



# Article Improving Elevated-Temperature Strength of an Al–Mn–Si Alloy by Strain-Induced Precipitation

# Yangyang Zhang, Wei Jin, Xuanzhang Hao, Feng Qiu 🕑 and Qinglong Zhao \* 🕑

Key Laboratory of Automobile Materials, Ministry of Education, and School of Materials Science and Engineering, Jilin University, No. 5988 Renmin Street, Changchun 130025, China; yangyangz16@mails.jlu.edu.cn (Y.Z.); jinwei1615@mails.jlu.edu.cn (W.J.); Haoxz1615@mails.jlu.edu.cn (X.H.); qiufeng@jlu.edu.cn (F.Q.)

\* Correspondence: zhaoqinglong@jlu.edu.cn; Tel.: +86-431-8509-4699

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**Abstract:** The coupled effect of strain-induced precipitation and stabilized substructure on the elevated-temperature strength of an Al–Mn–Si alloy and its thermal stability have been investigated. Prestrain significantly promotes the nucleation of nano-sized dispersoids, and strain-induced precipitation suppresses recrystallization, stabilizing substructure at elevated temperatures. Compared with the dispersoids formed during the heat treatment of as-cast alloy, substructure does not increase the coarsening rate of strain-induced precipitates. The strain-induced precipitation and stabilized substructure profoundly strengthen the aluminum alloy at the elevated temperature (300  $^{\circ}$ C).

Keywords: aluminum alloys; high temperature mechanical properties; strain-induced precipitation

# 1. Introduction

The current development of the aerospace and automotive industries demands cost-effective lightweight materials with high elevated-temperature strength [1,2]. Aluminum alloys are candidate structural materials for lightweight applications. Wrought aluminum alloys are usually strengthened by precipitation or work hardening. However, the precipitates in the conventional heat-treatable aluminum alloys are thermally unstable, and the coarsening of precipitates deteriorates the elevated-temperature strength. Recently, it is reported that the precipitation of thermally stable Mn-rich dispersoids during the long-term heat treatment could strengthen the Al-Mn alloy at elevated temperature, showing better thermal stability and creep resistance than typical 2xxx, 6xxx, and 7xxx wrought alloys [2]. Dispersion hardened Al–Mn alloys are suggested for potential applications at elevated temperatures. Extensive investigations are reported on the precipitation of dispersoids in Al–Mn alloys [3–8]. It is reported that Al6Mn is the dominate phase in binary Al–Mn alloys, and  $\alpha$ -AlMnSi becomes the dominate phase in Al–Mn–Si with a high content of Si [3,4]. The impurity Fe atoms partly substitute for Mn atoms in the dispersoids, forming Al<sub>6</sub>(Mn,Fe) or  $\alpha$ -Al(Mn,Fe)Si [9]. Liu and Chen optimized the content of Fe and Mn in Al–Mn–Mg for dispersion hardening [9,10]. The effect of Mg, Mo and Cu additions on the precipitation of Mn-rich dispersoids during the heat treatment of as-cast Al–Mn alloys has also been investigated [11–13]. A large quantity of uniformly distributed dispersoids are optimum for dispersion hardening. However, the dispersoids precipitated during the heat treatment of as-cast Al-Mn are heterogeneously distributed due to micro-segregation in the as-cast structure. It has been observed that the number density of dispersoids at the center of the dendrite arm was significantly lower than that in the periphery of the dendrite arm [6].

Plastic deformation could increase the yield strength and creep resistance of Al alloys at elevated temperatures [14,15]. However, the strain-induced substructure is thermally unstable,

and the occurrence of recrystallization softens the work hardened alloy. Our previous study shows that prestrain promotes concurrent precipitation during the hot deformation of the supersaturated Al-Mn-Mg alloy, which could retard dynamic softening [16]. The strengthening effect of prestrain and concurrent precipitation is not pronounced at 573 K due to the sluggish precipitation kinetics at the temperature lower than the typical precipitation temperature [16,17].  $\alpha$ -Al(Mn,Fe)Si dispersoids are partially coherent with the Al matrix [4]. Prestrain significantly facilitates the nucleation of  $\alpha$ -Al(Mn,Fe)Si dispersoids and accelerates the precipitation kinetics in the Al–Mn–Si alloy [17]. Dislocations and sub-boundaries facilitate solute diffusion, which might promote coarsening as well. It is unclear whether prestrain accelerates coarsening, which would reduce the thermal stability of dispersoids. The present work investigated the coupled effect of strain-induced precipitation and the stabilized substructure on the elevated-temperature strength and thermal stability of an Al–Mn–Si alloy. This may provide a simple strengthening principle for improving the elevated-temperature strength of dispersion hardened Al alloys.

