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Grain size effects on the fatigue response of nanocrystalline metals

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Abstract

The fatigue response of electrodeposited nanocrystalline pure Ni and a cryomilled ultra-fine-crystalline Al–Mg alloy was studied. It was found that grain refinement generally leads to an increase in resistance to failure under stress-controlled fatigue whereas a generally deleterious effect was seen on the resistance to fatigue crack growth.

Keywords: Nanocrystalline materials; Grain refining; Fatigue; Electroplating; Cryomilling

1. Introduction

The resistance of metals and alloys to fatigue crack initiation and propagation is known to be influenced significantly by grain size (e.g., [1]). On the basis of experimental results obtained in microcrystalline (mc) metals with grain sizes typically above 1 μm, it is widely recognized that an increase in grain size generally results in a reduction in the fatigue endurance limit. Here, with all other structural factors approximately held fixed, the endurance limit of initially smooth-surfaced specimens generally scales with the strength of the material, which increases with decreasing grain size. On the other hand, a coarse grain structure can lead to an increase in the fatigue crack growth threshold stress intensity factor range and a decrease in the rate of crack growth owing to such mechanisms as periodic deflections in the path of the fatigue crack at grain boundaries during crystallographic fracture [2], especially in the near-threshold regime of fatigue crack growth (e.g., [3]). The relevance of such broad trends extracted from conventional mc alloys to ultra-fine-crystalline (ufc) metals (grain size typically in the 100 nm to 1 μm range) and nanocrystalline (nc) metals (grain size typically less than 100 nm) is largely unknown at this time. Such lack of understanding is primarily a consequence of the paucity of experimental data on the fatigue response of metals with very fine grains. The fatigue response of metals produced by severe plastic deformation using equal channel angular pressing has been studied [4–6]. Here, cyclic softening and deterioration in low cycle fatigue response have been found with grain refinement, despite an improvement in the

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fatigue endurance limit seen in stress-life tests. However, conclusive general trends could not be extracted from such observations in that mc metals with severe initial cold work are also known to exhibit cyclic softening [7]. Comprehensive knowledge of the fatigue properties of nc and ufc metals and alloys is critical to the overall assessment of their usefulness in service applications involving structural components. Inadequate fatigue behavior would likely overshadow several potentially attractive characteristics [8–12] of fine-grain materials (i.e. enhanced strength, hardness, wear and corrosion resistance).

The difficulty associated with characterizing the fatigue properties of fully dense nc materials of relatively uniform purity and grain size stems from current limitations in producing samples with sufficient dimensions for "valid" fatigue testing. The thickness of the laboratory specimens produced for exploratory studies is often limited to a few hundred micrometers or less, which severely complicates conventional fatigue testing and analysis. To the authors' knowledge, detailed and systematic studies of the resistance of nc materials to fatigue damage and failure have not been reported in the open literature.

The present study was initiated with the objective of investigating the effects of cyclic loading on the fatigue resistance of fully dense nc metals. The stress–life (*S*–*N*) fatigue response and the fatigue crack growth resistance of nc electrodeposited pure Ni were compared with a similarly produced ufc pure Ni. A particular objective of the present experimental work was to investigate whether grain refinement in the ufc and nc regime results in (a) an enhancement in the fatigue endurance limit, and (b) an increase in the rates of fatigue crack propagation. For the latter effect, additional crack growth experiments were also conducted in a cryomilled ufc Al–Mg alloy for which sizeable quantities of bulk specimens were available so that conventional fatigue testing could be employed.

