Enhanced Age Hardening in an Al-Mg-Si Alloy Using High-Speed Compression

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The age hardening of an Al-Mg-Si alloy was enhanced by the effect of high-speed (10⁶ s⁻¹) compression prior to aging. This enhancement of the age hardening is brought about by the formation of vacancy clusters during high-speed compression. High resolution transmission electron microscopy reveals that these vacancy clusters form stacking fault tetrahedra. Following peak aging, vacancy clusters and aging precipitates coexist in the grain interior. The high aging hardness obtained following high-speed compression is most probably due to the combined effects of vacancy and precipitation hardening. [doi:10.2320/matertrans.L-M2015810]

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1. Introduction

Since aluminum and aluminum alloys have a higher stacking fault energy than other metals, vacancy clusters arranged as stacking-fault tetrahedra (SFT) should rarely arise from dislocations under deformation at conventional speeds, i.e. with a strain rate lower than 10⁻¹ s⁻¹.¹ However, Kiritani et al.² have reported that a large number of SFT vacancy clusters are generated, even in aluminum and aluminum alloys, under high-speed deformation, corresponding to strain rates greater than 10⁴ s⁻¹.³ They have also shown experimentally that the multiplication of dislocations was highly suppressed with vacancy clusters formed by high-speed compression.⁴ They claimed that these kinds of vacancy clusters are formed under a high internal stress field inside the aluminum matrix, thereby restricting the movement of dislocations.

A number of industrial manufacturing processes are available to control the microstructure of materials using the plastic deformation, such as forging and extrusion, amongst others. In practice, these processes are usually performed at low deformation speeds (< 10⁻¹ s⁻¹) and the mechanical properties of the materials are generally enhanced by dislocation (strain) hardening. However, low-speed plastic deformation is widely known to have a negative effect on precipitation hardening. This is because the dislocations introduced during low-speed deformation act as inhomogeneous coarse precipitation sites, reducing the number of fine and homogeneous precipitates. A perfect synergy between dislocation hardening and precipitation hardening is therefore difficult to achieve.

To date, few studies have been devoted to exploiting the effects of vacancy clusters induced by high-speed deformation to control the microstructure of aluminum alloys. Since the SFT induced by high-speed compression are expected to be small (on the nanometer scale),⁵ SFT should have much less of a detrimental effect on precipitation hardening than dislocations do. Additionally, the SFT themselves may afford strengthening in a manner similar to void hardening.⁶ The present paper examines the effects of high-speed deformation on the microstructural evolution and age-hardening behavior of Al-Mg-Si-base aluminum alloys.

2. Experimental Procedure

A 6061 aluminum alloy rod (Al-1.0Mg-0.68Si-0.28Cu, in mass%) was used as a starting material. Disk specimens 10 mm in diameter and 2 mm thick were cut from the original rod. The specimens were solution-heat-treated at 530°C for 90 min and quenched in water. The specimen surfaces were polished using emery paper (1200 grade) and buffed with an aqueous solution containing 0.3 µm alumina particles.

The disk specimens were placed on a jig and compressed at high speeds via the planar collision of projectiles from a single powder gun, as illustrated in Fig. 1. The projectiles consisted of a brass (φ 15 mm × 5 mm) bottom and a polyethylene body (φ 15 mm × 20 mm) containing a ferrite magnet (φ 7 mm × 4 mm), the latter allowing the projectile speed to be measured through the variations of the induced electromagnetic forces. The speed of the projectile was adjusted to between 450 m/s and 670 m/s, corresponding to strain rates of 2.3 × 10⁴ s⁻¹ and 3.4 × 10⁵ s⁻¹, respectively. The interval between the solution heat treatment and the high-speed compression lasted no more than 0.5 h. To compare the effects of different deformation speeds, disk specimens swaged by 30% at room temperature were also prepared as low-speed deformation standards. The solution-ized specimens without and with deformation were respectively aged at 100°C and 175°C in an air furnace. Age-hardening curves were obtained by following the variation of the Vickers hardness as a function of the aging time. For the transmission electron microscopy (TEM) observations, the surface of the disk specimens was polished with emery paper to a thickness of 0.5 mm. The polished disk specimens were punched out with a diameter of 3.0 mm, electropolished using the twin jet method in an aqueous solution (HNO₃ : CH₃OH = 1 : 4), and then cooled to between −40°C and

