Quantification of Volume Fraction of Precipitates in an Aged Al–1.0 mass % Mg₂Si Alloy

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An Al–1.0 mass % Mg₂Si alloy was aged at 473 K for 36 ks. Using the same sample, resistivity (ρ) measurements, tensile tests and TEM observations were carried out. The volume fraction (V_{fp}) of precipitates was estimated from the decrement in resistivity due to aging. Length (λ), diameter (d) and number density (N_p) per unit volume of the needle-shaped precipitates were measured on TEM micrographs. Perhaps because of initiation of overaging in the present aging condition, λ and N_p showed a wide scatter and differed significantly in two equivalent samples, independently heat treated and tested. However, proof stress and V_{fp} in the two runs well coincided with each other and the volume fraction estimated from TEM micrographs (V_{fTEM}) matched within a factor of 2 for the two runs.

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1. Introduction

It is well known that the peak age hardening of Al–Mg–Si alloys is provided by precipitates of the needle-shaped transition phase β'' .¹⁾ The strength of this alloy is determined by the volume fraction (V_f) and the size (length, λ , and cross-sectional diameter, d) of the precipitates.^{2,3)} Therefore it is our interest to estimate quantitatively the above parameters and to correlate them with the strength of these alloys.

Resistivity (ρ) measurement has been used as a powerful method to investigate the aging behavior of Al–Mg–Si alloys because of its high sensitivity to solute concentration,^{4–6)} although most of previous studies remained at a qualitative analysis. However, using recently re-investigated fundamental data,⁷⁾ the decrement in solute concentration or in other words, the total amount of solute consumed to form precipitates, can be estimated quantitatively with good accuracy and reproducibility. The detail will be mentioned later in 2.1. The measured ρ is the average value for the total volume of the specimen, e.g. about $2.55 \times 10^{-7} \text{ m}^3$ in the present work which is more than 10^{10} times larger than that per field of TEM observation. Using the densities of matrix and precipitates, the volume fraction of the precipitates (V_{fp}) can be estimated.

The size, shape and distribution of the precipitates are the parameters necessary to discuss the precipitation hardening. TEM micrographs give direct information about the above parameters of precipitates, though from a much smaller volume of the specimen, e.g. $10^{-20} \sim 10^{-18} \text{ m}^3$ per field of TEM observation in the present work.

In this research, quantification of the amount of precipitates in Al–1.0 mass % Mg₂Si alloy, slightly overaged, has been at-

tempted through ρ measurements and TEM observations.

2. Utilization of Resistivity

2.1 Interpretation of measured resistivity

Resistivity of multicomponent dilute solid solutions at temperature T ($\rho^{s.s.}(T)$) is represented by eq. (1):⁷⁾

$$\rho^{s.s.}(T) = \rho^{\text{PURE}}(T) + \sum \Delta\rho^i(T) \cdot C^i \quad (1)$$

where $\rho^{\text{PURE}}(T)$ is the resistivity of ideally pure solvent, $\Delta\rho^i(T)$ is the contribution to resistivity per unit concentration of “ i ”th solute or defect at temperature T and C^i is its concentration. Previously reported $\Delta\rho^i$ values scattered severely.⁸⁾ This fact made the resistivity unreliable for a quantitative study of aging phenomena. However, $\Delta\rho$ values of many binary solid solutions based on aluminium, magnesium or titanium, have been re-investigated by Komatsu *et al.* as reviewed elsewhere.⁷⁾ For several solutes in aluminium, Table 1 shows $\Delta\rho$ at 77 and 300 K with the ratio $\eta^i = \Delta\rho^i(300)/\Delta\rho^i(77)$ and the difference $\varepsilon^i = \Delta\rho^i(300) - \Delta\rho^i(77)$ as the parameters indicating the deviation from Matthiessen’s Rule (DMR).⁷⁾ Though the significant figure of chemical content of solutes is 3, using at least 5 alloys of different contents the scattering of chemical analysis was averaged. Therefore for convenience of readers, the $\Delta\rho$ values are given with 4 significant figures.

