3-Dimensional Microstructural Evaluation of Wear-Induced Layer in Al–Al₃Ti Functionally Graded Materials by Serial Sectioning

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The wear behavior of Al–Al₃Ti functionally graded materials (FGMs) ring was investigated by 3-dimensional microstructural observations. The Al–Al₃Ti FGMs ring was cast which contained platelet-shaped Al₃Ti particles. The plane normal of the particles was parallel to the radial direction of the ring. The wear resistance of the Al–Al₃Ti FGMs ring depended on the sliding direction of the wear tests because of the anisotropic distribution of the Al₃Ti platelet particles. A wear-induced layer with a fine microstructure was formed just below the worn surface, and the formation behavior of the layer also depended on the sliding direction. However, the equivalent Hencky strain required to form the wear-induced layer was about 5, regardless of sliding direction. The anisotropy of the wear resistance in the Al–Al₃Ti FGMs ring and the formation behavior of the wear-induced layer were explained by the shear strain distribution on the worn surface.

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1. Introduction

When metallic materials undergo wear, a huge shear strain is introduced on the worn surface; consequently, a subsurface wear-induced layer is often formed just below the worn surface. The wear-induced layer has a nanocrystalline microstructure and is harder than that of a non-deformed region.

In previous studies, the wear behavior of Al–Al₃Ti functionally graded materials (FGMs) fabricated by centrifugal casting has been investigated. The Al–Al₃Ti FGMs casts were ring-shaped, and the volume fraction of the Al₃Ti platelet particles was oriented parallel to the radial direction of the FGMs rings. Figure 1 shows a schematic illustration of the Al–Al₃Ti FGMs ring, where the directions labeled WA, WB and WC indicate the sliding directions of the wear tests performed in previous studies. When the outer surface of the Al–Al₃Ti FGMs ring was worn in the WA sliding direction with an S45C plate, a wear-induced layer with 100 µm in thickness was generated just below the worn surface for a sliding distance of 100 m and an initial applied stress of 0.5 MPa. The wear-induced layer consisted of fine fibrous Al₃Ti particles and a nanocrystalline solid solution matrix containing a partly amorphized phase because of the heavy fragmentation of the Al₃Ti platelet particles. In addition, the wear-induced layer was formed at a nominal shear strain of greater than 90. However, the nominal shear strain required to form the wear-induced layer in the Al–Al₃Ti FGMs ring has been investigated for wear in only the WA sliding direction on the outer surface of the FGMs ring. The nominal shear strain for the formation of the wear-induced layer in the WB and WC sliding directions has not been reported.

To investigate the anisotropy of the wear-induced layer formation in an Al–Al₃Ti FGMs ring, it is necessary to determine the initial distribution of the Al₃Ti platelet particles and the change in distribution during wear, because the fragmentation and realignment of the Al₃Ti platelet particles should affect the shear deformation on the worn surface during the wear tests. However, the distribution of the Al₃Ti platelet particles on the worn surface is very complicated due to its fragmentation and realignment.

3-dimensional (3D) microstructural observation has been proposed as a microstructural analysis method for materials with complex microstructures. Zaefferer et al. have investigated the 3D microstructure of pearlite in an Fe–0.49%C alloy by using scanning electron microscopy (SEM)-electron backscattered diffraction (EBSD) with a focused ion beam (FIB). They analyzed the habit plane of the ferrite-cementite lamellae in the pearlite colony by precisely reconstructing the 3D microstructure of the pearlite.
In addition, they mentioned that the 3D microstructural observations with EBSSD were useful for determining the boundary plane. However, the 3D microstructural observation by SEM-FIB is not suitable for analyzing large areas. 

