Effect of High-Speed Impact Compression on Natural Aging and Subsequent Artificial Aging of a 6061 Aluminum Alloy*1

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The authors have shown that vacancy clusters were induced using high-speed impact compression. The suppression of natural age-hardening was clearly seen in the alloy with high-speed impact compression. TEM observation showed that fine precipitates were formed due to artificial aging even after high-speed impact compression and natural aging. Maximum hardness of the peak-aged alloys with high-speed compression was almost the same as that without natural aging, showing that negative effect of two step aging was almost overcome by high-speed impact compression.

Keywords: aluminum-magnesium-silicon alloy, point defects, vacancy, high-speed deformation, age-hardening

1. Introduction

To enhance the mechanical properties of structural materials, strain hardening has been industrially applied using manufacturing technologies such as rolling, forging, and extrusion to induce dislocations in the material. Recently, it has been clarified that very large strain working (logarithmic equivalent strain of 4–5 or more) in metals such as severe plastic deformation (SPD) produces ultra-fine grain microstructures and significantly improves the mechanical properties. There have been many reports on aluminum alloys using the very large strain working technique1–4. It has also been reported that the movement of dislocations is restricted and the vacancy clusters (point defect clusters) are formed by high-speed compression within metallic materials using rapid planar impingement of flying objects (∼100 m/s)5–9. The production of high-functional materials can be expected by controlling the microstructure with the point defects introduced by high-speed plastic deformation.

The authors have shown that vacancy clusters were induced by high-speed impact compression on the order of 10⁵/s in aluminum alloys, and the hardness and tensile strength were significantly increased by precipitation strengthening during subsequent heat treatment10–12. Recently, to achieve weight reduction of automobile bodies, Al–Mg–Si alloys have been applied to the body sheet of automobiles. This is intended to utilize the bake hardenability characteristics of the Al–Mg–Si alloy to achieve strength by age hardening during paint baking. However, it is widely recognized that Al–Mg–Si alloys exhibit a complicated two-step aging behavior. There is a negative effect that cluster (1) is formed when holding at room temperature after solution heat treatment, and this cluster has difficulty growing into the β′ phase during the subsequent aging heat treatment13,14. To control the negative effects, various improvements such as pre-strain15, modification of the alloy composition and the addition of elements16,17, and heat treatment18 can be used. Vacancy clusters induced by high-speed impact compression can be regarded as a type of strain; therefore, these clusters affect the natural aging behavior and subsequent artificial aging behavior of Al–Mg–Si alloys. However, the relationship between high-speed impact compression and natural aging or subsequent artificial aging is not yet clear. Thus, in this study, the effects of high-speed impact compression on the natural aging behavior and subsequent artificial aging behavior of an Al–Mg–Si alloy were evaluated.

2. Experimental Procedure

The 6061-T6 extruded rod (φ10 mm) used in this study was provided by Furukawa Sky Co., Ltd. (currently UACJ Corporation). Table 1 lists the chemical composition of the alloy. The extruded rod material was cut into disk-shaped test pieces (φ10 mm, 2-mm thickness). The specimen surfaces were polished using emery paper (1200 grade) and an aqueous solution containing 0.3-μm alumina particles. The high-speed impact compression apparatus used in this study is shown in Fig. 1. More information about the high-speed impact compressive deformation method is provided in previous works10–12. The disk specimens were then impact-com-

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pressed using a single stage powder gun. The projectile used was approximately 12 g and consisted of a brass bottom and polycarbonate body containing a ferrite magnet. After setting the blasting cap located on top of the gun barrel, smokeless gunpowder was exploded. The projectile was accelerated with the explosion of gunpowder and collided with the disk specimen. Figure 2 shows the sample before and after high-speed impact compression. After high-speed impact compression with an initial strain rate of 2.3 × 10⁵/s, the thickness was reduced from 2.0 to 0.87 mm, and the diameter was increased from 10 to 15–16 mm. The impact pressure was approximately 5 GPa based on the impedance match method used in this study. The sample names for each process in this study are listed in Table 2. All the samples were solution heat-treated at 803 K for 5.4 ks followed by quenching in water. After that, the samples underwent (1) aging at 448 K; (2) natural aging at room temperature for 600 ks followed by aging at 448 K; (3) high-speed impact compression (5.4 GPa) and aging at 448 K; or (4) high-speed impact compression (4.7 GPa), natural aging at room temperature for 600 ks, and subsequent aging at 448 K. These samples are designated as (1) SA (solution + aging), (2) SNA (solution + natural aging + aging), (3) SIA (solution + impact + aging), and (4) SINA (solution + impact + natural aging + aging) alloys, respectively.