Using a wire or rod-shaped specimen, resistivity is obtained as a product of resistance and size factor, S/l , where l and S are the distance between two potential contacts and cross-sectional area of the specimen, respectively. In course of the re-investigation of $\Delta\rho$, the S was determined from the measured mass and density of the specimen.⁹⁾ This density-mass method gives smaller error in the measured S and size factor of the specimen, accordingly. Moreover, the following equation¹⁰⁾ deduced from Matthiessen’s Rule was utilised to detect the error in size factor:

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Table 1 Contributions of solute to resistivity ($\Delta\rho$) in aluminium at 77 and 300 K with $\eta = \Delta\rho(300)/\Delta\rho(77)$.

Solute	$\Delta\rho/\text{n}\Omega\cdot\text{m}\cdot\text{at}\%^{-1}$		η	$\varepsilon/\text{n}\Omega\cdot\text{m}\cdot\text{at}\%^{-1}$
	77 K	300 K		
Mg	5.275	5.506	1.044	0.231
Si	6.556	6.950	1.060	0.394
Cu	7.986	7.721	0.967	-0.265
Zn	2.442	2.401	0.983	-0.041
Fe	79.06	77.81	0.984	-1.25
Mn	73.94	63.33	0.856	-10.61
Zr	66.07	61.03	0.924	-5.04
Ti	55.63	56.19	1.010	0.56

$$\rho^{\text{s.s.}}(T_1) = (\rho^{\text{PURE}}(T_2) - \eta\rho^{\text{PURE}}(T_1))/(R - \eta) \quad (2)$$

where R is $\rho^{\text{s.s.}}(T_2)/\rho^{\text{s.s.}}(T_1)$ and $T_2 > T_1$. Usually 300 and 77 K are selected for T_2 and T_1 , respectively. According to an assessment and recommendation by Desai *et al.*,¹¹⁾ $\rho^{\text{PURE}}(300)$ and $\rho^{\text{PURE}}(77)$ of ideally pure aluminium are 27.33 and 2.200 n $\Omega\cdot\text{m}$, respectively. In a solution showing no DMR or $\eta = 1$ and $\varepsilon = 0$, the numerator in the eq. (2) becomes 25.13 n $\Omega\cdot\text{m}$. The $\rho^{\text{PURE}}(T)$ is also necessary for the estimation of residual solute concentration C in a binary solid solution by

$$C = (\rho^{\text{s.s.}}(T) - \rho^{\text{PURE}}(T))/\Delta\rho^i(T)$$

induced from eq. (1).

For multicomponent solutions, A (B, C, ..., n), eq. (2) is rewritten as:¹⁰⁾

$$\rho^{\text{s.s.}}(T_1) = [\rho^{\text{PURE}}(T_2) - \eta^B \rho^{\text{PURE}}(T_1) + \Sigma C^i \Delta\rho^i(T_1)(\eta^i - \eta^B)]/(R - \eta^B) \quad (3)$$

or

$$\rho^{\text{s.s.}}(T_1) = [\rho^{\text{PURE}}(T_2) - \rho^{\text{PURE}}(T_1) + \Sigma C^i \varepsilon^i]/(R - 1) \quad (4)$$

where Σ is the sum of solute species from C to n in eq. (3) and B to n in eq. (4). Numerators in eqs. (2) to (4) correspond to the gradient of regression lines of plots on the $1/(R - 1)$ or $1/(R - \eta)$ versus $\rho^{\text{s.s.}}(T_1)$ diagram, which is usually called as Matthiessen's plot (MP).⁸⁾ In case of a solute with unknown η , eq. (4) is often used to detect errors in size factor. Empirically the solute of positive DMR, or showing larger $d\rho/dT$ with increasing concentration, gives larger gradient of regression lines in MP than the ideal value 25.13 n $\Omega\cdot\text{m}$. Positive ε or DMR of both Mg and Si in Table 1 will give larger values of numerator than the above estimation.

When the resistivity is decreased by aging, the main cause is a decrease in solute concentration, which lowers resistivity significantly. At the same time, increase in volume fraction of precipitates slightly increases the measured ρ . Overlapping of these effects usually leads to lower gradient of regression line on the MP.

