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The influence of a multilayered metallic coating on fatigue crack nucleation

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Abstract

A thorough review of the literature on fatigue crack initiation indicates that for optimum resistance to fatigue crack initiation, a surface coating needs more than just a high hardness and that a combination of properties including toughness, cyclic work hardenability, residual compressive stresses, and adherence, in addition to a hardness higher than that of the substrate are required. Based on this assumption, it was hypothesized that nanometer-scale, multilayer coatings will posses a combination of these required properties enabling significant increases in fatigue crack initiation resistance. To test this hypothesis, fatigue experiments were conducted on Cu samples with different surface treatments including a nanoscale Cu–Ni multilayer. The fatigue lives of the multilayer coated samples were significantly greater than those of uncoated samples or samples coated with a monolithic coating of Cu or Ni indicating that the nanodimensional layering of the multilayer coating is responsible for retarding fatigue crack initiation and failure. The samples were examined with various analytical techniques including scanning and transmission electron microscopy and atomic force microscopy. Published by Elsevier Science Ltd.

Keywords: Fatigue; Multilayers; Nanomaterials; Electroplating; Surfaces; Crack initiation

1. Introduction

It is generally accepted that the principal mechanism of fatigue crack initiation in homogeneous ductile materials is strain localization produced by irreversibilities in the cyclic slip process [1–4]. In single crystals of face centered cubic (FCC) metals with high stacking fault energies, such as Cu, this process generates a characteristic surface morphology consisting of concentrated areas of notches and peaks, usually referred to as persistent slip bands (PSBs), surrounded by areas in which little or no deformation occurs [5–7]. While similar to that of a single crystal, the fatigue crack initiation process is more complex in polycrystalline materials. This is due to the influences of grain boundaries [8,9] as well as the variations in PSB morphology that occur between individual grains in polycrystalline materials caused by the strong dependence upon grain orientation and temperature [10–12]. During fatigue, small variations in the surface morphology can provide stress concentrations sufficient to promote crack initiation at even modest plastic strain amplitudes [13]. Hence, the process of crack initiation is quite sensitive to the conditions at the free surface. This sensitivity also makes it possible to alter the characteristics of fatigue crack initiation by modifying the surface properties or microstructure to create a surface that is more resistant to fatigue-induced surface roughening [14–16]. As a result, numerous investigators [17–23] have examined the influence of a wide variety of surface treatments (e.g., shot peening, anodizing, oxidizing, nitriding, boriding, plasma spraying, ion beam mixing, and ion implanting) that impart different properties at the surface of a material, and thereby, alter the mechanisms that promote the evolution of the critical surface morphology.

Alden and Backofen [17] examined the influence of thick anodic films on the fatigue behavior of Al and found they could extend the fatigue life for as long as the film could resist cracking by the fatigue loading. Their results were later expounded in a study by Hornbogen and Verpoort [21] who examined the influence of boriding, nitriding, and plasma spraying on

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fatigue crack initiation in austenitic stainless steel. Their results demonstrated that while these coatings suppressed surface roughening, the surface films eventually fractured resulting in slip localization and rapid initiation of fatigue cracks in the substrate which, in some cases, actually reduced the overall performance [21]. Clearly, most research into surface coatings for prevention of fatigue crack initiation has focused on suppressing the development of the critical PSB surface morphology via the use of high hardness coatings and dislocation image forces [22]. These films have demonstrated success in suppressing the formation of the critical PSB surface morphology, but their impact on fatigue life has been limited because cyclic loading can initiate cracks by another mechanism [8,24–26]. In most cases involving hard surface coatings, this secondary crack initiation mechanism is rupture of the film, presumably where subsurface PSBs intersect the surface film, followed by intense slip in the vicinity of the rupture [18].

Based on this review of the literature, it was concluded that for maximum resistance to fatigue crack initiation, a surface film should possess five different properties: (i) hardness — to suppress the development of the characteristic PSB-induced surface morphology, (ii) toughness — to resist cracking where subsurface PSBs intersect the film, (iii) cyclic work hardenability — to prevent slip localization, (iv) residual compressive stresses — to counter the effects of tensile stresses, and (v) adherence — to maintain surface film contact with the substrate. With the exception of shot peening, virtually all surface treatments for improved resistance to fatigue crack initiation studied to date have failed to provide a significant improvement due to an insufficiency in one or more of these five properties.