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—20°C using liquid nitrogen. The TEM observations were performed with a JEM-2100F microscope operating at 200 kV. Small tensile test pieces were also cut from the disk specimens without and with high-speed deformation by electro-discharge machining. These were 2.9 mm in gauge length, 1.0 mm wide, and 3.0 mm thick. Tensile tests were carried out at room temperature with a strain rate of 3.4 × 10⁻³ s⁻¹. The fracture surfaces after the tensile tests were observed by scanning electron microscopy (SEM).

3. Results and Discussion

Figure 2 presents the age-hardening behavior tested at 100°C or 175°C of 6061 aluminum alloy samples compressed at high-speed after solution heat treatment, where a part of data was shown in the previous paper. The age-hardening curves obtained for cold-swaged (low-speed deformed) and non-deformed specimens are also shown for the sake of comparison. The deformed samples are more than twice as hard as the original ones. The increase in hardness, ΔHV, due to the deformations is estimated to be 48–60 Hv. Initially, the increase in hardness is similar regardless of the deformation speed; however, following age-hardening at 100°C or 175°C, the dependence on the deformation speed becomes apparent. Indeed the hardness of the specimens deformed at high-speeds is clearly enhanced. With high-speed deformation, maximum hardnesses of 158 Hv and 136 Hv are obtained following peak aging at 100°C and 175°C, respectively. This difference should be due to the...
different solubility of the aging precipitates at these temperatures. In contrast, the aging-induced increase in hardness is much smaller for the specimens deformed at low speeds, and these samples are of a similar hardness to those obtained by aging without prior deformation. This suggests that the dislocations induced by low-speed deformation suppress age hardening.

Figure 3 shows magnified TEM images of 6061 aluminum alloy samples prepared without and with high-speed deformation ($10^5$ s$^{-1}$) after solution heat treatment, where a part of data was shown in the previous paper. Both coarse (> 10 µm in diameter) and fine (< 1 µm in diameter) grains are observed in Fig. 3(b) for the specimen deformed at high speed ($10^5$ s$^{-1}$). However, the effect of high-speed deformation on grain refinement is clearly weaker than obtained previously through equal channel angular pressing (ECAP) and high-pressure torsion (HPT). In addition, the dislocations do not accumulate in large numbers in spite of the high axial strain (0.5–0.6) induced by high-speed deformation. It is evident nonetheless that a significant increase of age hardening is brought about in these specimens with relatively little grain refinement. In addition, a number of white particles 2–10 nm in diameter are densely distributed inside the grains as shown in Fig. 3(d), which are not seen in Fig. 3(c) for the non-deformed specimen. Since these TEM observations were conducted at an acceleration voltage of 200 kV, these may be the result of electron beam radiation. However, these stacking fault tetrahedra were visible from the very beginning of the observations and did not change in morphology during several minutes of irradiation. This therefore suggests that the white particles observed in Fig. 3(d) are aggregates of vacancy clusters introduced by high-speed deformation.

Figure 4 shows the microstructure obtained by high resolution TEM of a 6061 aluminum alloy prepared with high-speed deformation ($2.3 \times 10^5$ s$^{-1}$) after solution heat treatment. A magnified image of the vacancy cluster reveals a triangular arrangement when viewed from the [110] direction. This suggests that triangular SFT are formed after high-speed deformation. The authors believe that the vacancy clusters observed here are similar to the SFT described by Kiritani.

