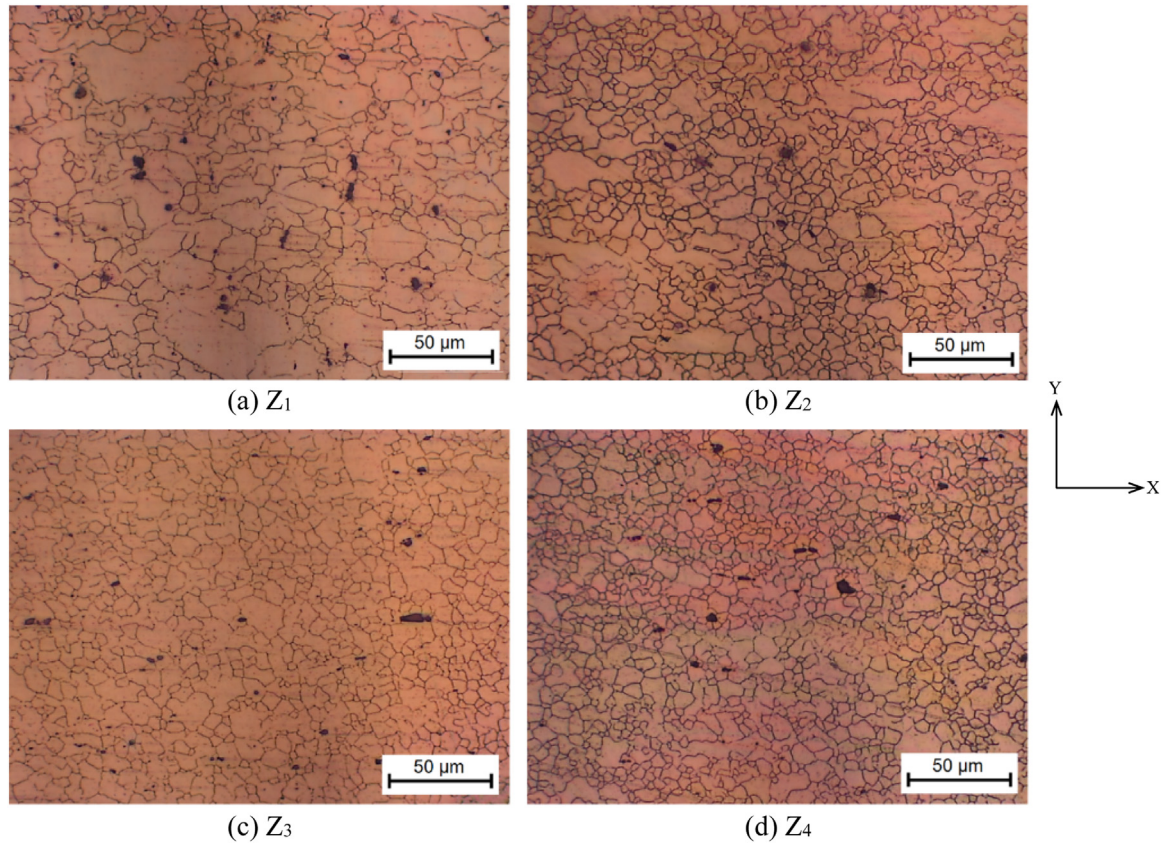


Fig. 7. Microstructure evolution along the flow line on the symmetry plane XY at 300 °C and 0.1 mm/s.

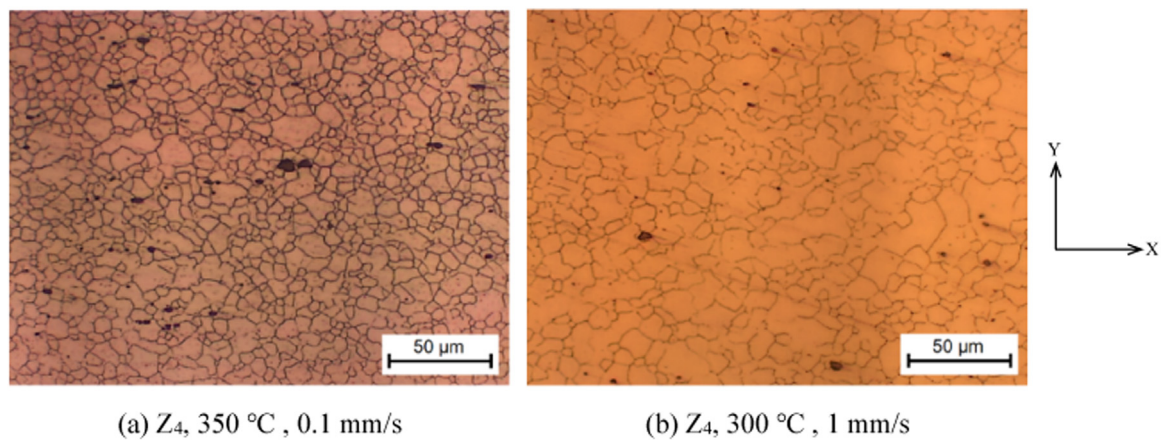


Fig. 8. Microstructure of zone Z_4 of curved profiles ($v_2/v_1 = 1/2$) formed at a higher temperature (350 °C) and strain rate (using velocity $v_1 = 1$ mm/s).

and (iv) 300 °C, 1 mm/s. The microstructures are consistent with the optical results.

