

# FATIGUE OF NANOSTRUCTURED METALS AND ALLOYS

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## ABSTRACT

The resistance of metals and alloys to fatigue crack initiation and propagation is known to be influenced significantly by grain size. In this work, the fatigue behavior of electrodeposited, fully dense, nanocrystalline pure Ni, with average and total range of grain sizes well below 100 nm, was examined. The fatigue response was also compared with that of ultra-fine-crystalline and microcrystalline Ni wherever appropriate. It was found that grain refinement to the nanocrystalline regime generally leads to an increase in the resistance to failure under stress-controlled fatigue whereas a deleterious effect was seen on the resistance to fatigue crack growth. To explore the generality of the above trends, similar experiments were performed in additional ultra-fine-crystalline material systems, produced using alternate processing techniques such as cryomilling and equal channel angular pressing.

## 1 INTRODUCTION

It is well established that the resistance to fatigue damage in most metals and alloys is significantly influenced by grain size (e.g., [1]). Based on experimental evidence from microcrystalline (mc) metals with grain sizes typically above 1  $\mu\text{m}$ , it is widely accepted that an increase in grain size, and attendant reduction in strength, generally leads to a decrease in the fatigue endurance limit. Conversely, a coarser grain structure can result in an increased fatigue crack growth threshold stress intensity factor range,  $\Delta K_{\text{th}}$ , and a decrease in the rate of crack growth as a result of periodic deflections of the fatigue crack at grain boundaries during crystallographic fracture [2], especially in the near-threshold regime of crack growth [3]. Previously, it has not been possible to extrapolate such broad trends, obtained from conventional mc metals, to ultra-fine-crystalline (ufc) metals, with average grain sizes ranging from 100 nm to 1  $\mu\text{m}$ , or nanocrystalline (nc) metals, with average grain sizes typically less than 100 nm. This is primarily a consequence of the difficulty associated with producing nc materials with sufficient dimensions to meet the requirements for valid fatigue testing. Previous studies have been conducted on ufc metals produced by severe plastic deformation [4-6]. Their results indicated cyclic softening and deterioration in low cycle fatigue response with grain refinement, despite an overall improvement in the fatigue endurance limit. However, conclusive general trends could not be extracted from such observations considering mc metals with severe initial cold work are also known to exhibit considerable cyclic softening [7]. A more complete understanding 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 of fine-grain materials [8-12] (i.e. enhanced strength, hardness, wear and corrosion resistance).

This investigation was initiated with the objective of probing 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 mc, ufc, and nc pure Ni were evaluated, with a primary focus on determining whether grain refinement in the ufc and nc regimes results in an enhanced fatigue endurance limit, and/or 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 and an equal channel angular pressed (ECAP) ufc Grade 2 pure Ti, for which sizeable quantities of bulk specimens were available such that conventional fatigue testing could be employed.

## 2 MATERIALS AND EXPERIMENTAL METHODS

While there are a number of methods currently available to fabricate nc materials, electrodeposited Ni was chosen as the model system for this investigation. Two notable advantages of the electrodeposition process are the ability to produce relatively large (in-plane) quantities of uniform, fully dense material (e.g. 80 mm x 80 mm), and the capacity to confine the grain size to an extremely narrow distribution within the nc regime. Although the attainable thickness resides in the millimeter range, samples in this investigation were limited to a thickness of approximately 100 – 150  $\mu\text{m}$ , to ensure through-thickness grain size uniformity. Electrodeposited Ni foils with two different grain sizes (nc Ni with an average grain size of 20–40 nm and a ufc equiaxed structure with an average grain size of approximately 300 nm) were procured from Integran Corporation, Toronto, Canada.

For the purpose of minimizing processing induced residual stresses, all fatigue specimens were extracted from the electrodeposited Ni foils by way of electro-discharge machining. Both low- and high-cycle fatigue experiments were carried out in a laboratory air environment, using a sinusoidal waveform in all cases. Full experimental details are provided in [13].

High cycle fatigue crack growth experiments for the nc, ufc, and mc Ni foils were conducted using single edge-notched specimens, where fatigue cracks were initiated in cyclic tension at load ratios ranging from 0.1 to 0.7 at a cyclic frequency of 10 Hz (sinusoidal waveform) at room temperature. The specimens were 39 mm long, 9.9 mm wide, and 100  $\mu\text{m}$  in thickness. Changes in crack length as a function of the number of fatigue cycles were monitored optically with a traveling microscope. The crack growth rate,  $da/dN$ , was monitored as the length of the crack increased under a constant range of imposed cyclic loads. To ensure small-scale yielding conditions prevailed, all data was truncated such that the remaining uncracked ligament length was always at least twenty times greater than the maximum plastic zone size at the crack tip.