2.2 Preparation of Al–Al₃Ti FGMs ring

A commercial ingot of an Al–5mass%Ti alloy containing Al₃Ti platelet particles was used. Because the relative atomic masses of Al and Ti are 26.98 and 47.90, respectively, the volume fraction of Al₃Ti in this alloy was about 11 vol%. The Al–Al₃Ti FGMs ring was fabricated by centrifugal casting. The Al–5mass%Ti alloy ingot was melted at 900°C. Because the liquidus temperature is much higher than 900°C, the melt of Al–5mass%Ti alloy at 900°C consisted of the liquid Al and Al₃Ti platelet particles. The Al–5mass%Ti alloy melt was poured into a spinning cylindrical mold with an inner diameter of 90 mm. The applied centrifugal force in the spinning mold was \( G = 80 \), where \( G \) is expressed in units of standard gravity. The remaining Al₃Ti platelet particles moved in the direction of the centrifugal force in the Al melt because the densities of Al₃Ti and Al melt are 3.4 and 2.2 Mg/m³, respectively. The Al–Al₃Ti FGMs cast fabricated by centrifugal casting was ring-shaped with an outer diameter of 90 mm, a wall thickness of about 20 mm and a length of 30 mm.

2.2 Wear tests

The samples for the wear tests were cut from the outer region of the Al–Al₃Ti FGMs ring, where the Al₃Ti platelet particles were most concentrated. The samples had a cross section of 5 × 5 mm and were 10 mm in length.

The wear tests were performed using a block-on-ring machine under a rotating counter-disk. The wear plane and the sliding direction for each wear test are indicated by the arrows marked WA, WB and WC in Fig. 1. The counter-disk was a Fe–0.45 mass%C alloy with a Vickers hardness of 190. The surface of the counter-disk was mechanically polished using SiC paper and liquid Al₂O₃ before the wear tests. The wear tests were performed with an initial applied stress of 0.5 MPa and a sliding distance of 100 m to investigate the anisotropy of the wear resistance in the FGMs ring. The initial applied stress and the sliding distance of the wear tests for the microstructural analysis around the wear-induced layer were 2.2 MPa and 1000 m, respectively. This is because large shear strain is required to form the wear-induced layer. For the both wear tests, the sliding speed was fixed at 1 m s⁻¹.  

5mass% Al₃Ti FGM samples were observed by OM for a cross section parallel to the sliding direction and perpendicular to the worn surface. In order to reconstruct the 3D distribution of the Al₃Ti particles and the wear-induced layer, serial sectioning was performed by repeating the polishing and the OM microstructural observation. The alignments of the OM images and the sectioning space were adjusted using the size of the Vickers indent. The sectioning space was ~10 μm. The 3D distribution was reconstructed from the sectioning images with commercial reconstruction software (AVIZO). The crystal orientation distribution of the α-Al matrix and the Al₃Ti particles around the worn surface were investigated by using SEM-EBSD. The microstructural observation plane was mechanically and electrically polished. The electro-polishing was carried out using Struers A2 electrolyte at 13°C and 39 V for 10 s in a Struers Electropol device. The EBSSD crystal orientation analysis was performed at an acceleration voltage of 15 kV and step sizes of 0.5 and 0.8 μm.

3. Results

3.1 Initial microstructure of the Al–Al₃Ti FGM

Figure 2 is a 3D photograph showing the distribution of the Al₃Ti platelet particles in the outer region of the Al–Al₃Ti FGMs ring before the wear tests. Coarse Al₃Ti platelet particles and small Al₃Ti particles were observed. The plane normal direction (ND) of the coarse Al₃Ti platelet particles was aligned parallel to the centrifugal direction of the FGMs ring. This distribution of the coarse Al₃Ti platelet particles in the FGMs ring is in agreement with that reported in a previous study. However, the centrifugal direction did not affect the distribution of the small Al₃Ti particles in the FGMs ring because the small particles were precipitated in the α-Al matrix after it solidified.
Figure 3(a) shows the inverse pole figure (IPF) map and the phase map showing the microstructure of the outer region of the Al–Al3Ti FGMs ring. The lattice directions of the coarse Al3Ti platelet particles, which have a D022 crystal structure, are also shown in this IPF map. In addition, the plane ND of the coarse Al3Ti platelet particles was parallel to the centrifugal direction of the FGMs ring. Moreover, the lattice direction of the coarse Al3Ti platelet particles in Fig. 3(a) shows that the plane ND of the Al3Ti platelet particles corresponded to 001/Al3Ti direction. Figure 3(b) shows the (001)Al3Ti pole figure of the Al3Ti platelet particles in Fig. 3(a). Almost all the Al3Ti platelet particles had the (001)Al3Ti plane ND parallel to the centrifugal direction (RD) of the FGMs ring. This suggests that the plane ND of the coarse Al3Ti platelet particles, which had a plane ND of (001)Al3Ti, was aligned parallel to the centrifugal direction of the FGMs ring. Yamashita et al. have investigated the crystal orientation relationship between the α-Al matrix and the Al3Ti particles in Al–Al3Ti FGMs ring.9) The plane ND of the Al3Ti platelet particles corresponded to the 001/Al3Ti direction, which is consistent with our results. 