2.2 Estimation of volume fraction of precipitates

The influence of precipitates on the resistivity should be

proportional to its volume fraction. However, since this influence is negligibly small in the present study where V_f is less than 1 vol%, the decrement in resistivity measured at 77 K ($\delta\rho(77)$), before and after an aging treatment, can be approximated as

$$\begin{aligned} \delta\rho(77) &= \rho^{\text{STQ}}(77) - \rho^{\text{AGED}}(77) \\ &= \Delta\rho^{\text{Mg}}(77) \cdot \delta C^{\text{Mg}} + \Delta\rho^{\text{Si}}(77) \cdot \delta C^{\text{Si}} \end{aligned} \quad (5)$$

from eq. (1) where $\rho^{\text{STQ}}(77)$ and $\rho^{\text{AGED}}(77)$ are the $\rho^{\text{s.s.}}(77)$ immediately after a solution treatment and after a given aging, respectively. δC^{Mg} and δC^{Si} are the solute concentrations decreased by the aging to form precipitates.

While there are several studies on the atomic ratio of the transition phase,¹²⁻¹⁷⁾ the data differ considerably from each other depending on, for example, the temperature and the time of the aging treatment. Since this issue seems to be still under discussion, stoichiometric compositions are assumed for both β'' and β' transition phases in this work. Namely the ratio $\delta C^{\text{Mg}}/\delta C^{\text{Si}}$ is assumed to be constantly 2. By expressing the sum of δC^{Mg} and δC^{Si} as $\delta C^{\text{Mg}_2\text{Si}}$ which is the amount of Mg_2Si precipitates, eq. (5) can be rewritten as

$$\begin{aligned} \delta\rho(77)/\text{n}\Omega\cdot\text{m} &= (2\Delta\rho^{\text{Mg}}(77) + \Delta\rho^{\text{Si}}(77)) \cdot \delta C^{\text{Mg}_2\text{Si}}/3 \\ &= 5.702 \cdot \delta C^{\text{Mg}_2\text{Si}}/\text{at}\% \end{aligned} \quad (6)$$

Atomic% of Mg_2Si ($\delta C^{\text{Mg}_2\text{Si}}$) obtained from measured $\delta\rho(77)$ using eq. (6) is then transformed into mass% and the volume fraction of precipitates (V_{fp}) is calculated using the densities of Al⁸⁾ ($2.70 \times 10^3 \text{ kg}\cdot\text{m}^{-3}$) and Mg_2Si ¹⁸⁾ ($2.00 \times 10^3 \text{ kg}\cdot\text{m}^{-3}$). The above density of Mg_2Si is a calculated value from structure and lattice parameter¹⁸⁾ of a bulk Mg_2Si .

3. Experimental

3.1 Specimen preparation and heat treatment

Ingots made from high purity Al, Mg and Si were hot rolled, furnace cooled after the intermediate annealing at 683 K for 3.6 ks and finally cold rolled to plates of 0.5 mm in thickness. The chemically analyzed composition of the specimen was Al-0.95%Mg₂Si-0.01%Si or 0.60%Mg-0.36%Si. Contents of Fe, Cu, Zn and other impurities were less than 0.01% (all in mass%). Specimen dimensions and sampling site are shown in Fig. 1.

Fig. 1 Specimen dimensions and sampling site.

A pair of specimens were solution treated at 823 K for 3.6 ks and quenched into iced water (STQ) followed by 473 K aging for 36 ks. To avoid the unintended room temperature aging, the quenched specimen was dipped into a liquid nitrogen bath within 10 s after the quenching. Only $\rho(77)$ was measured for the as-quenched state and both $\rho(77)$ and $\rho(300)$ were measured after aging. Then samples were cut for tensile tests and TEM observations as shown in Fig. 1.

3.2 Measurements

Resistivity was calculated from the resistance and the density-mass method size factor measured before the STQ. D in the expression of measured resistivity ($\rho_D(77)$) means density-mass method size factor. The resistance was measured by the direct current 4-contacts method, using a precision potentiometer equipped with a polarity switch, an electronic galvanometer and a standard resistance directly lined to the specimen.⁹⁾ Switching independently the polarities within the potentiometer and of the outer circuit into the specimen, each potential of the standard resistance and the specimen were measured 4 times as one set. From the average of the measured potentials, the resistance of specimen was calculated. When the resistance calculated from 2 sets of potential measurements did not coincide by more than 4 significant figures, one more set of the potential measurements was carried out.