The specific misorientation distribution is shown in Fig. 12. LABs in the profile zone Z_4 are calculated to occupy respectively 13.97%, 9.87%, 8.16% and 14.06%, for the cases: (i) 250 °C, 0.1 mm/s, (ii) 300 °C, 0.1 mm/s, (iii) 350 °C, 0.1 mm/s, and (iv) 300 °C, 1 mm/s. Fig. 12a shows a large number of LABs are formed after DVSE at 250 °C, 0.1 mm/s, which is a result of many incomplete DRX grains observed in Fig. 9e. By increasing temperature to 300 °C, as shown

in Fig. 12b, the number of LABs decreases, and homogeneous equiaxed refined grains are generated. This is due to the complete DRX that has taken place at a higher temperature. With a temperature increase to 350 °C, 0.1 mm/s, as depicted in Fig. 12c, the number of LABs slightly decreases whilst the fine DRXed grain size increases, suggesting that grain growth has occurred. It is also apparent that a number of HABs of ~ 86 – 88° exist, indicating that twin boundaries have occurred. A close inspection of the orientation reveals that $\{10\bar{1}2\}$ tension twins have been formed which can be

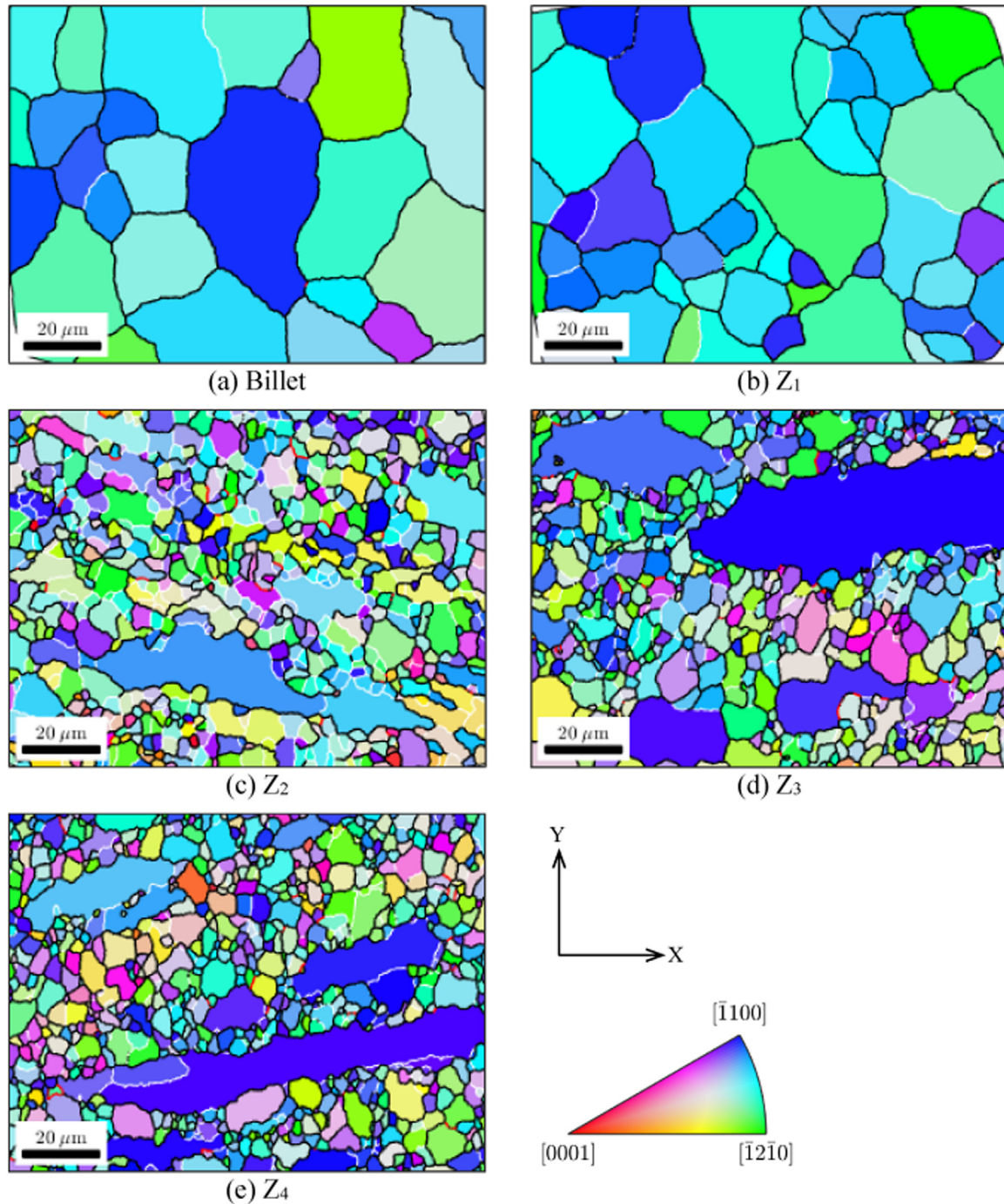


Fig. 9. EBSD IPF maps on the flow plane XY of the billet and along the flow line of the extruded shape at 250 °C, $v_1 = 0.1$ mm/s, $v_2/v_1 = 1/2$.

characterised by $86.2 \pm 5^\circ$ rotation around a $\langle 11\bar{2}0 \rangle$ axis, as shown in Fig. 10b (band contrast map highlighting boundaries of $86.2 \pm 5^\circ$ misorientation). It is known that tension twins can be caused by tension parallel to the c -axis, compression perpendicular to the c -axis and constraint imposed by deformation of neighbouring grains [51–53]. Actually, the c -axis of the majority of grains of the initial billet is distributed perpendicular to its extrusion direction (billet axial direction), this is detailed in Section 3.4. Tension twins are found in a number of coarse grains in Fig. 10b, possibly be-

cause some DRXed grains after grain growth have random orientation [5] with their c -axis parallel to the extrusion direction where tensile stress exists for DVSE [43], leading to tension twins. Actually tension twins in channel-die extrusion of magnesium has been found to be affected by temperature, the critical resolved shear stress (CRSS) of tension twins at elevated temperatures (150–450 °C) tends to decrease with the increase of temperature [54]. It is a widely accepted rule that as temperature increases, slip is favoured at the expense of twinning. However it cannot be taken as evidence that twin-

