
Experimental study and modelling of stress relaxation ageing behaviour and post-form mechanical properties in creep age forming of Al-Zn-Mg alloy

Yong Li^{1,*}, Fenggong Lyu², Zhusheng Shi³, Yuansong Zeng², Xia Huang² and Jianguo Lin³

¹School of Mechanical Engineering and Automation, Beihang University, Beijing 100191, China

²AVIC Manufacturing Technology Institute, Beijing 100024, China

³Department of Mechanical Engineering, Imperial College London, London SW7 2AZ, UK

*Corresponding author: liyong19@buaa.edu.cn (Y. Li)

Abstract

The stress relaxation ageing behaviour and post-form mechanical properties of an Al-Zn-Mg alloy, AA7B04, have been experimentally investigated and modelled. Stress relaxation ageing tests were carried out under different initial stress levels and durations at 165 °C and subsequent tensile tests were performed at room temperature. The detailed effects of stress and time on stress relaxation and main post-form mechanical properties (yield strength, ultimate tensile strength and uniform elongation) have been analysed and discussed. Based on the results and analysis, a set of unified constitutive equations has been developed for the first time to simultaneously predict stress relaxation ageing behaviour of the aluminium alloy during creep age forming (CAF) process and the main mechanical properties of the products after CAF. The model is comprised of three sub-models, including microstructure, stress relaxation ageing and post-form mechanical properties, and has successfully predicted corresponding behaviour. The developed model provides an effective tool to not only predict the forming process, but also support possible industrial applications for CAF products.

Keywords: Stress relaxation; Ageing; Post-form mechanical property; Al-Zn-Mg alloy; Constitutive model.

1. Introduction

Creep age forming (CAF) is an advanced sheet forming technology that combines creep/stress relaxation and artificial ageing processes to concurrently deform and strengthen heat-treatable aluminium alloys, making it an efficient and accurate method to manufacture large panels in the aerospace industry, such as wing skins [1, 2]. The integrated creep-ageing or stress relaxation ageing (SRA) behaviour of aluminium alloys is a key factor to determine both the final shape and post-form mechanical properties of creep age formed (CAFed) products. Hence, it is important to systematically understand and predict the relationship between creep/stress relaxation and key mechanical properties during and after CAF, so as to provide useful guidance for not only accurate forming but also successful applications of the CAFed products.

The creep-ageing or stress relaxation ageing behaviour, including both key microstructures and macro-properties, of various heat-treatable aluminium alloys, such as Al-Cu-Mg [3], Al-Zn-Mg [4] and

Al-Cu-Li [5], has been widely investigated in the last decades, based on which, many constitutive models have been proposed to predict the evolutions of precipitation, dislocation, creep/stress relaxation and yield strength during creep-ageing for CAF process simulation [3, 6]. Except for the yield strength, other post-form mechanical properties of the CAFed materials, such as tensile strength and elongation, which are also key factors concerned for industrial applications, however, have attracted less attention currently. Li et al. [7] and Chen et al. [8] have investigated the work hardening behaviour, tensile strength and uniform elongation properties of AA2050 and AA7050 material after CAF respectively, and the relationship between the microstructures and post-form mechanical properties have been discussed. However, corresponding modelling work is still unavailable.

Hence, this study tries to perform a systematic investigation of the SRA behaviour during CAF and key post-form mechanical properties after CAF in both experimental and modelling ways. SRA and tensile tests of an Al-Zn-Mg alloy, AA7B04, have been performed for detailed analysis and discussion, based on which, a unified constitutive model to simultaneously predict both stress relaxation and key post-form mechanical properties has been proposed and discussed.

2. Materials and experiments

The material investigated is AA7B04, with the main chemical composition of Al-5.97Zn-2.48Mg-1.51Cu (wt%). The material was prepared with a special initial temper, named as AA7B04-P, which had undergone solution heat treatment (SHT), water-quenching, pre-stretching and a single step artificial ageing to an under-aged state.

