Near-Surface Nanocomposite Structure on Commercial Purity Aluminum Induced by Fine Particle Bombardment

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Commercial purity aluminum (A1070) was subjected to fine particle bombardment (FPB) with 1.0 mass% carbon steel and pure nickel projectile particles. Nanocomposite structures formed at the near-surface region, which contained refined aluminum grains of less than 100 nm in diameter and dispersed projectile fragments several tens of nanometer to several micrometer in size. Beneath the nanocomposite structure was found a fine-grained matrix with approximately 1-μm-diameter grains.

Observation of the microstructural transition at the near-surface region suggested that the nanocomposite structures developed via severe plastic deformation (SPD), accompanied by a folding and imposing of the convex part of the surface, and by mechanical mixing with the fragments of the projectile particles.

The nanocomposite structures exhibited high hardness values of approximately HV200, which are superior to those of extra-super duralumin (A7075-T6). The relationship between the grain size and hardness value in the structures corresponds well to the Hall–Petch relationship extrapolated from previous studies. Therefore, the increase in hardness is thought to be mainly the result of grain refinement strengthening.

1. Introduction

Aluminum alloys are used in vehicle bodies to reduce weight and improve fuel-efficiency due to their low specific gravity. However, since aluminum alloys are susceptible to fatigue, wear, and heat, they cannot be directly used in components that must resist these factors.

In the past, fiber-reinforcement and partial alloying using an electron beam\(^1\)\(^-\)\(^3\) were developed to improve wear and high-temperature resistance. However, there are few practical uses of these techniques due to their low throughput and high cost. Recently, techniques for grain refining through severe plastic deformation (SPD) have been reported for improving the mechanical properties of aluminum alloys\(^4\),\(^5\) such as accumulative roll bonding (ARB),\(^6\)\(^-\)\(^8\) equal-channel angular pressing (ECAP),\(^9\)\(^-\)\(^12\) and high-pressure torsion (HPT).\(^13\),\(^14\) However, practical use of these techniques has not yet been accomplished.

In this study, fine particle bombardment (FPB) was performed on commercial purity aluminum (A1070). FPB is a type of shot peening treatment that projects particles several tens of micrometer in diameter onto the surface of materials at more than 100 m/s. This method is expected to improve the fatigue property and wear resistance of a material by providing compressive residual stress and a dimple profile on the material surface. Additionally, FPB can be used in low-cost mass production, and can be focused on only the required area of a component or on parts with complicated shapes.

FPB can be used to refine the grains of a near-surface region using severe plastic deformation (NS-SPD), which is caused by asynchronous particle collisions. The formation of nanocrystalline structures by such change in grain size through FPB treatment has been reported for steel\(^15\)\(^-\)\(^24\) but there have been few reports on nanocrystalline structure formation on aluminum alloys.\(^4\) In the present study, by treating aluminum with FPB, we found unique structures that should be called nanocomposite structures near the surface. These structures have yet to be reported for other materials. In this paper, we examine in detail the formation and strengthening mechanisms of these near-surface nanocomposite structures.

2. Experimental

The specimens used in these experiments were prepared from annealed commercial purity aluminum (A1070-O), the standard chemical compositions of which are shown in Table 1, and were shaped into disks 20 mm in diameter and 5 mm thick. The surfaces of the specimens were polished using #2000 emery paper prior to the FPB treatment.

The FPB treatment was carried out using an air blast shot peening apparatus. Fine particles of 1.0 mass% carbon steel and pure nickel were used. Each particle was less than 53 μm in diameter. The shot pressure was 0.8 MPa, and the arc heights correlating with the impact force were 0.20 mmN for the carbon steel particles and 0.03 mmN for the pure nickel particles. A 10 s treatment time was used to cover more than 200% of the surface area. To evaluate the formation process of the nanocomposite structures, we also prepared specimens treated for 0.5 s and 2 s, respectively.

The near-surface composite structures induced by FPB were evaluated using an electron probe microanalyzer (EPMA; JEOL JXA-8500F) and a transmission electron microscope (TEM; TOPCON EM-002BF) equipped for energy dispersive spectroscopy (EDS; Thermo Fisher Scientific).

Table 1 Standard chemical composition of A1070 (mass%).

