

Experiments and modeling of double-peak precipitation hardening and strengthening mechanisms in Al–Zn–Mg alloy

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Abstract: The aim of the present work is to develop a model for simulating double-peak precipitation hardening kinetics in Al–Zn–Mg alloy with the simultaneous formation of different types of precipitates at elevated temperatures based on the modified Langer–Schwartz approach. The double aging peaks are present in the long time age-hardening curves of Al–Zn–Mg alloys. The physically-based model, while taking explicitly into account nucleation, growth, coarsening of the new phase precipitations and two strengthening mechanisms associated with particle-dislocation interaction (shearing and bypassing), was used for the analysis of precipitates evolution and precipitation hardening during aging of Al–Zn–Mg alloy. Model predictions were compared with the measurements of Al–Zn–Mg alloy. The systematic and quantitative results show that the predicted hardness profiles of double peaks via adding a shape dependent parameter in the growth equation for growth and coarsening generally agree well with the measured ones. Two strengthening mechanisms associated with particle-dislocation interaction (shearing and bypassing) were considered operating simultaneously in view of the particle size-distribution. The transition from shearing to bypassing strengthening mechanism was found to occur at rather early stage of the particle growth. The bypassing was found to be the prevailing strengthening mechanism in the investigated alloys.

Key words: Al–Zn–Mg alloy; double-peak precipitation; precipitation hardening; modelling; mechanical properties; strengthening mechanisms

1 Introduction

Aluminum alloys with high strength are technologically, economically and ecologically attractive. They offer the potential use of high energy-efficiency usage in engineering applications. Extensive experimental work has been carried out to develop 7000 series aluminum alloys with enhanced properties at elevated temperatures. Previous investigation has shown that the addition of Mn [1], Cr, Cu and Zr [2,3] to ingot metallurgy processed Al 7075 significantly improves the weldability and the tensile strength without sacrificing the tensile ductility. It is indicated that although the 7000 series Al alloys with T6 treatment are of high strength, their localized corrosion resistance is poor. To increase their corrosion resistance, over-aging treatments such as T73, T76, T74 and retrogression and re-aging (RRA) have been developed. The hardness and strength

properties of Al–Si–Cu–Mg and 7075 alloys have double-peak characteristics [4,5].

The development of microstructural models for complex industrial alloys has become a major activity over the past twenty years. An area of particular focus has been devoted to model the precipitation kinetics and the relationship between the precipitation microstructure and the resulting mechanical properties of many industrially important alloys. Various models for precipitation kinetics, which are based on thermodynamics, kinetics and dislocation mechanics, were developed and calibrated for wrought aluminum alloys in recent years [6–13]. A model for the yield strength of multi-component alloys was applied to overaged Al–Zn–Mg alloys (7xxx series). The model is based on an approximation of the strengthening due to precipitate by-passing during precipitate coarsening and takes account of ternary and higher order systems [13].

Theoretical description of the precipitation and

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hardening processes in these alloys may assist in finding their optimal compositions and heat treatments for achieving the required mechanical properties. To the best of our knowledge, few reports about the model of double-peak aging time of 7000 series aluminum alloys have been investigated. In this work, we address optimization of both different types of precipitates and mechanical properties through developing and calibrating a model for precipitation hardening of the high-strength Al–Zn–Mg alloys. The predictions of the model are performed in order to improve the physical insight into the range of material behavior observed in the experiments.

2 Model development

The model used in the present work is based on the classical theory proposed by LANGER and SCHARTZ [14] and WAGNER [15] (LSW), which was then modified by Kampmann and Wagner numerical model (KWN) [16]. This model takes into account three mechanisms involved in the solid-state decomposition of a supersaturated solid solution taking place during aging: nucleation, growth and coarsening of precipitates [17,18].

2.1 Nucleation, growth and coarsening

Recently, this KWN model has been extended to predict the precipitation of metastable $L1_2$ Al_3Zr dispersoids in 7xxx aluminium alloys containing zirconium [17]. It should be noted that in this analytical model droplets of a new phase are considered spheres, and neither elastic energy nor anisotropy of interfacial energy effect is taken into account. Precipitates in the Al-matrix are often of rod/needle-like shape, as in the case of η . For such a case, some corrections should be introduced in the model, in particular for the law of steady state growth of precipitates and in the Gibbs–Thomson equation.