Nanometer-scale multilayered thin films offer a potential route to a surface coating that possesses all five of these properties. The basic strengthening mechanisms that operate in bulk materials also apply to thin films; however, the high density of interfaces present in the multilayered films give rise to some significant enhancements in the mechanical properties [16]. The fundamental basis of these property enhancements is the closely-spaced interfaces acting as barriers to the motion of dislocations through the film. The postulated strengthening mechanisms resulting from this structure are: (a) Orowan-type strengthening, resulting from a layer thickness that impedes the formation and motion of dislocation loops within the layer, (b) image force strengthening, resulting from modulus differences at the interfaces between the two materials, and (c) Hall–Petch type strengthening, resulting from the resistance to dislocation transmission across the interfaces within the film [27–33]. Surface, coherency, and intrinsic (or deposition) stresses, and compositional modulation effects have also been proposed as sources of strengthening in multilayered materials, but the literature suggests the magnitude of these influences may be on a smaller scale by comparison [34–38].

The range of synthesis techniques for multilayered films allows selection of constituents that maintain specific structural relationships within the layers, and also between the film and the substrate. Furthermore, the mechanical properties of multilayered thin film coatings can easily be modified or adjusted by simply changing the composition or thickness of the layers [33,39,40]. The resulting materials often display mechanical properties that greatly exceed those predicted by a rule-of-mixtures relationship of the individual constituents. In particular, a multilayer composed of thin layers of two, normally ductile, FCC metals may generate sufficient hardness to block surface slip yet retain the ductility, toughness, and cyclic work hardening capacity, required to suppress cracking of the film or other slip localization mechanisms that promote fatigue crack initiation. Thus, it may be possible to optimize the properties of multilayered coatings for fatigue crack initiation resistance [32]. This research was designed to determine if a multilayered thin film, with a higher hardness than the substrate, but a much lower hardness than the brittle films used in most previous studies, can extend the fatigue life of a substrate. Verification of this hypothesis would mean that thin, nanometer-scale multilayered surface films can be used to engineer the surface deformation behavior of materials and improve fatigue performance.

2. Experimental

For this work, cylindrical rotating beam fatigue specimens of the geometry shown in Fig. 1 were machined from extruded, polycrystalline electronic grade Cu (CDA101) rod stock. The specimens were given a two-stage heat treatment (2 h at 260°C, 0.5 h at 650°C, air cooled), similar to the one used by McGrath and Thurston [41], that was designed to produce a microstructure with a nominal grain size of 15 μm±5 μm and minimal residual strain. Surface preparation consisted of mechanical polishing to a 4000 grit finish followed by electropolishing in 85% ortho-phosphoric acid. A suitable evaluation of this hypothesis required experiments in each of four surface treatments: (i) uncoated, as-electropolished Cu — to establish the baseline fatigue behavior of the Cu under the selected loading conditions, (ii) a 5 μm thick monolithic Cu film — to evaluate the influence of a surface film that accounts for the increase in cross sectional area, but adds no appreciable contribution to the properties at the interface, (iii) a 5 μm thick monolithic Ni film with a composition and structure as close to that of the Ni layer in the multilayer as possible — to evaluate the change in the fatigue behavior of Cu with the addition of a surface film that has a higher elastic modulus and strength than the substrate, but contains no

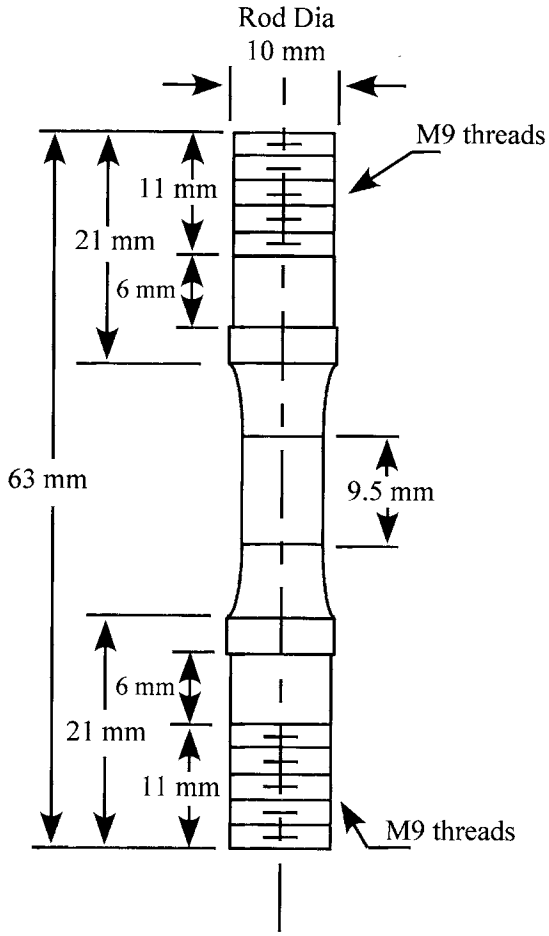

Fig. 1. The geometry of the specimen used for the rotating 4-point bend fatigue testing.

layering, and (iv) a 5 μm thick Cu–Ni multilayer with a 1:1 layer thickness ratio and a composition modulation wavelength (bilayer repeat length) of 40 nm — to evaluate the change in fatigue behavior with the addition of a hard surface film with enhanced properties of a multilayer.

