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Grain Structure at Crack Path in Fatigued Nano-Crystalline Ni

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The generally acceptable mechanisms of fatigued crack growth in nanocrystalline (NC) materials were based on empirical studies and molecular dynamics (MD) simulations of pure Ni films [1, 2]. MD simulation suggested that fatigued crack growth is due mainly to crack-tip blunting, nanovoid formation, subsequent decohesion at grain boundaries [2] while Meirom et al. suggested a crack propagation rate dependent and dislocation-slip mechanism [3]. However, these studies were based on two-dimensional thin films. The lack of 3-dimensional constraint and a high volume fraction of the surface, 2-D thin-film samples may not represent the mechanical behavior of bulk polycrystalline materials [4]. Furthermore, the investigations of fatigued crack structures in NC materials have been limited to surface studies using SEM; the lack of knowledge on the inner grain structure at the crack path impedes the further understanding of fatigued crack growth mechanism in NC materials. A crack propagation study of sufficient thick, full condensed 3-dimensional fatigued NC Ni with the emphasis of grain structure at crack path using TEM was thus undertaken.

Bulk NC Ni-99.9% sheets (0.5 mm in thickness with average grain size of 29 nm) were fabricated using a pulsed electrodeposition technique (Integran Technologies Inc., Canada). Testing specimens (37.5mm x 3.81mm x 0.5 mm) were machined from the as-deposited sheets by electrical discharged machining (EDM) (Misubishi DWC90C with 0.25 mm brass wire), followed by mechanical polishing along the longitudinal direction to flatten the surface (final thickness = 0.43 mm) and remove transverse scratches that may serve as crack precursors. Tests were conducted with a three-point bend fatigue system under deflection-controlled conditions. After examining the crack/fracture surfaces with a SEM, crack/fracture surfaces were protected by electrodepositing a thick Ni layer to prevent possible artifacts before/during preparing TEM specimens.

TEM investigation, as shown in Figs. 1 though 5, clearly depicts deformation-induced grain growth along the crack path, which asserts Yang's finding [5]. The grown grain morphology is dependent on the stress intensity factor range ($\Delta K = C\Delta\sigma\sqrt{\pi a}$ for opening crack mode, where C is a geometry-related value, $\Delta\sigma = \sigma_{\max} - \sigma_{\min}$ is stress range, a is crack length) which is associated with crack growth velocity [1]. At low ΔK, the noticeable grain growth zone is about one or two grains in depth from crack path (Fig. 2). As ΔK increases (Figs. 2 to 4 or Fig.5a to b), grain growth zone may reach at least a few microns in depth which is hundreds of times the average grain size (though TEM observation is limited by the size of electron transparent area). The increase of ΔK also leads to the change in crack behavior. The crack propagation behavior changed from intergranular at low crack velocity (ΔK =13, indicated by less or equal to average grain size, Fig. 6) to partially transgranular at high crack velocity (ΔK =21, indicated by large than average grain size, Fig. 7). Non-equilibrium grain boundaries (Fig. 7, curved arrows) resulting from grain growth activity may account for the facet cracks observed at crack tip advance direction. Twin crystals were believed to be a product of crack propagation in NC grains formed by Shockley partial dislocations due to stress. The absence of twins in the high ΔK region where noticeable grain growth occurred indicated that the crack behavior has changed. Thus, the mechanism of crack propagation is crack growth velocity/ ΔK dependent and related to grain growth mechanism.

[1] T. Hanlon et al., International Journal of Fatigue 27 (2005) 1147-1158.
[2] D. Farkas et al., Phys. Rev. Lett. 94 (2005) 165502.
[3] R.A. Meirom et al., Phys. Rev. Lett. 101 (2008) 085503.
[4] E. Ma, Scripta Mater. 49 (2003) 663-668.
[5] Y. Yang et al., Metall. and Mater. Trans. A 39A (2008) 1145-1156.
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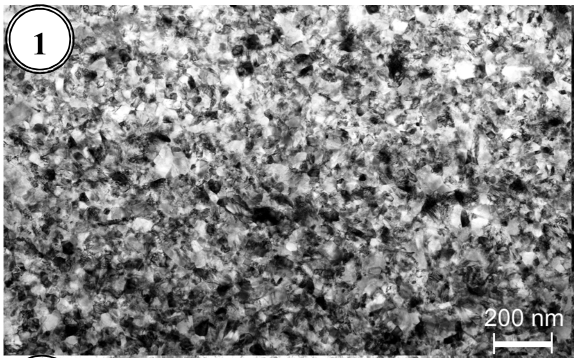

