



Multi-Dimensional Quantitative Analysis of Precipitates in the Hot-Rolled Al-1 % Mn Alloy^{*1}

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Precipitates formed during the hot-rolling process of Al-1%Mn alloy were quantitatively characterized by 3-dimensional (3-D) evaluation. The 3-D image was reconstructed using a serial sectioning technique by combining the focused ion beam and scanning electron microscopy (SEM). In the reconstructed 3-D volume, more than 10000 precipitates were extracted, and the log-normal distribution explained the size distribution well. We also quantitatively evaluated the precipitates from the 2-dimensional (2-D) SEM image taken during the serial-sectioning and from the scanning transmission electron microscopy (STEM) observation that the specimen was fabricated from the serial-sectioned sample. According to the data evaluated from the 2-D and the 3-D images, the average size of the precipitation measured by the 3-D image was larger than that of the 2-D images of SEM and STEM. That is presumed by the morphological anisotropy owing to hot rolling. We also discussed the effect of the number of the analyzed precipitates on the average size and distribution. The average size significantly depends on the number of analyzed precipitates. Besides, we evaluated the normality of the logarithmic distribution of the size by the Quantile-Quantile plot.

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1. Introduction

The mechanical and functional properties of materials are governed by their microstructure. Recently, combined studies have been performed to improve the understanding and prediction of microstructure; for example, such studies include the prediction of microstructure through computational simulation and experimental validation, as well as the integration of experimental results and modeling using a theoretical approach. Consequently, the importance of computational simulations has increased substantially. Olson emphasized the importance of designing materials on the basis of optimum computational simulations, which depend on the target structure [1]. Subsequently, Raabe summarized and introduced computational simulation techniques spanning from the atomic level to the macrostructural level, along with their underlying physical principles [2]. Computational simulations and modeling have also been widely applied to aluminum alloys. In particular, a modeling technique for thermomechanical processing is used to optimize the microstructure of aluminum alloys to achieve desirable properties [3]. Owing to improvements in computational performance and advances in modeling techniques, the accuracy of such models has improved considerably. However, most computational simulations require input parameters related to materials and processing conditions, and further refinement of these parameters is important for achieving high-precision modeling.

Microstructural parameters, such as grain size, that is, the microstructural factors, are obtained through characterization using microscopy and X-ray analysis. In microscopy, the area fraction of second-phase particles [4] and the grain size [5]

can be directly obtained from cross-sectional observations. The obtained two-dimensional (2D) data has been proposed to be converted into three-dimensional (3D) space as a volume fraction. This technique is known as “Quantitative Microscopy” [6] and is widely used for the quantitative characterization of microstructures. Quantitative characterization of metastable and stable precipitates, which are among the most important microstructural factors in aluminum alloys, has often been performed by X-ray small-angle scattering (SAXS) [7–10]. SAXS enables the characterization of nanosized particles in a bulk sample. To date, SAXS has been applied not only to static characterization but also to the study of the dynamics of nanostructural evolution with aging [10]. The characteristic structural size range accessible by standard SAXS is from a few to tens of nanometers; therefore, SAXS is a powerful tool for characterization of clusters in aluminum alloys. Cluster evaluation has also been performed using 3D atom probe (3DAP) [11]. 3DAP is a powerful tool for chemical analysis and is used to characterize fluctuating chemical structures. However, in 3DAP, only a very small specimen is analyzed, making characterization of precipitates with sizes ranging from a few tens to hundreds of nanometers difficult.

Recently, microstructures have been characterized from 2D to 3D representations through the development of advanced observation techniques and computational visualization methods [12–24]. The 3D microstructure has been characterized using optical microscopy (OM) [12–14], scanning electron microscopy (SEM) [15, 16], and transmission electron microscopy (TEM) [17–19]. In addition, 3D structures can be observed using X-rays at synchrotron facilities, enabling the experimental and quantitative evaluation of microstructural factors [20–24]. Using OM, the recrystallized grain structures of pure iron [12] and low-carbon steel [13] have been characterized in 3D by serial sectioning and discussed in relation to the grain growth behavior. Wang *et al.* characterized the microstructure of