The multilayer was electrodeposited on electropolished Cu samples starting and ending with a Ni layer from a single, 1.5 mol/l Ni nickel sulfamate-based, plating solution of the type described by Moffat and Searson [42,43]. In this technique, both layers are plated from the same electrolyte by switching potential from one where only Cu can deposit to another where both Ni and Cu deposit, but with Cu mass transport rate limited such that an almost pure Ni layer is deposited (≈0.5 atomic or mass fraction Cu). The thickness of the layers is controlled as a function of current and time through Faraday's Law and the Cottrell equation [16,44].

A special electrochemical apparatus was designed to enable deposition of uniform, high quality multilayers over the entire gage section of relatively large cylindrical fatigue samples [16]. The monolithic Ni coating was deposited from the same bath as the multilayer because the Ni layers in the multilayer will contain a small concentration of Cu and possibly Cu dendrites [16,42,43]. The monolithic Cu coating was deposited from a different electrolyte (1.5 mol/l $CuSO_4$) since the Cu layer will be essentially pure Cu, but the deposition of a 5 μm thick Cu layer at the mass transport rate limiting current of the multilayer plating solution would require a very long plating time. The quality and uniformity of the deposited layers were evaluated by optical microscopy and only films that reproduced the microstructure of the substrate, including grain boundaries and twin boundaries, were considered to be suitable for fatigue testing. Fig. 2 is a representative transmission electron micrograph showing the structure of the multilayered film in cross section after plating on a fatigue sample [16].

The fatigue crack initiation studies were conducted with a commercial 4-point rotating beam, bending fatigue machine where the sample is subjected to a sinusoidally varying, fully reversed, load cycle (i.e., $R=-1$) [45]. An initial series of fatigue tests was conducted to establish the fatigue life as a function of stress amplitude for uncoated, electropolished copper samples with this grain size [16]. The results were used to identify a single cyclic stress value (±90 MPa) and frequency (15 Hz) for testing the hypothesis. These loading conditions were specifically designed to be in the high-cycle fatigue (HCF) regime where the fatigue life is dominated by the crack initiation resistance and loading frequency. To enable a complete statistical analysis, five samples were tested in each of the four different surface treatments because the range of scatter in the fatigue lives for a surface treatment might be large compared to any improvement. The results were analyzed by creating a probability-life or Weibull plot of the results by estimating the failure probability from the ordered rank of the

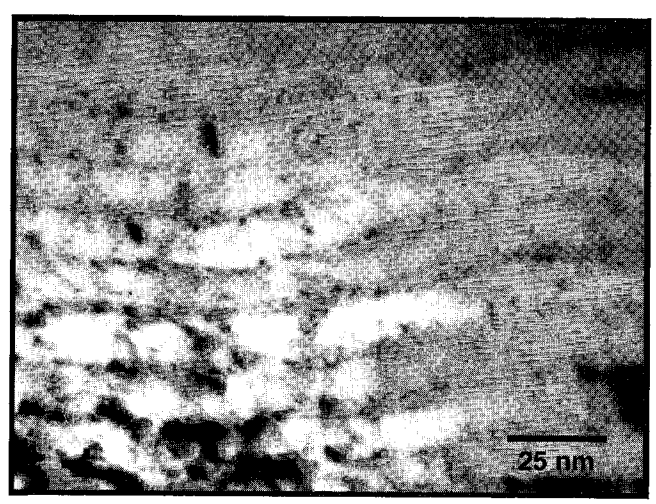

Fig. 2. TEM image showing the layering observed in the Cu–Ni multilayered films. The film has a 40 nm repeat bilayer thickness, a 1:1 constituent ratio, and contains 125 interfaces in a 5 μm thickness.

results and fitting the probabilities to a Weibull cumulative distribution function [46–50].