The rate of homogeneous nucleation of precipitates is treated within the framework of classical Becker–Döring theory [19]. In the case of multi-component alloys, different types of precipitates with different kinetic and thermodynamic parameters may develop simultaneously. Many attempts have been made to relax the conditions under which the LSW expression was rigorously derived for applications to real materials. In our simplified analysis for the present case of Al–Zn–Mg alloys, two types of binary precipitates, η and η' , are assumed to form independently.

Using dimensionless parameters, a system of four differential equations are derived.

$$\rho_i = \bar{R}_i / R_{Ni} \quad (i=1,2) \quad (1)$$

where $R_{Ni} = 2V_{mi} \gamma_0 / (X_{\beta i} RT)$ and γ_0 are the scaling length and interphase surface energy, respectively; $X_{\beta i}$ is the concentration in the precipitation; R is gas constant; T is the thermodynamic temperature; V_{mi} is the molar volume of the new phase i .

2.2 Modelling of hardness

Precipitate particles can impede the motion of dislocations through a variety of interaction mechanisms [20]. At the early stage during aging, dislocations cut through the precipitates due to the small size and weak of the secondary phase particles, and the mechanism changes to bypass the precipitates when the size of the precipitates reaches the critical value with the aging time [21]. When the precipitates are sheared, the dislocation encounters the obstacle, and it will bow out under the influence of the applied stress into an arc of constant radius. The dislocation will bow out until the obstacle can no longer sustain the force acting upon it, whereupon the dislocation will break free and move until it encounters a new obstacle [20]. The stress at which the obstacles are overcome is proportional to their average size and inversely proportional to the effective distance between particles along a dislocation line, λ :

$$\sigma_{sh} = \Gamma_1 \frac{\bar{R}_p}{\lambda} \quad (2)$$

where Γ_1 depends on the nature of the interaction between precipitates and dislocations, as well as on the type of dislocation, $\Gamma_1 = 2\gamma_a/b$, γ_a represents the force per unit length opposing the motion of dislocation as it penetrates the particle, b is the Burgers vector, and $\bar{R}_p$ is the mean radius of precipitate. In the following consideration Γ_1 was assumed as a parameter averaged over all types of dislocations interacted with precipitates. Taking into account a relationship between λ and the square lattice spacing, λ_s , between obstacles in the glide plane [20]: $\lambda = \lambda_s / \beta_c^{1/2}$, where $\beta_c = \Gamma_1 b \bar{R}_p / 2\Gamma$, one can obtain

$$\sigma_{sh} = \bar{\Gamma} \frac{\bar{R}_p^{3/2}}{\lambda_s} \quad (3)$$

where $\bar{\Gamma} = \Gamma_1^{3/2} (b/2\Gamma)^{1/2}$, Γ is the dislocation line tension. When the precipitates increase in size, and the distance between precipitates also increases, it becomes easier for the dislocation to bypass them. In this case the strengthening depends only on the distance between precipitates in the glide plane and on the dislocation line tension, thereby producing the simple result [20,22].

$$\sigma_{bypass} = 0.81 \frac{2\Gamma}{b\lambda_s} \quad (4)$$

This prevails for precipitates having a radius higher than a critical value, $\bar{R}_c$ given by the equality of two

stresses $\sigma_{\text{shear}} = \sigma_{\text{bypass}}$, thus

$$\bar{R}_c = 1.74 \frac{\Gamma}{b\Gamma_1} = 0.87 \frac{\Gamma}{\gamma_a} \quad (5)$$

Two strengthening mechanisms associated with particle-dislocation interaction (shearing and bypassing) should be considered operating simultaneously due to particle size distribution, $f(\bar{R}_p)$. The contribution of precipitates sheared by dislocations to hardness can be written as follows [8]:

$$\sigma_i^{\text{sh}} = \frac{\bar{\Gamma}_i}{\lambda_{si}} \cdot \int_0^{\bar{R}_{ci}} R_p^{3/2} \cdot f_i(R_p) dR_p \quad (i=1, 2) \quad (6)$$

and the contribution of by-passed precipitates as

$$\sigma_i^{\text{bypass}} = \frac{1.64\Gamma \cdot \bar{R}_{ci}}{b\lambda_{si}} \cdot \int_{R_{ci}}^{\infty} f(R_p) dR_p \quad (i=1, 2) \quad (7)$$

