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X-Ray Study on Mg-Sm Alloy Produced by HPT

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Abstract. The purpose of this study is to reveal the cause of hardening of magnesium base alloys by the high pressure torsion processing (HPT) using X-ray diffraction. HPT was applied to Mg base alloys of the Mg-Sm system (2.8-5.5 mass %Sm). HPT was performed under pressure 4GPa at 20 ^{o}C and 200 ^{o}C. HPT results in significant strengthening of the Mg-Sm alloys due to the formation of sub microcrystalline structure. The dynamic recrystallization was realized through the pole figure measurement and the photograph of X-ray back scattering. The Mg supersaturated solid solution decomposition during HPT was observed by the X-ray profile analysis and the calculation of lattice constants of the Mg phase.

Introduction

Severe plastic deformation (SPD) improves on strength of metals and alloys due to grain refinement down to nanoscale [1,2,3]. Recently, many materials by SPD have been investigated on their mechanical properties. Magnesium-based alloys are light materials, and they are hoped of using as structural materials. Therefore, improvement of strength of Mg alloys is very important. Hardening of Mg alloys occurs by not only grain refinement but also age hardening. Not only that the decomposition of supersaturated magnesium solid solution would be caused by SPD. In addition, X-ray diffraction analysis provides us the useful information to realize the structure of crystalline materials, for example, the quantity of chemical phase, the change of the lattice constant, the state of preferred orientation and etc [4,5]. In this study, Mg alloys including a few percent of samarium were given heavy deformation by the high pressure torsion processing (HPT). Structures of Mg-Sm Alloys depending on Sm content and on HPT were observed by using X-ray diffraction profiles. Especially, the preferred orientations of Mg-Sm alloys by HPT were investigated in detail before profile analysis, because the profile distortion by the orientation would disturb correctly measurement on X-ray profile analysis.

Experimental Procedure

Three binary alloys were used for the experiments. They contained 2.8, 4.5 and 5.5 mass %Sm. The Mg-Sm alloys were solution treated at about 520^{o}C for 5 hours and quenched in cold water. After quenching, the pieces that have the dimensions of 10mm in diameter and 0.6mm in thickness were subjected to SPD at room temperature (20^{o}C) and 200^{o}C by HPT under a pressure of 4GPa (5 revolutions) [6]. X-ray diffraction experiments were carried out for the structure investigation of specimens. (0002) pole figures and diffraction profiles were obtained from each specimen. Table 1

lists the measurement conditions for X-ray diffraction experiment. Figure 1 shows the experimental set up. The X-ray irradiated areas were determined 2 points on the disk specimen due to comparing the center position with the 2.5mm outer position from center. In the pole figure measurement, the inclination angle normal to specimen surface α was set from 30^o to 90^o in intervals of 10^o, and the rotation angle β was set from 0^o to 350^o in intervals of 10^o. In addition, the diffraction ring was observed by the back scattering optical system.

Table 1 X-ray measurement conditions.

Characteristic radiation	Cr-Kα
Tube voltage, kV	35
Tube current, mA	60
Collimator diameter, mm	1.0
Kβ filter	V
Detector	Curved PSPC

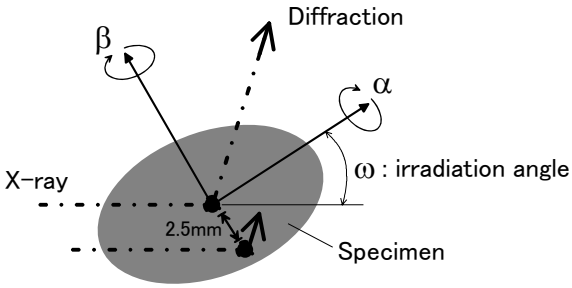

Fig. 1 Experimental set up.