The influence of the electrodeposited multilayered coating on the fatigue-induced surface roughening was evaluated by measuring the changes in the surface roughness as a function of the number of applied load cycles. Using stylus profilometry, roughness measurements were conducted on the Cu surfaces in both the as-electropolished and the multilayer-coated conditions. For the measurements in the uncoated condition, a fatigue test was run until the number of load cycles reached a 1×10^n or 3×10^n (i.e., a half-log cycle) increment. The sample was then carefully removed from the fatigue machine, and roughness traces were taken along the tensile axis at nominally 60° increments around the circumference of the gauge sections. The same measurement technique was used for the multilayer-coated surfaces, with the exception of longer fatigue intervals between roughness measurements. Contact-mode AFM topography measurements, similar to those performed by Schwab [51] on Ni single crystals, were used for additional high-resolution characterization of the surfaces in the same two conditions at the endpoint of the fatigue lives.

3. Results

The results of the Weibull statistical analysis performed on the data from the fatigue experiments are presented in Table 1. These data are also plotted in the probability-life diagram shown as Fig. 3. In this figure, the solid curve is the Weibull fit to the results for uncoated, electropolished Cu and the dashed lines represent the 90% confidence band estimated for uncoated Cu using the 5% to 95% rank confidence intervals of Little and Jebe [47]. Any point inside the interval bounded by the two dashed lines is not significantly different from that observed for uncoated Cu at this confidence level. The monolithic Cu coated samples had a slightly shorter fatigue life than the uncoated Cu samples and the monolithic Ni coated samples had a slightly longer fatigue life than the uncoated Cu samples. No multilayer coated

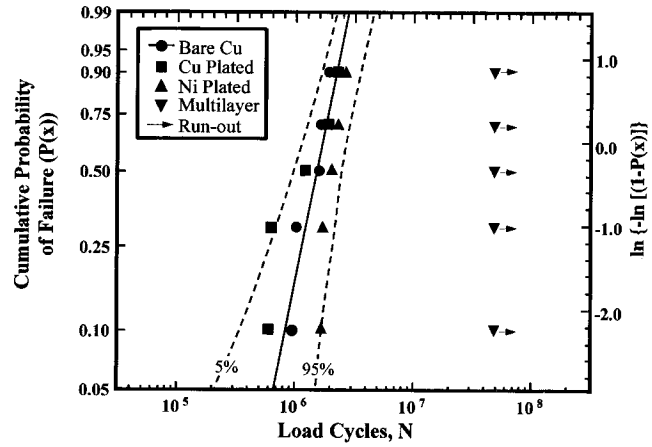

Fig. 3. Fatigue results in a Weibull scale probability-life diagram. The solid line represents a Weibull fit to the bare Cu data and dashed lines represent the confidence interval for fit to the bare Cu data [46–48].

samples failed at this cyclic stress amplitude, thus a limit on the testing time had to be imposed. After examining the mean and standard deviation determined from the data obtained from the other surface conditions, it was decided that at 5×10^7 load cycles, the statistical significance of the results would be unquestionable and this value was then used as the "run-out" point. While these run-out tests prove that the multilayer improved the fatigue life, it only allows for the observation of the early stages of the fatigue crack initiation mechanism in the multilayer-coated system.

Fig. 4 is an SEM micrograph of the fatigued surface of one of the uncoated, electropolished Cu samples. The classic surface morphology of PSBs showing the characteristic notches and peaks can be seen in this figure [1,3,10]. The surface of a sample coated with a monolithic Cu coating is shown in Fig. 5. While the surface morphology shown in this figure is not identical to that shown in Fig. 4, the differences present in the figure are due to variations in the crystallographic orientations of the grains shown and not the result of any changes in the slip behavior [16]. Fig. 6 is a SEM micrograph of the surface of a monolithic Ni-coated sample tested to failure. No evidence of slip could be found in the Ni

Table 1
Results of Weibull analysis performed on fatigue data

Surface treatment	Mean [$\log N_f$]	SD [$\log N_f$]	Weibull scale parameter (β)[a]	Weibull shape parameter (α)[a]
Uncoated Cu	6.16	0.16	$1.72(\pm0.09)\times10^6$	2.87±0.06
Cu w/ 5 μm thick monolithic Cu film	6.06	0.27	$1.55(\pm0.14)\times10^6$	1.64±0.33
Cu w/ 5 μm thick monolithic Ni film	6.31	0.07	$2.19(\pm0.06)\times10^6$	6.76±1.91
Cu w/ 5 μm thick Cu–Ni multilayered film	7.70	—	—	—

[a] Weibull equation for this analysis: $P(x)=1-\exp\left[\frac{N}{\beta}\right]^{\alpha}$.