A set of SRA tests at 165 °C was performed under different initial stress levels (200, 250, 300 and 350 MPa) and with different durations (2, 4, 8 and 16 h) to get the stress relaxation behaviour of the material. In addition, tensile tests at room temperature were also carried out on specimens after SRA tests to investigate the evolutions of main post-form mechanical properties of the material during SRA. The dog-bone specimens were prepared for both SRA and tensile tests, with the key dimension of 8 mm in radius and 50 mm in gauge length. Both kinds of tests were carried out on an Instron 5584 machine with a furnace to provide the high temperature environment. In SRA tests, specimens were firstly heated to 165 °C, then loaded to the initial stress level and held for designated durations for stress relaxation behaviour characterisation. The quasi-static tensile tests were performed with a strain rate of 10^{-4} s⁻¹. Further details of the material and corresponding test conditions can be found in a previous study [4].

3. Experimental results and discussion

Fig. 1 shows the stress relaxation curves of AA7B04-P at 165 °C. A higher initial stress leads to a more prominent stress relaxation behaviour, due to the larger creep strains generated at higher initial stress levels [4]. The remained stress after 16 h tests is generally larger with a higher initial stress level from 200 to 300 MPa, however, the remained stress for the initial stress of 350 MPa is lower than that for 300 MPa. This is because 350 MPa already exceeds the 0.01% offset yield point of the material (325 MPa) and the phenomenon may be explained by the additional initial dislocations introduced when loaded to the plastic region [4, 9].

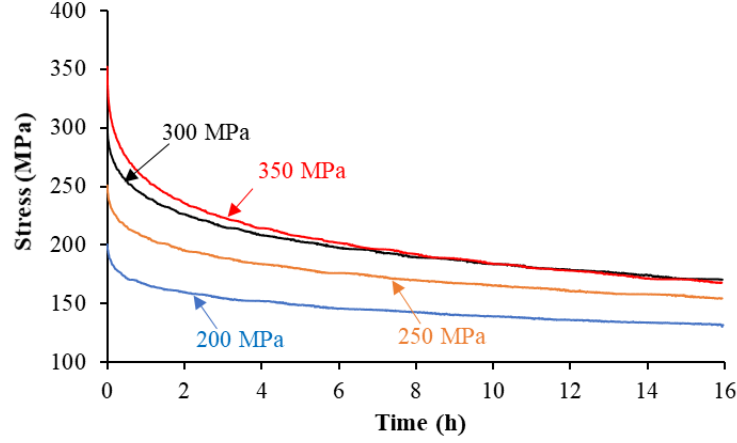


Fig. 1. Stress relaxation curves of AA7B04-P under different initial stress levels at 165 °C.

The room temperature flow stress curves of the material after SRA tests under different initial stress levels and with different durations have been plotted in Fig.2. All the flow stress curves of the SRAed materials show a fair level of work hardening behaviour, in which the highest level of work hardening exists in the as-received material. The flow stress level becomes lower with increasing SRA time under the initial stress of 200 MPa. In addition, Fig. 2 compares the flow stress of the material after 16 h SRA tests under 200 and 300 MPa, and the results show that with a higher initial stress, a lower flow stress is observed in the material after SRA test with the same duration.

