Cyclic strain hardening of nanocrystalline nickel

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Abstract

We demonstrate cyclic strain hardening and frequency-dependent fatigue life in electrodeposited nanocrystalline Ni subjected to tension–tension cyclic deformation. These observations are rationalized through mechanistic arguments based on the exhaustion of dislocation sources in the early stages of cyclic loading and on the domination of diffusive processes during later stages.
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1. Introduction

The cyclic deformation behavior of microcrystalline (mc) metals and alloys has been the topic of considerable research for the past several decades. For these metallic systems with average grain sizes typically larger than 1 μm, it is now recognized that significant cyclic hardening generally occurs in microstructures with low initial defect density and a relatively small number of initial impediments to dislocation motion (see, e.g., Ref. [1]). The mechanisms of hardening have been studied extensively by correlating the imposed cyclic stress or strain amplitude with stress–strain hysteresis and saturation responses, as well as with the evolution of energetically favorable dislocation structures (see, e.g., Refs. [1–5]). It has also been shown that face-centered cubic (fcc) pure metals can cyclically harden or soften to a common saturation steady-state flow stress from an initially annealed or cold-worked state, respectively, if the stacking fault energy is sufficiently high to promote significant cross-slip [6]. Such effects are considered to be independent of the imposed cyclic loading

frequency or waveform in the absence of environmental effects.

Recent interest in nanocrystalline (nc) metals and alloys, with average and peak grain sizes well below 100 nm, has been spurred by several appealing characteristics inherent to these materials: high strength/hardness [7], apparently greater resistance to scratch and wear damage [8], cyclic contact damage [9] and corrosion damage [10], and enhanced resistance to fatigue crack initiation [11], despite compromised ductility [12–14] and damage tolerance [11,15]. To date, however, the cyclic deformation characteristics of nc materials have not been reported in the open literature. Such information is vital for developing quantitative life prediction models and for assessing the viability of nc materials in structural applications, where repeated loading invariably occurs.

Studies of cyclic deformation in ultra-fine-crystalline (ufc) metals, with an average grain size typically on the order of several hundred nm, produced by equal channel angular pressing (ECAP), reveal cyclic softening and a relatively inferior low cycle fatigue response relative to coarser-grained metals [16–18]. However, the severe plastic deformation inherent to ECAP induces an extremely high initial defect density. As a result, conclusive trends isolating the effects of grain refinement on cyclic

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deformation behavior could not be extracted from such experiments. When electrodeposited ufc Ni specimens were subjected to cyclic deformation, a reduction in internal stress was observed with repeated loading, ostensibly due to interactions of mobile dislocations with grain boundaries [19].

In this paper, we report the first experimental evidence for significant cyclic hardening in electrodeposited nc Ni, with an average grain size of ∼30 nm. At room temperature in a laboratory air environment, this hardening behavior was found to depend on loading frequency and cycle number, indicating the importance of both time-dependent and cycle-dependent mechanistic processes on the observed deformation characteristics. Possible origins of such effects are postulated.

2. Material and experimental procedure

Electrodeposited nc Ni sheets, approximately 125 µm in thickness, were procured from Integran Technologies Inc. (Toronto, Canada). The as-received microstructure of the material was characterized by transmission electron microscopy. Plan view micrographs confirmed a narrow grain size distribution. The average grain size was ∼40 nm, with the maximum grain dimension well below 100 nm. The grain interior appeared to be clean and devoid of dislocations. Cross-sectional transmission electron microscopy (TEM) micrographs confirmed an aspect ratio of the grains greater than one. The major axis of these grains is, however, still very small compared to the thickness of the sheets. Microstructural details and monotonic deformation characteristics of this material can be found in our earlier companion papers [12,13,20].

A sub-sized 'dog-bone' specimen, measuring 53 mm in total length, 20 mm in gage length, 5 mm in gage width, and ∼125 µm in thickness was used for all cyclic tension–tension tests. Strain gages were mounted on both sides of the specimen to verify uniaxial loading conditions. Samples were mounted in a tabletop servohydraulic testing machine (DynaMight™ from Instron Corporation, Canton, MA) fitted with a 1 kN (dynamic) capacity load cell, and loaded in tension to 5 N, prior to soldering data acquisition cables to the strain gage solder tabs. Specimens were then held in tension at 5 N for 30 min to allow the gages to equilibrate, after which they were electronically balanced to a strain of zero per cent.

Tension–tension fatigue loading, with a sufficiently high load ratio ($R = 0.25$) to avoid specimen bending, was employed in all experiments. Samples were tested to failure in fatigue at a frequency of 0.2 Hz and 20 Hz (sinusoidal waveform) at room temperature. Each test was conducted under constant load amplitude and performed in a laboratory air environment (approximately 25 °C and 50% relative humidity). Load, displacement, and strain data were acquired at 0.5 kHz and 5 kHz for the 0.2 Hz and 20 Hz tests, respectively. Full details of the experimental procedure are reported elsewhere [21].