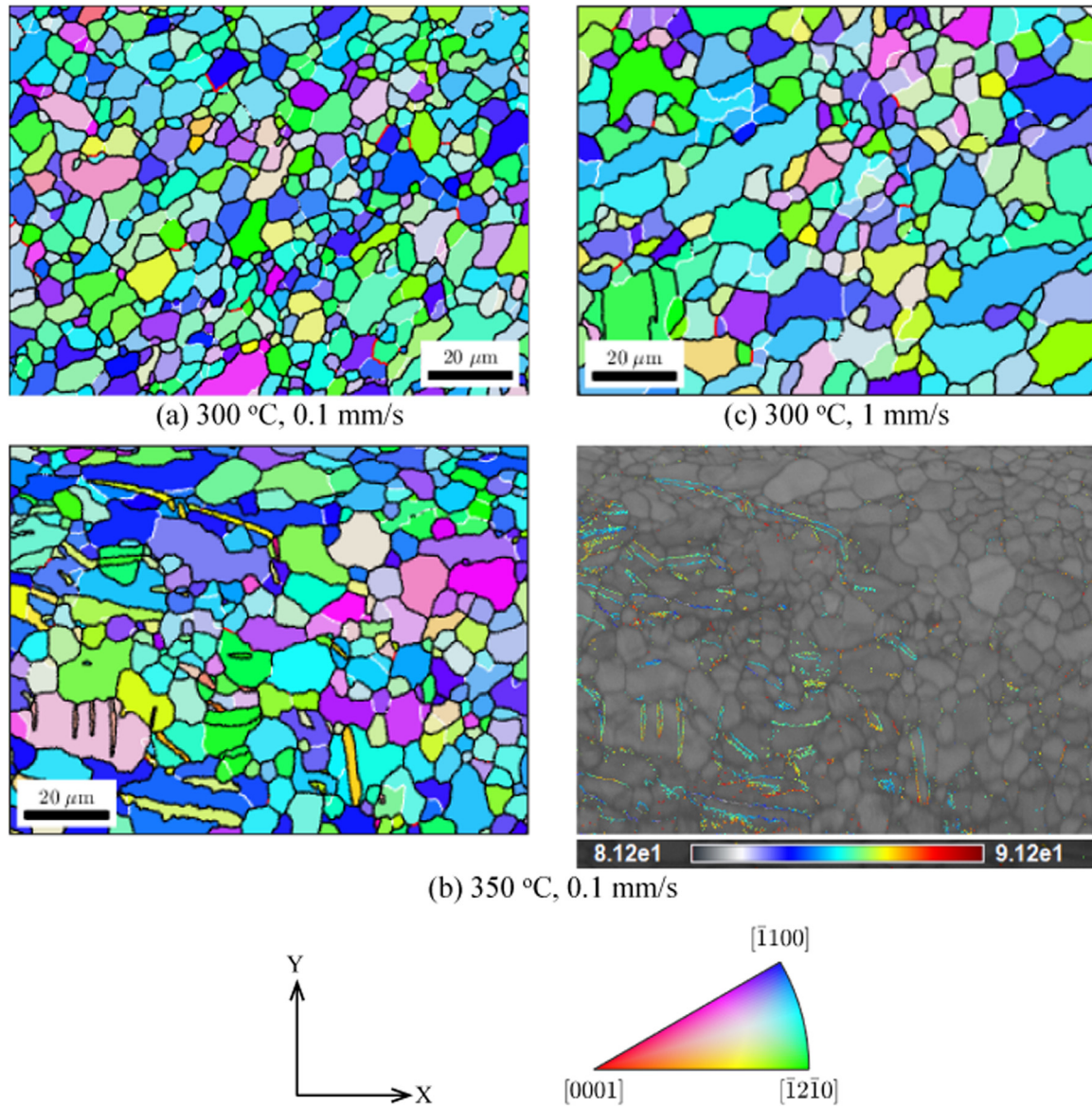


Fig. 10. EBSD IPF maps of zone Z₄ of curved profiles ($v_2/v_1 = 1/2$) formed at different temperatures (300, 350 °C) and strain rates (using velocity $v_1 = 0.1$, 1 mm/s).

ning becomes more difficult at higher temperatures, but only as showing that the stress needed for slip varies more sharply with temperatures than the stress needed for twinning [55]. In the case of magnesium there has been a diverse opinion, where an increase in twinning with increase of temperature and also an opposite tendency have been reported [55–57]. It is also noted that the activity of tension twins tends to be grain size dependent, which occurs mostly in regions where coarse grains exist. This is consistent with the reports that the activity of twinning decreases with the decrease of grain size and disappears when the grain size is sufficiently small [58,59]. The existence of twins reveals that the deformability of the coarse grains (due to grain growth) at 350 °C is lower than that of the fine grains. By increasing the extrusion velocity v_1 (hence strain rate) to 1 mm/s while maintaining the temperature of 300 °C, as shown in Fig. 12d, the LABs

fraction increases greatly. This leads to the conclusion that incomplete DRX occurs at 300 °C, 1 mm/s, due to greatly decreased deformation time.

