



Quantitative study of nanoscale precipitates in Al–Zn–Mg–Cu alloys with different chemical compositions



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ABSTRACT

The present work gives a quantitative study on the nanoscale precipitates in Al–Zn–Mg–Cu alloys with 6 different chemical compositions (all are in peak-aged state), by combining synchrotron-based small-angle X-ray scattering (SAXS) and transmission electron microscopy (TEM) techniques. Based on the TEM observations, the size, shape, interprecipitate distance, volume fraction and number density of precipitates are extracted from SAXS data through model fitting. The results show that after peak-aging treatment, the average precipitate sizes in different alloys are close to each other, whereas the volume fraction and number density of nanoscale precipitates increase with the increasing content of Zn+Mg+Cu as well as the Zn/Mg ratio. The results also show that the alloy with a higher number density/volume fraction of nano-scale precipitates tends to have a higher mechanical strength.

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1. Introduction

The Al–Zn–Mg–Cu alloys (7000 series) are extensively used in the aerospace and automotive industries, mainly due to their high specific mechanical strength. Over the years, to improve the mechanical strength of the alloys, scientists have been constantly adjusting the contents and ratio of the main alloying elements (Zn, Mg and Cu) [1,2]. And a lot of 7000 series aluminum alloys with different chemical compositions and performances have been developed. It is well known that the strengthening effect in Al–Zn–Mg–Cu alloys mainly comes from the nanoscale precipitates formed during the aging process [3–5]. Basically, the geometrical characteristics of the nanoscale precipitates (including the size, shape, interprecipitate distance, volume fraction, and number density) determine the mechanical strength of the alloy [6]. Thus, comparative study on the nanoscale precipitates in different Al–Zn–Mg–Cu alloys is very important for the understanding of alloy performance and the design of chemical compositions.

Over the years, different aging tempers have been developed for the 7000 series aluminum alloys, such as one-step T6, two-step T7X and three-step retrogression and reaging (RRA) treatments. Each aging temper provides the alloy with different performances

[7–9]. Normally, for alloys with different chemical compositions, the T7X and RRA tempers are different. However, the T6 peak-aging temper can be the same for different Al–Zn–Mg–Cu alloys (120 °C for 24 h). Thus, to compare the effect of chemical composition, Al–Zn–Mg–Cu alloys all in the peak-aged states will be chosen in the present study. This can eliminate the influence caused by different aging processes.

During aging, the usual precipitation sequence in Al–Zn–Mg–Cu alloys can be summarized as: supersaturated solid solution → Guinier–Preston zones → metastable η' → stable η phase [4]. In the past years, the crystal structures, shapes and sizes of the nanoscale precipitates have been well studied, mainly using the transmission electron microscopy (TEM) and tomographic atom probe (TAP) analyses [3,4,10–12]. The TEM and TAP methods offer direct observations of local structural characteristics for nano-scale precipitates. However, both the methods yield limited statistics when concerning the quantitative information on the volume fraction and number density of precipitates [13]. Small-angle X-ray scattering (SAXS), which studies nano-scale scatters in the reciprocal space, proves a complementary tool to the two techniques. By analyzing the nanoscale precipitates in the order of 10^{11} or above, SAXS approach can provide quantitative and statistically relevant data for characterization of the precipitates [6,14–16].

This work chooses Al–Zn–Mg–Cu alloys with 6 different chemical compositions (all in peak-aged state) as the objects, and gives a comparative and quantitative study on the nanoscale precipitates, by

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combining synchrotron-based SAXS and TEM techniques. The results will be important for the design of chemical compositions and the understanding of alloy performances.

2. Experiment

The specimens used in this study were cut from 6 hot-rolled Al–Zn–Mg–Cu alloy plates with different chemical compositions (shown in Table 1). All the specimens were first treated by solution sufficiently, followed by cold water quenching, and then T6 peak-aging treatments. The detailed solution treatment tempers for different alloys are listed in Table 2. The T6 peak-aging tempers for all 6 alloys are 120 °C for 24 h. The T6 peak-aged alloys were used for the following property and microstructure examinations.

The room temperature tensile properties of the 6 peak-aged alloys were measured by using a MTS-810 test machine at a constant crosshead speed of 1 mm/min. For each alloy, 3 tensile specimens were tested for an average value. The electrical conductivity of the alloys was measured using the 7501 eddy current meter (standard: GB/T12966-2008). For the measurements, specimens of 25 mm × 25 mm × 5 mm in size were mechanically polished with 2000 grit silicon carbide papers, and then 3 to 5 times of measurements were performed for each specimen to obtain an average electrical conductivity. The TEM examinations were conducted on a JEM-2010FX transmission electron microscope, operating at 200 kV. Three millimeter diameter disks for TEM studies were punched out directly from slices which were mechanically ground down to 50 μm thickness. These disks were electropolished using a twinjet machine with a 25% nitric acid solution in methanol at –30 °C and 15–25 V.