Tensile tests were carried out at room temperature with an initial strain rate of $6.7 \times 10^{-4} \text{ s}^{-1}$ and a chuck distance of 50 mm.

3.3 Parameters of precipitates from TEM observations

For TEM samples preparation, the 20 mm long plate obtained from the center of ρ specimen was ground to a thickness of about 0.1 mm and 5 to 6 disks were punched as shown in Fig. 1. These disks were polished by a double jet polisher using $\text{HNO}_3\text{--CH}_3\text{OH}$ electrolyte (volume ratio 1 : 3) at 243 K and a voltage of about 12 V. TEM observations were performed with a JEOL 2000FX at 200 kV.

The sample was aligned along the $\langle 001 \rangle$ matrix zone axis. At first, foil thickness (t) was measured by means of convergent beam electron diffraction (CBED) under 2 beam condition.¹⁹⁾ Then, keeping the same field of observation where the thickness was measured, the number (n) of precipitates grown along a $\langle 001 \rangle$ matrix zone axis was counted on bright field image (BFI). Although β'' and β' phases should coexist in the present specimens aged at 473 K for 36 ks,²⁰⁾ they were not distinguished. Assuming homogeneous distribution of precipitates to three different $\langle 001 \rangle$ axes, the counted n is about one third of the total. In order to obtain the average information of the specimen, the sets of n and t were measured with more than 15 fields of observation per TEM specimen. Since some of the precipitates crossing the specimen surface and centering in a missing volume may be counted, the foil thickness was corrected by adding the average precipitate length (λ_{ave}).^{19,21)} Then the number of precipitates per unit volume (N_P) was given as

$$N_P = 3n/[A(t + \lambda_{\text{ave}})] \quad (7)$$

where A is the area of the observed field.

In order to avoid overestimation of λ caused by overlapping of precipitates, λ measurements were carried out where the specimen was sufficiently thin, *i.e.* about 100 nm. d was measured under high magnifications between 500 k and 800 k. Contrast arising from the interfacial strain between matrix and precipitate was lowered as much as possible in order to avoid an overestimation of d .

For simplicity a cylindrical shape of the precipitates is assumed in this work. Then under the assumption of homogeneous distribution of the precipitates, the volume fraction of precipitates (V_{fTEM}) is calculated²²⁾ by eq. (8):

$$\begin{aligned} V_{\text{fTEM}}/\text{vol}\% &= 100 \cdot N_{\text{Pave}} \cdot V_{\text{Pave}} \\ &= 100 \cdot \pi \cdot N_{\text{Pave}} \cdot d_{\text{ave}}^2 \cdot \lambda_{\text{ave}}/4 \end{aligned} \quad (8)$$

where V_{Pave} is the average volume of the precipitates.

The parameters measured on TEM micrographs are largely affected by the heterogeneity of microstructure. To examine the reproducibility, all measurements were carried out independently in 2 specimens after the same heat treatment.

4. Results and Discussion

4.1 Resistivity change and age hardening

Figure 2 shows changes in resistivity ($\rho_D(77)$) and proof stress ($\sigma_{0.2}$) of individual specimens with aging at 473 K. The MP for $\rho_D(77)$ after aging is also shown on the right hand side. Most of $\rho_D(77)$ immediately after solution treatment ($\rho_D^{\text{STQ}}(77)$) are slightly smaller than the calculated value ($\rho_{\text{cal}}^{\text{STQ}}$). This small discrepancy is often observed and considered as coming from a slight difference in the composition, about 0.05 mass%Mg or Si in this case, between samples for the chemical analysis and resistivity measurement. The gradient of regression line in the MP was $25.19 \text{ n}\Omega\cdot\text{m}$ which is slightly smaller than the numerator in eq. (4), 25.58, obtained from a Σ term of $C^{\text{Mg}_2\text{Si}}$ ($2\varepsilon^{\text{Mg}} + \varepsilon^{\text{Si}}$) using 0.5 mass% of $C^{\text{Mg}_2\text{Si}}$ and ρ^{PURE} values of Desai *et al.* As expected in 2.1, precipitates after aging have raised the value of ρ to a small extent and lowered the gradient.