EBSD results confirm that curved profiles with well refined grains can be formed by DVSE from billets of $\sim 25 \mu\text{m}$ at the forming condition 300 °C, 0.1 mm/s. It has been reported that a critical limiting grain size of the initial billet plays a large part in determining whether the microstructure of magnesium alloys after few passes of ECAE/P is homogeneous or bi-modal [34,35]. It was shown that an initial grain size of $\sim 11.0 \mu\text{m}$ is still too large to obtain a homogeneous distribution of fine grains after two passes of ECAE/P at 200–300 °C and a pressing speed 20 mm/min, and the microstructure became reasonably homogenous after 4 passes [60]. Xu et al. [61] also found that an initial grain size of ~ 15.0 – $22.0 \mu\text{m}$ still leads to an inhomogeneous microstructure

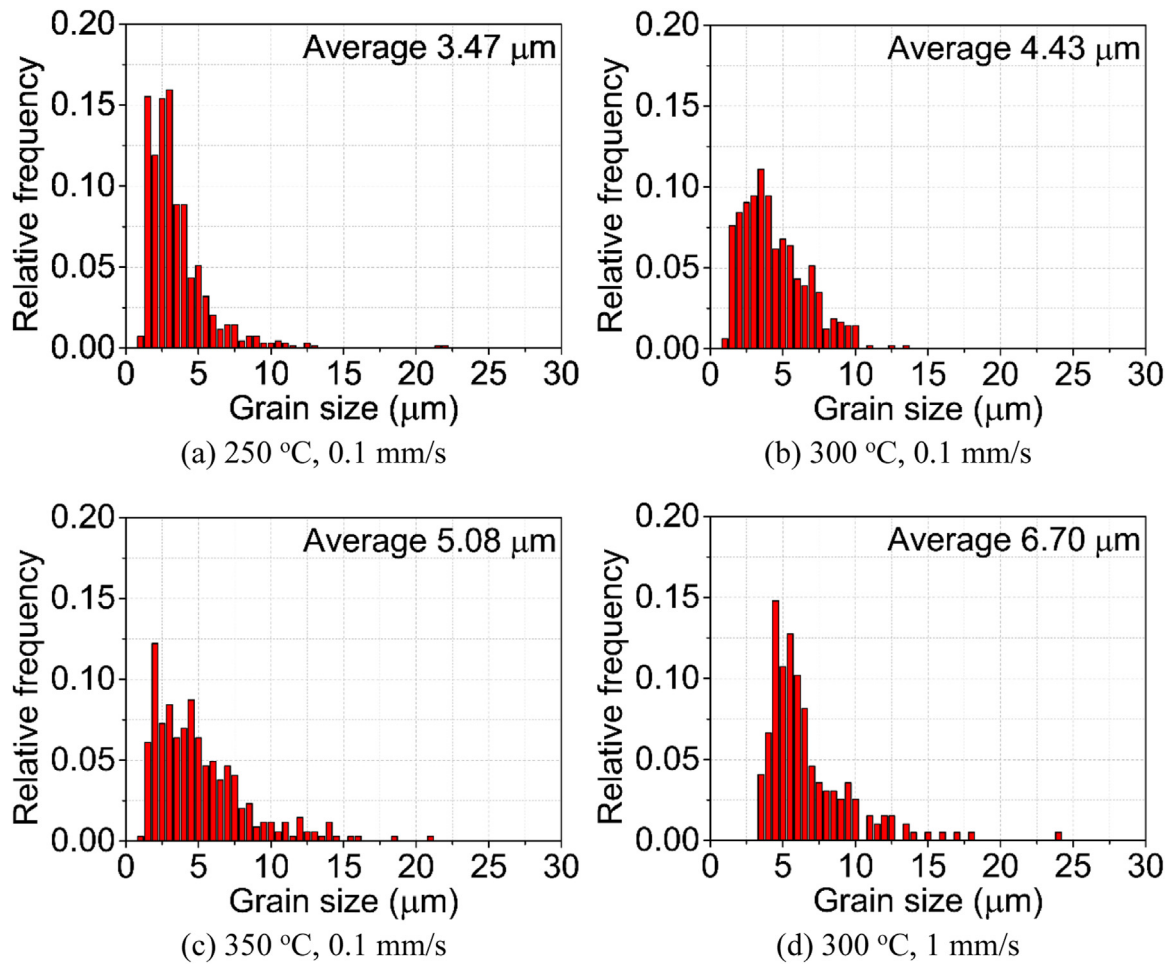


Fig. 11. Grain size distribution in zone Z₄ of curved profiles ($v_2/v_1 = 1/2$) formed at different temperatures (250, 300, 350 °C) and strain rates (using velocity $v_1 = 0.1, 1$ mm/s).

after one pass of ECAE/P, even with the assistance of significant back pressure. As discussed in [35], the grain refinement for Mg-based alloys processed by ECAE/P initiates in a necklace-like manner at the prior boundaries of coarse grains, which is similar to that in DVSE. For Mg-based alloys having a large initial grain size, a bi-modal grain structure will be initially developed after few passes of ECAE/P, the centres of the initial coarse grains are not yet consumed by the recrystallised fine grains, and homogeneous microstructure will be observed only after a sufficient number of ECAE/P passes. Here, it has been shown in our results that an initial billet with grain size of $\sim 25 \mu\text{m}$ can be formed into a curved profile with refined and homogeneous microstructure after a single operation of DVSE at proper temperature and strain rate (300 °C, $v_1 = 0.1$ mm/s). The reason is that DVSE results in more severe straining than that in ECAE/P per pass, as DVSE could be reasonably considered as two N-ECAE/P processes depending on the extrusion velocity ratio. This is compatible with the previous report that greater strains and grain refinement of AA1050 are induced in DVSE than that in ECAE/P per pass [42].