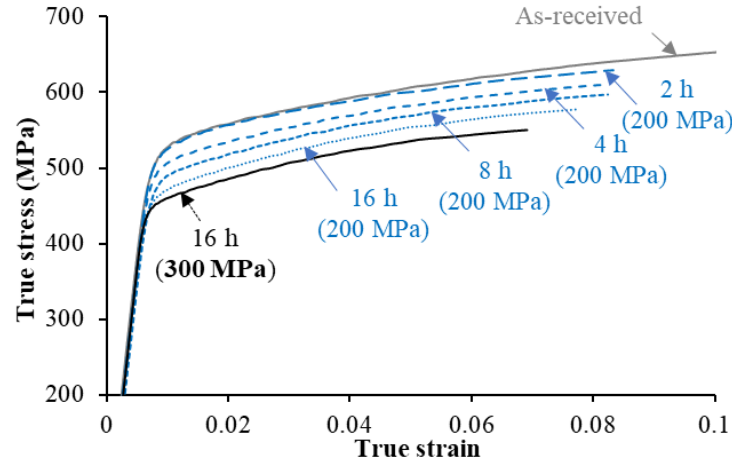


Fig. 2. The stress-strain curves of AA7B04-P after SRA tests at 165 °C for different time and under indicated initial stress levels.

Fig. 3(a) shows the detailed ultimate tensile strength (σ_{UTS}) and yield strength (σ_y) values of the materials SRAed under initial stresses of 200 and 300 MPa. Although the as-received material is in an under-aged condition [4], yield strength of the material decreases after 2 h SRA tests and experiences a continuously decreasing trend afterwards, which indicate that over-ageing has already started after 2 h. With a higher initial stress, a slightly lower yield strength is observed in the SRAed materials under the same testing time and temperature, as higher initial stresses lead to larger stress relaxation/creep, which can accelerate the coarsening of precipitates in the material [10]. Meanwhile, σ_{UTS} shows the same trend with σ_y results.

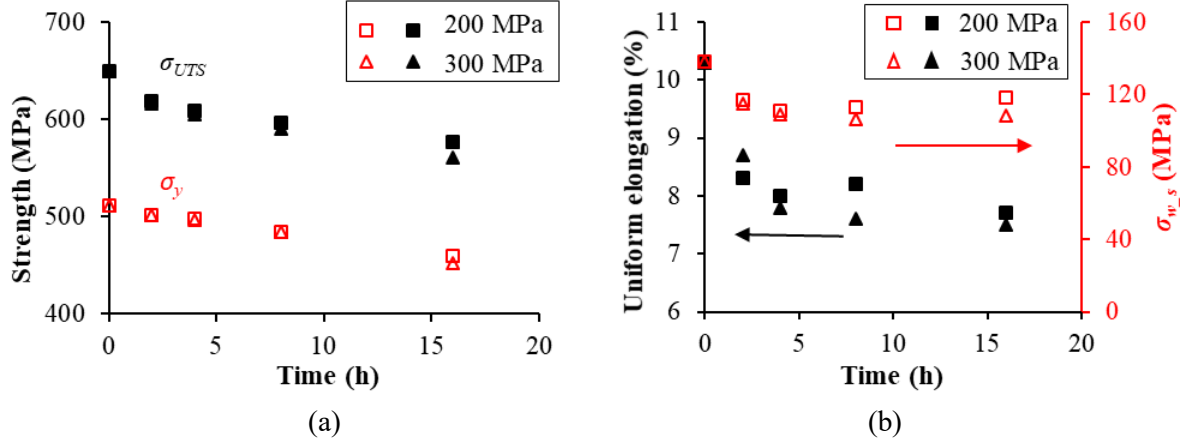


Fig. 3. Evolution of (a) ultimate tensile strength (σ_{UTS}) and yield strength (σ_y), and (b) uniform elongation and saturate work hardening stress ($\sigma_{w,s}$) versus SRA time for AA7B04-P at 165 °C.

The uniform elongation, as shown in Fig. 3(b), decreases significantly at the first 2 h, which could be related to the depleted solutes in the material from under-ageing to over-ageing within the first 2 h [11]. Fig. 3(b) also shows the saturate work hardening stress ($\sigma_{w,s} = \sigma_{UTS} - \sigma_y$) of the SRAed materials, which demonstrates a similar trend with the uniform elongation. It should be noted that a slight increase of $\sigma_{w,s}$ after 8 h SRA tests can be observed in Fig. 3 (b), which may be due to that the size of non-shearable precipitates grow to a high level after 8 h tests and the geometrical necessary dislocations accumulated around these fine precipitates become high enough to facilitate the work hardening of the material [12].