3.2 Microstructural evolution during wear

Figures 4(a)–4(c) show the cross-sectional microstructures of the Al–Al3Ti FGMs sample worn in the WA, WB and WC directions, respectively. The wear-induced layers were observed near the worn surface in all the samples. Figures 5(a)–5(c) are SEM micrographs showing the microstructures below the wear-induced layer of the Al–Al3Ti FGMs sample worn in the WA, WB and WC directions, respectively. Although the wear-induced layers are not observed in the photographs of the Al–Al3Ti FGMs sample worn in the WA and WC directions, this is because that these layers were delaminated during the electropolishing. Fine, fragmented Al3Ti particles and coarse Al3Ti platelet particles were locally dispersed just below the wear-induced layer in the worn samples for the WA and WC directions (Figs. 5(a) and 5(c)). In addition, the coarse Al3Ti platelet particles in the sample worn in WB direction were fractured in the sliding direction (Fig. 5(b)). The size of these fractured Al3Ti platelet particles near the wear-induced layer was large compared with those in the samples worn in the WA and WC directions. It can be expected that the wear tests in the WA and WC directions would induce larger local shear strain than would the test in the WB direction.

The 3D distributions of the Al3Ti particles and the wear-induced layer near the worn surface are presented in Fig. 6. In the samples worn in the WA and WC directions, very fine fragmented Al3Ti particles were present just below the wear-induced layer, and the unfragmented coarse Al3Ti platelet particles remained below the region containing the
fragmented Al3Ti particles. In contrast, in the WB sample the coarse Al3Ti platelet particles were fractured and tilted in the sliding direction over the whole region in the 3D photograph. Because the fragmentation of the coarse Al3Ti platelet particles in the WA and WC samples occurred below the wear-induced layer only, the wear tests for the WA and WC directions produced local shear strain on the worn surface. In addition, the wear-induced layer formed in the WA and WC samples continuously covered the worn surface, whereas the WB sample had a discontinuous wear-induced layer. Therefore, the formation of the wear-induced layer in the WA and WC directions was easier than in the WB direction.

To investigate the shear deformation behavior near the worn surface during wear, crystal orientation analysis was performed by using EBSD. Figure 7 shows the IPF and phase maps of the crystal orientation distributions for the worn surface. The arrows in these IPF maps indicate the deformation twins in the Al3Ti particles. The grains of the α-Al matrix just below the wear-induced layer were refined by the severe shear deformation caused by the wear. Deformation twins were also observed in the Al3Ti particles near the worn surface for all the samples. It has been previously reported that Al3Ti is plastically deformed by deformation twinning, and the Al3Ti deformation twin was formed on the \( (112)_{\text{Al3Ti}} \) plane.\(^{15,16} \) The twin boundaries shown in Fig. 7 are parallel to the \( (112)_{\text{Al3Ti}} \) trace. The deformation twins observed in the present study were formed by a large shear deformation caused by the wear. The plastic deformation region, indicated by the distribution of the fragmented Al3Ti particles and the refined or shear-deformed α-Al grains, showed that the samples worn in the WA and WC directions exhibited localized shear deformation, whereas widespread shear deformation occurred in WB sample. This local shear deformation of the worn surface enhanced the formation of the wear-induced layer. Therefore, the wear-induced layer in the Al–Al3Ti FGMs ring is easily formed in the WA and WC samples rather than the WB sample.

