Nano-structural refinement of metallic surfaces by using low-compression cyclical loading

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Abstract
Recent studies on very high cycle fatigue (VHCF) have observed a unique fracture surface with a fine granular area around the crack origin that arises from long-term repeated contacts of fracture surfaces (over $10^7$ cycles). A fine microstructure including nanocrystalline is known to form in the matrix just beneath the fracture surface. Focusing on these phenomena, this study devised a new surface modification technique, called Cyclic Press (CP), that creates a nanocrystalline layer using cyclic compressive loading. Repeated low-compression loadings were applied to the surfaces of SNCM439 high-strength steel and ADC12 die-cast aluminum alloy with an indenter for $5 \times 10^7$ cycles. Then, the surfaces and cross sections were analyzed by using a laser microscope, a scanning electron microscope, a scanning ion microscope, and energy dispersive X-ray spectrometry (EDS). As a result, a nanocrystalline structure very similar to that of the cross section beneath the fracture surface in the VHCF regime clearly formed in the surface layer of both materials. Additionally, an oxygen-rich layer formed at the surface of the CP-treated ADC12. This result was considered to be caused by diffusion of elemental oxygen in the atmosphere by a huge number of repeated contacts between the indenter and specimen. Overall, the results indicate that CP is a useful technique for forming nanocrystalline layers in metal surfaces and can also be expected as a new method for diffusing surrounding gaseous elements into surfaces at room temperature.

Keywords: Surface treatment, Structural refinement, Cyclic Press, Iron and steel, Aluminum alloy

1. Introduction

Severe plastic deformation (SPD) has been recognized as one of the important principles for nanocrystallization of metallic materials. Many techniques based on SPD, such as equal-channel angular pressing (ECAP), high pressure torsion (HPT), mechanical milling, accumulative roll-bonding (ARB), and high-speed shot peening, have been studied as useful processes for microstructure refinement (Horita, 2008, Umemoto et al., 2008). Metallic materials with fine structures, especially nanocrystalline structures, show not only high strength but also superplasticity and corrosion resistance. SPD can achieve these properties without additional alloying elements; therefore, it is recognized as a low-cost and recycling-efficient mechanical process. To refine microstructures by using SPD, a large plastic deformation with a true strain over 400 % is considered necessary (Umemoto et al., 2008).

On the other hand, a similar nanocrystallization is likely to be obtained by applying cyclical low-compressive stress. Research on very high cycle fatigue (VHCF) has observed a nanocrystallized region just beneath the fracture surfaces of fatigued specimens. On the fracture surface of high-strength steel obtained in the VHCF regime of over $1 \times 10^7$ cycles, a very fine convex-concave feature on the submicron scale has been observed near the fracture origin, as shown in Fig. 1 (Nakamura et al., 2010). The area has been variously named the optical dark area (ODA, Murakami et al., 1999), fine granular area (FGA, Oguma, N. et al., 2003), granular bright facet (GBF, Shiozawa et al., 2006), etc., and the mechanism behind the formation of this otherwise unique feature has been extensively discussed. The
important findings are that the nano-crystalline structure forms beneath the crack surface (Oguma, N. et al., 2003) and that a large number of compressive force applications during fatigue loading plays a crucial role in forming the structure (Hong et al., 2016) and the convex-concave fracture surface (Shiina et al., 2002, Shiina et al., 2004). This unique fracture surface has been observed not only in high-strength steel, but also in titanium (Oguma, H. et al., 2003, Oguma, H. et al., 2004), aluminum (Yamada and Miyakawa, 2009) and magnesium alloys (Shiozawa and Miyazaki, 2013). As a reason for the appearance of the fracture surface and the nanocrystallization beneath it, the authors have hypothesized that a huge number of repeated contacts of the crack surfaces has a similar mechanical effect as SPD (Nakamura et al., 2007, Nakamura et al., 2010, Nakamura and Oguma, 2011).

To clarify the validity of the hypothesis, this study devised a new experiment in which a relatively low compressive load is applied to the surface of the material specimen by using an indenter, as shown in Fig. 2. High-strength steel (SNCM439) and die-cast aluminum alloy (ADC12) were chosen as the experimental materials, since the nanocrystallization beneath the fracture surface was reported to occur in these materials in the VHCF regime (Oguma, N. et al., 2003, Sakai et al., 2015, Hong et al., 2016, Yamada and Miyakawa, 2009). After applying cyclic compressive loading, the surface feature of the contact area and the longitudinal section beneath it were investigated by using a laser microscope, a scanning electron microscope (SEM), a scanning ion microscope (SIM), and energy dispersive X-ray spectrometry (EDS) analysis, and the effect of cyclic compressive loading on the microstructure was qualitatively examined. We named the process shown in Fig. 2 “Cyclic Press” (CP), and, we examined the possibility of using it as a new surface modification technique.