3.4. Effects of temperature and strain rate on texture

Fig. 13a-e show the {0002} pole figure of the zone Z₄ of the curved profiles formed at different cases: (i) 250 °C, 0.1 mm/s, (ii) 300 °C, 0.1 mm/s, (iii) 350 °C, 0.1 mm/s, and (iv) 300 °C, 1 mm/s. The {0002} pole figure of the initial billet is also given. Here, ED represents the extrusion direction (axial direction for the initial billet and extruded profiles), ND represents the normal direction, where plane ED-ND coincides the flow plane XY depicted in Fig. 2. For comparison, Fig. 13f-g show the ideal positions and fibres in {0002} pole figure for hcp crystals subjected to simple shear [62] and one pass of ECAE/P with 90° channel intersection angle [63], respectively. The shear direction/plane of the simple shear in Fig. 13f is along the X' direction/axis. For ECAE/P with 90° channel intersection angle (Fig. 13g), the deformation mode is near simple shear where the orientation of the ideal shear plane X'Z' is tilted 45° to the ED, thus the ideal positions of typical texture components are rotated by an angle 45°, i.e. most grains have the {0002} basal plane oriented to the shear direction. In reality, for AZ31 alloy processed

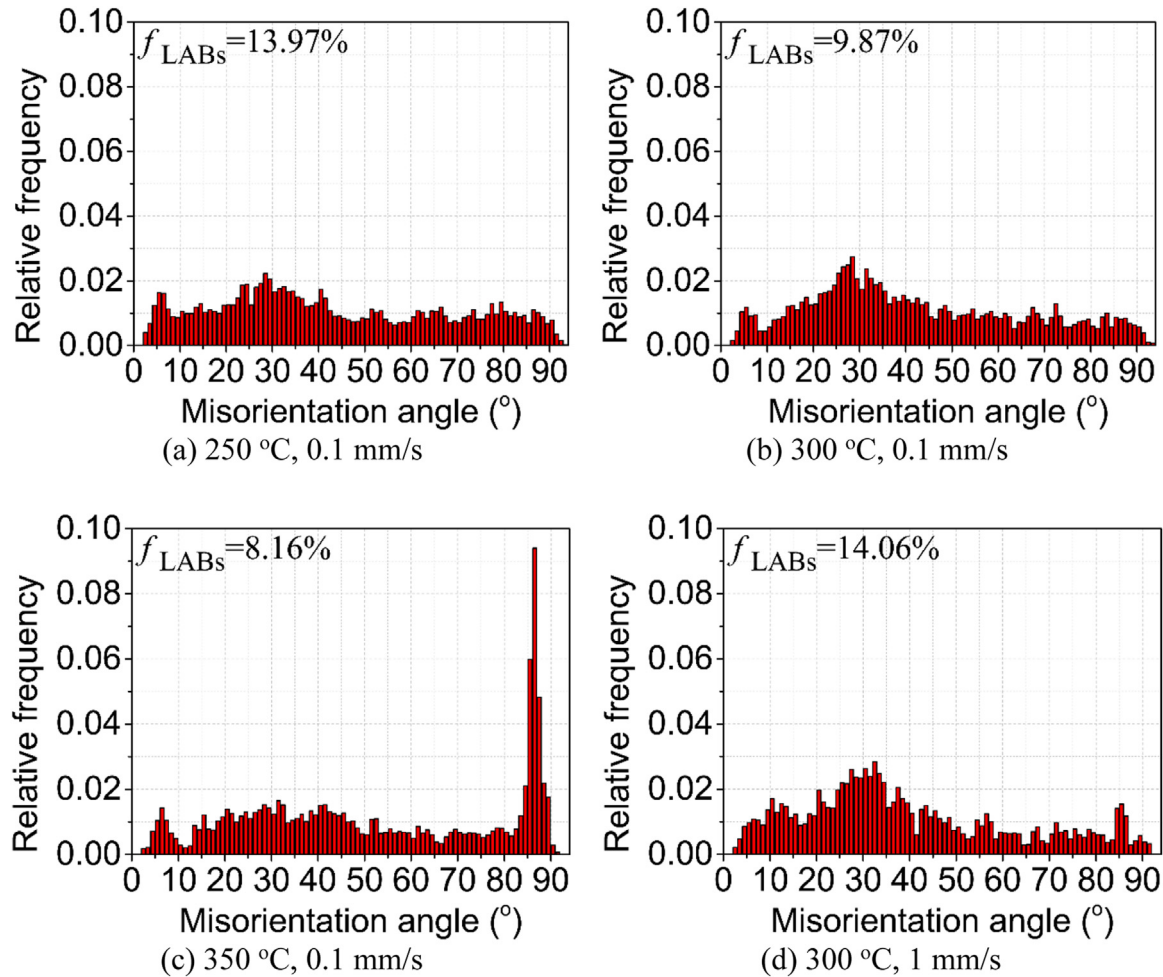


Fig. 12. Misorientation distribution in zone Z₄ of curved profiles ($v_2/v_1 = 1/2$) formed at different temperatures (250, 300, 350 °C) and strain rates (using velocity $v_1 = 0.1, 1$ mm/s).

by one pass of ECAE/P, the orientation of the {0002} basal plane ranges ~ 20 – 45° to the ED depending on the initial texture [64].

The high maximum texture intensity and the fibre-like distribution in Fig. 13a indicate that the initial billet exhibits a strong $\langle 10\bar{1}0 \rangle$ fibre texture, the basal plane of most grains is oriented parallel to the extrusion direction (billet axial direction), which has been typically observed in magnesium after conventional extrusion ('hard' orientation with low Schmid factor for basal slip). This can greatly limit its ductility near room temperature. On the other hand, the material processed by DVSE shows greatly weakened {0002} pole density, basal plane rotates to the position inclined by ~ 5 – 10° to the ED, though the inclination angle and the texture intensity are different in the developed texture for different cases (temperatures and strain rates). This angle is smaller than the previously reported texture of ECAE/P claiming ~ 20 – 45° to the ED. This may be attributed to the different shear plane direction in contrast to the ED [42]; larger shear strain is introduced in DVSE than that in ECAE/P, which makes the ideal shear plane (plane 'EB' shown in Fig. 2) or shear direction closer to the ED ($< 45^\circ$) compared with ECAE/P ($= 45^\circ$).

