Achieving Grain Refinement of Upsized Al–3Mg–0.2Sc (mass%) Round Rods Using Multi-Pass High-Pressure Sliding (MP-HPS)*¹

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In this study, the multi-pass high-pressure sliding (MP-HPS) process was applied for grain refinement of Al–3Mg–0.2Sc (mass%) rods with an upsized dimension of 16 mm in diameter. To achieve a homogeneous microstructure throughout the cross-section, the rod sample was rotated with 60° around the longitudinal axis (MP-HPS-R) for three times. A microstructure with an average grain size of 280 nm was developed around the center of the cross-section through the MP-HPS-R process. Superplasticity with total elongations of more than 400% was achieved in the 9 mm diameter range on the cross-section of the MP-HPS-R-processed rod. [doi:10.2320/matertrans.L-M2020828]

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1. Introduction

Severe plastic deformation (SPD) is attracting much attention because of the potential for achieving grain refinement to the submicrometer or nanometer level and thus leading to a significant improvement of the mechanical properties.^{1–5)} However, for practical application, scaling up of the SPD sample is still a challenging task. Recently, it was demonstrated that the high-pressure sliding (HPS) is an effective process for grain refinement in a form of strips with homogeneous microstructures.^{6–9)} The samples size was further increased when the HPS process was combined with sample feeding for sheet forms, as named incremental feeding HPS (IF-HPS),^{10,11)} and with sample rotation around the longitudinal axis for rod forms, as named multi-pass HPS with rotation (MP-HPS-R).^{12,13)} Both the IF-HPS and MP-HPS-R processes are promising for practical applications. Nevertheless, for the MP-HPS-R process, rods with a maximum of 10 mm in diameter were thus far processed and a homogeneously strained region with only a 6 mm diameter was attained around the center of the rod.

In this study, therefore, we challenge to apply the MP-HPS-R process for grain refinement of rods with a bigger diameter as 16 mm. An Al–3Mg–0.2Sc (mass%) alloy is used for this experiment as the alloy exhibits superplasticity when the grain size is refined by SPD processes and the total elongation can be regarded as a degree of the grain refinement.^{14–17)} Microstructure observation is performed using transmission electron microscopy (TEM) and mechanical properties are evaluated using micro-hardness measurement and tensile testing. It is shown that upsized rods with ultrafine-grained structures are successfully produced while maintaining the fundamental characteristics associated with SPD processing.

2. Experimental

This study used an Al–3Mg–0.2Sc (mass%) alloy in a form of rods with 16 mm diameter and 40 mm length. Each rod was solution-treated at 873 K for 3.6 ks (1 h) followed by quenching in water. MP-HPS-R was conducted at room temperature under an applied pressure of 1.0 GPa at a sliding speed of 1 mm/s for a sliding distance of 15 mm. To achieve a homogeneous development of microstructure on the cross section, the straining process using the MP-HPS-R followed the rotation procedure as illustrated in Fig. 1:⁹⁾ shear strain was introduced at the mid height of the cross section through the first sliding operation, then the rotation by 60° around the longitudinal axis followed for the second sliding operation and another rotation by 60° for the third sliding operation. Because this study used anvils in a closed type at both longitudinal ends to increase constraining condition, each sliding operation consists of forward and backward reciprocal motions to keep the rod shape as its original.⁹⁾

Specimens for hardness measurements and tensile tests were cut around the mid length along the longitudinal direction as depicted in Fig. 2(a). Vickers microhardness was measured on the cross sections, as shown in Fig. 2(b), with a polished mirror surface covering the entire area with 0.5 mm apart each other using a microhardness tester (Mitutoyo HM-102) with an applied load of 4.9 N for a dwell time of 15 s with an accuracy of ±2 HV. The tensile specimens were extracted as indicated in Fig. 2(b) with the dimensions of 0.6 mm gage length and 0.7 mm thickness shown in Fig. 2(c).

Fig. 1 Processing approaches for three passes: with 60° rotation around longitudinal axis.

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Fig. 2 Schematic illustrations of (a) sampling location for Vickers microhardness measurement and tensile testing, (b) positions for Vickers microhardness and cross sectional region at gauge part of tensile specimen, (c) dimensions of tensile specimen.

Fig. 3 Hardness variation throughout cross section at center of rod sample after MP-HPS-R processing with sliding distance of 15 mm.

