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A mechanism of grain growth-assisted intergranular fatigue fracture in electrodeposited nanocrystalline nickel–phosphorus alloy

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Abstract—Quantitative investigation of the grain growth and resultant change in grain boundary microstructure during high-cycle fatigue was performed to understand intergranular fatigue fracture in electrodeposited nanocrystalline Ni – 2.0 mass% P alloys by using FE-SEM/EBSD technique. Pre-fatigued specimens had an average grain size of 45 nm, a sharp {001} texture and a high fraction of low-angle boundaries and of twin, or $\Sigma 3$ coincidence site lattice (CSL) boundaries. The considerable grain growth occurred due to the migration of low-angle boundaries induced by shear stress during cyclic deformation. The misorientation angle of those low-angle boundaries increased covering the whole surface of fatigue-fractured specimen. A certain fraction of low-angle boundaries was transformed into high-angle random boundaries resultant from grain growth during high-cycle fatigue. Those random boundaries which surrounded the grown {001}-grains were aligned along shear bands at almost 45° to the stress axis, and formed the diamond-shaped grain configuration, as reported in the literature on high temperature fatigue. The reported increase of the fatigue limit by nanocrystallization is likely reduced due to the cyclic stress-induced grain growth associated with the migration of low-angle boundaries composing nanograin cluster. Moreover, the random boundaries transformed from low-angle boundaries can be preferential sites for crack nucleation and propagation at the positions of initially formed shear bands during fatigue.

Keywords: Nanocrystalline material; Fatigue fracture; Cyclic stress-induced grain growth; Grain boundary character distribution; Grain boundary geometrical configuration

1. Introduction

Since the first report on nanocrystalline materials presented by Birringer, Herr and Gleiter in 1986 [1], the unique physical properties and extremely high strength of nanocrystalline metals and alloys have been drawing strong interest of many researchers in the past 30 years [2–9]. From the engineering application point of view, the fatigue properties of nanocrystalline metals and alloys are an important issue for their future development. Hanlon et al. [10,11] have pointed out that the nanocrystallization can generate a beneficial effect on the increase of fatigue strength, but a detrimental effect as a decrease of the resistance to fatigue crack propagation was observed. In fact, nanocrystalline materials demonstrated almost 2–3 times higher fatigue limit than that for ultra-fine grain (100 nm–1 μm) and microcrystalline (>1 μm) materials [10–14]. However, the fatigue ratio in nanocrystalline materials was found to be limited to a much smaller degree than that in conventional microcrystalline materials. Unfortunately, the unique fatigue properties and possible fatigue fracture mechanism operating in nanocrystalline materials have not been fully understood yet, as discussed in detail and pointed out by Padilla and Boyce in their recent review [14].

In general, it is well known that nanocrystalline materials have a much higher density of grain boundaries and excess free volume than conventional polycrystalline materials. Accordingly, the grain boundary migration and grain growth, which are known to occur normally at high temperature, can do at even room temperature in nanocrystalline materials during high-cycle fatigue [15–19]. Witney et al. [16] have observed the grain growth under cyclic stress in nanocrystalline copper specimens ultimately resulted in an increase of grain size by about 30% from the initial average grain size of ca. 20 nm. Boyce and Padilla [19] have demonstrated that considerable grain growth locally occurred only around fatigue cracks in electrodeposited nanocrystalline Ni-based alloys during high-cycle fatigue.

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They suggested that the observed grain growth in the nanocrystalline Ni-based alloys was due to a non-diffusional and shear stress-driven process. Again, possible mechanisms of grain growth under cyclic stress in the alloys have not been much studied and not fully understood yet.

In recent years, the intergranular fatigue fracture in polycrystalline materials has been extensively studied from the view point of structural effects of grain boundaries, focusing different roles of different types of grain boundaries [20–29]. Zhang and Wang [26–28] have made a systematic investigation of the effects of the type and character of grain boundaries on intergranular fatigue cracking in copper and copper based alloy bicrystals, and also in polycrystalline metals, from the viewpoint of the interaction of persistent slip bands (PSBs) with grain boundaries. From their findings, random boundaries can always be preferential sites for fatigue crack nucleation, while fatigue cracks never nucleated at low-angle boundaries. The present authors [29] have evidenced that fatigue cracks in polycrystalline aluminum were preferentially nucleated at random boundaries, while low-angle boundaries never cracked because of their lower level of the stress concentration resulted from less dislocation pile up and easy slip transfer across this type of boundaries. The low-Σ CSL boundaries cracked only when the trace of grain boundaries on the specimen surface was parallel to PSBs. Moreover, it has been demonstrated by Yang et al. [17] that the fatigue fracture in nanocrystalline Ni–Fe alloys was caused by linking of microcracks along grain boundaries owing to joining nanovoids, but unfortunately, any possible effect of the grain boundary character on the nucleation site for intergranular nanovoids and cracks was not studied. From our previous studies of structure-dependent mechanical properties of grain boundaries in polycrystalline materials [30], the grain boundary microstructure which is quantitatively evaluated by the grain boundary character distribution (GBCD), grain boundary geometrical configuration, density (grain size) and grain boundary connectivity, has been increasingly proved to play dominant roles in bulk mechanical properties.