Results and Discussion

Orientation Observation. Figure 2 shows examples of the diffraction ring obtained from the specimens before HPT and after HPT. However, only Mg-2.8Sm is shown here. As a result, only the diffraction from Mg phase could be obtained due to very weak diffraction intensity of Mg-Sm chemical composition phase. Before HPT, it had spots in a ring, because the specimen had strong preferred orientation by casting process. After HPT, those spots disappeared, and the picture at HPT 20^oC was similar to that at 200^oC. Therefore the dynamic recrystallization could occur by SPD. Figure 3 shows examples of the pole figure. These figures also reveal the dynamic recrystallization by SPD. The point where the density was high locally disappeared after HPT. The diffraction ring and the pole figure of 4.5 and 5.5%Sm were also obtained similar to that of 2.8%. Therefore, in the X-ray profile analysis, the peak profiles obtained from the materials having preferred orientation are taken care. Especially in this study, the profiles from the as-received specimens were taken an average with angle α and β. Figure 4 shows the pole figure by the irradiation at 2.5mm outer position. Extension of pole density line from the center to the outside shows that the specimen has <0001> preferred orientation. Therefore, the dynamic recrystallization at the center should occur prior to the outside. However, the absolute value of the pole density is not so different at each position. Then the diffraction profiles of the center position would be used in the profile analysis.

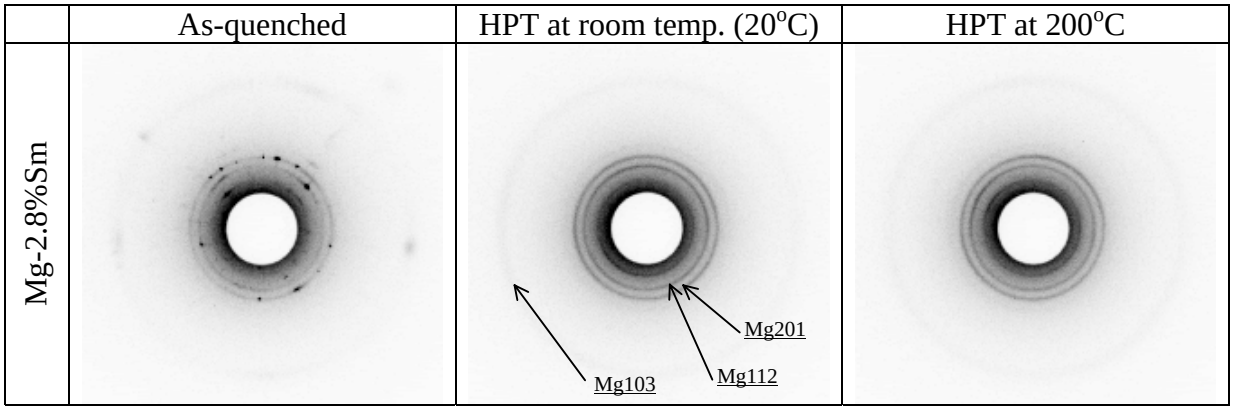

Fig. 2 Diffraction ring obtained by X-ray back scattering optical system.