#### 2. Materials and Methods

The chemical composition of the experimental alloy Al–Mn–Si was 1.2 Mn, 0.5 Si, 0.1 Fe, and 0.025 others (in wt.%), similar to the commercial AA3103 alloy. TiC nanoparticles (0.2 wt.%) were added into the melt as grain refiner. The alloy was casted into a steel mold with dimensions of 200 mm × 40 mm × 18 mm. The samples were rolled to the final thickness of 2.1 mm in multiple passes at room temperature, with the reduction of ~90%. The as-cast and rolled samples were heat treated at 673 K (400 °C) for various hours in a preheated air furnace, followed by air cooling. A slow heating rate affects the nucleation of dispersoids [17]. Thus, isothermal heat treatment was adopted to minimize the effect of the heating rate. The as-cast samples for the heat treatment and tensile tests were machined into the same thickness as the rolled samples to ensure the same heating rate. The as-cast sample heated for *x* h was labeled as Cast-xh, and the rolled samples heated for *x* h was labeled as Rolled-xh.

Microhardness tests were performed with a load of 200 g and dwell time of 10 s. The electrical resistivity was measured at room temperature using the eddy current method. The tensile tests were performed using an Instron 5869 material testing system (Instron, Norwood, MA, USA) at 573 K (300 °C) with a constant ramp rate (initial strain rate ~0.003 s<sup>-1</sup>). The tensile specimens were held at 300 °C for 10 min prior to testing. The tensile specimens were with a gauge length of 10.0 mm and a cross-section of  $4.0 \times 2.1 \text{ mm}^2$ .

The dispersoids were observed in a JEOL 2010F transmission electron microscope (TEM) (JEOL Ltd., Tokyo, Japan) operating at 200 kV. TEM foils were prepared using twin-jet electro-polishing in the 10% HClO<sub>4</sub> ethanol solution at 253 K and 20 V. The dispersoid size distribution was determined by ~300 measurements for each sample. The specimens for scanning electron microscopy (SEM) (Tescan, Brno, Czech Republic) were electro-polished using the similar parameters as the TEM specimens. The grain structure of specimens was characterized by electron backscatter diffraction technique (EBSD) with a step size of 0.2  $\mu$ m, and the results were analyzed using the Oxford HKL Channel5 software package (Version 5.11.20405.0, Oxford Instruments, High Wycombe, UK, 2012).

#### 3. Results

#### 3.1. Precipitation Kinetics

The  $\alpha$ -Al(Mn,Fe)Si dispersoids precipitate during heating at 400 °C. The depletion of solutes in solid solution results in the reduction of electrical resistivity ( $\rho$ ). The effect of solutes on electrical resistivity can be evaluated by a linear relationship as [3]:

$$\rho = 0.0267 + 0.032 \text{Fe}\% + 0.033 \text{Mn}\% + 0.0068 \text{Si}\%$$
(1)

Fe%, Mn%, and Si% are the solute concentrations in wt. %. Mn and Fe solutes significantly affect electrical resistivity, while the contribution of Si solutes is minor. The electrical resistivity of

the as-cast and as-rolled samples prior to heating is similar (Figure 1a), indicating that the influence of plastic deformation on electrical resistivity is minor compared to the influence of solutes. Thus, electrical resistivity could be used to indicate the precipitation. The electrical resistivity of the rolled samples decreases rapidly at the early stage of heating (<5 h), and becomes almost stable afterward (Figure 1a). The electrical resistivity of as-cast samples decreases slowly until heated for 24 h. The rapid decrease in the electrical resistivity of the rolled sample heated for short time suggests that plastic deformation promotes precipitation kinetics. Neglecting the minor contribution of Si on electrical resistivity, the volume fraction of dispersoids  $f_p$  can be estimated by [5]:

$$f_p = c\Delta(Mn,Fe)\%$$
, and  $\Delta(Mn,Fe)\% = (\rho_t - \rho_0)/0.033$ . (2)

 $\Delta$ (Mn,Fe) % is the change (wt.%) of Mn and Fe concentration in solid solution. The constant  $c \approx 2.5$  is given by ref. [5].  $\rho_t$  is the electrical resistivity as a function of heating time, and  $\rho_0$  is the electrical resistivity prior to heating. The precipitation kinetics is accelerated by prestrain, and a higher volume fraction of dispersoids were formed in the rolled samples. The saturation of volume fraction  $f_p$  implies the occurrence of coarsening instead of growth during the long-term heating at 400 °C.



**Figure 1.** (a) The electrical resistivity ( $\rho$ ) of the as-cast and rolled alloys after heating at 400 °C; (b) the volume fraction of dispersoids calculated according to Equation (1).