In the case of two kinds of strengthening mechanisms resulting from two precipitates phases the total contribution of four sets of discrete obstacles to precipitation hardening can be evaluated according to Pythagorean addition rule mentioned in Ref. [20]. In the present work the double peaks precipitation hardening can be estimated as

$$\Delta\sigma_{\text{prec}} = \sqrt{(\sigma_1^{\text{sh}})^2 + (\sigma_2^{\text{sh}})^2 + (\sigma_1^{\text{bypass}})^2 + (\sigma_2^{\text{bypass}})^2} \quad (8)$$

For the size distribution, $f(\bar{R}_p)$, as suggested by DESCHAMPS and BRECHET [8], the Gaussian law was used.

$$f(R_p) = \frac{2}{\Delta\sqrt{\pi}} \cdot \frac{1}{1 + \text{erf}(\bar{R}_p/\Delta)} \cdot \exp\left(-\frac{(R_p - \bar{R}_p)^2}{\Delta^2}\right) \quad (9)$$

where R_p is the most probable radius and Δ is the standard deviation of the distribution that will serve as the fitting parameters of the hardening model. For particle spacing, λ_s , the expression is obtained on the basis of computer simulations [23]:

$$\lambda_s = 1.15 \sqrt{\frac{2\pi}{3f_v}} \cdot \bar{R} \quad (10)$$

where f_v is the particle volume fraction, and $\bar{R}$ is the overall average radius of precipitates.

The strengthening of the alloy is usually considered the summation resulting from different contributions [23]:

$$\sigma_{\text{tot}} = \sigma_0 + \sigma_{\text{ss}} + \Delta\sigma_{\text{prec}} \quad (11)$$

where σ_0 is the friction stress of the matrix and is considered constant, while σ_{ss} is the solid solution contribution. If we assume that the contribution from each element is additive, the solution hardening potential can be calculated as follows [24]:

$$\Delta\sigma_{\text{ss}} = \sum k_i X_i^{2/3} \quad (12)$$

where X_i is the concentration of a specific alloying element in the matrix and k_i is the corresponding scaling factor. The contribution of the solid solution strengthening can be evaluated using the difference in hardness between the alloy and pure aluminum. Note that the hardness of the alloy is proportional to the total stress H_{tot} [25]. In order to clearly show the variation of hardness with any parameter of dimensionless or aging time, a referred hardness, H_0 , is chosen as the standard and this variation can be simply reflected as the variation of the normalized hardness, R_H , which is the ratio between the hardness with the parameter varying and the referred hardness as follows:

$$R_H = \frac{H_{\text{tot}} - H_0}{H_0} = \frac{\Delta\sigma_{\text{prec}} - k_i(X_{0i}^{2/3} - X_i^{2/3})}{\sigma_0 + k_i X_{0i}^{2/3}} \quad (13)$$

3 Experimental

The material tested was 7475 aluminum alloy rods with a diameter of 20 mm. The composition (mass fraction, %) of the prepared alloys is Zn 5.9 %, Mg 2.5 %, Cu 1.7 %, Ti 0.02 %, Mn 0.03 %, Cr 0.24%, Fe 0.05%, Si 0.04 % and the balance is aluminum. After continuous casting, the rectangular ingot was homogenized at 500 °C for 6 h followed by air cooling to room temperature. The specimens were solution treated at 470 °C for 1 h, quenched in water at room temperature and then immediately aged at 120 °C for 180 h.

Hardness specimens were lightly polished with SiC paper and aluminum powder polishing cloths. Mechanical property measurements were performed at room temperature on HR-150 and AG-10TA type testing machine. The reported experimental data in the present work are the mean values of three specimens which are in the same tempered condition. For each sample, at least 20 points were measured to obtain an average value with a typical uncertainty of $\pm 2\%$. All the specimens were mechanically polished prior to pitting exposure. The morphologies of specimens were investigated by scanning electron microscopy using a Philips XL30.

4 Results and discussion

The microstructure of Al-Zn-Mg alloy after aging is shown in Fig. 1. The variation of the hardness measurement (Fig. 2) indicates that the hardness curves of alloy after aging exhibit a double-peak variation and the minimum hardness appears at approximately 90 h. Precipitation hardening gives the main contribution to the hardness. It is found that this variation of calculations is similar to that of experimental hardness results, which validates the applicability and feasibility of this model

