FATIGUE AND FRACTURE BEHAVIOR OF NANOCRYSTALLINE COPPER AND NICKEL

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ABSTRACT

Nanocrystalline (nc) metals are being explored as potential interconnection materials to meet the needs of increased functionality and miniaturization in next generation electronic packages. Fatigue and fracture behavior of these materials is an important aspect in assessing their suitability for this application. This study focuses on evaluating the fracture and fatigue behavior of nc Cu and nc Ni of average grain size of about 50 nm produced by equichannel angular extrusion (ECAE). Fracture toughness tests have been performed using single edge notch bend (SENB) specimens and far-field J values for nc Cu and nc Ni have been found to be 21.66 kJ/m^2 and 12.13 kJ/m^2 respectively. The fatigue crack propagation rates also suggest superior fatigue resistance than microcrystalline (mc) Cu and Ni. An initial set of fatigue crack initiation experiments have also been carried out and a significantly higher fatigue resistance is observed compared to their microcrystalline counterparts.

1. Introduction

The mechanical properties of ultrafine-grained (UFG) materials (nanocrystalline and submicrocrystalline metals and alloys) have been a subject of growing interest over the last few years. This is largely due to promising combination of their mechanical characteristics such as a yield stress and hardness with fairly large plasticity, which makes these materials attractive for practical purposes. Although considerable experimental information is available on the fatigue response of metals and alloys with grain size typically larger than 10 µm, little is known about the fatigue characteristics of nanocrystalline (nc) metals. Several studies have examined the total fatigue life of UFG metals produced by equichannel angular extrusion (ECAE), where severe plastic deformation (SPD) was used for grain refinement [1-2]. In these experiments with average grain sizes of more than a hundred nm, repeated cyclic loading resulted in pronounced cyclic softening and a weakening of the resistance to low cycle fatigue. Hanlon et al. [3] compared the stress-life (S-N) fatigue response of a fully dense, electrodeposited nc Ni (grain size in the range 20-40 nm) with that of a similarly produced UFG Ni (grain size in the range 300 nm) and of a conventional microcrystalline (mc) Ni.

Low cycle fatigue (LCF) and high cycle fatigue (HCF) regimes are conventionally distinguished in accord with applied strain amplitude. At high strains corresponding to short lives, the plastic strain component is dominant in the total applied strain and the fatigue life is determined by ductility. At long fatigue lives, the elastic strain amplitude is more significant than plastic and the fatigue life is dictated by the fracture strength so that the fatigue limit increases with strength. The fatigue limit of UFG-Cu does show a noteworthy improvement depending on processing. The most impressive enhancement of the high-cyclic fatigue life has been observed in ECAP Ti and the peak-aged CuCrZr alloy [4] when compared with their conventionally fabricated counterparts. Small grain size results in a more homogeneous deformation that retards crack nucleation by reducing stress concentrators and raises the fatigue limit of the material. At larger grain sizes, dislocation-based plasticity dominates during straining and grain boundary sources are activated and the dislocations move through the grain interior. At very small grain sizes, the volume fraction of grain boundary region becomes comparable to the volume of grains and the deformation localizes in the grain boundary region. Although it is apparent that the grain boundaries play an increasingly important role in the deformation of nanostructured materials, Youngdahl et al. [5] have concluded that deformation of nanocrystalline copper with a mean grain size ranging from 30 to 100 nm produced by gas condensation and subsequent compaction, occurs due to dislocation activity. No evidence for grain boundary sliding or rotation was found in the in-situ experiments.

The first results of fatigue of nanocrystalline copper produced by powder compaction were reported by Witney et al. [6]. The significant enhancement of the HCF life in terms of fatigue limit is achieved for all materials with the exception of aluminum alloys. Markushev and Murashkin [7] reviewed the effect of SPD on sub-microcrystalline structure formation and mechanical properties of Al-alloys. They concluded that SPD is ineffective for the strength and fatigue improvement of Al-alloys. The presently available experimental data reveal that the ultimate tensile strength and the fatigue limit follow the Hall-Petch relationship. Very first experiments revealed that the cyclic response of ECAP materials is strongly dependent on materials purity, processing and the initial UFG structure. Vinogradov et al. [8] found that the cyclic hardening curve of UFG Cu was nearly flat during most fatigue life. No softening was observed under plastic strain amplitudes $\Delta\varepsilon_{pl}/2 = 5\times10^{-4}$ and 10^{-3}. However, Agnew and Weertman [2] have demonstrated pronounced cyclic softening in UFG Cu produced by ECAP. The mechanisms of cyclic softening of UFG fcc metals have been largely understood and associated with a complex effect of dislocation recovery, dynamic "recrystallization" and grain coarsening [2,10]. Thiele et al. [12] have performed the detailed structural investigations of the fatigue-induced structures in UFG Ni in the dependence of the grain size.