Figure 2a,b shows that the distribution of dispersoids in the as-cast samples is heterogeneous after heating. The string-like distribution is probably due to heterogeneous precipitation on dislocations, which was also observed in ref. [6]. The TEM and SEM images (Figures 2 and 3) show that the distribution of dispersoids in the rolled sample is homogeneous due to the high-density dislocations and sub-boundaries. The size distribution and mean diameter of dispersoids are demonstrated in Figure 2e,f. The size distribution is much narrower in the rolled samples than that in the as-cast samples, and the mean equivalent diameter in the rolled sample is still smaller than half of that in the as-cast sample after heating for 36 h. The number density per unit volume of dispersoids can be estimated from the volume fraction and mean diameter. The number density of dispersoids in the rolled sample after heating is at least one magnitude higher than that in as-cast sample.



**Figure 2.** The transmission electron microscope (TEM) bright field images of dispersoids precipitated during heating at 400  $^{\circ}$ C: as-cast sample heated for 16 h (**a**) and for 36 h (**b**); rolled sample heated for 12 h (**c**) and for 36 h (**d**), with the corresponding size distributions (**e**); and (**f**) the mean diameter and calculated number density per unit volume (ND) of dispersoids.

#### 3.2. Softening of the Deformed Alloy

Recovery and recrystallization might occur during thermal holding of the rolled alloy. Figure 4 indicates that the rolled alloy softens rapidly at the early stage of heating at 400 °C, while the decrease in hardness becomes much slower after 12 h. The EBSD mapping illustrates that recrystallization has occurred during heating for 12 h (Figure 5a). The partially recrystallized structure was retained after prolonged holding for 24 h (Figure 5b). Figure 5c shows that the recrystallization fraction increases from 19% to 24%, and implies that the growth of recrystallized grains consumed the recovered substructure mainly, while the deformation grains remained stable. The sluggish softening kinetics is attributed to Zener drag effect induced by the dispersoids. As shown in Figure 3, a large quantity of dispersoids are distributed on grain boundaries and sub-boundaries, retarding recovery and recrystallization.



**Figure 3.** Backscattered electron images of the prestrained alloy after thermal holding at 400 °C for 12 h (a) and 36 h (b), showing the homogeneous distribution of dispersoids and stabilized substructure.



Figure 4. The microhardness evolution of the as-cast and rolled alloys during heating at 400 °C.



**Figure 5.** The orientation imaging maps of the rolled alloy after heating for 12 h (**a**) and 36 h (**b**), showing the recrystallized (in blue), recovered (in yellow) and deformed (in red) grains, with the corresponding area fractions (**c**).

#### 3.3. Mechanical Strength at the Elevated Temperature

Figure 4 shows the hardness evolution of the as-cast samples during thermal holding, suggesting that the dispersion hardening reaches a maximum value at 16 h, and the hardness decreases after long-term thermal holding. The 0.2% proof stress  $\sigma_y$  at 300 °C of the sample Cast-16 h was 41 MPa, and it was almost unchanged when the thermal holding at 400 °C was prolonged for 20 h (Figure 6). The hardness of rolled samples was much higher than that of as-cast samples after heating at 400 °C, suggesting that substructure and strain-induced precipitation strengthen the alloy more significantly than dispersion hardening in the heat treated as-cast samples. The  $\sigma_y$  of the sample Roll-12 h decreased from 75 MPa to 71 MPa when the thermal holding was prolonged for 24 h. The yield strength of sample Roll-12 h is 34 MPa higher than that of sample Cast-16 h (peak hardened), and the strength increment is relatively stable during the prolonged thermal holding.



**Figure 6.** The engineering stress-strain curves at 300 °C of the as-cast and rolled alloys after thermal holding at 400 °C.

# 4. Discussion

# 4.1. Thermal Stability of Strain-Induced Precipitates and Substructure

The previous study [17] suggests that prestrain significantly promotes the nucleation kinetics of Mn-rich dispersoids rather than their growth due to the high density of heterogeneous nucleation

sites. However, the effect of prestrain on dispersoid coarsening was not studied. According to the Lifshitz-Slyozov-Wagner theory [18], the coarsening rate for precipitates is described as

$$r_m^{3}(t) - r_m^{3}(0) = Kt.$$
(3)