Tensile tests were conducted with an initial strain rate of $3.0 \times 10^{-3} \text{ s}^{-1}$ at 573 K. Microstructures of the HPS-processed rods were observed using TEM (Hitachi H-8100) at an accelerating voltage of 200 kV. The disks for TEM were ground mechanically to a thickness of 0.15 mm and further thinned by a twin-jet electropolishing technique using a mixture of 10 vol% perchloric acid, 20 vol% glycerol and 70 vol% methanol at $\sim 253 \text{ K}$.

3. Results and Discussion

Figure 3 shows hardness variations on the cross section after processing by the MP-HPS-R following the procedure (6 passes through reciprocal motion) given in Fig. 1. A mean hardness of $\sim 140 \text{ HV}$ was measured around the center of the cross section, and otherwise, it is of $\sim 110 \text{ HV}$, which is approximately twice as high as the value of $\sim 60 \text{ HV}$ in the solution-treated state. Nevertheless, the hardness variation appears to be almost homogenous in the angular direction and this is owing to the 60° rotation around the longitudinal axis.

Fig. 4 Results of tensile testing after MP-HPS-R processing with initial strain rate of $3 \times 10^{-3} \text{ s}^{-1}$ at 573 K: nominal stress-nominal strain curves for sliding distance of 15 mm and appearance of tensile specimens after deformation to failure including undeformed specimen.

Fig. 5 Total elongation plotted against distance from center in cross section of rod. Tensile tests performed at $3 \times 10^{-3} \text{ s}^{-1}$ at 573 K.

Figure 4 shows the nominal stress-strain curves and appearance of tensile specimens after deformation to failure with an initial strain rate of $3.0 \times 10^{-3} \text{ s}^{-1}$ at 573 K. Superplasticity was well attained with the elongations more than $\sim 400\%$ and with smooth and uniform deformation along the gage length. The superplasticity appeared in the specimens taken from the positions marked ③, ④, ⑤, ⑥, ⑦, ⑧ in Fig. 2(b). Note that the elongation of the specimen from position ⑤ is more than 1400%, but the elongations of the specimens from positions ①, ②, ⑨ near the periphery are of 170%, 240%, and 120%, respectively. These features are consistent with the hardness distribution shown in Fig. 3 and in particular the advent of superplasticity corresponds to the areas where the hardness is higher and thus the grain refinement was better achieved at the center on the cross section.

Figure 5 shows the total elongation plotted against the distance from the center on the cross section. The elongation decreases as the distance is away from the center. The superplastic elongation more than $\sim 400\%$ (dotted line in Fig. 5) is attained around the center within a range of 9 mm

Fig. 6 TEM bright-field image (left) and dark-field image (right) with selected-area electron diffraction patterns (center) after MP-HPS-R processing for sliding distance of 15 mm.

diameter. This superplastic range is twice as large as the earlier report.¹³⁾ Nevertheless, the highly strained region with the advent of superplasticity is confined in the central region which is approximately a half of the rod diameter. This inhomogeneity of strain distribution occurs due to the fact that the introduction of strain around the center is more likely to be overlapped during the three consecutive sliding processes but in the outer areas, the overlap is less likely to occur because of the increasing separation between the sheared zone. Thus, to enlarge the homogeneously strained region, it is suggested that the sliding operation should be performed with a smaller angular rotation as 45° .

Figure 6 shows TEM bright-field image (left) and dark-field image (right) with selected-area electron diffraction (SAED) patterns (center) after MP-HPS processing through the sliding distances of 15 mm in the Al–Mg–Sc alloy. The dark-field images were taken using the diffracted beams indicated by the arrows in the SAED patterns. Diffraction spots in the SAED patterns form well-defined rings, indicating that most of the grain boundaries are of high-angle nature. The average grain size of the specimens with the sliding distance of 15 mm is 280 nm.

4. Conclusions

In this study, grain refinement of an Al–3Mg–0.2Sc (mass%) rod was successfully processed by MP-HPS-R. At the center of the cross section, the grain size was reduced to 280 nm and the hardness increased to ~ 140 HV. The Al–Mg–

Sc rod after processing by MP-HPS-R exhibited superplasticity with total elongations more than $\sim 400\%$ around the center of the cross section within a range of 9 mm diameter.

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