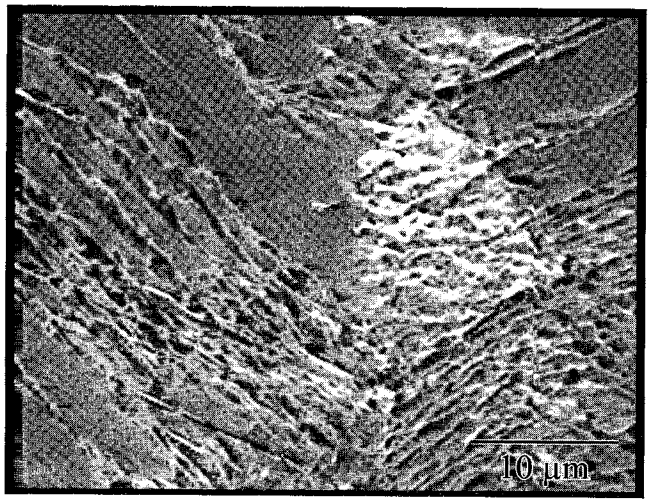

Fig. 4. SEM image of bare Cu surface after fatigue (N_f=1.5(±0.5)×10^6 cycles).

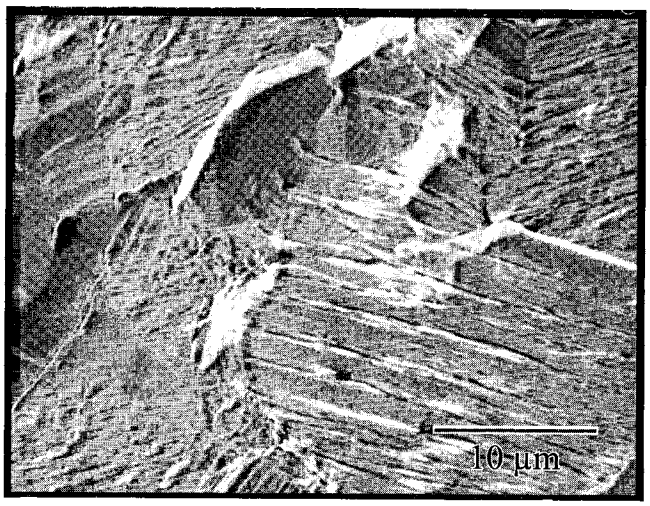

Fig. 5. SEM image of monolithic Cu-plated surface after fatigue (N_f=1.3(±0.7)×10^6 cycles).

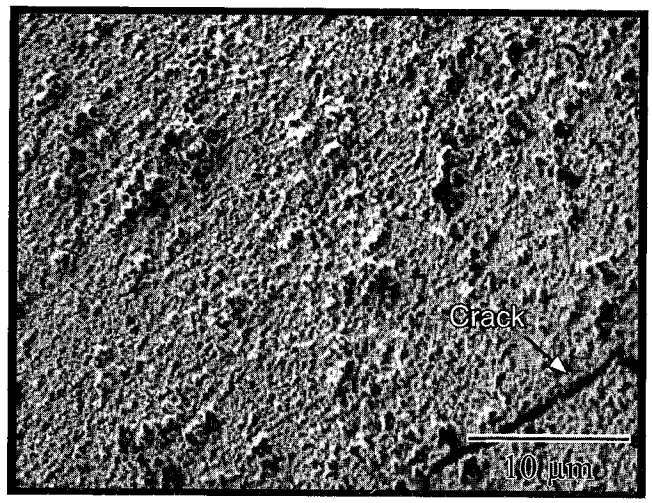

Fig. 6. SEM image of monolithic Ni-plated surface after fatigue (N_f=2.0(±0.3)×10^6 cycles).

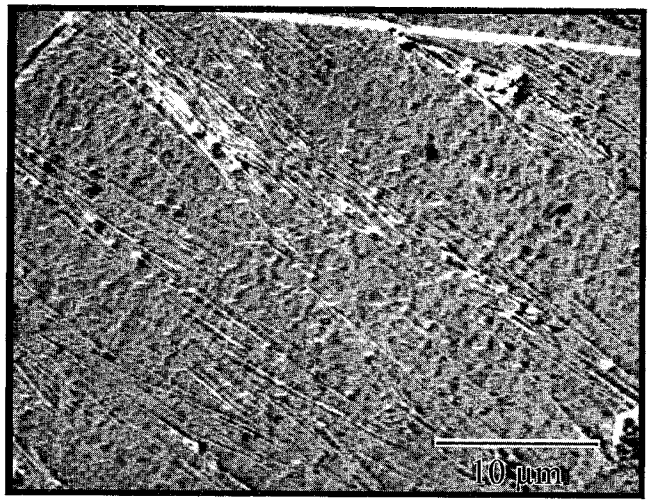

Fig. 7. SEM image of Cu–Ni multilayer-plated surface after fatigue (N>5×10^7 cycles).

coatings on these samples. On the other hand, cracking of the Ni layers was observed and the arrow in Fig. 6 points to a secondary crack.