2. Experimental procedure
2.1 Materials

The materials of this study were low-temperature-tempered Ni-Cr-Mo steel (SNCM439) and Al-Si-Cu die-cast aluminum alloy (ADC12). The chemical components of SNCM439 are C: 0.40, Si: 0.22, Mn: 0.78, P: 0.022 S: 0.013,
Cu: 0.18, Ni: 1.78, Cr: 0.83, Mo: 0.20 (mass %). The supplied SNCM439 was a round bar heat-treated by normalizing (1133 K for 3.6 ks followed by air cooling), quenching (1123 K for 3.6 ks followed by oil cooling), and tempering (433 K for 7.2 ks followed by air cooling). The tensile strength and Vickers hardness were $\sigma_B = 2106$ MPa and 640Hv, respectively. The chemical components of ADC12 are Cu: 2.22, Si: 10.7, Mg: 0.22, Zn: 0.70, Fe: 0.73, Mn: 0.24, Ti: 0.04, Ni: 0.07, Al: Bal. (mass %). The tensile strength and Vickers hardness of ADC12 were $\sigma_B = 289$ MPa and 90Hv.

2.2 Conditions of cyclic press

By using a hydraulic servo fatigue testing machine, uniaxial sinusoidal compressive loading was applied to the specimen’s surface in ambient air at room temperature, as shown in Fig. 2. The maximum and minimum values of the cyclic loading were 392 N and 0 N, and the frequency was 200 Hz. The number of cyclic loadings was $5 \times 10^7$ cycles in total. The material of the indenter was alloy tool steel (SKD11) whose hardness was about from 697Hv to 772Hv.

2.3 Surface and microstructure observation method

As a result of applying the cyclic loading, an indentation formed on the contact surface of the specimens, as shown in Fig. 3(a). The indentation was observed using an SEM (VE-9800, KEYENCE). In addition, the surface roughness and depth of the indentation were measured using a color 3D laser scanning microscope (VK-9700/9710 Generation II, KEYENCE). The areal roughness parameter (arithmetic mean roughness $R_a$) was determined from the 3D image. The measurement points were set at the center of the indentation and at the non-contact surface around the indentation.

To investigate the microstructure beneath the indentation, the observation samples were prepared as shown in Fig. 3. The CP-treated specimen was cut through the center of parallel to the loading direction (Fig. 3(a)). The cut faces were finished by polishing with emery papers to remove cutting burrs. The observation surface was created using focused ion beam (FIB) technique as shown in Fig. 3(b). In the FIB process, the surface of the observation area was covered by a very thin layer of carbon to avoid damage to the surface layer of the sample. After that, FIB sectioning using gallium ions was performed perpendicular to the contact surface of the specimen. After the FIB processing, the microstructure was observed using a scanning ion microscope (SIM).

3. Experimental results

3.1 Observation results at contact surface

Figure 4 shows an SEM image of the contact surface of SNCM439. A 500-µm-diameter indentation formed. A few deposition substances were observed around the indentation (the black arrows in Fig. 4). They seem to be fretting oxide debris due to repeated contacts between the surfaces of the indenter and the specimen. The surface roughness $R_a$ was 0.227 µm at the center of the indentation and 0.171 µm at the non-contact surface. The depth of the indentation was around 1 µm. These results showed that the deformation at the surface during the CP process was not so significant.

Figure 5 shows an SEM image of the contact surface of ADC12. A 1-mm-diameter indentation formed; it was larger than the indentation in SNCM439. The surface roughness $R_a$ was 1.866 µm at the center of the indentation and 2.196 µm at the non-contact surface. The depth of the indentation was 15 µm. These changes at the contact surface in...
ADC12 were larger than those in SNCM439; however, they still seem to be small compared with those of the ordinary SPD process.

### 3.2 Microstructure observation of longitudinal section of CP-treated sample

#### 3.2.1 Results of SNCM439

Figure 6 shows the microstructure beneath the center of the indentation in the CP-treated SNCM439. The dark gray thin area at the top of the images is a carbon layer deposited during the FIB processing, and the longitudinal section of the sample is below this layer. The structure occupying most of Fig. 6(a) is tempered martensite; however, at the top layer has granular features. This finer microstructure is clearly visible in Fig. 6(b), which is a magnified image of the rectangular area framed by the red dotted line in Fig. 6(a). The grain size of the fine microstructure was from 200 nm – 500 nm and the depth from the surface reached about 1 µm (Fig. 6(b)). This fine microstructure was similar to the nanocrystallized region observed in longitudinal sections beneath the fracture surfaces of SNCM439 in the VHCF regime (Oguma, N. et al., 2003, Sakai et al., 2015, Hong et al., 2016).