2. Materials and experimental methods

The material investigated in this work is an electrodeposited, fully dense Ni for which two different grain sizes were produced using similar processing methods: an nc material with an average grain size of 20–40 nm and a ufc equiaxed structure with an average grain size of approximately 300 nm. The former had a columnar grain structure with an aspect ratio of 7–10, whereas the ufc material was nearly equiaxed. The as-received structures of both materials were characterized by electron microscopy (Fig. 1) and/or X-ray diffraction. Foils of both samples, with dimensions of approximately 150 mm × 100 mm × 100 μm, were procured from Integran Corporation, Toronto, Canada. Full details of the processing and structure of these electrodeposited Ni specimens are reported elsewhere [13,14], and will not be addressed here because of space restrictions. For comparison purposes, an mc pure Ni with an equiaxed grain size of 10 μm (procured from Goodfellow, Berwyn, PA) was also studied.

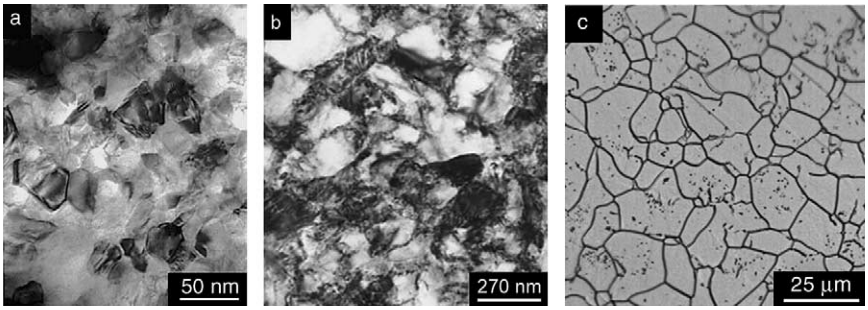

Fig. 1. Micrographs showing the grain structure of (a) nc Ni [8], (b) ufc Ni, and (c) mc Ni.

For the purpose of investigating the stress–life response, the nickel foils were electro-discharge machined into "dog-bone" type specimens, 25 mm in length. After mechanical polishing, these specimens were subjected to zero–tension–zero ($R = 0$) loading at a frequency of 1.0 Hz with a sinusoidal waveform in a laboratory air environment. Multiple samples of the nc and ufc specimens were tested to failure at a given stress range, and the number of cycles to failure was recorded. Tensile tests performed on the nc and ufc specimens revealed 0.1% offset yield strength values of 820 and 425 MPa, respectively, and strain to failure values of 3% and 10%, respectively. The mc Ni had a yield strength of 180 MPa, tensile strength of 450 MPa and tensile strain to failure of 35%.

Fatigue crack growth experiments for the nc, ufc, and mc Ni foils were conducted using edge-notched specimens, where tensile fatigue cracks were initiated in cyclic tension at a load ratio of 0.3 and cyclic frequency of 10 Hz (sinusoidal waveform) at room temperature at an initial stress intensity factor range $\Delta K = 9.5\ \mathrm{MPa\,m^{1/2}}$; crack growth data were gathered when ΔK increased to 11 $\mathrm{MPa\,m^{1/2}}$ after initial crack growth. The specimens were 39 mm long, 10.5 mm wide, and 100 μm thick. An edge notch was end milled to a depth of 1 mm, and its tip was sharpened using a razor blade sprayed with a 3 μm diamond polishing suspension. Changes in crack length as a function of number of fatigue cycles were monitored using optical microscopy. The crack growth rate $\mathrm{d}a/\mathrm{d}N$ was calculated as a function of ΔK as the length of the crack increased under a constant range of imposed cyclic loads. Two tests were performed under identical conditions for each material and the results plotted here contain data from both tests (except in the case of mc Ni for which only one set of data was obtained).