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perlite in 3D and quantitatively analyzed its features, such as the curvature of the ferrite/cementite phase boundary [14]. Based on these results, they discussed the relationship between microstructural evolution and cementite spheroidization. For 3D characterization using SEM, serial sectioning combined with a focused ion beam (FIB) has been employed. Among these studies, Hara *et al.* succeeded in 3D observation of the microstructure of precipitates in the heat-resistant steel and ω -phase in Ti alloys at a nanoscale [15, 16]. In TEM, 3D tomography is widely used and is based on the acquisition of images over a wide tilt angle range followed by computational reconstruction. As an example of TEM-based 3D tomography in Al alloys, Kaneko *et al.* characterized individual Si and Ge precipitates in aged Al-Si and Al-Ge alloys [17, 18]. Furthermore, X-ray tomography is advantageous because it is a nondestructive method and has been applied to the study of microstructural evolution during crack propagation [21] and to the evaluation of pore and void formation and growth [22]. Quantitative 3D characterization has thus been widely performed, depending on the spatial resolution of the probe. However, quantitative analysis of precipitates in aluminum alloys has mainly been conducted for the clusters nucleated at the early stage of precipitation using SAXS. Although case studies in which the size of the precipitates in bulk samples has been evaluated by X-ray tomography have been reported [23, 24], to the best of the authors' knowledge, no previous studies have quantitatively and statistically characterized individual precipitates.

In this study, we report the quantitative analysis of the precipitates nucleated during hot rolling in an Al-Mn alloy using a serial sectioning technique combined with orthogonally arranged FIB-SEM. Tanaka *et al.* investigated the substructure of Al-Mn alloys formed by the plane strain compression method and suggested the importance of dynamic precipitates formed during hot deformation for substructure stability [25]. In addition, precipitates are important microstructural factors that govern interactions with dislocations, and their size and distribution are important parameters for thermomechanical modeling. Therefore, we focused on precipitates in Al-Mn alloys formed through hot rolling. The 2D characterization was conducted using SEM and TEM on samples thinned from those used for serial sectioning, and the obtained features were compared.

2. Experimental Procedure

The material used in this study was an Al-Mn alloy consisting of pure Al with a composition equivalent to 1050 Al and 1 mass% Mn. The alloy composition is listed in Table 1. The ingot was cast using a standard semicontinuous direct-chill technique and hot-rolled at 773 K. The hot-rolled

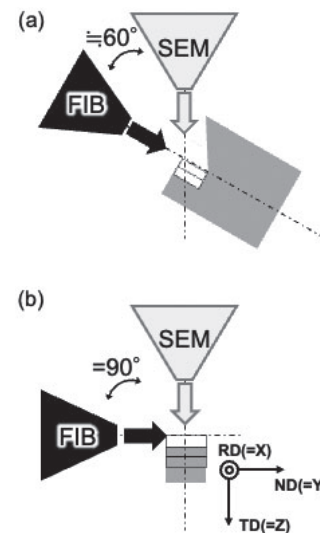


Fig. 1 Schematic drawing of typical configurations of the FIB-SEM system. (a) Conventional arrangement and (b) Orthogonally configured system. The geometry of the specimen in the serial sectioning conducted in this experiment is also indicated in (b).