The SAXS experiments were carried out at the BL16B1 beam-line, Shanghai Synchrotron Radiation Facility (SSRF) of China. X-rays of 15 keV ($\lambda=0.8266$ Å) generated by a bending magnet were irradiated on the sample foils of about 100 μm in thickness. The scattered X-rays were collected by using a two-dimensional charge-coupled device (2-D CCD) detector at a sample-to-detector distance of ~2 m. This provided data in a scattering vector range of $0.02 \text{ Å}^{-1} < Q < 0.3 \text{ Å}^{-1}$. The measured SAXS data were corrected for the incident beam intensity, transmission factor, the thickness of the sample, background noise, and then converted to absolute units by measuring a glassy carbon standard sample [17]. All these data reductions and calibrations were performed using the Nika package within IGOR Pro software [18]. Such absolutely calibrated intensity data is essential for many important aspects of quantitative SAXS analysis, such as obtaining the volume fraction, number density, as well as size distribution of the nanoscale precipitates. The volume fraction of the aging-induced precipitates is calculated based on Eq. (1) using the integrated intensity Q_0

$$Q_0 = \int_0^\infty I(q)q^2 dq = 2\pi^2(\Delta\rho)^2 f_v(1 - f_v) \quad (1)$$

where f_v is the volume fraction and $\Delta\rho$ is the electron density contrast between the precipitates and matrix. In this work,

Table 1
Main chemical compositions for the 6 different Al–Zn–Mg–Cu alloys.

Alloy	Chemical compositions						
	Zn	Mg	Cu	Zr	Al	Zn+Mg+Cu	Zn/Mg
7B85	7.50	1.70	1.40	0.12	Bal	10.6	4.4
7050	5.93	2.23	2.16	–	Bal	10.3	2.7
7B04	6.23	2.88	1.58	0.12	Bal	10.7	2.2
7150	6.56	2.25	2.10	0.12	Bal	10.9	2.9
7055	8.30	2.05	2.16	0.12	Bal	12.5	4.0
7A93	10.80	2.90	1.80	0.12	Bal	15.5	3.7

Table 2

Solution and peak-aging tempers for the 6 Al–Zn–Mg–Cu alloys, and the tensile properties and electrical conductivities for the 6 peak-aged aluminum alloys.

Alloy	Solution temper	Peak-aging temper	Ultimate tensile strength (MPa)	Yield strength (MPa)	Electrical conductivity (MS/m)
7B85	475 °C/2 h	120 °C/24 h	578 ± 8	520 ± 10	19.4 ± 0.1
7050	460 °C/1 h + 474 °C/2 h		620 ± 15	540 ± 15	17.9 ± 0.1
7B04	470 °C/1.5 h		645 ± 0	577 ± 3	16.9 ± 0.0
7150	470 °C/1.5 h + 480 °C/1 h		660 ± 0	582 ± 3	17.4 ± 0.0
7055	470 °C/2 h		673 ± 3	595 ± 5	17.5 ± 0.1
7A93	450 °C/1.5 h + 475 °C/2 h		795 ± 3	770 ± 3	16.6 ± 0.0

quantitative structural information were obtained by model fitting of the SAXS data, with the help of Irena curve fitting package within IGOR Pro [19].

3. Results and discussions

3.1. Tensile strength and electrical conductivity

The room temperature tensile properties and electrical conductivities of the 6 peak-aged Al–Zn–Mg–Cu alloys are summarized in Table 2. The deviations are also shown in the table, which are the difference between the average value and the measured largest (or smallest) value. It can be seen that among the 6 different alloys, the 7B85 alloy has the lowest mechanical strength but highest electrical conductivity. On the contrary, the 7A93 alloy has the highest mechanical strength but lowest electrical conductivity. The tensile and yield strengths increase in a sequence of 7B85, 7050, 7B04, 7150, 7055 and 7A93. On the other hand, the electrical conductivity increases in a sequence of 7A93 (16.6 MS/m), 7B04 (16.9 MS/m), 7150 (17.4 MS/m), 7055 (17.5 MS/m), 7050 (17.9 MS/m) and 7B85 (19.4 MS/m).

3.2. TEM characterizations

Fig. 1(a)–(c) shows the selected area diffraction patterns (SADPs) in $\langle 100 \rangle_{\text{Al}}$, $\langle 112 \rangle_{\text{Al}}$ and $\langle 111 \rangle_{\text{Al}}$ zone axes for the peak-aged 7150 aluminum alloy. The main strong diffraction spots from the Al matrix have been indexed. The weak sharp diffraction spots at $\{100\}$ and $\{110\}$ positions in $\langle 100 \rangle_{\text{Al}}$ and $\langle 112 \rangle_{\text{Al}}$ projections came from spherical Al_3Zr dispersoids with a cubic unit cell structure. The spots at $1/3\{110\}$ and $2/3\{110\}$ positions in $\langle 112 \rangle_{\text{Al}}$ and $\langle 111 \rangle_{\text{Al}}$ projections came from the semicoherent η' precipitates, which are the major precipitates for the Al–Zn–Mg–Cu alloy under T6 temper [4]. Also, some weak diffuse GPII spots can be found near $1/3\{422\}$ positions in $\langle 112 \rangle_{\text{Al}}$ projection. Besides, very weak diffraction spots at $\{1, (2n+1)/4, 0\}$ positions can be found in $\langle 100 \rangle_{\text{Al}}$ projection, which came from the GPI zone.

Fig. 2(a)–(c) displays the bright-field (BF) TEM micrographs in $\langle 100 \rangle_{\text{Al}}$ (a), $\langle 112 \rangle_{\text{Al}}$ (b) and $\langle 110 \rangle_{\text{Al}}$ (c) zone axes for the peak-aged 7150 aluminum alloy [6]. Fig. 2(d) gives the high-resolution transmission electron microscopy (HRTEM) image for the alloy in the $\langle 110 \rangle_{\text{Al}}$ direction. It can be seen that the precipitates show different appearances in the three different zone axes. In $\langle 100 \rangle_{\text{Al}}$ zone axis, most of the precipitates are elliptical (close to spheres). In $\langle 112 \rangle_{\text{Al}}$ zone axis, both elliptical and elongated precipitates can be seen, and the long axis of the elongated ones are all along one certain direction.

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