In addition to the fatigue crack growth experiments conducted on the nc and ufc Ni foils, the fatigue crack growth response of an Al–7.5wt%Mg alloy was also studied. This choice was motivated by the fact that grain size effects in the alloy could be assessed in the ufc regime using specimens whose dimensions are sufficiently large (35 mm × 35 mm × 5 mm) to meet the requirements of conventional standards for fatigue testing of bulk materials. Furthermore, the mechanical properties of this alloy system have been the subject of considerable experimental research, although its fatigue crack growth response has not been assessed to date. The Al–Mg alloy was produced by cryomilling. A complete review of the powder production and consolidation techniques, as well as microstructure for this alloy, is given in [15–18]. Transmission electron microscopy (TEM) images reveal that the Al–Mg alloy presented in this study has an equiaxed grain structure, with a grain size of ~300 nm [18]. Its yield and tensile strengths were measured experimentally to be 540 and 551 MPa, respectively. An extruded billet of the ufc Al–7.5Mg was sectioned to obtain 5 mm thick compact specimens in the C–R (circumferential–radial) configuration. The notch tip was machined to a radius of 0.09 mm, and the specimen faces were polished to a surface finish of 0.25 μm. The through-holes, located on either side of the notch to pin-load the specimens, were machined after the fatigue pre-crack was introduced.

Fully compressive far-field cyclic loads were used to introduce a self-arresting fatigue pre-crack in each sample [1,19,20]. These loads were transmitted through two parallel plates fitted in a servohydraulic test frame. Each sample was cyclically loaded at 10 Hz in compression with an incrementally increasing stress range, $\Delta\sigma$, until a crack initiated and grew from the notch (to a distance of at least fifteen times the notch radius). Crack growth was monitored in-situ with a telescopic video camera module, and ex-situ with an optical microscope. Following the pre-cracking stage, tension–tension fatigue experiments were performed at load ratios ranging from 0.1 to 0.5, at a frequency of 10 Hz. Crack growth was sufficiently slow to facilitate load-controlled (rather than K-controlled) experiments. The initial ΔK imposed on each sample was well below the anticipated threshold stress intensity factor range, ΔK_{th}. The stress intensity factor range ΔK was increased in 10% increments until measurable crack growth had occurred, at which point ΔK_{th} could be determined. Data for the remainder of the crack growth curve was collected in a similar fashion. The fracture surfaces were subsequently examined under a scanning electron microscope (SEM).

3. Results and discussion

The effects of grain size on the resistance to total fatigue life of pure Ni is plotted in Fig. 2 in terms of the stress–life (*S–N*) diagram. It is seen that the nc Ni with an average grain size of approximately 30 nm has a slightly greater resistance to stress-controlled fatigue loading than the ufc Ni with an average grain size of approximately 300 nm. This trend is observed both in the stress range at a given number of cycles to failure and in the endurance limit. It is also seen that the range of endurance limit values observed for mc pure Ni [21] is significantly below those of nc and ufc pure Ni. The results shown in Fig. 2 thus clearly illustrate that grain refinement leads to an enhancement in the resistance to *S–N* fatigue.

Fig. 3 shows the variation of crack length as a function of the number of fatigue cycles for the nc, ufc, and mc Ni, where constant cyclic load experiments were conducted with an initial stress intensity factor range of 11.5 MPa m$^{1/2}$. It is clearly seen that the fatigue crack length increases at a much faster rate with fatigue cycling in the nc Ni than in the ufc or mc Ni under identical loading conditions. The variation of fatigue crack growth rate da/dN with ΔK in the Paris regime is plotted in Fig. 4, where fatigue crack growth occurs up to four times faster in the nc Ni than in ufc Ni. Among the three materials, the mc Ni showed the slowest fatigue crack growth rate at a given ΔK; this crack growth rate was several times smaller than that of ufc Ni.

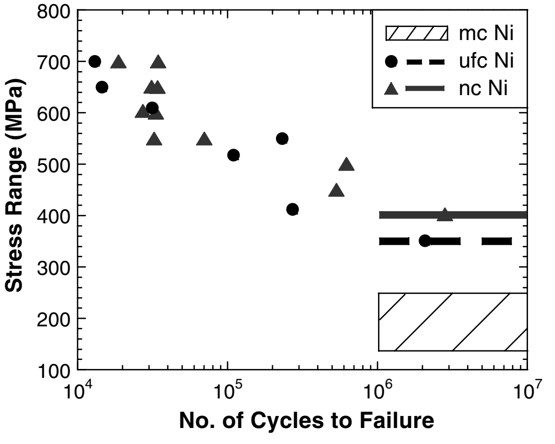

Fig. 2. A comparison of the *S–N* fatigue response showing the stress range versus number of cycles to failure for the nc, ufc and mc pure Ni [21].