As a result, the inclination angle of the {0002} basal plane to the ED in DVSE is less than that in ECAE/P. This inclination angle is slightly affected by the temperature, an increased temperature tends to lead to a lower angle, i.e. the {0002} basal plane rotates towards the ED as processing temperature increases. This is similar to the situation found in ECAE/P [60,65]. It is attributed to the sharply decreased critical resolved shear stress for pyramidal slip at elevated temperature, making the basal plane inclined to the principal stress axis [65–67]. As slip systems of the basal plane are operative at elevated temperature, the crystals rotate around the Z axis by basal slip as the principal stress axis rotates, making the basal plane rotate towards the ED [65]. In the meantime grain boundary sliding occurs and the low angle boundaries continue to turn into high angle boundaries. The contribution of grain boundary sliding to crystal rotation is greater at the case where more DRXed grains are generated [65], eventually for the case of 300 °C, 0.1 mm/s the basal plane of a majority of grains is orientated more parallel to the ED compared with the incomplete DRXed cases as a result of basal slip and grain boundary sliding. The existence of texture slightly deviated from the ED in the case of 350 °C, 0.1 mm/s is

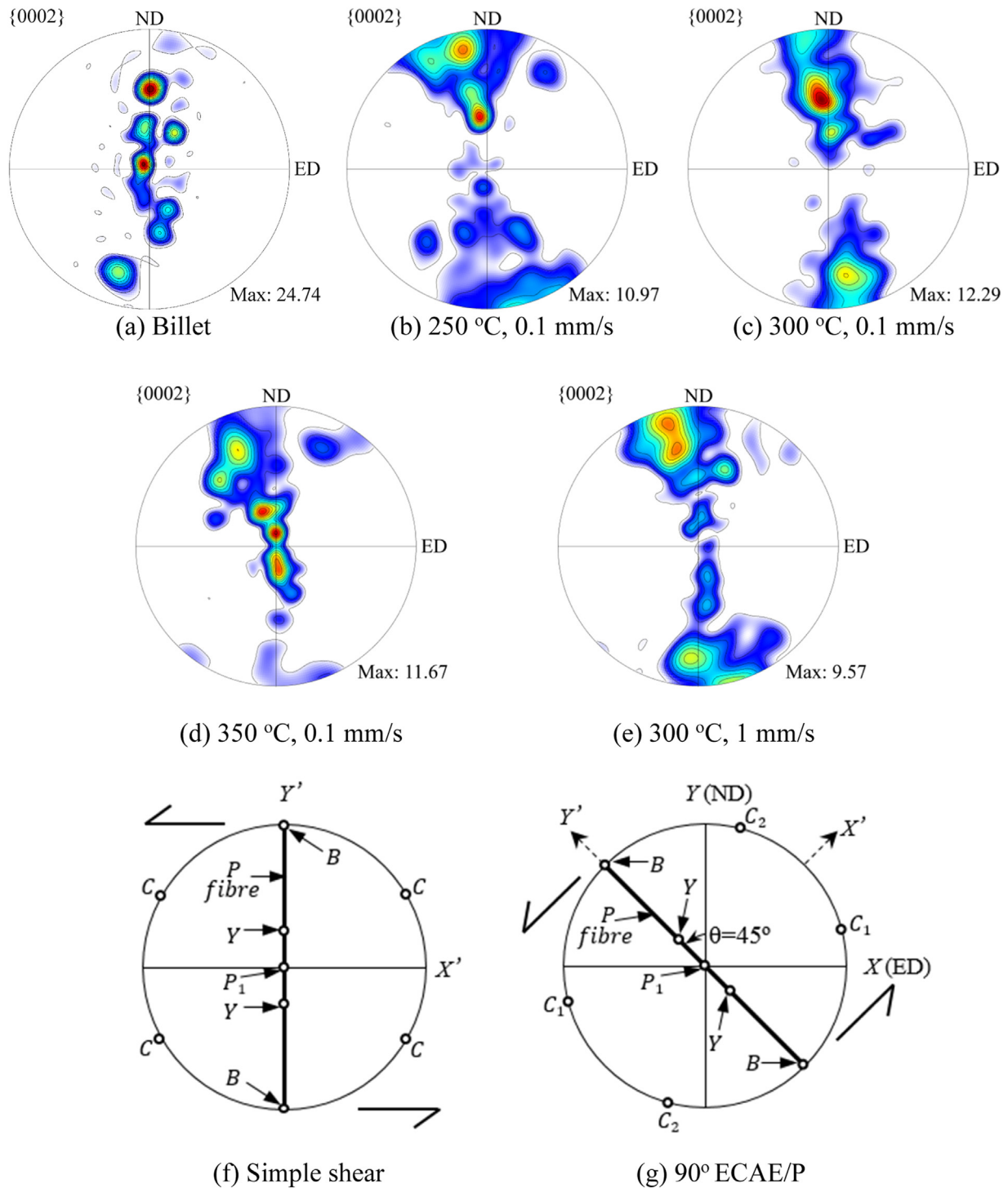


Fig. 13. The {0002} pole figure of the initial billet and zone Z₄ of curved profiles ($v_2/v_1 = 1/2$) formed under different conditions.

possibly caused by the random orientation of some DRXed grains after grain growth [5].