In this work, fracture and fatigue properties of nc-copper and nickel are investigated and are compared with that of microcrystalline copper and nickel. Nanocrystalline copper and nickel used in this study have been prepared by equichannel angular extrusion (ECAE). The purpose of this work is to assess the suitability of nanocrystalline copper and nickel for electronic packaging applications. Fracture toughness and preliminary fatigue tests have been carried out.

2. Experimental Procedure

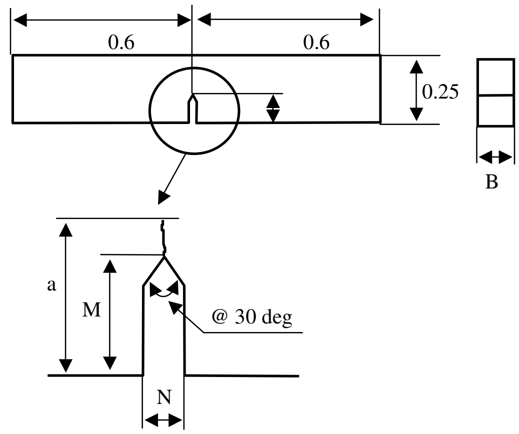


Figure 1: Specimen geometry and dimensions of the SENB fracture toughness specimens.

Fracture toughness testing was performed using a single-edge-notch bend (SENB) geometry with a width of 6.3 mm and a span of 25.4 mm as shown in Figure 1. The W/B ratio was two and the crack length, a (crack starter notch plus fatigue crack) is approximately equal to the thickness, B, and is about 0.548 for both copper and nickel specimens. All fracture toughness specimens were fatigue precracked using a 3- point bend fixture. The test fixture is designed such that the line of action of the applied load passes midway between the center of the support rollers within 1 % of the distance between these centers. The fatigue precracking was conducted under load control at a frequency of 10 Hz. The waveform was sinusoidal, and a constant stress ratio (minimum load/ maximum load) of $R = 0.1$ was maintained. A 500 lbs load cell was used for this purpose. The crack size was measured as a function of number of cycles during precracking using replica technique.

The fatigue test specimen geometry is shown in Figure 2. These tests were carried out under load-controlled mode at a frequency of 15 Hz at load levels of 0.85 times the yield strength. The number of cycles to failure was noted.

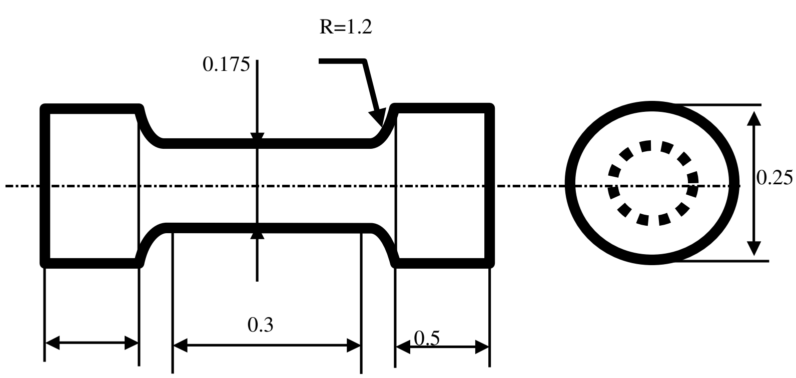
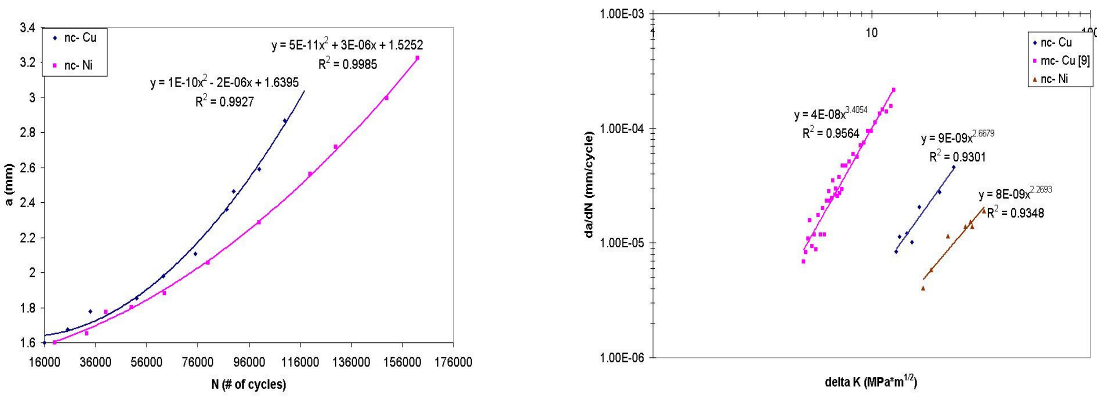


Figure 2: Specimen geometry and dimensions for fatigue test specimens.