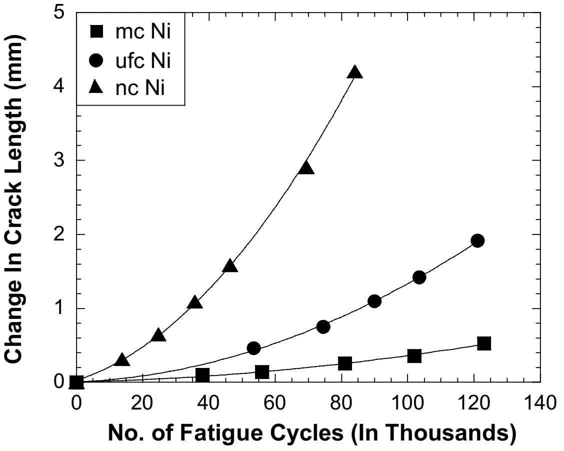

Fig. 3. A comparison of the variation of fatigue crack length as a function of the number of fatigue cycles for mc, ufc, and nc pure Ni subjected to an initial stress intensity factor range of 11.5 MPa m$^{1/2}$ at $R = 0.3$ at a fatigue frequency of 10 Hz at room temperature.

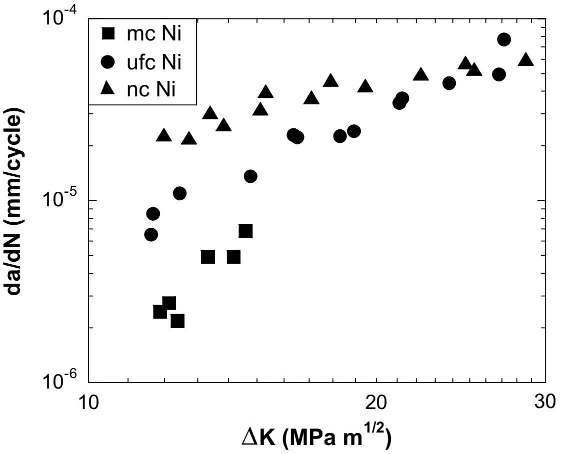

Fig. 4. Variation of fatigue crack growth rate, da/dN, as a function of the stress intensity factor range, ΔK, for mc pure Ni and for electrodeposited ufc and nc pure Ni at $R = 0.3$ at a fatigue frequency of 10 Hz at room temperature.

Fig. 5 shows da/dN versus ΔK for the ufc Al–Mg alloy tested at $R = 0.1$–0.5 over the entire range of fatigue crack growth rate, from ΔK_{th} to

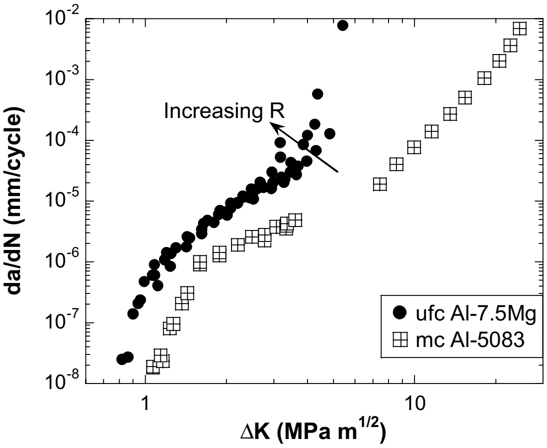

Fig. 5. Variation of fatigue crack growth rate, da/dN, as a function of the stress intensity factor range, ΔK, for the cryomilled Al–7.5Mg at $R = 0.1$–0.5 at a fatigue frequency of 10 Hz at room temperature. Also shown are the corresponding crack growth data for the commercial mc 5083 aluminum alloy at $R = 0.33$ [22,23].