plate was cut into rectangular pieces a few centimeters in size and mechanically and electrically polished to achieve a mirror surface. For the serial sectioning, a cubic specimen with a side length of approximately $30\text{ }\mu\text{m}$ was extracted from the plate using a FIB. Serial sectioning was performed using an orthogonally arranged FIB-SEM (Hitachi High-Tech Science Co., SMF 1000). A schematic of the conventional and orthogonally arranged FIB-SEM, together with the specimen, is shown in Fig. 1. As shown in Fig. 1(a), in the conventional system, the FIB and SEM were arranged at an angle of 60° . In contrast, in the system used in this study, the FIB and SEM were arranged at 90° shown in Fig. 1(b), such that the incident beam is always perpendicular to the specimen surface. High-quality SEM images were acquired through serial sectioning. In this study, room-temperature FIB slicing was performed using Ga^+ ions at an acceleration voltage of 1 kV, a beam current of 2 nA, and a slice pitch of 5 nm. SEM images are acquired at a resolution of 2000×2000 pixels over a field of view of $20 \times 20\text{ }\mu\text{m}^2$ at an acceleration voltage of 1 kV. Accordingly, a voxel in the reconstructed 3D data, representing the minimum unit for the quantitative analysis, corresponds to $10 \times 10 \times 5\text{ nm}^3$. The image contrast was optimized by combining signals from secondary and backscattered electrons to observe the precipitate clearly. Approximately 1000 SEM images were acquired during serial sectioning to construct the 3D image. The 3D image was reconstructed using commercially available software (Image Pro, Media Cybernetics, Ver. 10), and the precipitates were extracted on the basis of the contrast value of each voxel using the same software. The sizes of individual precipitates were measured. The serially sectioned specimen was further sliced using a FIB to prepare a thin film for scanning TEM (STEM) (JEOL Co., JEM-2800) observation. STEM was operated at an acceleration voltage of 200 kV. Energy dispersive X-ray spectroscopy (EDS) analysis was performed to chemically characterize the precipitates.

Table 1 Chemical composition of the specimen used in this study.

(mass%)								
Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Al
0.16	0.34	<0.01	0.99	<0.01	<0.01	<0.01	0.01	Bal.

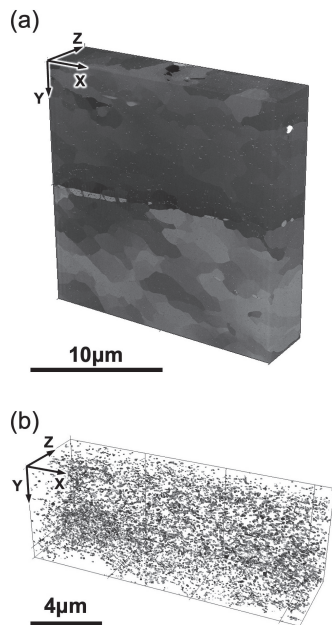


Fig. 2 (a) 3D volume of the hot-rolled Al-Mn alloy reconstructed from the series of images taken by the serial sectioning technique, and (b) precipitates extracted from the 3D volume in (a).

3. Results and Discussion

3.1 FIB-SEM serial sectioning and 3D image reconstruction

A 3D image of the hot-rolled Al-Mn alloy obtained by serial sectioning followed by the image reconstruction is shown in Fig. 2(a). X, Y, and Z directions correspond to the rolling direction (RD), normal direction (ND), and transverse direction (TD), respectively. The upper region of the 3D images exhibits a dark contrast, whereas the lower region appears bright. This contrast difference arises from the crystal orientation during data acquisition. In addition, markedly bright features elongated along the RD are observed in the central region of Fig. 2(a). These features correspond to secondary phases formed during the casting process. In contrast, precipitates exhibiting relatively weak contrast are uniformly distributed. A white-contrast region is clearly visible in the upper part of Fig. 2(a). As shown in Fig. 2(b), the precipitates analyzed in this study were selected from this upper region of Fig. 2(a). For ease of recognition, the precipitates were visualized as dark contrasts. Although precipitates were present throughout the entire image, as shown in Fig. 2(a), the contrast between the matrix and precipitates in the lower part of Fig. 2(a) was similar, making identification of precipitates difficult. Therefore, this region was excluded from the present analysis, and only a part of Fig. 2(a) was used for the quantitative analysis of precipitates. The size of the analyzed volume was approximately $16.0 \times 5.7 \times 4.6 \mu\text{m}^3$, and the number of analyzed precipitates was 11186.