Figure 8 shows a \((001)_{\text{Al3Ti}}\) pole figure and a schematic illustration of the distribution of Al3Ti particles near the worn surface. In the sample worn in the WA direction, \([001]_{\text{Al3Ti}}\) was almost parallel to the centrifugal direction (RD), which meant the coarse Al3Ti particles in the WA sample were fragmented but did not rotate during wear. In Fig. 8(b), \([001]_{\text{Al3Ti}}\) changed from the transverse direction (TD) to the RD during the wear test in the WB direction. This is in good agreement with the Al3Ti particle distribution in the 3D photograph in Fig. 6(b). Because the Al3Ti platelet particles...
were tilted toward the sliding direction by the simple shear deformation caused by the wear, [001]$_{Al_3Ti}$ moved toward the RD. However, in the sample worn in the WC direction, [001]$_{Al_3Ti}$ was concentrated in the ND. Because the Al$_3$Ti platelet particles in the WC sample initially had [001]$_{Al_3Ti}$ in the ND, the Al$_3$Ti particles were fragmented and [001]$_{Al_3Ti}$ also did not move, as in the WA sample. These results show that the Al$_3$Ti particles fragmented during the wear tests in the WA and WC directions but did not rotate, whereas the particles fragmented and rotated during the wear test in the WB direction. Thus, the fragmentation of the Al$_3$Ti particles was similar for wear tests in the WA and WC directions.

3.3 Anisotropy of the wear resistance

Figure 9 shows the relationship between the weight loss caused by the wear tests and the sliding direction. The Al–Al$_3$Ti FGMs ring exhibited anisotropic wear resistance; the weight loss for the wear tests in the WA and WC directions were smaller than that for the WB direction. Thus, the wear resistance in the WA and WC directions was higher than in the WB direction. Although the wear resistance in the WC direction was the highest, the difference between WA and WC was smaller than that between WA and WB. This indicates that the wear behaviors for WA and WC were similar. The anisotropic wear resistance is consistent with previously reported results.\(^6,8\)

4. Discussion

4.1 Critical shear strain required to form the wear-induced layer

The wear-induced layer was observed in all the samples after the wear test. The amount of shear strain required to form the wear-induced layer was calculated to investigate its anisotropy.

Sato et al. proposed the method used for evaluating the amount of shear strain induced by wear.\(^3\) Figure 10 is a schematic illustration of the simple shear deformation of a spherical grain. The circle (solid line) and the ellipse (broken line) represent an undeformed grain and a sheared grain,
respectively. When a grain with grain size $D$ undergoes simple shear deformation during wear and its shape changes from a sphere to an ellipse with a thickness $t$, the nominal shear strain, $\delta_{\text{nominal}}$, can be written as

$$\delta_{\text{nominal}} = \left( \left( \frac{D}{t} \right)^2 - 1 \right)^{\frac{1}{2}} \approx \frac{D}{t} \quad (D \gg t).$$  \hspace{1cm} (1)$$

Onaka proposed that the large strain induced by severe plastic deformation could be described by the Hencky strain, $\delta_{\text{hencky}}$, because it is logarithmic with a tensor component.$^{17,18}$ Using the Hencky strain and eq. (1), the equivalent Hencky strain, $\delta_{\text{hencky}}$, for the simple shear deformation in Fig. 10 can be written as$^5$

$$\delta_{\text{hencky}} = \frac{2 \ln \delta_{\text{nominal}}}{\sqrt{3}} \quad (\delta_{\text{nominal}} \gg 1).$$  \hspace{1cm} (2)$$

Equations (1) and (2) show that the critical shear strain required to form the wear-induced layer can be calculated by measuring the thickness of the shear-deformed grain just below the wear-induced layer.