Fig. 7 is a SEM micrograph of the surface of a multilayer-coated sample after reaching the 5×10^7 load cycles run-out point. Two significant features are exhibited in this figure. The first is the evidence of slip in the multilayer film. The quantity and magnitude of the slip steps on the surface are substantially smaller than those shown in Figs. 4 and 5 even though over 1.5 orders of magnitude additional load cycles have been applied to this sample. The second is the lack of cracking in the film. No evidence was present anywhere on the surface that indicated the coating fractured in a fashion similar to that observed in the monolithic Ni-coated samples (Fig. 6).

There is a wide range of numerical parameters and measurement techniques available to characterize the roughness of a surface [52]. Height-based parameters tend to be the most commonly used single-parameter descriptors for roughness characterizations because they can easily be obtained with stylus profilometry. Roughness parameters of this type usually require the establishment of a mean[1] (or reference) line prior to calculation [53]. The arithmetic mean roughness, or R_a value, is defined as the arithmetical mean of the areas of all profile values within the roughness profile length. The R_a parameter was determined to be appropriate for this analysis because it can account for the variations in the overall roughening behavior of a polycrystalline material in fatigue loading.

[1] A line parallel to the geometrical profile such that the sum of the squares of the deviations from it to the effective profile is a minimum.

The results of the stylus profilometry analyses are presented in Fig. 8. At the lower fatigue cycle values, the roughness of the electropolished Cu did not exhibit appreciable deviations from the as-polished value. While the R_a values did not change significantly, increases were observed in the magnitude of the scatter present in the data. Optical microscopy was performed on those surfaces and the results indicated that slip activity was present in some grains and not in others. This difference was attributed to the fact that those grains were better oriented for slip. As the number of load cycles increased, the magnitude of the R_a increased sharply and then approached a value where cracking was observed. In contrast, the multilayer exhibited a higher initial roughness than the as-electropolished condition, which is most likely due to the presence of Cu dendrites on the surface and to differences in the crystallographic orientations during electrodeposition [16], but no significant changes in the roughness or the scatter were observed during any of the subsequent measurements.

The AFM analyses produced similar results to those obtained by the profilometry. Figs. 9 and 10 are topographic images produced by contact-mode AFM showing the Cu surfaces at the end point of the fatigue testing. The surface of the as-electropolished Cu shown in Fig. 9 after 1×10^6 fatigue cycles has a topography that is virtually identical to the one shown in Fig. 4. On the other hand, the surface of the specimen with the Cu–Ni multilayered coating shown in Fig. 10 exhibits no distinguishable evidence of slip after the application of more than 1.5 orders of magnitude (5.3×10^7) additional fatigue cycles.

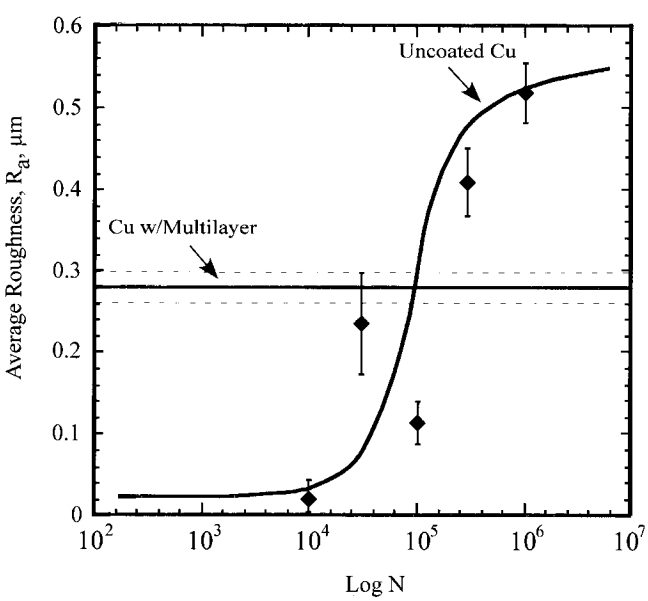

Fig. 8. Change in measured surface roughness with load cycles in rotating bending fatigue. The horizontal and dashed lines show the mean and uncertainty in the surface roughness for the multilayer which did not change significantly from the as-plated condition after 50 million load cycles.