Fig. 2 Changes in resistivity ($\rho_D(77)$) and proof stress ($\sigma_{0.2}$) of an Al–1.0Mg₂Si alloy with 473 K aging and a Matthiessen's plot.

Resistivity monotonously decreases with aging proportionally to the increasing amount of precipitates. $\sigma_{0.2}$ increases until the peak strength around 18 ks and then decreases with overaging. Good reproducibility for $\rho_D(77)$ and $\sigma_{0.2}$ was found in Run-2 and the correlation coefficient (0.9999) of the regressed line in the MP suggests a high accuracy of the measured resistivity.^{9,10} The V_{fp} calculated from decrement in ρ for each of Run-1 and Run-2 were both 0.43 vol%.

4.2 TEM observations

4.2.1 Length

Figure 3 shows examples of TEM micrographs used to measure the length (λ) of the precipitates. The λ in Run-2 looks longer than in Run-1. Figure 4 shows the frequency distribution curves of λ on a logarithmic scale for each magnification of observations and for the total. Also shown in the figure are the standard deviation, 2σ , and the weighted average for total, $b \cdot \exp\{\Sigma[n_i \ln(\lambda_i/b)]\} / \Sigma n_i$. b in the weighted average is the Burgers vector or atomic diameter (0.29 nm) as the minimum limit of λ , n is the total number of measured precipitates and i ($=1, 2, 3, \dots$) is the class of the histogram. Distribution of λ shows negligible dependence on the magnification within the same specimen.

(a) λ_{ave} 33nm, min-max 13-87nm, 200k 50nm

(b) λ_{ave} 116nm, min-max 25-307nm, 100k 50nm

Fig. 3 Examples of TEM micrographs of Al-1.0Mg₂Si aged at 473 K for 36 ks, used for measuring the length (λ) of the precipitates for Run-1 (a) and Run-2 (b).

The weighted average and standard deviation of λ are shown in Table 2 with magnifications and the numbers of observation fields and measured precipitates. The range of measured λ is quite wide, especially in Run-2 and the average in Run-2 was 2.6 times larger than that in Run-1. This great difference should not be caused by the difference in heat treatment ($T_{Sol.}$), for example quenching rate from temperature of solution treatment, because the size of the present specimen was enough small and water quenching or air cooling from $T_{Sol.}$ did not show any difference in the subsequent aging behavior.²³ As the aging condition of the present specimens was slightly over the peak age hardening, a partial shift of precipitates from β'' to β' is considered to be proceeding. A slightly lower solute concentration in the observation fields of Run-2 specimen, caused by microsegregation, may accelerate the partial shift of precipitates. Thus more β' precipitates were measured in the Run-2 sample.

Fig. 4 Frequency distribution on a logarithmic scale of the length (λ) of the precipitates of Al-1.0Mg₂Si aged at 473 K for 36 ks with a weighted average, $b \cdot \exp\{\Sigma[n_i \ln(\lambda_i/b)]\} / \Sigma n_i$.

Table 2 Weighted average and standard deviation (σ) of the length (λ) of the precipitates of Al-1.0Mg₂Si with magnification (Mag.) and the numbers of observation fields (Obs. field) and measured precipitates (n).

Run	Mag. ($\times 10^3$)	Obs. field	n	λ_{ave} ($\lambda_{ave} \pm \sigma$) /nm
1	150	1	38	33
	200	4	160	(23–48)
2	80	1	65	85
	100	2	97	(42–171)
	120	4	208	

$$\lambda_{ave, Run-2} / \lambda_{ave, Run-1} = 2.6$$

Harkness *et al.* empirically found a symmetric distribution of the zone size on a logarithmic scale in Al–Zn and Al–Ag alloys.²⁴⁾ Occasionally a better symmetry of distribution curves was obtained on a root scale in the present work. However since the physical meaning to take the root scale is not clear, the logarithmic scale is employed in this paper.

4.2.2 Cross-sectional diameter

Figure 5 shows examples of TEM micrographs used for measuring the cross-sectional diameter (d) of the precipitates. Since the large scatter of d was found depending on the observation fields, 2 photos taken at different observation fields are shown for each specimen. The larger scatter of d was found in the Run-2 specimen.