Figure 5 shows the microstructure of the 6061 aluminum alloy specimen prepared with high-speed deformation ($2.3 \times 10^5$ s$^{-1}$).
10^5 s^{-1}) plus peak aging at 175°C for 8 ks (Hv = 136). The bright field image in Fig. 5(a) shows that even after peak aging at 175°C, the vacancy clusters remain distributed densely inside the grains with their shape and size almost unchanged. This suggests that the vacancy clusters themselves are not responsible for the hardening, having weak diffusivity even at 175°C. In the dark field images of Fig. 5(a), aging precipitates are homogeneously distributed inside the grains. Based on the diffraction pattern and its bright field image, the aging precipitates in Fig. 5(b) are believed to be β′-Mg2Si phases. Thus, the microstructures shown in Fig. 5 suggest that the high hardness obtained through high-speed deformation plus aging (Hv = 136 at 175°C) is the synergistic result of vacancy and precipitation hardening. In this context, Kim et al.9) found that a 6061 aluminum alloy processed by ECAP showed no age-hardening at 175°C. Furthermore, Masuda et al.10) have shown that the hardness of a 6061 aluminum alloy processed by HPT did not change when aged at 170°C. Both for ECAP and HPT, aging precipitates presumably nucleate and grow preferentially at the grain boundaries of the nanograins. On the other hand, the microstructures in Fig. 5 demonstrate the coexistence of vacancy clusters and aging precipitates, which is clearly different from the results obtained for the aged ECAP- or HPT-processed samples.9,10)

Figure 6 shows stress-strain curves obtained for the specimens prepared without and with high-speed deformation. The data in Fig. 6 represent the highest of the three measurements performed on each sample. The yield stress and tensile strength of the specimen processed with high-speed deformation are 395 MPa and 420 MPa, respectively, compared with 285 MPa and 340 MPa without. The yield stress and tensile strength of the specimen processed with high-speed deformation are both much higher than the standard values for the 6061-T6 aluminum alloy.13) High-speed deformation reduces the fracture strain from 0.23 to 0.15, and the specimen processed with high-speed deformation indicates slightly low ductility.

Figure 7 shows the fracture surfaces after the tensile tests of the specimens processed without and with high-speed deformation. In the low-magnification images (Figs. 7(a), 7(b)), the center of both samples is covered mostly with dimple patterns; however, shear lips are discernable on the edges of the deformed sample only. The dimples in the two samples are of a similar size. This indicates that conventional ductile fractures occur in both samples, accompanied by the nucleation and growth of voids.

Since increased age hardening is brought about here through the superposition of two different hardening mechanisms—namely vacancy hardening and precipitation hardening—the effect of the vacancy clusters on the strength of the alloy is discussed in the following using eq. (1), proposed to explain for void hardening, \( r_v \).14,15)

\[
\sigma_v = M r_v = \frac{GbM}{2\pi L_v} \left[ \ln (D^{-1} + L_v^{-1})^{-1} + B \right]
\]

Here, \( G \) is the shear modulus (28 GPa for aluminum), \( b \) is the magnitude of the Burgers vector (0.28 nm), \( M \) is the Taylor factor for fcc materials (3.06), \( D \) is the average diameter of obstacles, \( L_v \) is the distance between voids, and \( B \) is a material constant, 1.52 for voids.14) Based on the morphology shown in Fig. 5(a), a \( \sigma_v \) value of 116 MPa is obtained with \( D \) and \( L \) taken respectively to be 5 and 50 nm. Figure 6 shows that the actual increase in the yield stress caused by high-speed deformation is \( \Delta \sigma_v = 110 \) MPa, almost the same as the calculated value, 116 MPa. This means that eq. (1) can represent well the increase of the yield stress in the present case. An accurate evaluation of the superposition of the two different hardening mechanisms is beyond the scope of this study; however, the interaction between vacancy clusters and precipitates should affect the superimposed hardening.
4. Summary

The effects of high-speed compressive deformation were studied on the age hardening and microstructure of 6061 aluminum alloys. The results obtained may be summarized as follows. (1) Dense aggregates of vacancy clusters were observed inside grains in the 6061 aluminum alloy sample deformed at high speed (at a strain rate of $10^5 \text{s}^{-1}$) following solution heat treatment. (2) Hardening was evidenced following aging at both 100°C and 175°C, even for the samples processed by high-speed deformation. (3) The high hardness of the samples processed using the high-speed deformation ($10^5 \text{s}^{-1}$) and then aged, is the result of the coexistence of vacancy clusters and precipitates.

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