for double-aged aluminum alloys containing η' and η precipitates. The parameters used in the simulation are listed in Table 1. The simulation parameters $k_{\gamma 1}$ and $k_{\gamma 2}$ were chosen by fitting the shapes of the calculated relative hardness peaks, with experimental hardness maxima corresponding to the evolution of given precipitates [26–28]; the location of peaks on the dimensionless time axis is determined by parameters c_{w1} , c_{w2} and D_2/D_1 . Additional temperature dependence of results is connected with variations in the limit solubility, hence in the initial supersaturation ξ_{0i} . Mole fractions of experimental alloy, $X_{0,Zn}=1.82\%$ and $X_{0,Mg}=1.31\%$, and the limit solubilities of Zn and Mg in Al taken from the Al–Zn and Al–Mg phase diagrams were used to obtain the initial supersaturations, $\xi_{01}=X_{0,Zn}/X_{e,Zn}$ and $\xi_{02}=X_{0,Mg}/X_{e,Mg}$. The chosen values $c_{w1}=c_{w2}=1.5$ correspond to incubation times $\tau_i \approx 2\tau_{i,min}$ needed to first particles of η and η' to become observable.

Critical values, $\rho_{Ci} = \bar{R}_{Ci} / R_{Ni}$, determining the transition from shearing to bypassing strengthening mechanism were chosen as 0.17 and 0.21 for $i=1$ and $i=2$, respectively, which gives the best fitting with the initial growth of the hardness peaks ($\sigma \sim f^{1/2} \bar{R}_p^{1/2}$) followed by a behavior that corresponds with the

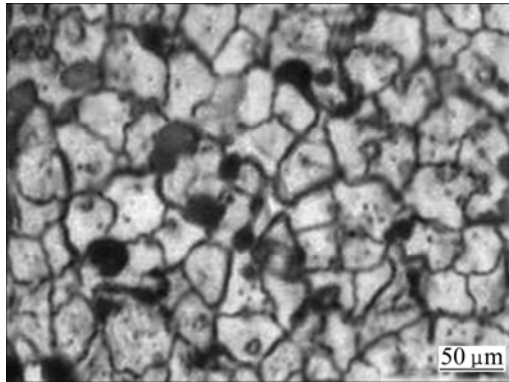


Fig. 1 Microstructure of Al–Zn–Mg Alloy after aging

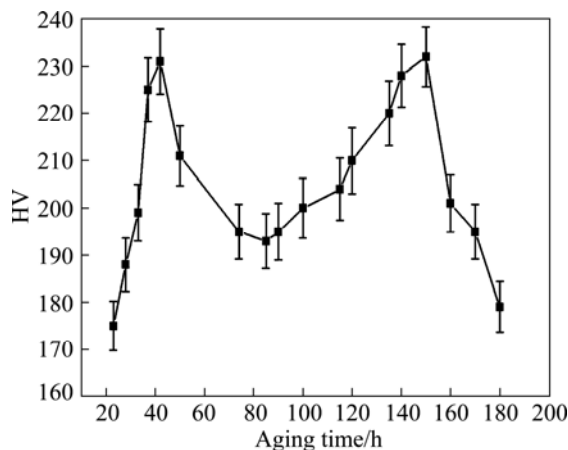


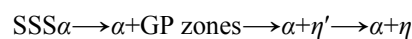
Fig. 2 Measured hardness of Al–Zn–Mg Alloy during age hardening according to present work

Table 1 Parameters of double-peak hardness model

Parameter	Significance	Value	Origin
$X_{e,Zn}/\%$	Mole fraction of Zn in Al matrix	1.3	Ref. [32]
$X_{e,Mg}/\%$	Mole fraction of Mg in Al matrix	1.2	Ref. [32]
ξ_{01}	Initial supersaturation of η	1.47	Ref. [32]
ξ_{02}	Initial supersaturation of η'	8.7	Ref. [32]
c_{w1}	Dimensionless parameter	1.5	This work
c_{w2}	Dimensionless parameter	1.5	This work
$k_{\gamma 1}$	Fitted parameter	0.08	This work
$k_{\gamma 2}$	Fitted parameter	0.50	This work
R_{N1}/nm	Fitted parameter	14±3	This work
R_{N2}/nm	Fitted parameter	60±14	This work
R_1^*/nm	Scaling length of η	2.4±0.7	This work
R_2^*/nm	Scaling length of η'	13.2±3.6	This work
$D_1/(10^{-14}m^2 \cdot s^{-1})$	Diffusion coefficient of η	7.4±3.8	Refs. [32,33]
$D_2/(10^{-14}m^2 \cdot s^{-1})$	Diffusion coefficient of η'	0.5±0.2	Refs. [32,33]
$(t/\tau)/h$	Scaling ratio	0.8±0.1	This work
t_{w1}/h	Incubation period of η	2.4±0.3	Ref. [8]
t_{w2}/h	Incubation period of η'	14.3±1.7	Ref. [8]
$\gamma_1/(mJ \cdot m^{-2})$	Surface energy of η	60±17	Refs. [32,33]
$\gamma_2/(mJ \cdot m^{-2})$	Surface energy of η'	400±120	Ref. [32]