4. Modelling

A unified constitutive model that utilises and modifies both the conventional creep-ageing and work-hardening equations has been proposed in this study to simultaneously model and predict the stress relaxation behaviour during CAF and main post-form mechanical properties of the alloys after CAF. The developed model is divided into three sub-models, from microstructures to macro-properties, including microstructure sub-model, stress relaxation ageing sub-model and post-form mechanical properties sub-model.

4.1 Microstructure sub-model

The main microstructures related to the stress relaxation ageing behaviour of aluminium alloys include precipitates, solid solutes and dislocations. The effect of precipitates on SRA behaviour has been generally characterised by its size (r) and volume fraction (f), while the effects from solid solutes and dislocations are represented by solute concentration (c) and dislocation density (ρ) respectively. All the microstructural variables are modelled as normalised values in this study to eliminate the high difficulties in obtaining the actual microstructure values. The evolutions of these main microstructural variables during SRA can be modelled by the following equations [6, 13].

$$\dot{\bar{r}} = C_r(Q - \bar{r})^{m_1}(1 + \gamma_0\bar{\rho}^{m_2}) \quad (1)$$

$$\dot{\bar{c}} = -A_1(\bar{c} - \bar{c}_a)(1 + \gamma_0\bar{\rho}^{m_2}) \quad (2)$$

$$\dot{\bar{\rho}} = A_2(1 - \bar{\rho})|\dot{\bar{c}}|^{m_3} - C_p\bar{\rho}^{m_4} \quad (3)$$

$$\bar{f} = \frac{1-\bar{c}}{1-\bar{c}_a} \quad (4)$$

$\bar{r}$ in Eq. (1) is the normalised precipitate radius defined as $\bar{r} = r/r_c$, where r_c is the critical radius at peak-ageing state. $\bar{r}$ then evolves from 0 at SHT state to 1 at peak-ageing state, and $\bar{r} \geq 1$ indicates over-ageing states. Q represents the maximum size of precipitates during SRA of the alloys. $\bar{c}$ in Eq. (2) is the normalised solute concentration defined as $\bar{c} = c/c_s$, in which c_s is the solute concentration at SHT temperature. $\bar{c}_a$ is the normalised value at the test temperature. $\bar{\rho} = (\rho - \rho_0)/(\rho_s - \rho_0)$ is the normalised dislocation density, in which ρ_0 and ρ_s are dislocation density of the material before SRA tests and the its maximum level during SRA tests, and thus, $\bar{\rho}$ generally evolves from 0 to 1 during SRA tests. The accumulation and dynamic recovery effects of dislocations during SRA are considered in the first term on the right hand side of Eq. (3), while the second term represents the static recovery effect. In addition, the normalised volume fraction of precipitates $\bar{f} = f/f_s$, in which f_s is the final equilibrium value of f during ageing, can be directly modelled by corresponding solute concentrations according to Eq. (4). Other parameters in Eqs. (1) to (4) are material constants and will be determined in Section 5.1.

4.2 Stress relaxation ageing sub-model

The stress relaxation ageing sub-model contains two parts, one for the modelling of stress relaxation curves and the other for yield strength evolutions during SRA tests. It is generally believed that the stress relaxation during SRA comes from creep strains, and can be directly modelled according to the evolutions of creep strains. Creep behaviour is determined by related microstructural variables and can be modelled by the following equations [6]:

$$\dot{\epsilon}_c = A_4 \sinh[B_1(\sigma - \sigma_{th})(1 - H)] \text{sign}(\sigma) \quad (5)$$

$$H = k_1 \bar{\rho} + k_2 \bar{c} + k_3 \bar{r} \quad (6)$$

$$\dot{\sigma} = -E \dot{\epsilon}_c \quad (7)$$

$\dot{\epsilon}_c$ is the creep strain rate, which is modelled by the sinh function of applied stress σ , creep threshold stress σ_{th} and a variable H representing the combined effect from key microstructural variables in Eq. (6). E is the Young's modulus of the material, and the stress relaxation rate can be directly related to creep strain rate by Eq. (7). A_4 , B_1 , k_1 , k_2 and k_3 are material constants to be determined in Section 5.1.