Figure 6 shows the frequency distribution of d on a logarithmic scale with the weighted average and standard deviation, 2σ . Because the negligible dependence of distribution curves on the magnification was found in λ (Fig. 4), distribution curves are shown as a total measurements within a specimen. The weighted average and standard deviation of measured d values are shown in Table 3. A better symmetry of distribution was found on a logarithmic scale than on a linear scale. The scattering of d is much smaller than that of λ , which can be understood as the growth of the precipitates

in the longitudinal direction (λ) is occasionally disturbed or stopped by the other precipitates perpendicular to the direction of growth, resulting in the larger scattering of distribution. The growth rate along the short axis (d) should be very small and nearly the same for individual precipitates.

The weighted average of d was 2.62 nm and 2.99 nm for Run-1 and Run-2, respectively, yielding a difference only by 14%. This is a fair reproducibility, but even this small difference would contribute to $V_{f\text{TEM}}$ as a function of d^2 , yielding the difference of 31% between Run-1 and Run-2. Both the average and range of scatter was larger in Run-2 than in Run-1, which is the same tendency as found in λ .

4.2.3 Number density

Figure 7 shows examples of TEM micrographs used for counting the number of precipitates (n). Number densities of precipitates (N_p) were obtained using n and λ_{ave} in Table 2. In TEM work, the number of precipitates projected on the screen increases proportionally with the foil thickness (t), increasing an apparent N_p . Paying attention to this, photos of the areas where t 's were almost the same were chosen in Fig. 7. Since 300 k and 100 k of magnification were used for Run-1 and Run-2, respectively, the observation area

Fig. 5 Examples of TEM micrographs of Al–1.0Mg₂Si aged at 473 K for 36 ks used for measuring the cross-sectional diameter (d) of the precipitates for Run-1 (a and b) and Run-2 (c and d).

Fig. 6 Frequency distribution on a logarithmic scale of the cross-sectional diameter (d) of the precipitates of Al–1.0Mg₂Si aged at 473 K for 36 ks with a weighted average, $b \cdot \exp\{\Sigma[n_i \ln(d_i/b)]\} / \Sigma n_i$.

Table 3 Weighted average and standard deviation (σ) of the cross-sectional diameter (d) of the precipitates of Al–1.0Mg₂Si with magnification (Mag.) and the numbers of observation fields (Obs. field) and measured precipitates (n).

Run	Mag. ($\times 10^3$)	Obs. field	n	$d_{\text{ave.}}$ ($d_{\text{ave.}} \pm \sigma$) /nm
1	500	6	97	2.62 (2.17–3.16)
	500			
2	600	5	61	2.99
	800	3	20	(2.15–4.17)

$$\frac{d_{\text{Run-2}}}{d_{\text{Run-1}}} = 1.1$$

$$\frac{d_{\text{Run-2}}^2}{d_{\text{Run-1}}^2} = 1.3$$

in Run-1 is 9 times smaller than that in Run-2. The difference in N_P between specimens is apparent in these photos. Table 4 shows the average, range and standard deviation of N_P values with several specifications. The scattering in Run-2 (max/min = 5.75) is much larger than that in Run-1 (1.94) which is the same tendency as found in λ and d , and the average of N_P in Run-2 is almost 1/2 of that in Run-1.

(a) N_P $24.9 \cdot 10^3 \mu m^{-3}$, t 151nm, 300k 50nm

(b) N_P $4.1 \cdot 10^3 \mu m^{-3}$, t 152nm, 100k 50nm

Fig. 7 Examples of TEM micrographs of Al-1.0Mg₂Si aged at 473 K for 36 ks, used for counting the number of the precipitates (n) for Run-1 (a) and Run-2 (b).

4.2.4 Volume fraction

Figure 8 shows the average and standard deviation (2σ) of the measured parameters λ , d and N_P and those of the V_{fTEM} estimated from the 3 parameters. Although the weighted averages of λ and d (Tables 2 and 3) are recommended to obtain the V_{fTEM} , simple averages of λ and d and the V_{fTEM} calculated using them are also shown (open marks). The 2σ of V_{fTEM} , for example expressed by right bar, was calculated by applying the 2σ of λ using the average values for the other 2 parameters, d and N_P .