bypassing mechanism ($\sigma \sim f^{1/2} / \bar{R}_p \sim 1/\lambda$). So, the latter mechanism prevails during most of the growth period of precipitates. After solution treatment followed by quenching to room temperature, there will be a supersaturation of vacancies and alloying elements in this alloy. Two types of zones or clusters are formed at RT, i.e., GP(I) zones and ‘vacancy rich clusters’ called VRC. The VRC are thought to be formed right after or during the quenching to RT and to be quite stable at this temperature. They are assumed to constitute the main formation route to η' with GP(II) zones as an intermediate phase [26,27]. At higher aging temperatures, above the GP zone solvus, GP(II) transforms into η' while GP(I) either dissolves or transform into η' if it reaches critical size. LI et al [29] found the similar results in Al–Si–Cu–Mg alloy and concluded that the first aging peak is the result from the high-density GP zone, while the second one is from metastable phases.

It is the precipitation strengthening which provides by far the strongest contribution to the strength of aged Al–Zn–Mg alloys. The main precipitation sequence which dominates hardening in most commercially used 7xxx alloys is [8]



where SSS α represents supersaturated solid solution; GP

zones are Guinier Preston zones; η is a quaternary phase containing Al, Zn and Mg, which can be considered to be based on a solid solution of MgZn_2 [8]. For 7000 series alloys, it is common to apply two step artificial aging treatments to achieving high strength while maintaining good stress corrosion cracking resistance. The compositions of the binary phases η' and η are dependent on heat treatment and alloy composition. The calculated density and average size of precipitates correspond to experimental values according to Eq. (10). The increased dislocation density has two effects on the precipitation reaction. First, the nucleation rate of precipitates can be enhanced owing to the lower barrier to nucleation on dislocations and, second, the kinetics of the reaction can be affected by an increased effective diffusion coefficient owing to the presence of short circuit diffusion along dislocation lines. Using the available experimental data on volume fractions of η -precipitates, found by SAXS [26,27] (Figs. 3, 4) and comparing it with the calculated volume fraction $f_1 = n_1 \rho_1^3$, one can find the scaling ratio

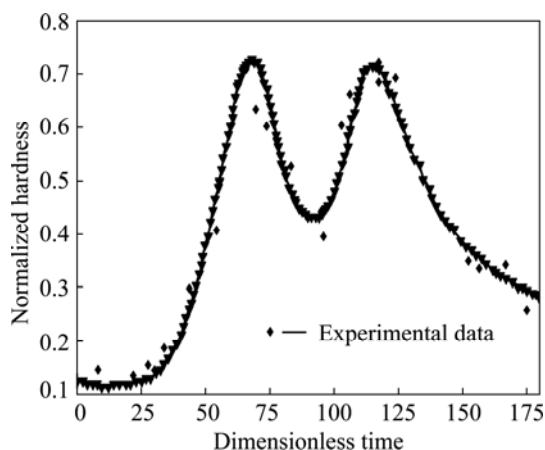


Fig. 3 Modeled dependence of precipitation hardening of Al-Zn-Mg alloys as function of normalized hardness and dimensional time with measured values

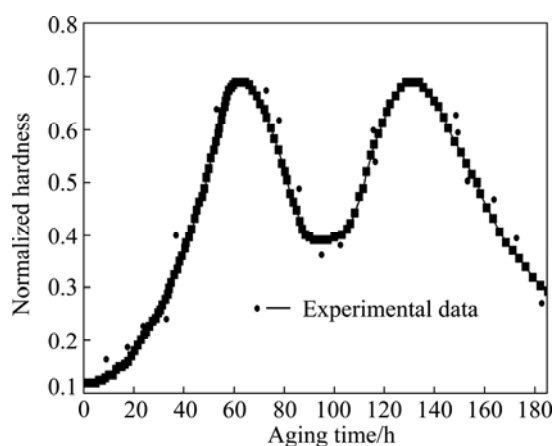


Fig. 4 Modeled dependence of precipitation hardening of Al-Zn-Mg alloys as function of normalized hardness and aging time with measured values

$(t/\tau) = R_{\text{NI}}^2/D_1$. The first aging peak is the result from the high-density GP zone (especially GP II zones). On the other hand, metastable phases formed at the intermediate stage of aging and kept semi-coherence with the matrix are effectively resistant to the movement of dislocation, thus have certain strengthening effect and may be the main reason for the formation of double aging peaks. Thus, a reasonable agreement between calculated and experimental precipitation hardening was found at 120 °C. In general, physically-based modelling approaches, such as the one presented in this work, can predict the composition and processing dependency of mechanical properties in complex aging hardened Al-Zn-Mg alloys.