The critical shear strains required to form the wear-induced layer for each sliding direction are shown in Table 1. The initial grain size of the $\alpha$-Al matrix in the Al–Al$_3$Ti FGMs ring was $D = 110 \mu m$. The critical equivalent Hencky strain for Al–Al$_3$Ti FGMs ring reported in the previous studies$^3,5$ is 90 and this corresponds to $\delta_{\text{hencky}} \approx 5.2$. Because of this, the critical shear strain required to form the wear-induced layer obtained in the present study is close to the reported strain in those studies. Furthermore, the critical shear strain required to form the wear-induced layer did not depend on the sliding direction.

In previous studies, the microstructural evolution of commercial pure Al during accumulative roll bonding (ARB) has been investigated.$^{19,20}$ The grains of pure Al were refined as the number of ARB cycles increased. In addition, Pirgazi et al. reported that the fraction of high angle grain boundaries in pure Al that had undergone ARB
increased to more than 72% after 6 cycles, and that polycrystalline Al with ultrafine grains was obtained. This number of ARB cycles corresponds to an equivalent Hencky strain of 4.8, which is similar to the critical strain required to form the wear-induced layer. Therefore, we propose that the equivalent Hencky strain of about 5 is the strain required to form ultrafine grains.

4.2 Anisotropy of the wear behavior

The microstructure near the wear-induced layer and the wear resistance in the Al–Al₃Ti FGMs ring depended on the sliding direction of the wear tests. The anisotropic behavior arose from the fracture behavior of the coarse Al₃Ti platelet particles, which depended on the sliding direction. Figure 11 shows a schematic illustration of the shear-stress directions, indicated by arrows, of a coarse Al₃Ti particle for each sliding direction. For the WA direction, the shear stress was introduced in the plane of the Al₃Ti platelet particles, and the contact area of the coarse Al₃Ti platelet particles with the worn surface was the largest. However, for the WC direction, the shear stress was introduced on the edge plane of the particle, which would make it difficult to fracture the particle. Because of this, the coarse Al₃Ti platelet particles were only fragmented near the worn surface and the fine fragmented particles were distributed there. As a result, the shear deformation during the wear tests in the WA and WC directions was local and the shear strain was high close to the worn surface only. However, the wear test in the WB direction caused the shear deformation of the coarse Al₃Ti platelet particles (Fig. 11), and the particles were easily broken. Consequently, the strain was introduced over a wide area. Therefore, the wear resistance of the Al–Al₃Ti FGMs ring was higher for wear tests in the WA and WC directions and the wear-induced layer formed easily in these wear directions. Finally, the anisotropy of the wear behavior also arose from the local shear strain at the worn surface because of the distribution of the coarse Al₃Ti platelet particles.

5. Conclusions

Microstructural evolution near the worn surface of the Al–Al₃Ti FGMs ring was investigated in three dimensions by the serial sectioning and crystal orientation analysis of the Al₃Ti particles using EBSD. The main conclusions about the anisotropy of the wear resistance and the formation behavior of the wear-induced layer in the Al–Al₃Ti FGMs ring are summarized below.

(1) When the Al–Al₃Ti FGMs ring was worn along the longitudinal direction of the ring on its outer surface or its thickness plane (WA and WC direction), the coarse Al₃Ti platelet particles were severely fragmented near the worn surface only. However, when the wear test was performed along the radial direction of the ring on its thickness plane (WB direction), the coarse Al₃Ti platelet particles were broken into relatively large fragments over a wide region.

(2) The wear resistance and the formation of the wear-induced layer depended on the sliding direction. The anisotropy was explained by the local shear deformation caused by the Al₃Ti particle distribution.

(3) The critical equivalent Hencky strain required to form the wear-induced layer was ~5, and it did not depend on the sliding direction. The equivalent Hencky strain of about 5 is the strain required to form ultrafine grains.

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