3. Results and Discussion

Figure 3 shows the plots of crack size vs. number of cycles and the $da/dN - \Delta K$ curves for nc Cu and nc Ni. The fatigue crack propagation rates in nc Cu were much slower at equal ΔK values compared to its microcrystalline counterpart as seen in Fig. 3b. Figure 4 shows a fatigue-cracked specimen. The fatigue crack grew at an angle of about 45^o to the loading axis. A possible reason for this observation may be the grain size distribution obtained during ECAP. The crack finds a path of least resistance. The precracked specimens were then monotonically loaded and the loads vs. load-line displacement curves were recorded. The far-field J-values were calculated were found to be 21.66 kJ/m^2 and 12.13 kJ/m^2 for nc Cu and nc Ni respectively. Significant plastic deformation was observed and the test was stopped when the plastic load-line displacement exceeded 3 times the elastic displacement.



(a) (b)

Figure 3: (a) Plot of crack length vs. number of cycles for nc Cu and nc Ni.
(b) $da/dN - \Delta K$ curves for copper and nickel.

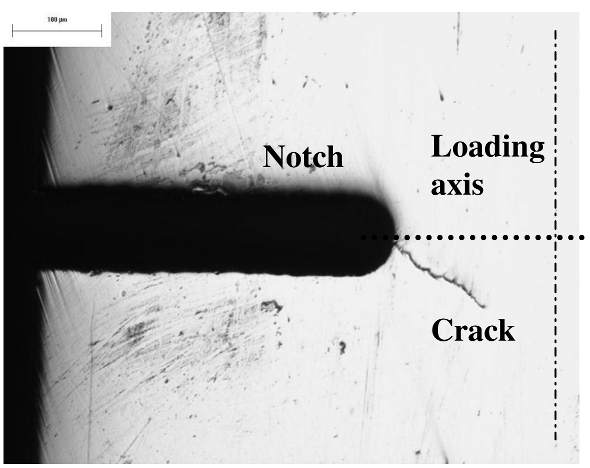
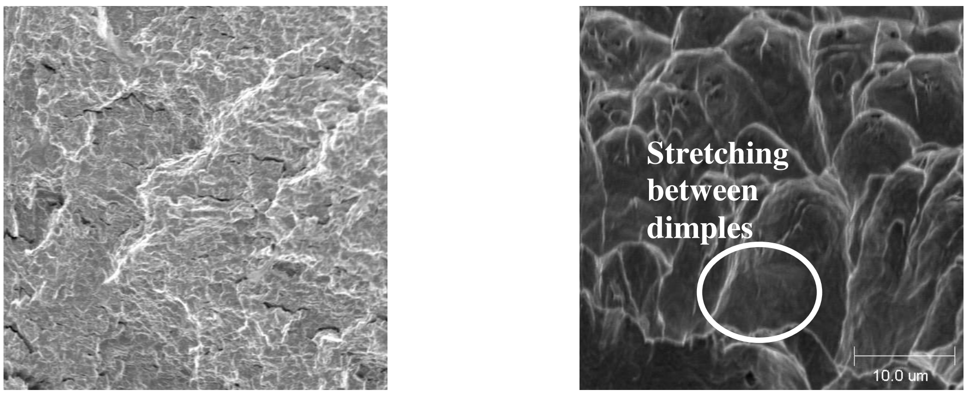


Figure 4: Optical micrograph of fatigue precrack at an angle to the loading axis.

Figure 5 shows SEM micrographs of the fracture surfaces. Fracture surfaces from both copper and nickel have shown dimpled rupture with the dimple diameter (~ 1 µm) significantly larger than the average grain size. Similar observations have been reported for fracture surfaces from tensile tests for nc-Ni, nc Al-Fe and nc-Cu [13-15]. Furthermore, the presence of significant stretching of the ligaments between the dimples has been observed that are indicative appreciable of local plasticity [16]. Also, the dimple size is uniform and extends across most of the specimen cross-section.

Figure 6 shows the plot of stress range vs. number of cycles to failure for nc Cu and nc Ni. Results for their microcrystalline obtained from counterparts have also been shown. It can be clearly noticed that the fatigue resistance of these materials is higher as compared to coarse grained copper or nickel.



(a) (b)

Figure 5: SEM micrographs of fracture surface for copper at (a) lower and (b) higher magnifications

Figure 6: Fatigue test results for nc Cu and nc Ni. Also shown are the values for mc Ni from literature for comparison [11].

Conclusions

Research into the structure, deformation and failure of nanocrystalline metals and alloys has provided a wealth of experimental information during the past decade. Such knowledge forms a critical foundation for the development of newer generations of structural materials whose mechanical properties can be modulated through the judicious design of grain boundaries. A systematic study of the fracture and fatigue properties of nc Cu and nc Ni is being carried out and some preliminary results have been stated here. Results indicate that grain refinement with nc metals can impart improved resistance to fatigue crack nucleation under predominantly high cycle fatigue loading. However, such grain refinement can lead to a detrimental effect on subcritical crack growth [11]. These materials show a potential for a wide variety of applications including that in electronic packaging. A lot still needs to be explored including low cycle fatigue life and effect of processing route on the properties. Results from future experiments will also be discussed in the final presentation.

Acknowledgements

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