The modelling of age hardening during industrial processing of 7000 series aluminium alloys is a challenge owing to the complicated aging history and special effects of processing of the alloys experience. Macroscopic properties of metallic materials strongly depend on their microstructures, i.e. on the shape and spatial arrangement of the different phases that compose the materials. It is thus important to understand the role of the different driving forces that may influence microstructural evolutions. The phase field method has emerged as the most powerful method for tackling microstructure evolutions at mesoscale [30]. The description of microstructures in terms of phase fields allows for complexities at a level close to that encountered in real materials. It would not be surprising to see in the near future a significant increase in the attempts of exploring various kinds of complex coherent phenomena with phase field method owing to these benefits. As discussed earlier, when using measured data for the precipitate diameter and thicknesses produced reasonably accurate results for hardness property, it would be beneficial to eventually be able to predict the precipitate morphology based on alloy composition and thermal history without experimental measurements. Although this seems like an ambitious goal, a recent advance [31,32] in multiscale modeling techniques provides the possibility that this goal may be realized soon. Phase-field modeling is a versatile tool for modeling microstructural evolution and has been used to model the evolution of solid-state transformations, such as precipitate nucleation, growth, and coarsening. The phase-field methodology is based on a free-energy driving force, consisting of bulk, interfacial, and strain energy contributions. Recent work [32] has demonstrated that it is possible to compute these energetic contributions using atomistic first-principles density functional calculations, hence creating a truly predictive tool for studying microstructural evolution. Multiscale modeling could also be used in the future to obtain a more realistic GP zone strengthening model via

calculated GP zone morphologies as a function of R_N , t_w , and T . Future work should be focused on modeling the temperature and time dependence of the diameter and thickness of η and η' phases, as well as the composition dependence of the precipitate growth kinetics. Advances in multiscale modeling may help in this regard.

5 Conclusions

1) A systematic and quantitative study of the precipitate evolution at the aging temperature in an Al–Zn–Mg alloy was performed. A model for the precipitation hardening process in 7000 series aluminum alloys was developed based on the modified LSW approach, which can handle the formation of different types of precipitates simultaneously via adding a shape dependent parameter in the growth equation for growth and coarsening.

2) Two strengthening mechanisms associated with particle-dislocation interaction (shearing and bypassing) were considered operating simultaneously in view of the particle size-distribution. A reasonable agreement between the calculations and observations was found. The transition from shearing to bypassing strengthening mechanism was found to occur at rather early stages of the particle growth. The bypassing was found to be the prevailing strengthening mechanism in the investigated alloys.

3) The normalized hardness was evaluated quantitatively in terms of precipitate size and volume fraction. The predictions of the model were performed in order to improve the physical insight into the range of material behavior observed in the experiments. Model predictions were compared with measurements of Al–Zn–Mg alloy. The predicted hardness profiles of double-peak generally agree well with measurements.

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Al–Zn–Mg 合金中的双峰时效析出硬化建模

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摘 要: Al–Zn–Mg 合金在长时间的时效中存在双峰硬化现象, 为了模拟析出硬化动力学, 基于改进的 Langer–Schwartz 方法发展了不同析出物形成的双峰硬化模型。该物理模型考虑了 Al–Zn–Mg 合金在时效过程中新析出物的形核、生长、粗化, 同时考虑了颗粒相互作用的 2 种机制(剪切和绕过)。将模型预测结果和实验结果进行了对比, 结果表明两者吻合很好。系统和定量模拟研究结果表明: 通过在生长方程中添加形函数, 硬度曲线与测量值吻合; 模型根据析出物的尺寸和体积分数定量评价该合金的力学性能; 强化机制包括剪切和绕过 2 种机制, 剪切机制向绕过机制的转变发生在颗粒生长的早期, 而绕过机制是主要强化机制。

关键词: Al–Zn–Mg 合金; 对峰时效; 析出硬化; 建模; 力学性能; 强化机制

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