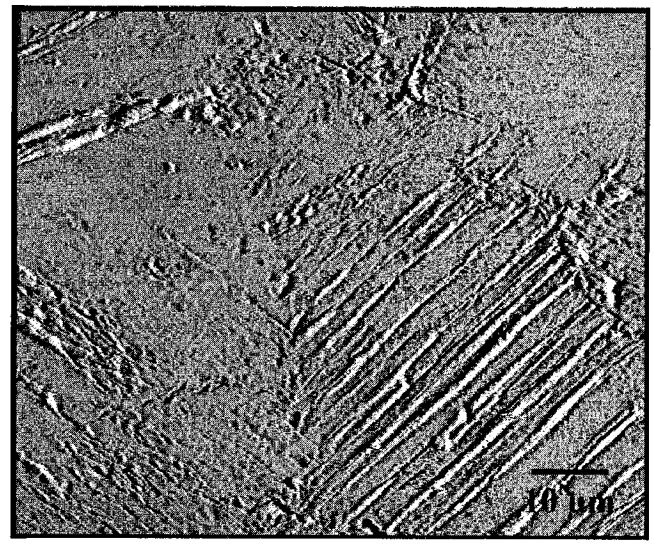

Fig. 9. Contact mode AFM image showing the surface topography of the uncoated Cu after 1.5×10^6 cycles in rotating bending fatigue.

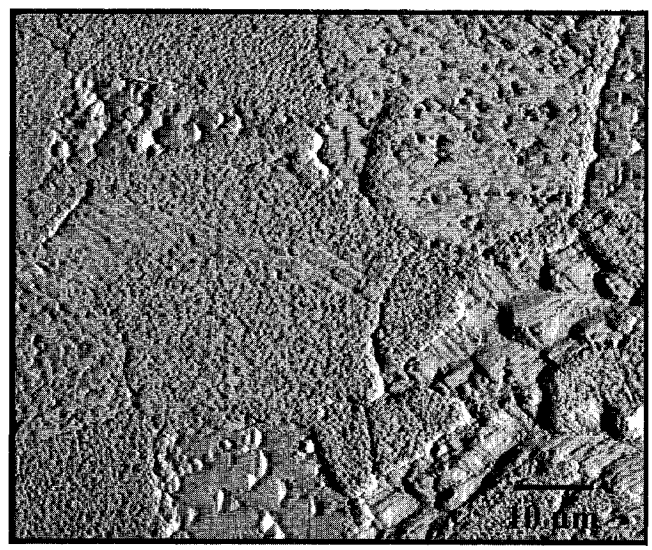

Fig. 10. Contact mode AFM image showing the surface topography of the Cu–Ni multilayer after 5.3×10^7 cycles in rotating bending fatigue.

4. Discussion

The fatigue crack initiation mechanism observed for uncoated Cu in this study is essentially identical to that reported for this material in the literature and the fatigue lives are in the range expected for this grain size [1,3,10,54,55]. In marked contrast to the monolithic Cu and Ni coatings, which had no numerically significant influence on the observed fatigue lives, the Cu–Ni multilayer coating resulted in a dramatic improvement in the

fatigue lives of the Cu substrates. A single low-amplitude cyclic stress level in the high-cycle fatigue (HCF) regime was selected for these experiments for two reasons: (a) crack initiation resistance is the most significant factor determining fatigue life in this stress range and (b) stresses in HCF range are more commonly used in engineering design for fatigue resistance. Because of all the potential influences in the fatigue crack initiation process, fatigue tests in the HCF regime typically have a large scatter. This is why the scatter required careful quantification, but as indicated by the confidence band in Fig. 3, the magnitude of the improvement provided by the multilayer coating far exceeds that required for significance by any statistical test [46–50]. As shown in the literature, the modulus in a multilayer does not deviate significantly from a rule of mixtures [39], yet the yield strengths and hardnesses of these structures can greatly exceed those of the constituent materials [33,39,40]. The deposition of a pure Cu layer of the same thickness as the multilayer had no similar effect on slip behavior as illustrated by Fig. 3. Therefore, the enhanced hardness and resulting toughness produced by nanometer-scale layering of two ductile constituent materials is the most likely source of the slip suppression and homogeneous deformation that prevented fatigue crack nucleation in these samples.