Hardness tests were performed with a load of 300 gf and time of 20 s using an Akashi MVK-G1 Vickers hardness testing machine. Measurements were performed at 5 locations, and the average value was calculated. Transmission electron microscopy (TEM) samples were prepared using the twin jet electrolytic polishing method. TEM observation was performed at an accelerating voltage of 200 kV using a Hitachi H8000 and JEOL JEM-2100F. Differential scanning calorimetry (DSC) was performed from −50°C to 500°C at a heating rate of 0.17 K/s in an Ar atmosphere using a Rigaku DSC8230; the standard material used was 99.99% pure aluminum.

3. Results and Discussion

3.1 Natural aging and subsequent age-hardening behavior of 6061 aluminum alloy subjected to high-speed impact compression

Figure 3 presents the hardness results for the SNA and SINA alloys. The hardness of the SNA alloy increased monotonically with aging time, with an increase of 18 HV after aging for 600 ks, because cluster (1) was formed during natural aging. In contrast, the hardness of the SINA alloy first increased by 47 HV with the high-speed impact compression. Then, the hardness increased by 10 HV during the subsequent natural aging for 600 ks, which is approximately half the increase for the SNA alloy. This finding indicates that the subsequent formation of cluster (1) was suppressed by high-speed impact compression.
The hardness changes of the samples subjected to each process are shown in Fig. 4. The SA alloy was peak-aged at 20 ks and exhibited a hardness of 119 HV. The hardness increased during natural aging in the SNA alloys, as observed in Fig. 3. Then, the subsequent age hardening at 448 K resulted in a hardness of 100 HV for the peak-aged condition, which was less than that of the SA alloy. This result demonstrates the negative effect of room-temperature aging. In addition, the prolonged time to peak aging was also recognized.

In the SIA alloy, the hardness first increased to 93 HV after impact compression and then reached 136 HV after peak aging; the maximum hardness was almost the same as that of the SA alloy. The peak aging time was also shortened. In the SINA alloy, natural aging after high-speed impact compression first occurred, as illustrated in Fig. 3. The hardness reached 137 HV after peak aging at 448 K, which is almost the same peak hardness as that of the SIA alloy.

The formation behavior of cluster (1) was evaluated using thermal analysis. Figure 5 presents the DSC measurement results. Data for 9M6S (Al–1.009Mg–0.586Si, mol%) \(^{17}\), for which the Mg and Si contents are almost the same as those in the present study, are also shown for comparison. The endothermic peak near 500 K (indicated by arrows) represents the solid-solution heat of cluster (1) \(^{19}\), and the higher endothermic peak indicates that more cluster (1) was formed. The amount of cluster (1) in the SA alloy (precipitated \(\beta^t\) at 448 K) is very small; however, the SNA alloy exhibited a large endothermic peak because of the larger amount of cluster (1). The endothermic peak in the SINA alloy is smaller than that of the SNA alloy, which indicates that the formation of cluster (1) of the SINA alloy is less than that of the SNA alloy; this finding is in good agreement with the hardness results. These results indicate that high-speed impact compression after solution heat treatment results in almost the same age hardening after natural aging for 600 ks as that without natural aging, and the negative effects of natural aging were suppressed.

### 3.2 TEM microstructure of 6061 aluminum alloy subjected to natural aging after high-speed impact compression

Figure 6 presents TEM images of the as-quenched alloy, SA alloy, and high-speed impact-compressed alloy. In the as-quenched alloy, a few dislocations and the intermetallic compound added for grain refinement were observed (Fig. 6 (a)). In the SA alloy, a high density of \(\beta^t\) was finely precipitated in the matrix (Fig. 6 (b)). In the high-speed impact-compressed alloy, dislocations introduced by the high-speed impact compression were observed, whereas the dislocation density was not as high as that of the alloys prepared by low-speed plastic working \(^{9,12,20}\) (Fig. 6 (c)). A nanometer-scale grain size was sparsely observed; however, the amount was not as large as that observed using the SPD method. Previous studies have demonstrated the appearance of vacancy clusters and grain refinement with high-impact compression \(^{10–12}\).