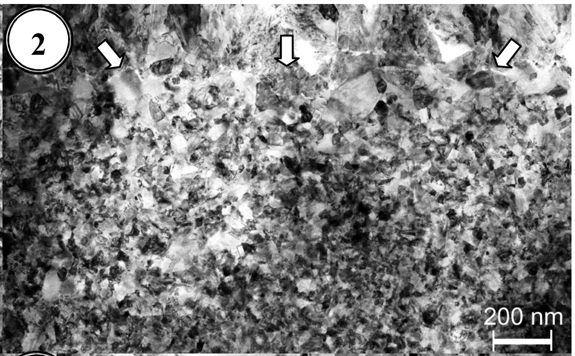

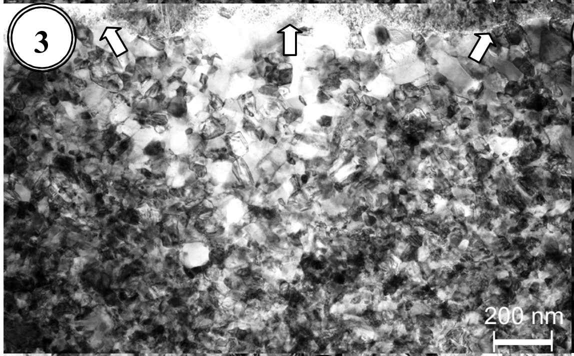

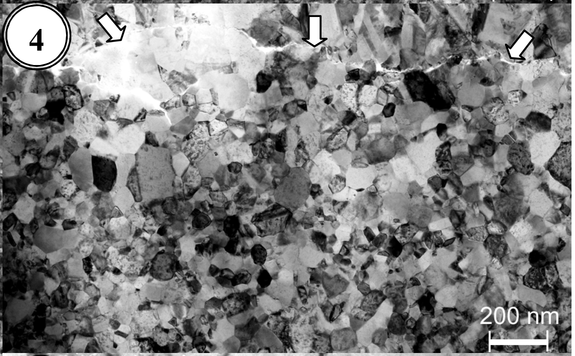

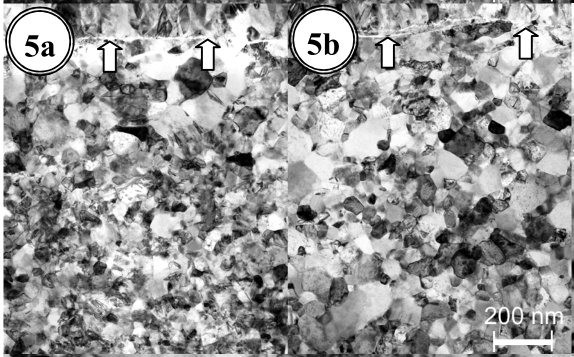

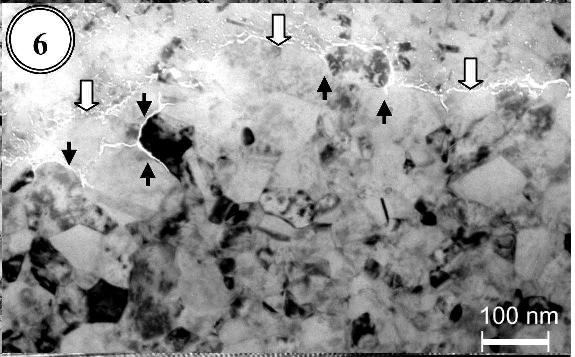

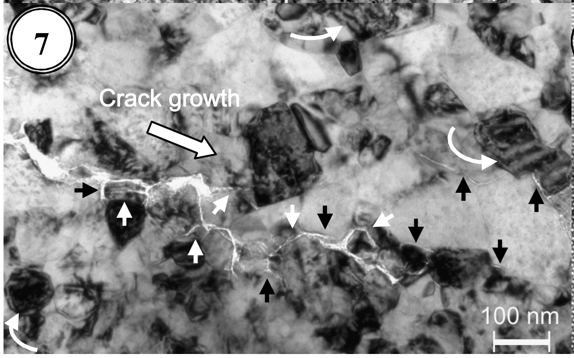

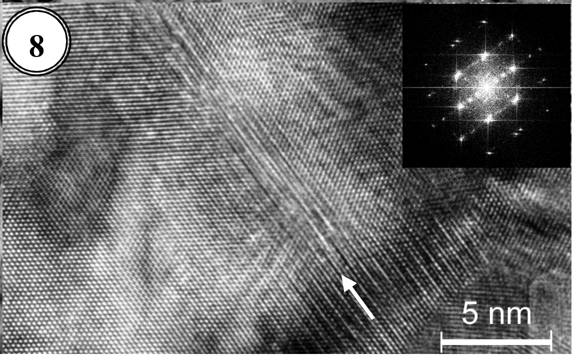

Fig. 1. STEM image of an as-received pulse electron-deposited Ni sheet showing nano-grain morphology.
Figs. 2 to 4. STEM images of fatigued samples at various stress range. STEM images were taken at the same distance (~100 μm) from crack initiation site. ΔK at each image was ~ 6.5, 13, and 20, respectively. The crack paths, i.e., fracture surfaces (arrows) are right below the deposited Ni protection layers.
Fig. 5. STEM images showing grain growth in the same TEM foil: (a) ~20 μm (ΔK ~10), and (b) ~100 μm (ΔK ~20) away from crack initiation site. The crack paths are indicated by open arrows.
Fig. 6. TEM image of crack path (open arrows, ΔK ~13) revealing intergranular cracks (dark arrows).
Fig. 7. TEM image of crack tip area (120 μm from the crack initiation site, ΔK ~21) showing transgranular (white arrows) and intergranular (black arrows) cracks with non-equilibrium gain boundary (curved arrow).
Fig. 8. High resolution image of crystal of twins found at crack path where ΔK ~ 6.5. Inset: FFT pattern.