While for yield strength (σ_y), it is comprised of three strengthening components from different microstructures, including dislocation hardening (σ_{dis}), precipitation hardening (σ_p) and solid solution hardening (σ_{ss}), which can be modelled as [14]:

$$\sigma_{dis} = A_3 \bar{\rho}^n \quad (8)$$

$$\sigma_p = \sigma_r \bar{f}^{m_5} \quad (9)$$

$$\dot{\sigma}_r = C_a \dot{\bar{r}}^{m_7} (1 - \bar{r}^{m_8}) \quad (10)$$

$$\sigma_{ss} = C_{SS} \bar{c}^{m_{10}} \quad (11)$$

$$\sigma_y = \sigma_{ss} + \sigma_a = \sigma_{ss} + \sqrt{\sigma_{dis}^2 + \sigma_p^2} \quad (12)$$

in which A_3 , n , m_5 , C_a , m_7 , m_8 , C_{SS} and m_{10} are material constants to be determined. A classic law of mixtures is utilised to combine the strengthening components for yield strength in Eq. (12), in which σ_a represents the combined effect from dislocations and precipitates.

4.3 Post-form mechanical properties sub-model

Work-hardening behaviour determines the post-form mechanical properties of ultimate tensile strength and uniform elongation. It has been reported that work hardening behaviour of aluminium alloys during plastic deformation is generally attributed to dislocation hardening: the statistically stored dislocations (ρ_{ss}) from interactions with weak obstacles (shearable precipitates) and solid solutes, and the geometrically necessary dislocations (ρ_{gn}) stored around strong obstacles (non-shearable precipitates) [15]. Both parts of dislocations would contribute to the work-hardening behaviour during SRA of AA7B04-P in this study according to the results in Section 3, and the following equations are utilised to model their evolutions and effects on work-hardening [14, 16]:

$$\dot{\bar{\rho}}_{ss} = k_4 * \bar{c}^{n_1} * (1 - \bar{\rho}_{ss}) \dot{\epsilon}_p^{n_2} \quad (13)$$

$$\bar{\rho}_{gn} = \frac{k_5}{\bar{r}^{n_3}} [1 - \exp(-\dot{\epsilon}_p^{n_4})] \quad (14)$$

$$\sigma_d = C_d \sqrt{\bar{\rho}_{ss} + \bar{\rho}_{gn}} \quad (15)$$

$$\sigma_f = \sigma_{ss} + \sqrt{\sigma_d^2 + \sigma_a^2} \quad (16)$$

$$\sigma_w = \sigma_f - \sigma_y \quad (17)$$

The normalised values of ρ_{ss} and ρ_{gn} are used for modelling in Eqs. (13) and (14), and their contributions to work-hardening has been represented by σ_d with the classic law of mixtures in Eqs. (15) and (16). The actual work-hardening stresses shown in the stress-strain curves in Fig. 2 then are represented by σ_w in Eq. (17). In addition, k_4 , n_1 , n_2 , k_5 , n_3 and n_4 are material constants.

5. Modelling results and discussion

5.1 Calibration of the model

The AA7B04-P material has been reported to achieve the peak-ageing state after artificial ageing at 165 °C for about 4 h, with an average precipitate radius of about 4.5 nm at as-received state and 5 nm after 165 °C/4 h artificial ageing [4], hence, the initial value of normalised precipitate radius $\bar{r}_0$ is determined as 0.9. A cell assumption was used to relate the volume fraction and radius of precipitate, as $\bar{f} = (r/r_c)^3$ [6], and the initial value of normalised volume fraction $\bar{f}_0$ was approximately 0.72. $\bar{c}_0$ was then determined by Eq. (4). The initial values of the strengthening components were approximately determined according to [17]. The initial values of all the variables are listed in Table 1.