Figure 7 presents a TEM image of the SIA alloy peak-aged at 448 K after high-speed impact compression. Figure 8 presents dark-field images of the peak-aged SIA and SINA alloys. In Fig. 7, the precipitation of fine \(\beta^t\) in the matrix is observed in addition to the heterogeneous nucleation at dislocations in the SIA alloy. In Fig. 8, no significant difference is observed in the formation of \(\beta^t\) or the precipitation density or size re-
3.3 Effect of high-speed impact compression on natural aging and subsequent 448 K aging

In 3.1 and 3.2, hardness measurements, DSC analysis, and TEM were performed on 6061 aluminum alloys that underwent natural aging and aging at 448 K after high-speed impact compression. Here, the hardness changes for the various processes are discussed. Vacancy clusters and dislocations are induced and grain refinement occurs during high-speed impact compression. Therefore, the hardness increase due to high-speed impact compression $\sigma_I$ can be expressed as

$$\sigma_I = \sigma_G + \sigma_V + \sigma_D. \quad (1)$$

Here, $\sigma_G$, $\sigma_V$, and $\sigma_D$ are the hardness increases associated with grain refinement, vacancy clusters, and dislocations, respectively. In this study, the hardness increase due to high-speed impact compression is summarized as $\sigma_I$ because it is difficult to divide $\sigma_I$ into $\sigma_G$, $\sigma_V$, and $\sigma_D$. However, the effect of $\sigma_G$ and $\sigma_D$ on $\sigma_I$ is considered to be small because high-speed impact compression produces less dislocations and grain refinement than low-speed plastic working$^{9,12,20}$ and SPD$^{12,21}$, respectively. As described in the introduction, in 6061 alloy, it is well known that cluster (1) and $\beta'$ are formed during natural aging and aging at 448 K, respectively. Therefore, the hardness increase due to natural aging and 448 K age-hardening are expressed as $\sigma_C$ and $\sigma_P$ due to cluster (1) and $\beta'$, respectively. Therefore, the hardness changes $\Delta HV$ due to each process can be expressed as

$$\Delta HV = \sigma_I + \sigma_C + \sigma_P. \quad (2)$$

Figure 9 presents the hardness changes for each process described in Fig. 4. For the SA alloy, the hardness increase is expressed by $\sigma_P$, and the hardness is 74 HV. For the SNA alloy, $\sigma_C$ is 14 HV for the natural aging, and $\sigma_P$ is 40 HV for subsequent 448 K aging. $\sigma_P$ of the SNA alloy is reduced...
Compared with that of the SA alloy, which indicates the negative effect due to natural aging of the alloy.

In the SIA alloy, the hardness increase and σ_f and σ_P are 48 and 40 HV, respectively. Although precipitation upon artificial aging in the SIA alloy was suppressed compared with that in the SA alloy, this value is almost the same as that for the SNA alloy. The total hardness increase ΔHV was observed to be larger than that of the SA alloy. Furthermore, the time for peak aging was shortened for the SIA alloy compared with that for the SA alloy. This result occurred because the precipitation was accelerated by heterogeneous nucleation on dislocations in addition to the precipitation in the matrix, as observed in Fig. 7. Therefore, the effect of high-speed impact compression on the aging behavior is that the formation of cluster (1) was suppressed by high-speed impact compression in the SINA alloy, as observed in Fig. 3 and Fig. 5. σ_P was 44 HV for the 448 K aging, which is approximately the same as that for the SNA and SIA alloys. Therefore, it can be observed that the formation of β' in the SINA alloy is substantially the same as that in the SIA alloy. This finding is in good agreement with the microstructural results presented in Fig. 8. In addition, the total amount of hardness increase ΔHV was similar as that for the SIA alloy. This finding clearly indicates that the negative effects of the two-step aging would not substantially occur after high-speed impact compression. In terms of the effect of work hardening on the natural and artificial aging of the alloy, it has been reported that in 5% cold-rolled samples subsequently exposed to natural aging, the hardness increase during artificial aging is reduced to 30% of that of the single-aged alloy. In contrast, the amount of σ_P in the SINA alloy was calculated to be 59% of that of the SA alloy in this study. This finding indicates that the high-speed impact compression method increases age-hardening compared with the conventional cold-rolling method, demonstrating the usefulness of this approach. Based on these findings, it can be clarified that performing high-speed impact compression after solution heat treatment results in almost the same age hardening as that observed for the alloy without natural aging, even after 600 ks, thereby suppressing the negative effects of the two-step aging process.

4. Conclusions

The effects of high-speed impact compression on the natural aging and subsequent artificial aging of a 6061 alloy were investigated. The following conclusions were drawn:

1) The suppression of natural age-hardening was clearly observed in the alloy that underwent high-speed impact compression after solution heat treatment compared with that that did not undergo high-speed impact compression.

2) By performing the high-speed impact compression after solution heat treatment, almost the same age hardening was achieved as without natural aging, even after natural aging for 600 ks, thereby suppressing the negative effects of the two-step aging of the alloy.

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