In order to assess the overall generality of the trends observed in electrodeposited Ni, the fatigue properties of two additional material systems, for which larger bulk specimens could be produced, were evaluated. First, a ufc cryomilled Al-7.5Mg alloy was fabricated in billet form, 50 mm in diameter, and several inches in length. This choice was motivated by the fact that grain size effects could be assessed in the ufc regime using specimens whose dimensions are sufficiently large (35 mm x 35 mm x 5 mm) to meet the requirements of conventional standards for fatigue testing of bulk materials. A complete review of the Al-7.5Mg powder production and consolidation techniques is given in [14, 15]. In addition, a ufc ECAP pure Ti was also investigated, with direct comparisons made to its mc counterpart.

## 3 RESULTS AND DISCUSSION

The effect of grain size on the fatigue resistance of initially smooth-surfaced pure Ni is fully described in [13]. From these previous results, it is evident that nc Ni has a slightly higher resistance to stress-controlled fatigue loading than ufc Ni. Additionally, the endurance limit of the mc Ni is significantly below that of both the nc and ufc material, clearly illustrating the beneficial effects of grain size reduction on the resistance to S-N fatigue.

The variation in fatigue crack growth rate with respect to  $\Delta K$  for pure Ni at load ratios of  $R = 0.1$ ,  $R = 0.3$ , and  $R = 0.7$  is plotted in Figure 1. In order to enforce the assumptions

inherent to linear elastic fracture mechanics, all data was truncated such that the remaining uncracked ligament was at least 20 times larger than the plastic zone size at the tip of the crack. Due to the relatively limited strength of the mc Ni, valid fatigue crack growth experiments using  $\Delta K$  as the characterizing parameter were not possible under the abovementioned requirements. It is evident from Figure 1 that the resistance to fatigue crack growth is substantially lower in the nc Ni at all levels of applied loading, over a wide range of load ratios.

To further explore the validity and generality of the above fatigue crack growth trends, the fatigue properties of ufc Al-7.5Mg and ufc ECAP pure Ti were examined, with comparisons made to their mc counterparts. Since the supersaturated Al-7.5Mg alloy could only be fabricated via cryomilling, which ultimately results in a very fine grain structure, a direct comparison to mc Al-7.5Mg could not be made. However, Al-5083 is a close mc counterpart, and is often used for comparison purposes [15].

Consistent with the results obtained for electrodeposited Ni, it was found that the ufc Al-7.5Mg fatigue crack growth rate over the entire  $da/dN$  range, from threshold to final failure, was substantially higher than that in the mc Al-5083 (Figure 2). The threshold stress intensity factor range was also considerably lower in the ufc material.

The fatigue crack growth response of pure mc and ufc Ti was also fully characterized from threshold to final failure. Figure 3 depicts the effects of grain size on the variation of  $da/dN$  as a function of  $\Delta K$  at load ratios of  $R = 0.1$  and  $R = 0.3$ . Here, grain refinement from the mc to the ufc regime leads to a reduction in  $\Delta K_{th}$  by a factor of 2.5. The rate of fatigue crack propagation is more than an order of magnitude higher in the ufc Ti, over a wide range of applied loading, further validating the trends captured in the electrodeposited Ni.

The primary mechanism responsible for the observed accelerated fatigue crack growth rate in nc and ufc specimens is a general lack of crack path deflection and deflection-induced closure. Microstructural size scales can play a dominant role in crack morphology as well as the mode of fracture, especially near the threshold regime. Periodic deflections in the fatigue crack at grain boundaries during crystallographic fracture can lead to a relatively tortuous crack path in coarser grain materials.

Full details of a model that captures the effects of deflection and closure in periodically deflected cracks is reported elsewhere [16]. Because the surfaces of a crack do not generally mate perfectly during the unloading phase of a fatigue cycle, some level of crack closure is normally anticipated, which can significantly affect the rate of fatigue crack growth. Fatigue crack examination revealed a dramatic decrease in crack path tortuosity with grain refinement, which ultimately resulted in the observed accelerated fatigue crack growth rate. Based on the analysis in [16], the increase in crack path tortuosity with increasing grain size resulted in a reduction in the effective driving force for propagation, as well as increased levels of crack closure. Both of these tend to reduce the fatigue crack growth rate.

#### 4 CONCLUSIONS

The effects of grain size on the fatigue behavior of nc, ufc, and mc materials were systematically investigated. Grain refinement in the nc and ufc regimes has been shown to have a substantial effect on stress-controlled fatigue life, as well as the fatigue crack growth behavior. Specifically, crack growth results obtained in electrodeposited, fully dense pure Ni indicate that grain refinement in the nc regime can have a deleterious effect on the resistance to subcritical fatigue fracture. On the other hand, a beneficial effect of grain size reduction was observed with respect to the total life under stress-controlled fatigue. Experimental results obtained for a cryomilled ufc Al-7.5Mg alloy and a ufc ECAP pure Ti also corroborate the

above interpretations of the effects of grain size on fatigue crack growth. A crack deflection/closure model [16] was employed to justify the decrease in fatigue crack growth rate in coarser grain materials.