**Fig. 4** SEM image of indentation formed at the contact surface of SNCM439.

**Fig. 5** SEM image of indentation formed at the contact surface of ADC12.

**Fig. 6** SIM observation of longitudinal section beneath the indentation in CP-treated SNCM439.

#### 3.2.2 Results of ADC12

Figure 7 shows the microstructure beneath the center of the indentation at the CP-treated ADC12: Fig. 7(b) is a magnified image of the portion framed with the red dotted line in Fig. 7(a). The light gray thin area at the top of the images is the carbon deposition layer formed during the FIB processing. As shown in Fig. 7(a), the longitudinal section under the carbon overcoat is divided into three different layers: ① a dark layer, ② bright layer, and ③ microstructure including refined grains. The gray contrasts in the bright layer seem to be fine grains several hundred nanometers in size. The fine grains in the third layer under the bright layer were around 500 nm in size, as shown in the area framed by the red box in Fig. 7(b). This feature is very similar to the one observed at the cross section under the crack surface obtained in the VHCF regime (Yamada and Miyakawa, 2009). Namely, grain refinement occurred not only in SNCM439 but also in ADC12 during the CP process.
Unlike SNCM439, dark and bright layers were observed in ADC12. Figure 8 shows the results of the EDS analysis of the components in these layers. In Fig. 8(a), the dark and bright layers are framed by a black dotted box. The area corresponds to the one framed by the white dotted box in Fig. 8(b) and (c). As shown in the element map of Fig. 8(b), aluminum (Al) was detected in the entire analysis region; however, the color density of Al inside the dotted box was slightly lower than in the surrounding area. In contrast, oxygen (O) was mainly detected inside the dotted box, and it existed just at the surface layer (Fig. 8(c)). The EDS line analysis along the red arrow in Fig. 8(a) is shown in Fig. 8(d). From point A to point B, Al was at a concentration of about 100 mol%; however, it decreased gradually from point B to point C and tended to saturate from point C to point D. In contrast, the amount of O increased from point B to point C and saturated from point C to point D. As shown in Fig. 8(a), the regions from point B to point C and from point C to point D corresponded to the bright and dark layers, respectively. Namely, the ratio of Al to O in the bright layer changed with depth, but the ratio in the dark layer seemed to be constant.

Research investigating the influential factors on SIM images (Kim et al., 2010) indicated that non-conductive matter shows a dark contrast. In that study, the surface of the non-conductive matter, which was positively charged by gallium ion implantation, attracted secondary electrons emitted from the surface. As a result, the intensity of the signal of the secondary electrons decreased, and the SIM image became dark. In our study, similar phenomena may have occurred; the dark layer in Fig. 7 was likely nonconductive aluminum oxide. On the other hand, the bright contrast in
the SIM image was due to the high intensity of the signal of the secondary electrons emitted from this region. So, the bright layer in Fig. 7 may have been the conductive matter including diffused oxygen. In addition, the above SIM observations showed that the bright layer included nanocrystalline structure. The results suggest that the bright contrast is the channeling contrast resulting from the nanocrystalline structure whose crystal axis is significantly inclined from the direction of the gallium ion beam. Therefore, the bright layer in Fig. 7 was likely a finer aluminum crystal in which oxygen diffused.

4. Discussion

The results described in 3.2.1 and 3.2.2 showed that the CP process clearly changed the microstructure beneath the surface of SNCM439 and ADC12 into nanocrystalline layers which were similar to the ones observed in the longitudinal section beneath the unique fracture surfaces in the VHCF regime. In addition, especially in ADC12, the CP process created an oxygen-rich layer at the surface. These results are likely due to the huge number of repeated contacts between the indenter and specimen. In this section, we qualitatively discuss the reason for the surface modifications caused by the CP process.

As shown in Fig. 4 and Fig. 5, the indentation was formed by applying a relatively low compressive loading to the specimen surface. The formation of the indentation showed that the surface was locally plastically deformed. The maximum contact pressure between the indenter and the specimen under the compression load of 392 N calculated using Hertzian contact stress theory was 3.88 GPa in SNCM439 and 2.56 GPa in ADC12 when the indenter and the specimen’s surface were regarded as a sphere and a semi-infinite body. Additionally, the maximum shear stress of 1.20 GPa was generated at the depth of 106 µm in SNCM439, and that of 794 MPa was generated at the depth of 130 µm in ADC12. These values are much larger than the yield stress of SNCM439 or ADC12. Therefore, the indentations may have formed during the first cycle of the CP process. This estimation was experimentally confirmed because the size and the depth of the indentation in SNCM439 after the first loading was almost the same as that shown in Fig. 4. Namely, the plastic deformation nearly saturated at the first cycle in the CP process, and elastic deformation likely repeated in the following loading cycles.