In addition, creep tests were performed at room temperature using the same specimen geometry and hydraulic testing machine.

3. Results

An example of the stress–strain response during cyclic loading ($\sigma_{max} = 1000$ MPa, $R = 0.25$, $v = 0.2$ Hz) is shown in Fig. 1. For the purpose of clarity, only the initial loading and selected cycles (1, 5, 30, 120, 600 and 1800) are included. The material progressively hardens and the hysteresis loop area progressively decreases with increasing cycle number. The general shape of this curve is representative of all cyclic curves recorded during this study. Upon the completion of a test, the strain amplitude and the hysteresis mid-loop width were measured in each recorded cycle. Their evolution as a function of cycle number is shown in Fig. 2. It is evident from these curves that both the strain amplitude and hysteresis mid-loop width decrease with increasing number of cycles, with the most rapid decline occurring over the first 30 cycles. At a fatigue frequency of 20 Hz, 30 cycles are completed in ∼1.5 s. At 0.2 Hz, the same number of cycles requires 2.5 min.

Fig. 2 also illustrates the effect of loading frequency on hardening behavior. The strain amplitude and mid-loop width for specimens cycled at 20 Hz are consistently smaller than those for similar specimens cycled at 0.2 Hz. While the magnitude of the mid-loop width reduction is similar over the first 30 cycles for the two frequencies examined, there appears to be a frequency effect on the decline in strain amplitude over the same 30 cycles.

Fig. 3 indicates that an increase in frequency from 0.2 Hz to 20 Hz results in prolonged fatigue life. It should be noted, however, that the total time consumed for specimens cycled to failure at 0.2 Hz was considerably greater than that for specimens tested to failure at 20 Hz. At a
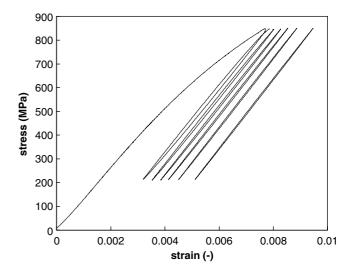

Fig. 1. Stress–strain response of cyclically loaded electrodeposited nc-Ni ($\sigma_{max} = 1000$ MPa, $R = 0.25$, $v = 0.2$ Hz). Only cycles 1, 5, 30, 120, 600 and 1800 are shown for clarity.
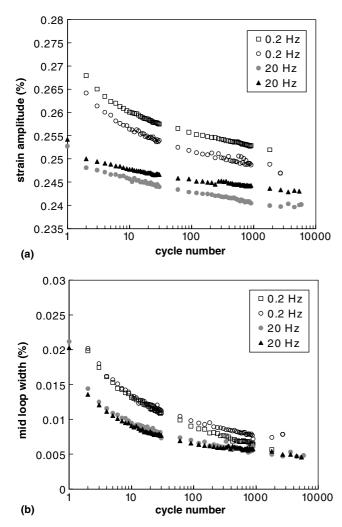
(a)

(b)

Fig. 2. (a) Strain amplitude and (b) hysteresis mid-loop width as a function of cycle number for electrodeposited nc Ni at a maximum stress of 1000 MPa and $R = 0.25$.
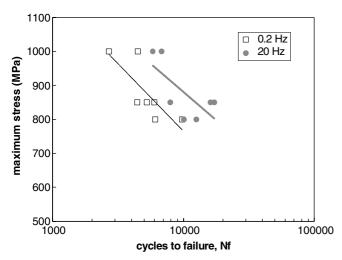

Fig. 3. Number of cycles to failure as a function of maximum stress for nc Ni. All tests were conducted in a laboratory air environment at room temperature.

maximum stress of 850 MPa, Fig. 3 indicates the total fatigue life at 0.2 Hz and 20 Hz is ~6000 cycles and
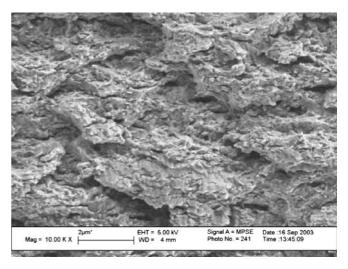

Fig. 4. Fracture surface morphology of a nc Ni specimen cyclically loaded to failure at a maximum stress of 850 MPa ($R = 0.25$) and a frequency of 0.2 Hz at room temperature in air.

~15,000 cycles, respectively. Corresponding elapsed times to failure were 8 h and 0.2 h, respectively. We also note here that the stress–life (S–N) response observed in Fig. 3 is in line with earlier results [11] on the same material subjected to slightly different loading conditions ($v = 1$ Hz, $R = 0$, and stresses ranging from 700 MPa to 400 MPa). Post-deformation TEM showed no evidence of grain growth or dislocation debris that could account for the observed hardening. Fracture surface examination revealed a frequency-independent brittle fracture morphology (Fig. 4), in contrast to the more ductile dimpled morphology found for monotonic loading conditions in this and other studies [12,13,20].