<table>
<thead>
<tr>
<th>Element</th>
<th>Cu</th>
<th>Si</th>
<th>Mg</th>
<th>Zn</th>
<th>Fe</th>
<th>Mn</th>
<th>Ti</th>
<th>Others</th>
<th>Al</th>
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<tbody>
<tr>
<td>A1070</td>
<td>&lt;0.04</td>
<td>&lt;0.2</td>
<td>&lt;0.03</td>
<td>&lt;0.04</td>
<td>&lt;0.25</td>
<td>&lt;0.03</td>
<td>&lt;0.03</td>
<td>&lt;0.03</td>
<td>&gt;99.70</td>
</tr>
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</table>
NORAN System 7). Thin foils for TEM observation were shaped using a focused ion beam apparatus (FIB; FEI FIB-2000). The hardness of the cross-sectional composite structures was measured using a micro Vickers hardness tester (Akashi MVK-H2-V32) at 0.049 N.

3. Results and Discussion

3.1 Observation of the structures formed through FPB treatment

Figure 1 shows cross-sectional backscattered electron micrographs of the near-surface region of FPB-treated A1070 using carbon steel and pure nickel particles. Continuous white regions approximately 10 µm thick were observed along the rough surfaces, which were formed by the particle collisions, as shown in Figs. 1(a) and (c). The magnified views in Figs. 1(b) and (d) reveal that the white regions are composite structures in which fragments of the projectile particles were dispersed. Plastic flows were observed in these composite regions. Therefore, this suggests that the composite structures were formed due to plastic deformation caused by the particle collisions. Note that although the projectile particles used in the FPB were less than 53 µm in diameter, the dispersoids in the composite structure were remarkably finer. The carbon steel projectiles produced dispersoids that were less than 2 µm in size, and the pure nickel projectiles produced dispersoids less than 5 µm in size, which included mechanically elongated shapes. Also, cracks were observed locally in the composite structures. Furthermore, we found that the aluminum matrix beneath the composite region was remarkably refined to a grain size of approximately 1 µm, as can be seen in Fig. 2. The fine-grained matrix region formed a layer approximately 15 µm thick and was separated from the nanocomposite structure by a well-defined interface.

Next, we demonstrate the nanoscale features of the composite structure region through the use of TEM. Figure 3 shows TEM micrographs and selected area diffraction patterns (SADP) of the composite structure. The black areas in Figs. 3(a) and (d) are fine dispersoids less than 100 nm in diameter, which were undetectable with the EPMA. Since each SADP taken from a 300 nm area had a ring-like shape, the matrix of this area is thought to consist of many grains with a large distribution of crystal orientations. The pure nickel particles afforded a finer-grained matrix than the carbon steel particles. The aluminum grain sizes in the matrix were evaluated for bright grains in dark-field TEM micro-
graphs using image analysis software. The grain sizes were calculated as the square root of the grain area. In the results, the average grain sizes from FPB using carbon steel and pure nickel particles were 83 and 36 nm, respectively. Since the matrix grains of the nanocomposite structures were refined to less than 100 nm, and a considerable number of dispersoids smaller than 100 nm were observed within them, it should be emphasized that nanocomposite structures were formed by the FPB treatment.

Figures 4 and 5 show elemental maps of the nanocomposite structures obtained through our TEM-EDS observations. The dark regions shown in Figs. 4(a) and 5(a) are typical examples of the dispersoids, which consist of only the bombarded element, Fe or Ni, and do not contain Al or O.
Comparing Figs. 4(b) and 4(c), as well as Figs. 5(b) and 5(c), the contrasts can be clearly seen to complement each other. This implies that intermetallic aluminum compounds were not formed using the projected element. Furthermore, as shown Figs. 4(d) and 5(d), oxygen was indentified in layers within the matrix. Therefore, because oxygen was undetected at the dispersoid region, it was presumed that the oxygen was dissolved or dispersed within the matrix as aluminum oxide.

3.2 Formation process of the nanocomposite structure

First, we will examine the early stages of the FPB treatment. Figures 6 and 7 show cross-sectional backscattered electron micrographs of the near-surface regions of the FPB-treated specimens when pure nickel particles were projected for 0.5 s and 2 s, respectively. Although a rough surface was formed from the particle collisions, a nanocomposite structure was not formed during the shorter 0.5 s treatment. After the longer 2 s treatment, fine dispersoids were observed near the surface, as indicated by the arrow in Fig. 7(a), which shows that a nanocomposite structure was formed. In addition to the rough surface, the oxygen elemental map from the EPMA (Fig. 7(b)) revealed an oxygen-concentrated area embedded in the specimen that formed a band along the plastic flow. This implies that the
initial surface was folded and imposed by plastic deformation resulting from the FPB treatment, and that oxide film on the initial surface, or oxygen in the atmosphere, was taken into the nanocomposite structure.