3.5. Effects of temperature and strain rate on micro-hardness and tensile properties

Micro-hardness tests were conducted for the initial billet and zone Z₄ of the curved profiles formed under the condi-

tions: (i) 250 °C, 0.1 mm/s, (ii) 300 °C, 0.1 mm/s, (iii) 350 °C, 0.1 mm/s, and (iv) 300 °C, 1 mm/s. The obtained hardness values are given in Fig. 14 in relation to the average grain size for each case. After DVSE, the hardness significantly increases, which is consistent with the measured decreased grain size shown in Fig. 11.

Fig. 15 shows the engineering stress-strain curves of the initial billet and extruded profiles, which are obtained from

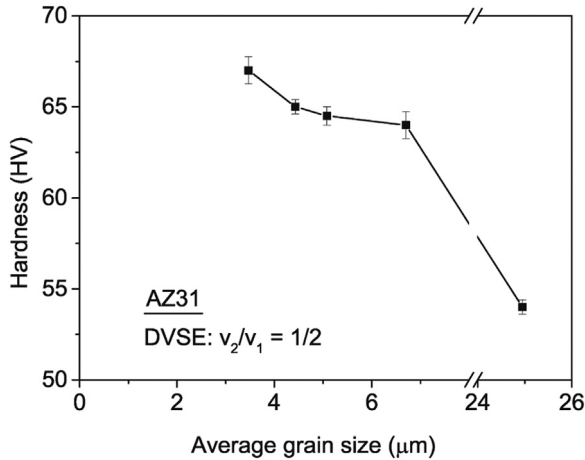


Fig. 14. Hardness of the initial billet and curved profiles in relation to their average grain size.

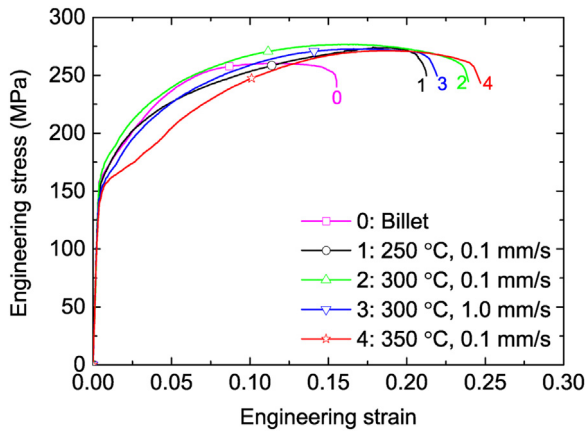


Fig. 15. Tensile stress-strain curves of the initial billet and DVSEd profiles ($v_2/v_1 = 1/2$) at different temperatures (250, 300, 350 °C) and strain rates (using velocity $v_1 = 0.1, 1$ mm/s).

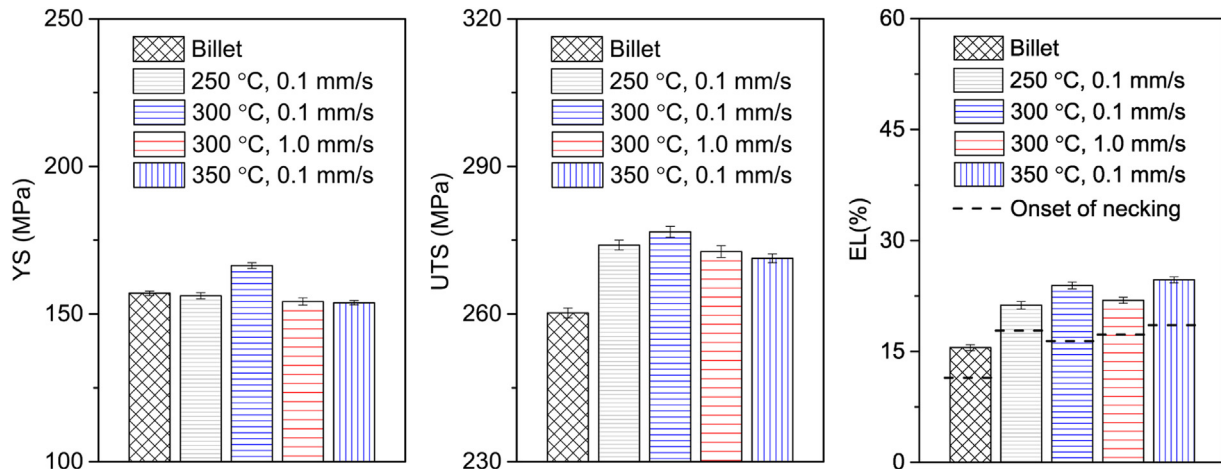


Fig. 16. Material properties of the initial billet and DVSEd profiles ($v_2/v_1 = 1/2$) at different temperatures (250, 300, 350 °C) and strain rates (using velocity $v_1 = 0.1, 1$ mm/s). The dashed line indicates the onset of necking, i.e. uniform elongation before necking.

room temperature tensile tests using a load parallel to the axial direction. Fig. 16 shows the yield strength (YS, 0.2% proof stress), ultimate tensile strength (UTS), and elongation to fracture (EL). After DVSE, the ductility (EL) has all increased without significantly compromising the strength. The UTS of the DVSE-processed (DVSEd) profiles has all increased as well and the yield strength is only slightly lower than that of the billet, except for the complete DRXed case (300 °C, $v_1 = 0.1$ mm/s) where a slight increase has been found. Grain refinement is not able to explain the yield strength variation for the material prior to and after DVSE, in which texture modification has occurred. As discussed above, a strong fibre texture with $\langle 10\bar{1}0 \rangle$ aligned basal plane has been generated in the initial billet after conventional extrusion, i.e. the grains of the initial billet are in a 'hard' orientation, thus the basal plane slip is difficult to be activated. DVSE results in the $\{0002\}$ basal plane of the profiles oriented $\sim 5\text{--}10^\circ$ to the ED (i.e. towards the orientation for easier slip). Owing to the modified basal texture induced by DVSE and the increased Schmid factor for the $\{0002\}$ basal plane caused by the pole rotation [26], stress for yielding could be low on the basal plane. This significantly modified texture also contributes to the related elongation improvement. This is similar to that in ECAE/P, which has been recognised as the texture softening effect [60,68].

