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Surface crack nucleation and propagation in electrodeposited nanocrystalline Ni-P alloy during high cycle fatigue

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Abstract. The morphology of specimen surface after fatigue fracture was evaluated in connection with grain orientation distribution and grain boundary microstructure to reveal a mechanism of fatigue fracture in nanocrystalline materials. The electrodeposited and sharply {001} textured Ni-2.0 mass% P alloy with the average grain size of ca. 45 nm and high fractions of low-angle and $\Sigma 3$ boundaries showed 2 times higher fatigue limit than electrodeposited microcrystalline Ni polycrystal. The surface features of fatigued specimen were classified into two different types of morphologies characterized as brittle fracture at the central area and as ductile fracture at the surrounding area.

1. Introduction
The fatigue properties of nanocrystalline materials have received a great interest of researchers [1-6], because fatigue property is particularly important for micro electro mechanical systems (MEMS) materials. However, unfortunately, there has been only a little experimental work on fatigue property and behavior of nanocrystalline materials up to now. The fatigue property and behavior of nanocrystalline materials has been discussed mostly by focusing only the effect of grain refinement. The study of the roles of other microstructural factors such as texture and grain boundary microstructure has been very scarce, although grain boundaries very likely play some key roles in fatigue crack nucleation and propagation depending on boundary character. Recently, we have found that the intergranular fatigue cracks nucleated predominantly at random boundaries in polycrystalline Al, while the fatigue cracks never occur at low-angle boundaries [7]. The low-$\Sigma$ boundaries showed the higher resistance to fatigue crack nucleation than random boundaries [7]. Thus the effect of grain boundary microstructure on the fatigue fracture in the nanocrystalline materials may become more significant than that in microcrystalline materials, because the volume fraction of grain boundaries becomes remarkably high in nanocrystalline materials.

This work was performed to investigate the fatigue fracture process in electrodeposited nanocrystalline Ni-P alloy in connection with grain boundary microstructure. The formation of surface damage closely related to fatigue crack nucleation under cyclic stress condition were observed and discussed in comparison with the case of static tensile stress condition on the basis of the results of FE-SEM/EBSD analyses for grain boundary microstructure.
2. Experimental procedure
A nanocrystalline Ni-2.0 mass% P alloy was produced by electrodeposition using an electrolytic bath of 150 g/l nickel sulphate, 45 g/l nickel chloride, 80 g/l phosphoric acid and 0.5 g/l phosphorous acid with pH 1.5 at 338 K and a current density of 2.0 mA/mm² for 10.8 ks. Flat dog-bone shaped tensile and fatigue test specimens whose gage zone dimension was 5.0 mm long, 2.0 mm wide and 0.2 mm thick were cut out from the electrodeposited sheets mechanically stripped from the Ti substrate. The specimen showed a yield stress of 920 MPa, the tensile strength of 1550 MPa and the plastic elongation of 7.5 %.

X-ray diffractometer with monochromatic Cu-Kα radiation was utilized to analyze the initial microstructure of the specimen. The automated FE-SEM/EBSD/OIM was employed to quantitatively analyze of grain boundary microstructures on the surface of pre- and post-deformed specimens.

Fatigue tests were carried out by using a servo-hydraulic machine in air at room temperature. The sinusoidal load was applied at the stress amplitude between 290 MPa and 550 MPa at the stress ratio of 0.1 and the frequency of 10Hz.

3. Results and Discussion
3.1. Initial microstructure of electrodeposited nanocrystalline Ni-P alloy
Figures 1 (a)-(b) show an OIM micrograph with the inverse pole figure, the grain boundary characterization (GBC) micrograph with the average grain size and the grain boundary character distribution (GBCD) for the initial specimen, respectively. The OIM micrograph shows individual grain orientations in distinct colors corresponding to those indicated in the stereo-triangle of the inverse pole figure. In the GBC micrograph, low-angle (L, \( \theta = 2°-15° \)), low-\( \Sigma \) and random (R) boundaries are shown by distinct colors shown in color bars. Although the Ni-P binary phase diagram \[8\] shows that the solubility limit of P into Ni matrix is 0.17 mass\%, the secondary Ni₃P phase did not precipitate in the specimen. According to Boylan et al., the nanocrystalline Ni-P alloy is electrodeposited as a supersaturated solid solution \[9\].

From the OIM micrograph shown in figure 1 (a), it was found that the surface orientation of the initial specimen strongly localizes around \{001\}. The other grains tend to have surface orientations around \{112\}, \{123\}. The \{001\} grains had the grain size ranging from 10 nm to 180 nm and the average grain size of 56 nm. The grain size distribution of the \{001\} grains were more widely spreading than that of the grains with other surface orientations. It was found that the initial specimen had a considerably high fraction of low-angle and \( \Sigma 3 \) boundaries, as shown in figure 1 (b). The fractions of low-angle and \( \Sigma 3 \) boundaries were 30 % and 28 %, almost 13 and 15 times higher than that theoretically predicted for a random polycrystal shown in the brackets, respectively. Judging from OIM micrograph and GBC micrograph, \( \Sigma 3 \) boundaries tend to lie between the grains with \{001\} surface and the grains with \{112\} or \{123\} surface. These \( \Sigma 3 \) boundaries may be of nonequilibrium state, because these \( \Sigma 3 \) boundaries showed sluggish and irregular shape.