The frequency-dependence of fatigue life (Fig. 3) is suggestive of either creep- and/or environment-assisted material degradation. Although experiments were conducted at room temperature, creep mechanisms are considered non-negligible as a result of the extremely fine grain size, which tends to reduce relevant diffusion distances. Fig. 5 illustrates the evolution of creep strain as a function of time for several constant-load creep tests performed at room temperature in air. Creep rates range from 6.1×10^{-10} s^{-1} to 7.2×10^{-8} s^{-1} for stress levels ranging from 900 MPa to 1300 MPa, respectively. For comparison purposes, Fig. 5 also includes the evolution of maximum strain as a function of time in fatigue experiments conducted at 0.2 Hz, with $\sigma_{max} = 1000$ MPa and 850 MPa. A direct comparison between the two data sets is, however, not feasible, as samples subjected to cyclic loading conditions experience peak loads for a relatively short period of time, compared to constant-load creep tests. While the evolution of maximum cyclic strain at $\sigma_{max} = 1000$ MPa and $v = 0.2$ Hz is similar to the strain evolution in constant-load creep at a nominal stress of 900 MPa (Fig. 5), a similar comparison cannot be satisfactorily drawn for specimens loaded at a frequency of 20 Hz, as these tests last only a fraction of an hour.
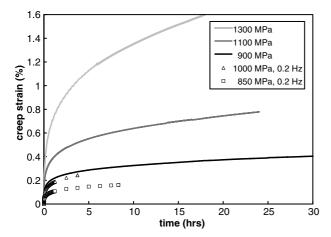
Fig. 5. Constant stress creep deformation at room temperature in air for nc Ni. Data points representing maximum strain evolution as a function of time for fatigue tests conducted at 0.2 Hz are superimposed.

4. Discussion

During plastic deformation dislocations may interact with each other and form locks. This process is irreversible and is seen as the most important contribution to cyclic hardening in conventional metals with grain sizes in the micrometer range and above. Considering the lack of dislocation debris in fatigued nc Ni specimens, this mechanism cannot be seen as the origin of the observed cyclic hardening behavior and an alternate explanation is sought. A variety of non-equilibrium dislocation sources, which can be activated at various stress levels, likely exist at the grain boundaries of as-deposited materials. Such sources are assumed responsible for the reduced hardness of these materials, relative to those with a comparable grain size subjected to an intermediate anneal [22]. Separate studies have confirmed that a mild anneal, without significant grain growth, can produce an increase in strength or hardness [23,24]. Indirect evidence for this phenomenon has also been provided by computational means [25].

Dislocation source exhaustion at a particular stress level would then require an increment in applied stress to activate another set of sources, further sustaining plastic deformation. Assuming, therefore, that dislocation source exhaustion is prevalent, such a phenomenon could account for the hardening behavior observed in Figs. 1 and 2. This also supports the contention that initially (<30 cycles), deformation is accommodated primarily by dislocation motion, while concurrent dislocation source exhaustion accelerates material hardening (steep initial reductions in strain amplitude and mid-loop width). In the later stages (>30 cycles), plastic deformation may rely more heavily on diffusion based mechanisms such as grain boundary sliding, creep and grain rotation, reducing the rate of material hardening (reduced slope in Fig. 2). This increased dependence on diffusion based mechanisms may also account for the dramatically different fracture surface morphology observed in fatigued versus monotonically loaded specimens (i.e. Fig. 4 versus results shown in Ref. [12]).

Additionally, grain boundary sliding is unlikely to be fully reversible, which may also contribute to the observed hardening behavior. No conclusion can be drawn from the current data set regarding the frequency-dependence of this exhaustion process, as detailed local atomic rearrangement processes at the grain boundary are not well characterized.

A time-dependence in mechanical response, manifested through a cyclic frequency-dependence of fatigue life is indicated in Fig. 3. This supports the above supposition that diffusive processes dominate the later stages of fatigue deformation. Still, times to failure in fatigue tests are considerably smaller than those in constant-load creep tests at comparable maximum stress levels. This suggests that creep–fatigue interaction has only a small influence on fatigue life. The observed frequency effect on fatigue life requires further investigation.

5. Conclusions

1. Cyclic hardening has been demonstrated for the first time in nc Ni, the majority of which occurs during the early stages of cyclic deformation.
2. Fatigue life exhibits a positive correlation with loading frequency.
3. Non-negligible creep deformation is observed at room temperature in nc Ni.
4. Possible mechanistic origins of such effects include dislocation source exhaustion in the early stages of fatigue life, followed by time-dependent, irreversible grain-boundary processes during the later stages.

Acknowledgements

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