A histogram of the sizes of 11186 precipitates is shown in Fig. 3. The Feret diameter, defined as the length of the circumscribed boundary of a precipitate, was used, and the average of the longest and shortest Feret diameters was taken as the precipitate size. Assuming a log-normal distribution

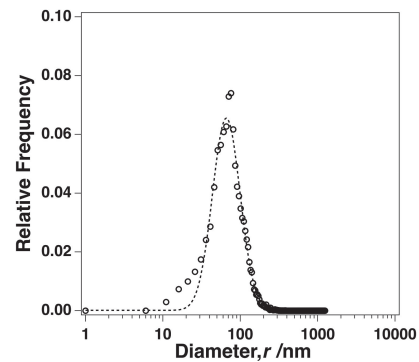


Fig. 3 Histogram of the diameter of precipitates observed in Fig. 2(b). The dashed line is the fitting profile of the histogram based on the log-normal distribution.

of the histogram, the fitted curve agrees well with the experimental histogram. Therefore, the size distribution of the precipitates can be explained by a log-normal distribution. As mentioned previously, regarding the size and distribution of precipitates, Harkness *et al.* [8] and Osamura *et al.* [9] have reported metastable Guinier–Preston (GP) zones with sizes of a few nanometers using SAXS. However, to the best of our knowledge, this is the first study in which the size of the stable phase with dimensions of a few hundred nanometers has been evaluated through individual observation and statistical analysis. A slight deviation of the fitted curve from the experimental data was observed at approximately 20 nm. This deviation arises from the SEM resolution of 10 nm/pixel. Noise smoothing over a single pixel can extend over a few pixels, corresponding to approximately 20 nm, which may lead to erroneous counting of precipitates.

3.2 STEM observation and EDS analysis

A characteristic feature of serial sectioning using an orthogonally arranged FIB-SEM system is that a thin film from the same region can be fabricated by further slicing after serial sectioning. Therefore, nanoscale structures can be observed by TEM following the 3D observation. The annular dark field (ADF) image is shown in Fig. 4(a), and the EDS maps obtained by STEM-EDS are shown in Figs. 4(b)–(d). As shown in Figs. 4(b)–(d), Mn, the main constituent, and

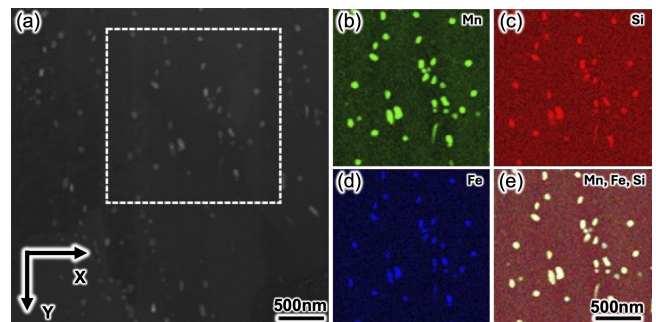


Fig. 4 STEM-EDS analysis of Al-Mn alloy. The specimen was taken from the serial-sectioned piece. The electron beam is parallel to the TD direction. (a) STEM-ADF image. (b)–(e) EDS maps showing the distribution of (b) Mn, (c) Si, (d) Fe, and (e) overlaid Mn, Fe, and Si, respectively. (online color)



Fig. 5 The image of the TD plane on the surface of the reconstructed 3D volume viewed from the Z-direction in Fig. 2(b).

Fe and Si, which are typical impurities in Al, were detected. The EDS map in which these three elements are superimposed is shown in Fig. 4(e). The ellipsoidal precipitates exhibiting bright contrast contain both Fe and Si in addition to Mn; therefore, these precipitates are presumed to be the quaternary α -phase compound $\text{Al}(\text{Mn}, \text{Fe})\text{Si}$, which is frequently observed in the low-purity Al–Mn alloy. Li *et al.* have reported the existence of the α -phase in 3003 Al alloys annealed at 773 K [26]. The observation of the α -phase in the present study is reasonable considering the thermomechanical conditions employed. In addition, the existence of the α -phase has also been reported by Tanaka *et al.* [27].

3.3 Quantitative analysis of precipitates

As shown in Fig. 3, the quantitative analysis of precipitate size was successfully performed in 3D, and the size distribution was well described by a log-normal distribution. To date, particle size distribution have been generally measured in 2D, even when the particles are distributed in 3D space [4–6]. Because the precipitates were directly measured in 3D in this study, we compared the size distributions obtained from the 3D SEM image, a 2D SEM image extracted from the 3D image that is shown in Fig. 5, and the 2D STEM image in Fig. 4(a).