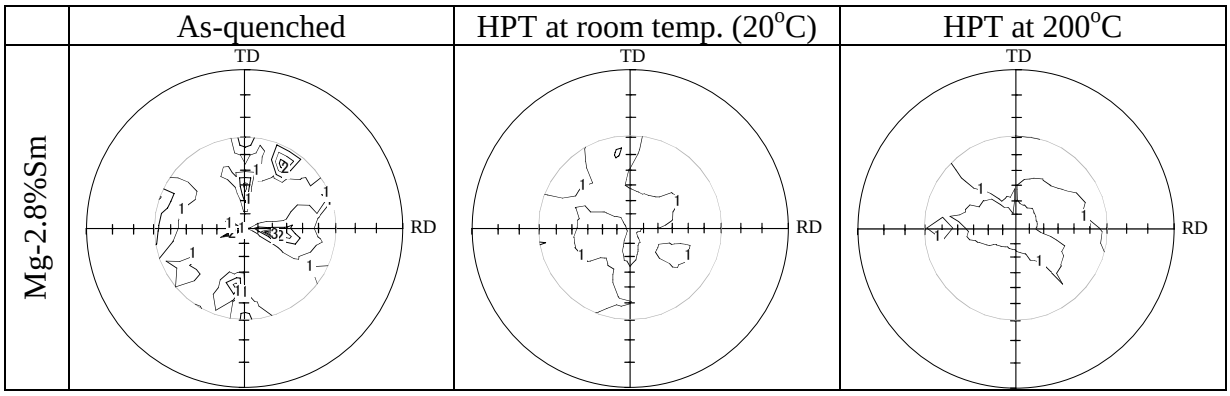

Fig. 3 (0002) pole figures obtained by Mg-2.8Sm specimens at center position.

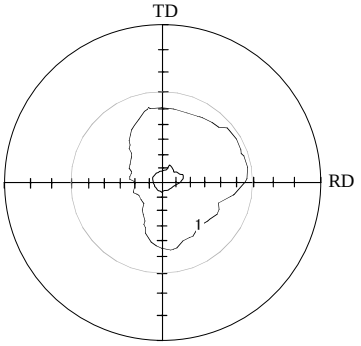

Fig. 4 Pole figure of HPT 200°C. (Mg-2.8%Sm, outer position)

Effect of Sm Content and HPT. Figure 5 shows an example of the diffraction profile obtained from the Mg-5.5%Sm specimen without HPT processing. In addition, the powder diffraction data of Mg phase also are indicated here. Since it calculated the average from the experiment data sets, the deviation of relative intensity from the powder diffraction standard data became small. Here, the diffraction profiles from $Mg_{41}Sm_5$ phase were able to be seen in this figure. Figure 6 presents the effect of Sm contents on the diffraction profile from the second phase. The diffraction intensity becomes high as the Sm content increases. This describes that the second phase particles are generated corresponding to the initial Sm content. Increases of the second particles should be mainly caused by the Mg solid solution decomposition during aging at room temperature and by the Mg-Sm compounds generated by the remainder of Mg solid solution. Here, lattice constants of the Mg phase are able to be calculate by Cohen's method [4,7] from each X-ray diffraction profiles. The Mg hexagonal crystal needs two lattice constants, a_0 and c_0. The equation is determined by:

$$\sin^2\theta = C\alpha + B\beta + A\delta. \quad (1)$$

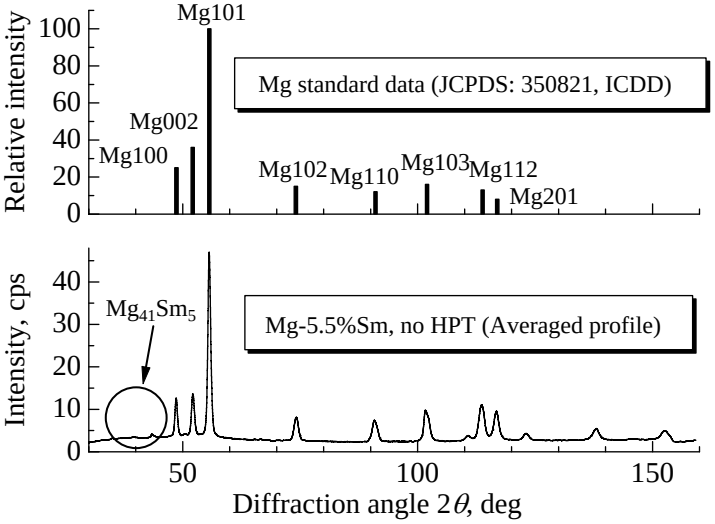

Fig. 5 X-ray profile from specimen (Mg-5.5%Sm) and powder diffraction data by JCPDS, ICDD.