In order to clarify the details of characteristic features of fatigue behavior and fracture mechanism in nanocrystalline materials, the quantitative evaluation of initial grain boundary microstructure and its change resulting from cyclic stress-induced grain growth is indispensable and urgently required. However, according to the authors' knowledge, this kind of quantitative microstructural studies for nanocrystalline materials is non-existent in the literature, although some recent TEM observations have confirmed considerable grain growth induced by static and cyclic stress [31–34].

The present investigation has been attempted to obtain better understanding of possible mechanisms of cyclic stress-induced grain growth and intergranular fatigue fracture in nanocrystalline materials. For this purpose, the quantitative examination of grain boundary microstructure and its modification during fatigue in electrodeposited nanocrystalline Ni – 2.0 mass% P alloy specimens was carried out using orientation-imaging microscopy (OIM) technique with the use of a field emission gun-scanning electron microscope (FEG-SEM) equipped with an electron backscatter diffraction (EBSD) unit.

2. Experimental procedure

2.1. Specimen preparation

A nanocrystalline Ni – 2.0 mass% P alloy was produced by electrodeposition onto a Ti substrate as in our previous work [13]. The electrodeposition was carried out using an electrolytic bath composed of 150 g l^{-1} nickel sulfate, 45 g l^{-1} nickel chloride, 80 g l^{-1} phosphoric acid and 0.5 g l^{-1} phosphorous acid with pH 1.5 at 338 K and a current density of 2.0 mA mm^{-2} for 10.8 ks. The Ni–P alloy sheet was mechanically stripped from the Ti substrate and stand-alone nanocrystalline Ni–P alloy thin sheets were obtained.

The fatigue test specimens with gage zone dimensions of 5.0 mm long, 2.0 mm wide and 0.2 mm thick were cut from the Ni–P alloy thin sheet. The specimen surface was mechanically polished using emery papers of 320–1500 grade and 0.06 μm alumina powder slurry, and electrolytically polished in an electrolytic solution of 6 vol.% perchloric acid, 15 vol.% methanol and 79 vol.% acetic acid at 280 K and at a current density of 4 mA mm^{-2} for 15 s. The Ni–P alloy specimen showed extremely high yield stress of 920 MPa, ultimate tensile strength of 1550 MPa and tensile elongation of 7.5%.

2.2. Characterization of grain boundary microstructure

An X-ray diffractometer (XRD) with monochromatic Cu Kα radiation was used to analyze the crystal structure of the pre- and post-fatigued specimen.

The FEG-SEM/EBSD/OIM system was applied for quantitative evaluation of grain boundary microstructure, particularly focusing on the change in the average grain size (including annealing twins), grain orientation distribution, misorientation angle distribution and GBCD in the pre- and post-fatigued Ni – 2.0 mass% P alloy specimens. In this work, statistical and quantitative evaluation of different types of grain boundaries was made according to our standard definition and procedure used so far [35]. Those grain boundaries with $1 \leqslant \Sigma \leqslant 29$ were defined as low-Σ CSL boundaries by the standard method based on the CSL concept after the Brandon's criterion. The fraction of different types of grain boundaries was evaluated by the length of specific type of grain boundaries based on the results from the EBSD/OIM analyses.

It has been well established that bulk mechanical and physico-chemical properties of polycrystalline metals and alloys are strongly affected by GBCD and grain boundary connectivity, and that the control of grain boundary microstructure is powerful for improvement in bulk properties of engineering materials and for development of high performance materials [35–37].

2.3. High-cycle fatigue tests

High-cycle fatigue tests were carried out using a servo-hydraulic machine in air at room temperature. The sinusoidal load wave was applied at a frequency of 10 Hz and the stress amplitude between 300 MPa and 550 MPa giving a stress ratio of 0.1. These values of stress amplitude were much smaller than the yield stress of the pre-fatigued specimen ($\sigma_y = 920$ MPa) and fell into the category of high-cycle fatigue. The Al sheets with 1 mm thickness were pasted on