 $r_m(t)$  and  $r_m(0)$  are the mean radius of dispersoids at the time *t* and 0, respectively. Dislocations and sub-boundaries are rapid diffusion paths, and hence it is generally expected that prestrain accelerates coarsening rate. However, Figure 2f shows that the increase in the mean diameter of precipitates in the sample Rolled-12 h after thermal holding for 24 h was smaller than that for the sample Cast-16 h after thermal holding for 20 h at the same temperature, suggesting that the coarsening rate *K* for the rolled sample is lower. It is reported that the mean diameter of dispersoids in the as-cast Al–1.0Mn–0.5Fe–0.5Si increased from 40 nm to 50 nm when the thermal holding at 375 °C was prolonged from 12 h to 24 h [5]. The reported dispersoid diameter [5] is comparable to the results of samples Roll-12 h and Roll-36 h in the present work. According to Equation (3), the coarsening rate *K* for the rolled alloy heated at 400 °C is similar to that for the as-cast alloy heated at 375 °C. The diffusion coefficient of Mn in Al at 400 °C is 4.3 times as high as that at 375 °C according to ref. [19]. It implies that the dispersoids formed by strain-induced precipitation show good coarsening resistance compared to those dispersoids formed during the heat treatment of the as-cast alloy, which is attributed to the promoted nucleation in the prestrained alloy.

Dispersoids induce Zener drag stress on the grain boundary and sub-boundary migration, which can be calculated by:

$$P_z = 3f_p \gamma \eta / 2r_m \tag{4}$$

where  $\eta$  is a factor considering the effect of dispersoid distribution [20].  $\gamma$  is the boundary energy. Based on the measurements of dispersoids (Figures 1b and 2f), the  $P_z$  value is calculated to be  $0.103\gamma\eta$ for the sample Roll-12 h, and becomes  $0.072\gamma\eta$  for the sample Roll-36 h. The Zener drag stress decreased by 30% due to the coarsening of dispersoids during prolonged holding at 400 °C for 24 h, leading to the minor growth of recrystallized grains by 5%. The substructure was thermally stabilized by strain-induced precipitation.

#### 4.2. Strengthening Effect

The dispersoids act as obstacles to dislocation movement. Glide dislocations circumvent dispersoids by Orowan bypass mechanism at room temperature. The Orowan stress at room temperature due to dispersion hardening can be calculated by [21]:

$$\sigma_{oro} = 0.81 \frac{MAGb}{2\pi\lambda} \ln(\frac{\pi r}{2b}).$$
(5)

 $M \approx 3$  is the Taylor factor for aluminum, G = 27 MPa is the shear modulus of aluminum, and b = 0.286 nm is the Burgers vector.  $\lambda$  is the dispersoid spacing on a slip plane.  $A \approx 1.2$  is a constant related to the Poisson's ratio [22]. The calculated  $\sigma_{oro}$  for the sample Roll-12 h is three times as high as that for the sample Cast-16 h. When the tension testing temperature is elevated to a higher temperature (e.g., 300 °C), dislocation climb and cross slip are more activated, reducing the strengthening effect of dispersoids. The pinning effect of dispersoids at elevated temperatures is attributed to their attractive interaction with the dislocation [23]. The detachment stress can be estimated as a fraction of Orowan stress, i.e.,  $\sigma_d = \kappa \sigma_{oro}$  ( $\kappa < 1$ ) [23].  $\kappa = 0.3$ –0.7 was fitted in literature [24], which decreases with rising temperature. The coarsening of dispersoids reduces the strengthening effect. The calculated  $\sigma_{oro}$ decreases by 31 MPa for the sample Roll-12 h after thermal holding prolonged for 24 h at 400 °C, and decreases by 5 MPa for Cast-16 h after holding prolonged for 20 h. The substructure contributes to the elevated-temperature strength [15,16]. Generally, the stress contribution is a function of dislocation density and subgrain size, but the contribution is difficult to quantitatively estimate. The coarsening of dispersoids reduces dispersoid nade substructure stability. The coarsening rate depends on the slow diffusion of Mn solutes according to Equation (3). The diffusion coefficient of Mn in Al increases by  $7.3 \times 10^2$  times when the temperature increases from 300 °C to 400 °C [19]. According to Equation (3), the thermal holding for 24 h at 400 °C is approximately equivalent to  $1.8 \times 10^4$  h at 300 °C for the consideration of dispersoid coarsening. Thus, the strengthening principle of strain-induced precipitation stabilizing substructure can be potentially applicable at elevated temperatures such as 300 °C.

# 5. Conclusions

Prestrain profoundly promotes the nucleation of nano-sized dispersoids, leading to the pronounced depletion of solid solution. Compared to the dispersoids formed during the heat treatment of the as-cast alloy, strain-induced precipitation shows similar or even lower coarsening rate due to the increased number density of nucleation sites. The large quantity of dispersoids formed by strain-induced precipitation suppress recrystallization, stabilizing substructure at elevated temperatures. Strain-induced precipitation and stabilized substructure significantly strengthens the aluminum alloy at the elevated temperature (300  $^{\circ}$ C). This processing principle should be applicable for improving elevated-temperature strength of other dispersion hardened aluminum alloys.

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Conflicts of Interest: The authors declare no conflict of interest.

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