Table 1. Initial and equilibrium values of variables in the constitutive model for SRA of AA7B04-P.

$\bar{r}_0$	$\bar{c}_0$	$\bar{\rho}_0$	$\bar{f}_0$	σ_{dis_0}	σ_{p_0}	σ_{ss_0}	σ_{th}	$\bar{c}_a$
(-)	(-)	(-)	(-)	(MPa)	(MPa)	(MPa)	(MPa)	(-)
0.9	0.356	0	0.72	0	386	120	57.8	0.105

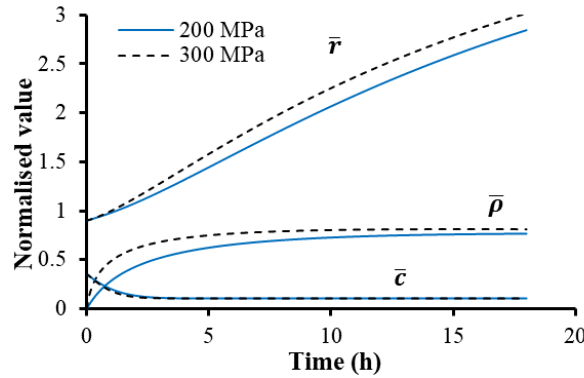
Based on the initial values determined in Table 1, a combination of manual adjusting and computational curve fitting method was utilised to calibrate all the material constants in the developed constitutive model according to the experimental data in Section 3, as detailed in [6, 14]. The determined material constants are listed in Table 2.

Table 2. Determined material constants in the constitutive model for SRA of AA7B04-P at 165 °C.

C_r (h^{-1})	Q (-)	γ_0 (-)	m_1 (-)	m_2 (-)	A_1 (-)	A_2 (-)	m_3 (-)	C_p (-)	m_4 (-)
0.0126	4.8	2.7	1.3	1.88	0.86	68	0.65	0.02	1.02
A_4 (h^{-1})	B_1 (MPa)	k_1 (-)	k_2 (-)	k_3 (-)	A_3 (MPa)	n (-)	m_5 (-)	C_a (MPa)	m_7 (-)
4.2E-5	0.025	0.9	0.3	0.04	128	0.5	0.5	30	0.5
m_8 (-)	C_{SS} (MPa)	m_{10} (-)	k_4 (-)	k_5 (-)	n_1 (-)	n_2 (-)	n_3 (-)	n_4 (-)	C_d (MPa)
0.8	239.7	0.67	1.2	4.6	0.07	0.7	0.08	0.65	282

5.2 Microstructural evolutions

Fig. 4 shows the evolution of normalised microstructural variables during SRA tests under 200 and 300 MPa from modelling results according to the material constants in Table 2. The normalised precipitate radius $\bar{r}$ increases continuously along the SRA time, and achieves to 1 (peak-ageing state) within 2 h, which shows the same trend with yield strength results obtained in Fig. 2, indicating the rationality of the modelling results [4]. The accelerated coarsening of precipitates at higher initial stress levels has also been successfully predicted in Fig. 4 [1]. Meanwhile, the normalised solute concentration $\bar{c}$ decreases to a stable level before 2 h, indicating an over-ageing behaviour after 2 h as well. The normalised dislocation density $\bar{\rho}$ increases significantly in the first couple of hours and gradually reaches a saturate level afterwards, which shows the same trend with many previous studies [1, 3]. In addition, more dislocations under higher initial stresses in SRA tests have also been well predicted by the developed model.