The size distribution of the precipitates obtained from the (a) 2D STEM and (b) 2D SEM images is shown in Fig. 6. The precipitates were extracted from each image using the same procedure as that applied to the 3D SEM image. A total of 147 and 203 precipitates were identified in the 2D STEM image and 2D SEM image, respectively. Because the resolutions of 2D STEM and SEM images are 3.38 nm/pixel and 10 nm/pixel, respectively, features smaller than 3.38 nm (STEM) and 10 nm (SEM) were excluded from the analysis as precipitates. Consequently, 129 precipitates in the 2D STEM image and 160 in the 2D SEM image were quantitatively analyzed. Although both datasets could be fitted with a log-normal distribution, the fitted curves and experimental data show greater scatter than the 3D data because of the smaller number of analyzed precipitates. The results are summarized in Table 2. The average size measured from the 3D SEM image was 81.24 nm; however, the average sizes obtained from the 2D images were approximately 53 nm and showed no dependence on the observation method. To clarify the origin of the differences in average sizes, we examined the reconstructed 3D SEM image from different viewing directions. The reconstructed microstructure viewed from the (a) Y-direction (Z–X plane) and (b) the X-direction (Y–Z plane), corresponding to the ND and RD of the rolled plate, respectively, is shown in

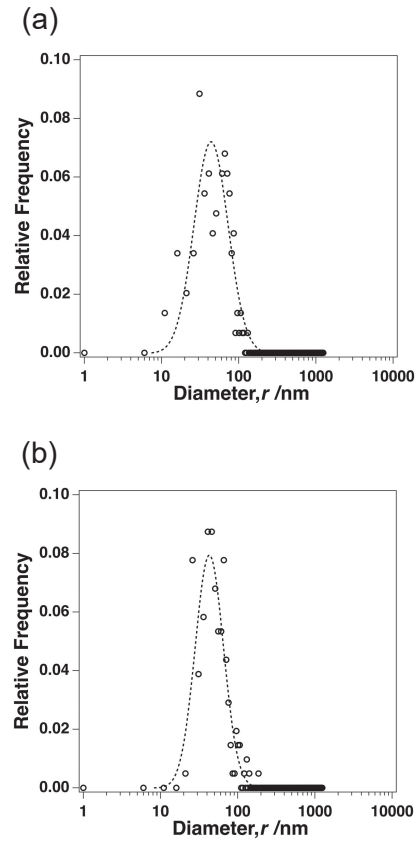


Fig. 6 Histograms of the diameter of precipitates observed in (a) STEM image in Fig. 4(a), (b) SEM image shown in Fig. 5. The dashed lines are the fitting profile based on the log-normal distribution.

Table 2 Statistic data of the precipitates obtained from the images of 3D-SEM in Fig. 2(b), 2D-SEM in Fig. 5, and 2D-STEM in Fig. 4(a), respectively. N is the number of analyzed precipitates, $\bar{D}$, σ , and SE are the average diameter, standard deviation and standard error measured from the data, respectively.

	N (-)	$\bar{D}$ (nm)	σ (nm)	SE (nm)
3D SEM	11186	81.24	34.842	0.003
2D SEM	160	53.60	25.324	2.002
2D STEM	129	53.02	23.837	2.099



Fig. 7 (a) ND (Z–X) and (b) RD (Y–Z) planes of the 3D volume shown in Fig. 2(b), each plane corresponds to (a) $Y = 0$ and (b) $X = 0$, respectively.

Fig. 7. Notably, the SEM image in the RD was rotated 90° anticlockwise so that the Z-direction is oriented consistently in both (a) and (b). Moreover, the uppermost image of the Z–X plane and the leftmost image of the Y–Z plane in Fig. 2(b) are shown in Figs. 7(a) and (b), respectively. As