Figure 1. (a) OIM and (b) GBC micrographs of electrodeposited nanocrystalline Ni-2 mass% P alloy specimen.
3.2. High-cycle fatigue property of electrodeposited nanocrystalline Ni-P alloy

Figure 2 shows the relationship between the stress amplitudes and the logarithm of the number of cycles to fracture for the nanocrystalline Ni -2.0 mass% P alloy specimens. The data obtained from electrodeposited nanocrystalline pure Ni with an average grain size in the range 20-40 nm [2] and electrodeposited microcrystalline Ni with grain size around 1-7 \( \mu \text{m} \) [10] are shown together in this figure. The fatigue limit for the nanocrystalline Ni-P alloy in the present work was about 360 MPa. Accordingly, the fatigue limit is 2 times higher than that for the microcrystalline pure Ni, but slightly lower than that reported for the nanocrystalline pure Ni. Thus, it is evident that the fatigue limit of polycrystalline materials is improved by grain refinement to nanocrystalline level of grain size. However, the effect of nanocrystallization for improvement in the fatigue limit was lower than that for improvement in static tensile strength and hardness. Change in grain boundary microstructure and surface morphology under cyclic stress condition may affect the moderate fatigue limit of nanocrystalline Ni-P alloy.

![Figure 2. S-N curve of electrodeposited nanocrystalline Ni -2.0 mass% P alloy subjected to cyclic deformation of 290 MPa – 550 MPa in stress amplitudes at frequency of 10 Hz and at room temperature.](image)

3.3. Surface morphology and microstructure after static tensile and dynamic fatigue tests

Figure 3 (a) is a SEM micrograph of the specimen surface after tensile fracture test. Figure 3 (b) and (c) are the higher magnification images of the areas (i) and (ii) in figure 3 (a), respectively. The stress direction is the horizontal in the micrographs. The specimen surface fractured by tensile stress was characterized as ductile fracture mode resulting in the formation of steps pattern which probably associated with formation of shear bands in the whole area of specimen. The spacing of neighboring shear steps was 1.0 \( \mu \text{m} \) – 5.0 \( \mu \text{m} \). The surface cracks were nucleated and propagated along these shear steps. The fracture surface was also characterized as dimple pattern typical for ductile tensile fracture.

Figure 4 (a) is a SEM micrograph of the surface of fatigued specimen. Figure 4 (b) and (c) are the higher magnification images of areas (i) and (ii) in figure 4 (a), respectively. The stress direction is the horizontal in the micrographs. The morphology of fatigued surface can be classified into two distinct types. The area (i) around the crack source showed a brittle fracture mode. The specimen surface was rather flat and waving micro-cracks were observed in area (i). The nucleation of these micro-cracks was found to be the source of fatigue fracture. The fracture surface of the area (i) was planar and the fracture propagates toward the peripheral area of the specimen, and the adjacent area shows striation pattern of characteristic of fatigue fracture. On the other hand, the surrounding area (ii) showed a ductile tensile fracture mode. Steps which probably resulted from the formation of shear bands were observed. The spacing of neighboring shear steps was 1.0 \( \mu \text{m} \) – 5.0 \( \mu \text{m} \). Several experimental studies of the formation of shear bands during cyclic deformation have been recently reported for nanocrystalline metals [11]. The surface cracks nucleated and propagated along these shear steps. Therefore, the formation of shear steps is very important in the fracture resulted from fatigue crack propagation in nanocrystalline Ni-P alloy. The OIM analysis of the surface of fatigued specimen suggested that the area among the shear steps was formed by group of grains with similar surface orientation and high fraction of low-angle boundaries. It is thought that these shear steps were formed by sliding at connected random boundaries i.e. cooperative grain boundary sliding by shear of groups of grains. The morphology of fracture surface of the area (ii) was characterized as dimple pattern.
typical for tensile ductile fracture. Moreover, the results of OIM analyses suggest that the grain growth which is associated with random boundaries, probably occurred up to the grain size about 300 nm during high cycle fatigue tests, though the grain boundaries surrounding fatigue deformed grains were not clear. Until recently, studies of the stress-induced grain growth under cyclic deformation in nanocrystalline materials is very scarce, although a moderate increase in grain size (the order of 30%) after cycle deformation has been reported for nanocrystalline copper [1]. The further studies are required for full understanding of mechanisms of room temperature grain growth under cyclic stress in nanocrystalline materials.

4. Conclusions
The electrodeposited and {001} textured nanocrystalline specimens with high fractions of low-angle (30 %) and Σ3 (twin) (28 %) boundaries showed the fatigue limit of 360 MPa. The surface features of fatigued specimen were classified into two different types of morphologies. The central area was characterized as brittle fracture and the adjacent area as ductile fracture mode resulting in the formation of steps pattern due to shear banding. On the other hand, the specimen surface after static tensile test was only characterized by ductile fracture. It was also found that stress-induced grain growth occurred. The fatigue fracture in nanocrystalline metals was found to be strongly affected by grain boundary microstructure, not simply by the grain size.

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