The differences in the character of fatigue-induced roughness present on the surfaces may provide an explanation for the substantial enhancement in fatigue performance of the multilayer-coated specimens. First, there is less evidence of slip on the surface of the Cu–Ni multilayer-coated sample shown in Fig. 5 even though the surface of the multilayer was subjected to over 1.5 orders of magnitude more load cycles than the uncoated Cu sample shown in Fig. 2. The topographic analyses performed on the uncoated Cu samples revealed two basic surface morphologies; both of which are represented in Fig. 2 and in Fig. 9. On grains oriented such that multiple slip systems were active, cross slip and work hardening promoted a relatively random distribution of slip. The result was a highly roughened surface exhibiting the classic "wavy slip" appearance [56]. On grains oriented such that slip occurred primarily on a single plane, regions essentially free of slip were separated by bands of intense slip activity. These bands contain the deep intrusions that eventually spawn fatigue cracks. In contrast, the topography of the multilayer-coated samples revealed sparsely distributed arrays of fine slip steps as shown in Fig. 5. Second, the morphological differences in the cyclic slip present on the surface of the multilayer may be an indication that the deformation promoted a more homogeneous distribution of slip. In addition, the slip present on the multilayer surface suggests that the multilayer did not cyclically soften during fatigue loading. The AFM analyses did not reveal the presence of cyclic slip on the surface of the multilayer, but this is most likely due to the sparse nature of the slip and the fact that the 100 μm×100 μm scan areas used for the AFM measurements are not ideally suited for high resolution characterization of large surface areas.

Even though a surface film must possess hardness for preventing the normal surface roughening mechanisms of fatigue crack nucleation, numerous investigators have found that simply applying a hard film and blocking slip at the surface can only be successful in preventing fatigue failures if secondary crack initiation mechanisms are also prevented. The examples presented earlier by Hornbogen and Verpoort [21] and by Alden and Backofen [17] demonstrated that the applied load and the stresses imposed by dislocation pileups where PSBs intersect the film were sufficient to break these films and nucleate fatigue cracks. In addition, cyclic softening where PSBs intersect a surface film could prevent surface alloying techniques from realizing significant improvements in fatigue resistance [18–20].

The hardness arising from the nanometer-scale layering alone is not sufficient to extend the resistance to fatigue crack initiation. As shown by Su [57], the lack of ductility in TiN multilayered films prevented those films from improving the fatigue resistance of a steel substrate. Unlike the behavior of traditional hard, brittle surface coatings, the Cu–Ni multilayer in this evaluation did not completely block slip activity at the surface. Instead, this particular hard film merely "delayed" the evolution of slip on the surface. In addition to simply providing a hard surface to resist fatigue-induced roughening, the layering of two ductile metals generated a film that appears to have the capacity to deform when the subsurface strains reach critical levels. As a result, the likelihood of crack initiation by secondary mechanisms has been substantially reduced. Several factors could be responsible for the observed resistance to secondary fatigue crack initiation mechanisms with the Cu–Ni multilayer. First, this multilayer is composed of layers of ductile metals, thus it should retain sufficient toughness so that brittle fracture should not occur easily. Second, the high density of interfaces in the multilayer form a barrier to slip that is not readily defeated by the passing of dislocations thereby minimizing the likelihood of localized cyclic softening. Third, the interfaces in the multilayer and work hardening should help to disperse slip near the surface thus creating more a homogeneous change in morphology during fatigue. Fourth, the presence of any residual compressive stresses in the electrodeposited layers may help reduce the magnitude of stresses at critical locations in this microstructure. At this time, the relative significance of each of these factors is unclear and additional experimental evaluations are planned to acquire more information about the role each has in the manner in which multilayer suppresses fatigue crack nucleation.

5. Conclusions

A thorough review of the literature indicated that an ideal surface layer should possess five properties to resist failure by cyclic loading: hardness, toughness, cyclic work hardenability, residual compressive stresses, and adherence. The wide range of properties achievable through the use of nanometer-scale multilayering may enable the synthesis of surface coatings that possess all of these properties necessary to suppress fatigue cracking.

The objective of this work was to evaluate whether a nanometer-scale multilayered thin film coating composed of two normally ductile FCC metals could significantly extend the fatigue life of a high stacking fault energy FCC metal by (a) suppressing the surface roughening that normally causes fatigue crack initiation in these materials and (b) avoiding the subsequent crack initiation mechanisms (i.e., brittle cracking, delamination and cyclic softening) that prevent most hard coatings from realizing significant improvements. This hypothesis was tested by electrodepositing a Cu–Ni multilayer on cylindrical Cu fatigue samples which were tested in a rotating beam fatigue testing machine. The results unambiguously demonstrate that the Cu–Ni multilayer coating significantly increases the fatigue crack initiation resistance of a Cu substrate. Neither of the ductile metals used in the multilayer could induce any significant improvement in fatigue resistance when applied as a monolithic layer with the same total thickness as the multilayer. Therefore, the observed improvement in fatigue life must be a direct result of the structure and properties of the multilayer. It is also concluded that a surface film that possesses a combination of the aforementioned properties provides better resistance to fatigue crack initiation than a hard, brittle barrier layer.

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