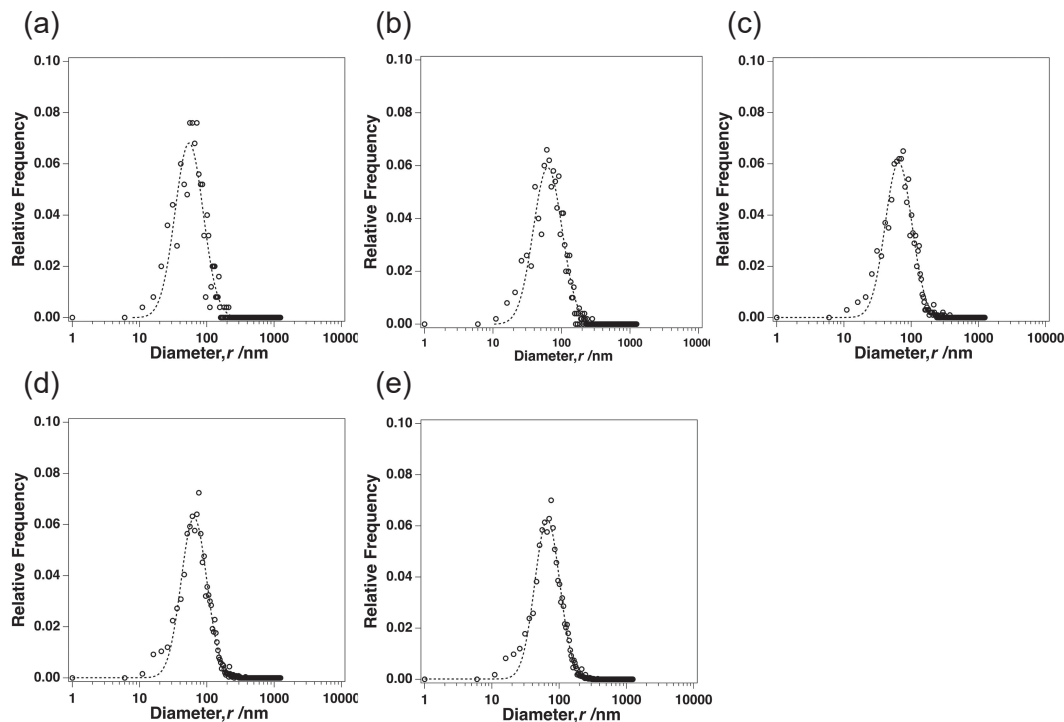


Fig. 8 Effect of the number of analyzed precipitates, N , on the histogram. In each histogram, N is (a) 250, (b) 500, (c) 1000, (d) 2500, and (e) 5000, respectively.

shown in Figs. 7(a) and (b), a few precipitates that elongated along the Z direction can be observed. The precipitates in the 3D SEM image were measured by considering the length in the Z direction, whereas this dimension was not captured in the 2D SEM and STEM images. Therefore, the precipitates measured in the 3D SEM images were larger than those measured in the 2D SEM and STEM images. Based on these results, because the microstructure of the rolled plate is expected to exhibit anisotropy, observing it from various directions is important.

Thus far, we have analyzed the precipitates observed in the 3D SEM image and compared the results with those obtained from 2D SEM and STEM images, thereby clarifying the microstructural features such as the average size and data scatter. However, the number of precipitates analyzed differed substantially: 11186 in the 3D SEM image and 160 and 129 in the 2D SEM and STEM images, respectively, differing by more than two orders of magnitude. Therefore, we examined the dependence of the results on the number of analyzed precipitates. The histograms obtained for different numbers (N) of precipitates selected from the 3D SEM images are shown in Fig. 8. The analyzed precipitates were identified based on their center positions and selected from $Z = 0$. This procedure corresponds to the analysis of the precipitates within an arbitrary volume in the 3D image projected onto the X–Y (TD) surface. As shown in Fig. 8(a), the histogram for $N = 250$ is not smooth and is scattered from the log-normal fitting. In contrast, the experimental data and fitted curve become similar to each other with increasing N from 500 to 2500 (Figs. 8(b)–(d)), and for $N = 5000$ (Fig. 8(e)), it is in good agreement, except for the data for sizes of approximately 20 nm. The relationship between the average precipitate size and N is shown in Fig. 9. With an