Range of V_{fTEM} is wide especially in Run-2, because the V_{fTEM} is largely affected by individual parameters of precipitates. The V_{fTEM} of Run-1 (0.39 vol%) shows a fairly good

Fig. 8 Average and standard deviation of the length (λ), cross-sectional diameter (d), number density (N_P) and volume fraction (V_{fTEM}) of precipitates of Al-1.0Mg₂Si aged at 473 K for 36 ks.

Table 4 Average, range and standard deviation (σ) of the number density of precipitates (N_P) with magnifications (Mag.), number of observation fields (Obs. field), foil thickness (t), number of counted precipitates (Σn_i), total volume of Obs. field and average length of the precipitates ($\lambda_{ave.}$) of Al-1.0Mg₂Si.

Run	Mag. ($\times 10^3$)	Obs. field	t /nm	Σn_i	$\Sigma A_i t_i / \mu m^3$	$\lambda_{ave.}/nm$	$N_P / 10^3 \mu m^{-3}$		
							min-max	σ	$N_{Pave.}$
1	250	2	167–178	136	0.02	33	14.0–17.1	2.2	21.6
	300	10	121–307	682	0.08		16.0–27.2	4.2	
	400	3	148–298	128	0.02		17.0–23.5	3.3	
2	100	4	126–157	499	0.24	85	3.9–4.1	0.1	11.3
	150	1	110	44	0.02		3.6	—	
	200	5	141–192	461	0.08		5.7–14.6	3.4	
	250	7	128–338	793	0.10		13.4–20.5	2.6	

observation field $i = 1, 2, 3, \dots$

$N_{PRun-2} / N_{PRun-1} = 0.52$

coincidence with V_{fp} (0.43 vol% in both Run-1 and Run-2), but the V_{fTEM} of Run-2 (0.68 vol%) is 1.6 times larger than the V_{fp} . In Run-2, the size of the precipitates, λ and d , is larger and the N_p is smaller than in Run-1.

Though the Ostwald ripening assumes constant solute concentration, the coarsening of precipitates in the present case is accompanied by a slow decrease in solute concentration and should be distinguished from the Ostwald ripening. The above results can be explained as that this type of coarsening has taken place more rapidly in the observation fields of Run-2. Precipitates grow both in its size (λ and d) and total amount (V_f) with aging, increasing the strength until the peak strength. However by aging longer than the peak strength, large precipitates begin to grow consuming smaller ones nearby, thus the N_p decreases. The systematic difference in the parameters of precipitates found between Run-1 and Run-2 suggests that the results obtained by TEM observations are affected by the heterogeneity of the microstructure. Therefore we should conclude that resistivity measurements give more accurate estimation of V_f than TEM observations. Although it is believed that the TEM observations could give a better accurate V_f by getting enough large number of measurements, this will consume much more time and energy compared to resistivity measurements.

Considering that the V_{fTEM} is obtained by the 3 parameters, λ , d and N_p (eq. (8)) and that the V_{fTEM} should be equal to the V_{fp} , an inverse operation of the above parameters is possible. By using the V_{fp} and 2 parameters including d which is less affected by the heterogeneity, the value of the last residual parameter should give a better average value, avoiding the effect of the heterogeneity. Evaluation of precipitation hardening by dislocation theory would be improved by using the V_{fp} , d and either of N_p or λ obtained by an inverse operation.

5. Conclusion

Although proof stress, volume fraction estimated by resistivity measurements and cross-sectional diameter of the precipitates showed a good reproducibility, length and number density of the precipitates differed greatly between two equivalent samples. This is partially understood by a wide range of scatter found in the size and distribution of the precipitates from TEM observations. The differences found in the dimensions and number of precipitates between the two samples were well explained by assuming that the coarsening of precipitates occurred more rapidly in one sample than the other. Therefore it is concluded that the heterogeneity of the microstructure was still reflected in the TEM results. To quantify the volume fraction of the precipitates, in general, the reproducibility of the resistivity measurement is better than the TEM observations. Notwithstanding more than 10^{10} times difference in inspected volume, the volume fraction of pre-

cipitates from resistivity and TEM coincides by a factor less than 1.6.

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