To explain the yield strength variation, both the strengthening effect caused by grain refinement and softening effect caused by texture modification need to be considered. At 250 °C, 0.1 mm/s, due to greater texture softening caused by a larger angle of basal planes rotating away from the ED (favourable orientation for slip) and weaker texture intensity, its yield strength is lower compared with that at 300 °C, 0.1 mm/s. The yield strength at 300 °C, 1 mm/s is lower than that at 300 °C, 0.1 mm/s mainly due to lower grain-refinement strengthening caused by the relatively large average grain size. Incomplete DRX occurs and bi-modal grains exist at 250 °C, 0.1 mm/s and 300 °C, 1 mm/s, which lead to a lower elongation than that at 300 °C, 0.1 mm/s. This is probably related

with the lower fraction of HABs ascribed to the incomplete DRX, resulting in reduced plasticity in the tensile tests [26]. Yield strength at 300 °C, 1 mm/s is slightly lower than that at 250 °C, 0.1 mm/s, it could be mainly because of its greater average grain size (6.70 and 3.47 μm , respectively, for the cases 300 °C, 1 mm/s, and 250 °C, 0.1 mm/s) while they have similar LABs fractions due to the incomplete DRX. Elongation at 300 °C, 1 mm/s is slightly larger than that at 250 °C, 0.1 mm/s, probably due to the slightly lower basal texture intensity. At 300 °C, 0.1 mm/s, complete DRX occurs, yield strength and elongation both increase compared to the billet and incomplete DRXed cases (250 °C, 0.1 mm/s and 300 °C, 1 mm/s), indicating the strengthening by grain refinement is greater than the softening by texture modification. After complete DRX, as increasing temperature only coarsens the grain and no obvious texture modification occurs (texture intensity slightly decreases), a reduced yield strength of the profile at 350 °C, 0.1 mm/s is obtained compared with that at 300 °C, 0.1 mm/s. At 350 °C, 0.1 mm/s, a number of $\{10\bar{1}2\}$ tension twins characteristic of orientations occur (Fig. 10b), mostly in regions where there are coarse grains, this might facilitate the uniform elongation (indicated in Fig. 16 as the dashed line) in room temperature tensile tests [53,58,69]. Also, slightly lower basal texture intensity and higher HABs fraction are found, which contribute to elongation [69].

4. Conclusions

A novel process, differential velocity sideways extrusion (DVSE), was utilised to form curved AZ31 profiles at elevated temperatures. Effects of extrusion temperature and strain rate on profile curvature, microstructure evolution and mechanical properties were studied, the correlation between microstructural parameters and mechanical properties were revealed. The following conclusions have been drawn:

- (1) DVSE enables the manufacture of curved AZ31 profiles with grain refinement in a single extrusion-bending operation, the average value of grain size decreases from $\sim 25 \mu\text{m}$ of the billet to $\sim 4\text{--}7 \mu\text{m}$ of the curved profile, depending on extrusion temperature and velocity. Profile curvature is strongly dependent on extrusion velocity ratio of the bottom ram v_2 to the top ram v_1 , although it is also slightly affected by the extrusion temperature (250, 300, 350 °C) and velocity ($v_1 = 0.1, 1 \text{ mm/s}$) for a given extrusion velocity ratio.
- (2) Grain refinement in DVSE is attributed to the dynamic recrystallisation (DRX), which initiates in a necklace-like manner at the prior boundaries of coarse grains in the channel intersection region. This is a result of the severe plastic deformation (SPD) occurring in DVSE similarly to channel angular extrusion. Complete DRX occurs after DVSE ($v_2/v_1 = 1/2$) at 300 °C, $v_1 = 0.1 \text{ mm/s}$, where homogeneous equiaxed grains ($\sim 4.5 \mu\text{m}$) are obtained. An increase in temperature (350 °C, $v_1 = 0.1 \text{ mm/s}$) causes grain growth ($\sim 5 \mu\text{m}$). Relatively low temperature (250 °C, $v_1 = 0.1 \text{ mm/s}$) and

high strain rate domain (300 °C, $v_1 = 1 \text{ mm/s}$) result in incomplete DRX, which leads to inhomogeneous bimodal necklace pattern grains ranging $\sim 1\text{--}25 \mu\text{m}$ of the former and $\sim 2\text{--}20 \mu\text{m}$ of the latter.

- (3) Micro-hardness and ultimate tensile strength of the profiles have been enhanced compared with the initial billet, mainly due to grain refinement. Nevertheless, a slight decrease in the yield strength is obtained except for the complete DRXed case (300 °C, $v_1 = 0.1 \text{ mm/s}$) where the yield strength is slightly improved. The yield strength variation is a result of the strengthening effect caused by grain refinement and softening effect caused by texture modification. Weaker basal textures are generated in the majority of grains of the profiles extruded by DVSE and the $\{0002\}$ basal plane is deviated from the extrusion direction (ED) by $\sim 5\text{--}10^\circ$. This significant texture modification undermines the yield strength of the profiles in the ED (tensile direction) whereas improves the uniform elongation.

Overall, the present work shows that it is feasible to manufacture curved magnesium profiles directly from billets by DVSE. Although only lab-scale magnesium profiles with a limited length have been produced due to the limitations of current extrusion tooling, the possibility to produce large-scale profiles by DVSE exists for commercial applications, which will be explored in the future with upscaled extrusion tooling and integration of extrusion, cooling and finishing operations in the production line.

Declaration of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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