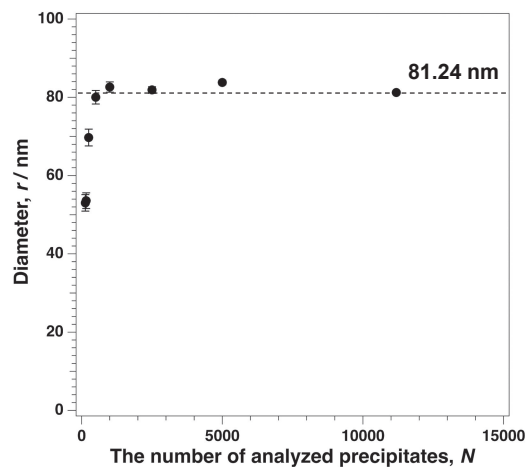


Fig. 9 Effect of the number of analyzed precipitates, N , on the average diameter of the precipitates. The average diameter of all precipitates detected in the 3D volume shown in Fig. 2(b), 81.24 nm, is indicated in this graph with the dashed line. Error bars represent a standard error.

increase in the number of analyzed precipitates, the average size increased and eventually saturated at the average size of all precipitates analyzed in this study, for N exceeding 1000. Therefore, quantitative analysis of the precipitation size requires evaluation of more than 1000 precipitates. However, further analysis is necessary to assess the effectiveness of using 1000 precipitates as the number of objects.

3.4 Normality of the size of precipitates

As discussed in the previous sections, dissimilarities were observed among the analytical results obtained under various conditions. In particular, variations in the experimental data and the fitted profile were qualitatively described on the basis

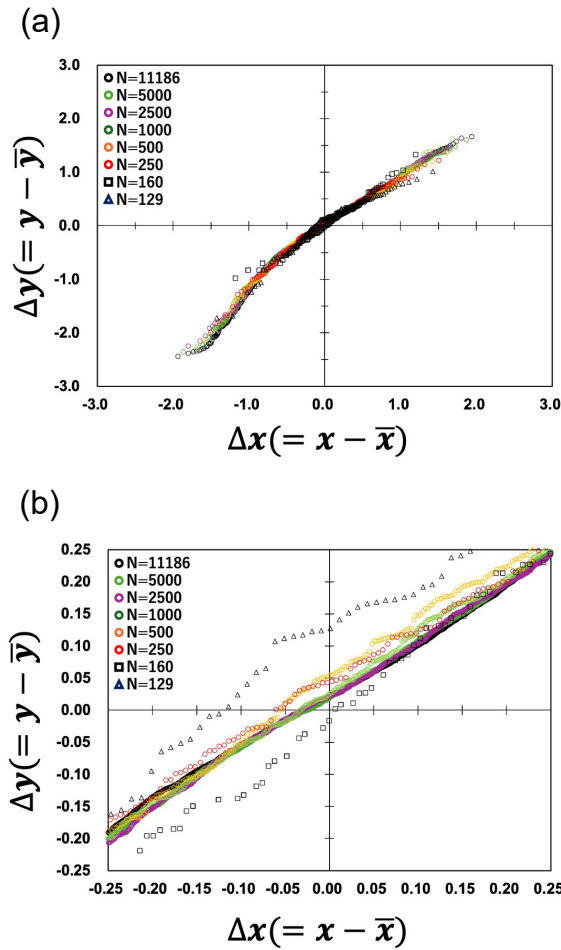


Fig. 10 (a) Q-Q plot of the distribution of the measured diameter, y , and the theoretical one, x , based on the assumed log-normal distribution with the average and standard deviation obtained from measurement. The enlarged graph around the origin point is also shown in (b). In these graphs, vertical and horizontal axes represent the difference from the average value of $\bar{x}$ and $\bar{y}$, respectively. (online color)

of a log-normal distribution. In this section, the normality of the data is evaluated and discussed quantitatively. Normality is assessed by comparing the experimentally obtained data with theoretically expected data under the assumption that the experimental dataset has a normal distribution using a quantile-quantile (Q-Q) plot [28, 29]. If the measured data follows a normal distribution, the Q-Q plot exhibits a linear relationship with a slope of 1 that passes through the origin, that is, $y = x$. In this study, because the measured precipitate sizes were described by a log-normal distribution, the normality of the logarithmic precipitate size was examined. The Q-Q plots obtained from the data shown in Figs. 3, 6, and 8 are shown in Fig. 10. In these plots, the x and y axes represent the theoretical and experimental values, respectively. In addition, because statistical parameters such as the average and standard deviation differ among the datasets, the deviations from the averages, Δx and Δy , are plotted in the Q-Q plots. The Q-Q plots for all datasets are shown in Fig. 10(a). The plots near $\Delta x = 0$ for all datasets are close to zero; however, the upper and lower edges, which correspond to the data far from the average, deviate from linearity. The enlarged Q-Q plots near the origin are shown in Fig. 10(b). The Q-Q plots for smaller N values deviate from the linear