A nanocrystalline layer was formed just beneath the indentations, as shown in Fig. 6 and Fig. 7. As mentioned in the introduction, the SPD process also provides a similar nanocrystallization. The reason for the nanocrystalline in the SPD process has been extensively investigated, and dynamic continuous recrystallization (Takaki, 2006) and grain subdivision (Hansen, 2001, Tsuji, 2008) have been recognized as major mechanisms of grain refinement. Here, accumulation of irreversible dislocations is necessary in order to refine the microstructure. During plastic deformation, a certain percentage of the active dislocations accumulate in the crystal grains, and these irreversible dislocations make a cell block structure consisting of the dislocation boundaries and regions with relatively low dislocation density. Further accumulation of dislocations increases the angle of the dislocation cell walls and causes the misorientation in the crystal grains. As a result, the crystal grains are divided finely. In the CP process, the subsequent cyclic deformation after the first contact between the indenter and specimen was almost elastic, and the fraction of irreversible dislocations seemed to be extremely low. However, the number of repeated contacts in this experiment was $5 \times 10^7$ and quite huge. Therefore, the total number of accumulated dislocations during the CP process is likely to reach the same amount which is necessary for the nanocrystallization in the SPD process.

On the other hand, in recent years, ultrasonic nanocrystal surface modification (UNSM) (Wu et al., 2014) and ultrasonic impact treatment (UIT) (Togasaki et al., 2010) have attracted attention as new types of SPD. UNSM and UIT create nanocrystalline structure at the surface by striking workpiece surface using ultrasonic vibration. The CP process does not use the ultrasound; however, the nanocrystallization mechanism appears superficially similar to that of UNSM and UIT. Further investigations will be necessary in order to investigate the similarities between the CP and UNSM or UIT, a huge number of repeated contacts between the indenter and workpiece may have the crucial role for the nanocrystallization.

The EDS analysis results showed that the dark and bright layers in the CP-treated ADC12 with elemental oxygen (O) in them. Since an oxygen is not a chemical component of ADC12, this result was caused by diffusion during the CP process. A similar phenomenon was reported in mechanical alloying of the elemental powder mixtures of Fe, Cr and Mn under a nitrogen gas atmosphere (Amini et al., 2009). This research showed that nitrogen atoms diffused in the Fe-Cr-Mn alloy powders. Additionally, in our previous study which applied CP to S25C low carbon steel in air, the
nanocrystallization at the surface layer was also observed and O element was detected from this layer (Nakamura et al., 2016). These results suggest that gaseous elements surrounding the sample could be gradually introduced into the surface layer by a huge number of repeated contacts between the indenter and the specimen. Considering that diffusion of surrounding gas elements into the surface layer was not reported in the ordinary SPD process, a huge number of contacts would be important for this phenomenon. In the CP-treated SNCM439, dark and bright oxygen-rich layers were not observed; however, O element diffused in the surface layer of CP-treated S25C (Nakamura et al., 2016). Since S25C and ADC12 have lower strength than SNCM439, the formation of an oxygen diffusion layer may be related to the ease of dislocation mobility. Although further investigation is needed to clarify the reason, CP can be regarded as a useful method for nanocrystallization, and also be expected as a new method for diffusing surrounding gaseous elements into the surface layers of metallic materials at room temperature.

5. Conclusion

Focusing on a formation of nanocrystalline structure in the longitudinal section beneath a unique fracture surface obtained in very high cycle fatigue regime, this study devised a new surface modification technique, called Cyclic Press, to create a nanocrystalline structure in metallic surfaces without causing large plastic deformation. Low-compression loading was cyclically applied to surfaces of SNCM439 high-strength steel and ADC12 die-cast aluminum alloy using an indenter, and the surfaces and cross sections were observed using a laser microscope, an SEM, a SIM, and EDS. The main findings of this study are as follows:

(1) A shallow and small indentation formed at the contact surfaces of both materials; however, the changes in the surface state were small in terms of plastic deformation.

(2) A nanocrystalline structure around 200 nm – 500 nm in grain size clearly formed in the surface layer of both materials. The nanocrystalline structure was very similar to the longitudinal section beneath the fracture surfaces in the VHCF regime. These similarities suggest that the microstructure of metallic materials becomes smaller as a result of applying cyclic compressive loading.

(3) EDS analysis showed that an oxygen-rich layer formed at the surface of the CP-treated ADC12. This result was considered to be due to diffusion of elemental oxygen from the atmosphere by a huge number of repeated contacts between the indenter and specimen.

(4) From the above results, CP can be regarded a useful technique for forming nanocrystalline layers in metal surfaces, and also be expected as a new method for introducing surrounding gas elements into the surface layer at room temperature.

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