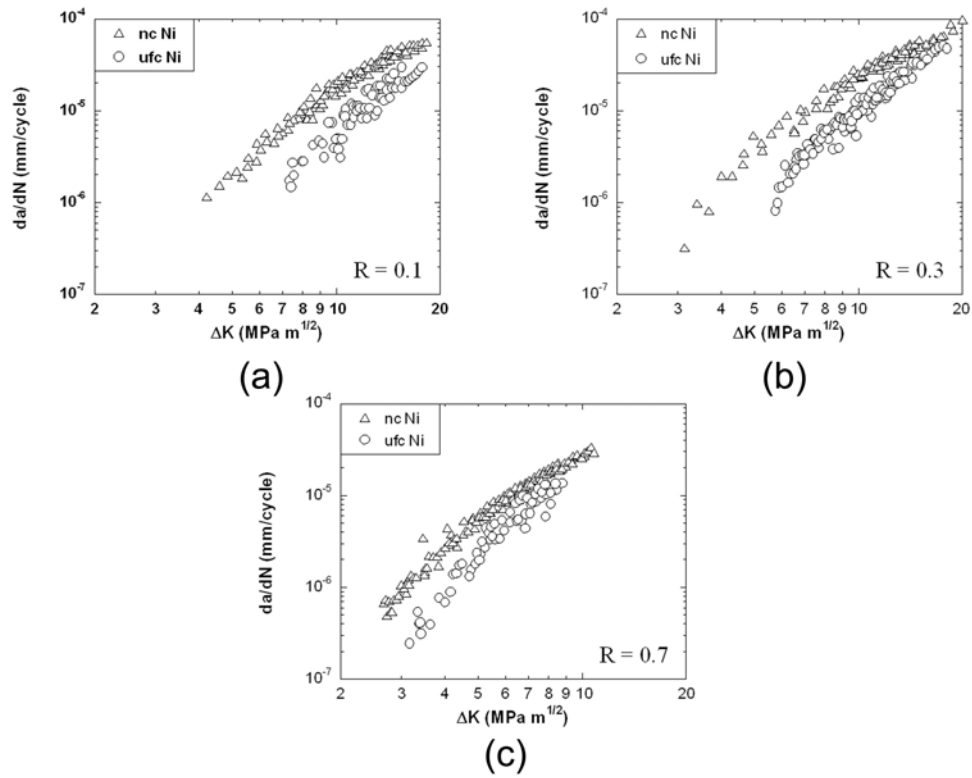


Figure 1. Variation of the fatigue crack growth rate,  $da/dN$ , as a function of  $\Delta K$  for pure electrodeposited ufc, and nc Ni at load ratios (a)  $R = 0.1$ , (b)  $R = 0.3$ , and (c)  $R = 0.7$ , at a fatigue frequency of 10 Hz at room temperature.

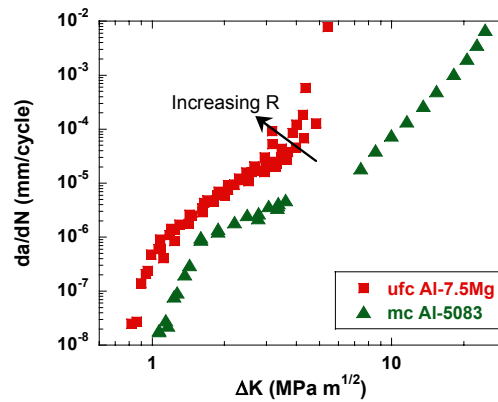


Figure 2. Variation of the fatigue crack growth rate,  $da/dN$ , as a function of the stress intensity factor range,  $\Delta K$ , for ufc cryomilled Al-7.5Mg at a fatigue frequency of 10 Hz at room temperature. Also shown are the corresponding crack growth data for a commercial mc 5083 aluminum alloy at  $R = 0.33$  [14].

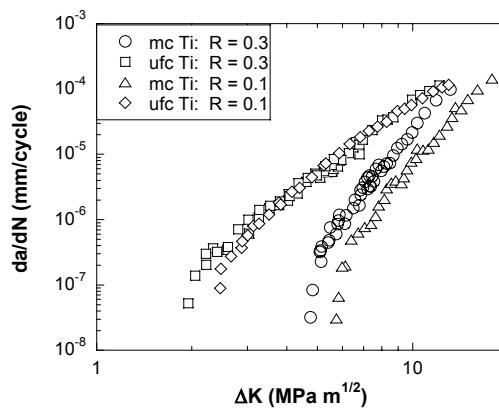


Figure 3. Variation in fatigue crack growth rate as a function of  $\Delta K$  for commercially pure mc Ti and ECAP ufc Ti at a fatigue frequency of 10 Hz at room temperature.

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