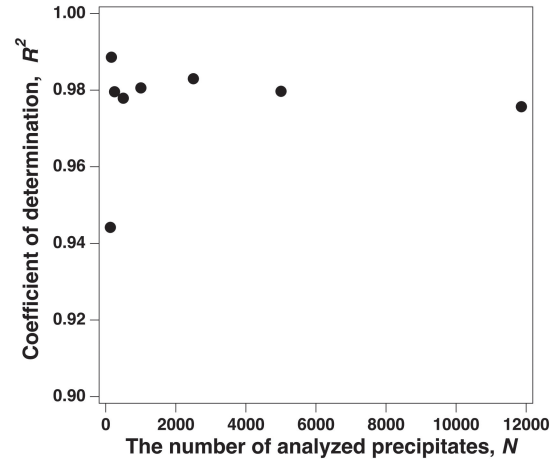


Fig. 11 Relationship between the coefficient of determination, R^2 , obtained from the least squares fitting of the Q-Q plot in Fig. 10 and the number of analyzed precipitates, N .

relationship, indicating that normality does not depend on the dimensions. In addition, the plots approach a linear relationship as N increases. To quantitatively evaluate these deviations, we show the relationship between the definition coefficient, R^2 , and the number of analyzed precipitates in Fig. 11. Even for the data obtained from the analysis of 2D STEM image analysis, in which the minimum number of precipitates was 129, the R^2 was more than 0.94. However, R^2 increases with increasing N , which is the same trend observed for the statistical analysis of the average size shown in Fig. 9.

In this study, we observed the precipitates in the Al-Mn alloy by serial sectioning using a FIB-SEM system and statistically and quantitatively evaluated the precipitate sizes. Although the present study reports results from only one specimen, comparison of material dependence would enable analysis not only of specific parameters, such as the average size, but also of the size distribution. As future work progresses, the method developed in this study will be applied to other microstructures. By obtaining additional microstructural parameters, such as shape, number density, and interparticle distance, we expect to further clarify the nature of the microstructure and contribute to improvements in modeling.

4. Concluding Remarks

In this study, we observed precipitates in hot-rolled Al-1 mass% Mn using multidimensional characterization and evaluated their sizes quantitatively. The results are summarized as follows.

- (1) A total of 11186 precipitates were selected from the 3D image obtained by FIB-SEM serial sectioning and reconstruction. The size distribution was well described by a log-normal distribution.
- (2) The precipitate size measured from the 3D image was 81.24 nm. However, the sizes measured from the 2D image obtained from SEM and STEM observations were approximately 53 nm. Examination of the 3D image viewed from the ND and RD revealed that the precipitates were elongated along the TD. Because

observation from the ND and RD is not possible in 2D images, the use of 3D analysis accounts for the differences in the measured sizes.

- (3) The dependence of precipitate size distribution on the number of analyzed precipitates was also investigated. The average precipitate size increased with the increasing number of analyzed precipitates and reached a constant value when more than 1000 precipitates were included. This finding indicates that quantitative statistical analysis requires more than 1000 precipitates.
- (4) The normality of the size distribution was evaluated using a Q–Q plot. The regression analysis of the Q–Q plots yielded a high R^2 value, with the lowest being 0.94 at $N = 129$. However, R^2 increased with increasing numbers of analyzed precipitates.
- (5) 3D observation is highly effective and important for microstructural characterization. In particular, a quantitative analysis of the anisotropic microstructures, such as those in as-rolled plate, which need to be observed from various directions, was demonstrated experimentally.

Acknowledgments

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