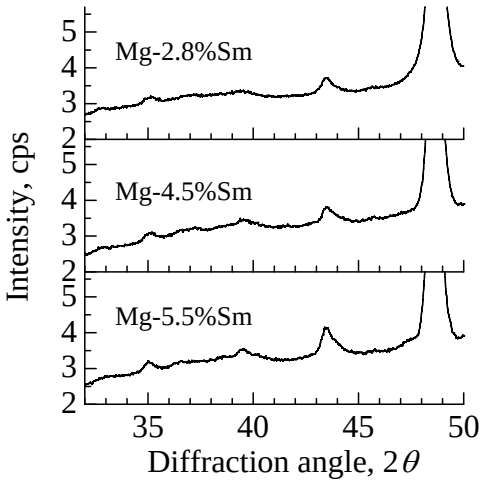

Fig. 6 Relation between Sm contents and $Mg_{41}Sm_5$ phase.

where, $C=\lambda^2/3a_0^2$, $\alpha=(h^2+k^2+l^2)$, $B=\lambda^2/4c_0^2$, $\gamma=l^2$, $A=D/10$, $\delta=10\sin^2 2\theta$. Parameters h, k, l are Miller index and λ is a wavelength of X-ray radiation. Calculation results are a_0=3.214Å, c_0=5.212Å at 2.8%Sm, a_0=3.215Å, c_0=5.210Å at 4.5%Sm and a_0=3.213Å, c_0=5.210Å at 5.5%Sm, respectively. Thus, any specimen was almost the same as the lattice constants. Figure 5 shows changing the diffraction profile of $Mg_{41}Sm_5$ phase according to HPT processing. The diffraction intensity becomes high after HPT processing. This fact described that the Mg solid solution decomposition is cause by SPD processing. Here, the Mg lattice constants are calculated using these profile data sets again, and results are a_0=3.214Å, c_0=5.208Å at 20°C HPT and a_0=3.212Å, c_0=5.209Å at 200°C HPT, respectively. Thus, the shortening of *c*-axis in lattice reflects the Mg solid solution decomposition though a detailed estimation of the error is necessary. The microhardness of the specimen has been measured by one of the authors [6]. Results are summarized in table 2. The hardness became high as the Sm content increased. The hardness also increased after HPT. Therefore, the hardness of Mg-Sm alloys mainly could be developed by the Mg solid solution decomposition generated on SPD processing.

Table 2 Microhardness of the Mg-Sm alloys after SPD[6].

	HV, kg/mm^2		
	Without SPD	20°C SPD	200°C SPD
Mg-2.8%Sm	76.8	90.6	100.7
Mg-4.5%Sm	78.4	100.6	104.3
Mg-5.5%Sm	88.4	107.7	109.4

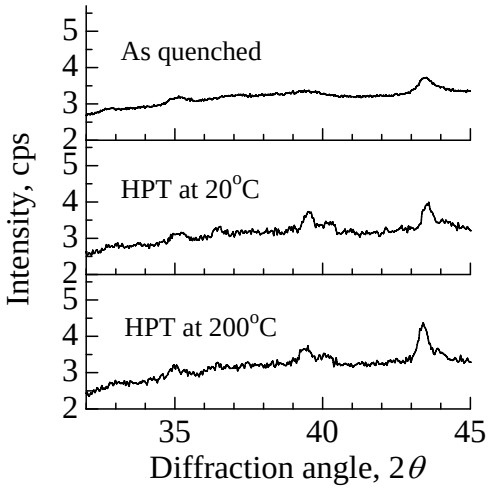

Fig. 7 Relation between HPT processing and $Mg_{41}Sm_5$ phase (Mg-2.8%Sm).

Summary

The Mg-Sm alloys that were given the severe plastic deformation by the high pressure torsion processing were investigated using X-ray diffraction. The pole figures of the specimens were also measured by X-ray diffraction. The specimen before HPT processing had a preferred orientation by casting. After HPT, the point where the density was high locally disappeared. Therefore the dynamic recrystallization could occur by HPT. The behavior of the second phase particles during HPT on Mg-Sm alloys was investigated using the X-ray profile and the calculation of lattice constants of the Mg phase. As a result, HPT intensifies the decomposition of Mg supersaturated solid solution.

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