On the basis of the microstructural transition observed, we propose the following formation mechanism for the nanocomposite structures (Fig. 8): A convex part formed on the rough surface at an early stage during the particle collisions, and was subsequently folded and imposed deeper into the matrix by subsequent particle collisions. FPB treatment provides multi-stage, multi-directional, and severe asynchronous plastic deformations. Thus, FPB treatment provides an extremely large amount of plastic strain in the folded and imposed regions, which leads to the formation of nanocrystalline structures.20–24) Figure 9 shows a cross-sectional backscattered electron micrograph of the near-surface region of a specimen FPB-treated for 10 s using carbon steel particles. Oxygen is detected only in the nanocomposite region and not in the fine-grained matrix (Fig. 9(b)). This proves that the folding and imposing process is essential to the formation of the nanocrystalline structures. Although oxygen may assist in the formation of nanocrystalline structures by suppressing the recovery and/or grain growth during FPB treatment, its role in the formation process has not been clearly detailed. On the other hand, in the fine-grained matrix region, a certain amount of grain growth occurs. A smaller amount of plastic strain or the absence of oxygen is thought to be the reason for this grain growth. Cracks in the nanocomposite region, shown in Fig. 1(d), are considered to be generated where incomplete bonding occurs during the folding and imposing processes.

This dispersion of fine particles from a FPB treatment has yet to be reported. We observed that the sizes of the dispersoids were remarkably finer than the projected particles even at early stages of the nanocomposite structure formation, as shown in Fig. 7(a), and we did not notice any large particles that were comparable in size with the projected particles. Therefore, it is believed that the projected particles were not simply embedded into the matrix, nor were they crushed after particle collision. Kameyama et al. reported that steel particles stick onto titanium alloy substrates during FPB.25) Similarly, the surface of the colliding particles were presumably stuck and peeled onto the aluminum substrate. Consequently, the stuck fragments were taken into the matrix as dispersoids.

### 3.3 Strengthening mechanism of the nanocomposite structures

Table 2 shows the Vickers hardness values of the nanocomposite structures obtained in the present study as compared to extra super duralumin (A7075-T6), which is one of the hardest aluminum alloys. Note that the nanocomposite structures formed through the FPB treatment were harder than A7075-T6. Also note that the nanocomposite structures formed from pure nickel particles were harder than those formed using carbon steel particles. Since the grain of the FPB-treated nanocomposite region using pure nickel particles was finer in the above TEM micrographs, it suggests that grain refinement strengthening contributed to a hardening of the nanocomposite structure. The relationship between grain size and hardness value is generally known as the Hall–Petch relationship.26,27) Figure 10 shows the relationship between Vickers hardness value (HV) and the inverse of the square root of the grain size ($D^{-1/2}$) for aluminum in a
nanocomposite structure revealed through a TEM micrograph. Tsuji et al., reported relationship between yield strength and the grain size of ARB-treated A1100.\textsuperscript{28} The solid line in Fig. 10 is calculated from the yield strength using the formula $\sigma_0 = 3.34 \text{ HV}$. The dotted line is an extrapolation of the solid line toward the fine-grain-size region. The hardness values for each nanocomposite structure correspond well to the Hall–Petch relationship in the fine-grain-size region. This implies that the principal strengthening mechanism of the nanocomposite structure was grain refinement strengthening caused by severe plastic deformation during the particle collisions.

4. Conclusion

An evaluation of near-surface nanocomposite structures formed on commercial purity aluminum by FPB treatment revealed the following results: The nanocomposite structures consist of aluminum grains less than 100 nm in size and dispersoids tens of nanometer to several micrometer in size. The evaluation also revealed that the nanocomposite structures were formed through a multi-stage, multi-directional, and severe asynchronous plastic deformation, which was caused by folding and imposing during the particle collisions. The nanocomposite structures exhibited high hardness values of approximately HV200, which is superior to those of extra-super duralumin. The relationship between grain size and hardness value corresponds well with the Hall–Petch relationship extrapolated from previous studies. The high hardness value of the nanocomposite structures was mainly caused by grain refinement strengthening.

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REFERENCES


Table 2  Vickers hardness values of the nanocomposite structures obtained in the present study compared with extra-super duralumin.

<table>
<thead>
<tr>
<th>Measurement area</th>
<th>Nano composite microstructure</th>
<th>Extra-super duralumin (A7075-T6)</th>
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<tbody>
<tr>
<td>FPB-treated with carbon steel particle</td>
<td>min. 155.4</td>
<td>ave. 200</td>
</tr>
<tr>
<td>Pure nickel particle</td>
<td>max. 299.0</td>
<td>ave. 159.1</td>
</tr>
</tbody>
</table>

Fig. 10 Relationship between the Vickers hardness value (HV) and inverse of square root of the grain size ($D^{-1/2}$) for aluminum.