**Fig. 4.** Modelled evolution of normalised values of dislocation density ($\bar{\rho}$), precipitate radius ($\bar{r}$) and solid solution concentration ($\bar{c}$) in AA7B04-P during 16 h SRA tests at 165 °C under different initial stresses.

5.3 Stress relaxation behaviour

The stress relaxation curves under different initial stress levels from experiments and corresponding modelling results are compared in Fig. 5. A good correspondence between them has been achieved, especially for initial stress levels from 200 to 300 MPa, indicating the effectiveness of the model and

determined material constants in modelling the stress relaxation behaviour of the alloys in the elastic region. When the initial stress increases to 350 MPa, exceeded the 0.01% offset yield stress, the model predicts a larger remained stress compared with experimental results, the reason is that the different stress relaxation mechanisms in the plastic region than those in elastic region has not been considered in the model developed in this study [5].

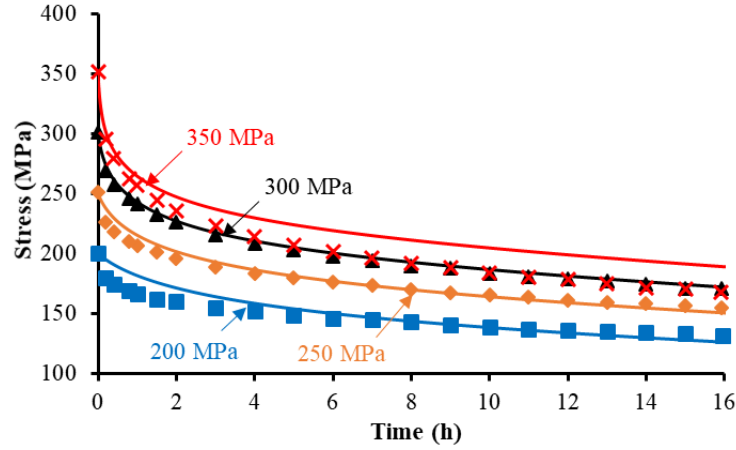


Fig. 5. Comparison of stress relaxation curves of AA7B04-P after 165 °C/16 h SRA tests under indicated initial stress levels from experiments (symbols) and modelling (solid lines).

5.4 Yield strength behaviour

Fig. 6 compares the experimental and modelling results of the evolutions of yield strength and strengthening components during SRA tests. A good agreement between experiments and models in yield strength has been achieved, indicating the effectiveness of the developed model. A slight increase of yield strength σ_y has been well predicted within the first 2 h of SRA tests, due to the combined effect from precipitation hardening σ_p , dislocation hardening σ_{dis} and solid solution hardening σ_{ss} , as shown in Fig. 6. In addition, the effect of higher initial stress levels in accelerating the softening of the over-aged material has also been successfully modelled in Fig. 6.

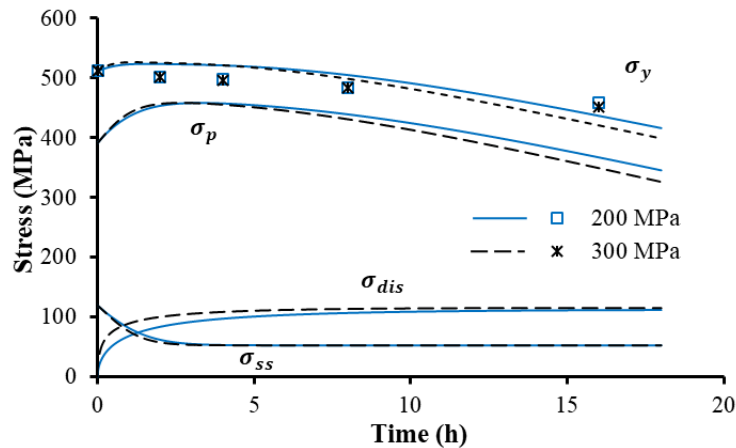


Fig. 6. Evolutions of yield strength σ_y and strengthening components from precipitates σ_p , dislocations σ_{dis} and solid solutes σ_{ss} of AA7B04-P during 16 h SRA tests at 165 °C from experiments (symbols) and modelling (solid lines).