final failure. Also shown in this figure, for comparison purposes, are the corresponding fatigue crack growth characteristics at $R = 0.33$ of the commercial 5083 Al–Mg alloy [22,23] which is a close mc counterpart of the present cryomilled Al–7.5Mg alloy. Although the processing conditions for these two alloys are different, it is evident from Fig. 5 that consistent with expectations, grain refinement from the mc to the ufc range results in a noticeable reduction in ΔK_{th} and a significant increase in the rate of fatigue crack growth from threshold to final failure. The stress intensity factor range for catastrophic failure, which is representative of the fracture toughness and static-mode failure mechanisms, for the Al–7.5Mg alloy is also several times smaller than that for the 5083 aluminum alloy. A scanning electron fractograph of the fatigue fracture surface of the Al–Mg alloy at a growth rate in the upper end of the Paris regime is shown in Fig. 6. Here, ductile transgranular fracture is mixed with growth around cracked inclusion particles, which were introduced into the microstructure by the cryomilling process. The presence of such particles is believed to contribute to the lowering of the fracture toughness and of the ΔK values at which catastrophic fracture is instigated during fatigue crack growth in the Al–Mg alloy.

4. Concluding remarks

We have demonstrated that grain refinement in the nc and ufc regimes can have a substantial effect on total life under stress-controlled fatigue and on fatigue crack growth. Specifically, fully dense nc and ufc Ni produced by electrodeposition exhibit substantially higher resistance to stress-controlled fatigue compared to conventional mc Ni. However, fatigue crack growth results obtained in this study for nc and ufc Ni also appear to indicate that grain refinement in the nc regime can have a deleterious effect on the resistance to subcritical fatigue fracture. These trends seen in this study are thus consistent with expectations of the role of grain size in influencing fatigue crack initiation and stable crack growth, see e.g., [1]. Experimental results obtained for a cryomilled Al–7.5Mg alloy also corroborate such interpretations of the effects of grain size on fatigue crack growth.

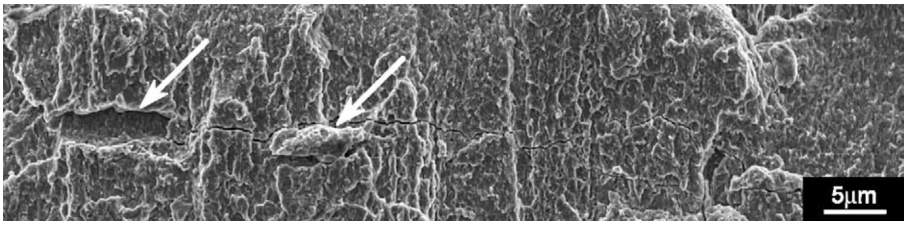

Fig. 6. Scanning electron fractograph showing the fatigue fracture features of the Al–7.5Mg alloy. Transgranular crack growth is accompanied by cracking of inclusion particles (indicated with arrows) introduced during the mechanical alloying process.

Although the results presented here pertain only to a limited set of nc metals and experimental conditions, it is interesting to speculate about possible implications for potential applications. It appears from the results obtained here and from those found for severely deformed ufc metals [4–6] that grain refinement into the nanoscale regime offers the possibility of enhanced resistance to high cycle fatigue, such as that characterized by the stress-life approach. Such an approach to tailor surfaces with nanoscale grains may offer enhanced resistance to fatigue crack initiation under low amplitudes of cyclic stresses. Possible microstructural designs (see, e.g., [24]) involving graded transitions from a nanostructured surface grain morphology to a relatively coarser interior grain morphology may also provide gradual transitions from a surface layer resistant to high-cycle fatigue to a core which is more resistant to fatigue damage and crack growth.

Acknowledgements

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