5.5 Work hardening behaviour

The work hardening stress σ_w vs. plastic strain ϵ_p curves from experiments and modelling results are compared in Fig. 7. Based on the predicted microstructures in Fig. 4 at different time states of SRA tests, the predicted work hardening results all correspond well with experimental results for the SRAed alloy with different durations in Fig. 7. In addition, the increase of work hardening stress after 8 h SRA tests has also been well predicted in Fig. 7(b). All these results help to demonstrate the effectiveness of the developed model in predicting both microstructures and macro properties of the alloys during and after SRA tests.

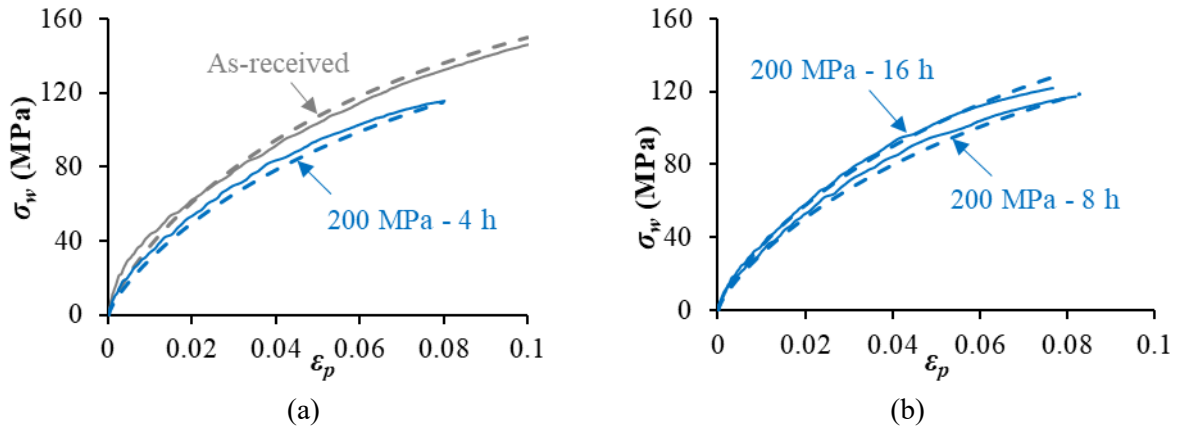


Fig. 7. Comparison of plastic stress vs. plastic strain curves of AA7B04-P after SRA tests at 165 °C under initial stress of 200 MPa with indicated time durations from experiments (solid lines) and simulations (dashed lines).

6. Conclusions

The stress relaxation ageing behaviour during CAF and post-form properties after CAF of AA7B04-P material at 165 °C have been experimentally investigated in this study. A higher initial stress leads to more apparent stress relaxation behaviour. Over-ageing occurs within the first 2 h of SRA tests, after which both lower yield strength and ultimate tensile strength can be observed with a higher initial stress. The uniform elongation and saturate work hardening stress show similar trends during SRA, decrease first and experience a minor increasing trend after 8 h, which may attribute to the accumulated geometrically necessary dislocations from the well-developed non-shearable precipitates.

A unified constitutive model, which is divided into three sub-models (microstructure, stress relaxation ageing and post-form mechanical properties sub-model), has been developed in this study. The model, for the first time, simultaneously model and predict the stress relaxation ageing and key post-form mechanical properties in both micro and macro sides during and after SRA tests, providing a solid and effective tool to predict the forming process and support potential industrial applications. It should be noted that the current model excellently predicts the SRA related behaviour in the elastic region, however, more work need be done for accurately predicting the SRA behaviour in both elastic and plastic regions.

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