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Fatigue Behavior of Electrodeposited Nanocrystalline Nickel Films

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Abstract

Material characterization of thin film for MEMS/nano structures is a building block for the reliability assessment. One of the most significant barriers for reliable MEMS/nano structure is the long term reliability which is different from those of bulk materials. Nano-indenter has been used widely to get the elastic modulus of materials in a very simple way but other testing machines are required for tensile and endurance properties. For the long term reliability assessment of micro/nano structures, a new micro fatigue testing machine was developed to obtain the high cycles fatigue behavior of thin films. Nanocrystalline nickel thin films exhibited significant rate dependency on tensile and fatigue behavior even in room temperature due to grain size related behavior in contrast to coarse grained wrought nickels.

Keywords: nanocrystalline nickel; fatigue; rate dependancy; thin films; tensile; grain size

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1. Introduction

Electrodepositions make polycrystalline films submicron sized grains, thus the mechanical behavior of coarse grain sized one cannot substitute the nanocrystalline (NC) metals due to size effects. In former studies to date, NC metals exhibited unique mechanical behavior, including improved mechanical strength by Hall-Petch relation, suppression of dislocation-mediated plasticity and substantial increase of volume fraction of grain boundary region, suggesting grain-boundary sliding [1].

For MEMS applications, long-term reliability with more than thousand cycles is required, thus plain specimens are needed to assess the fatigue lifetime of structure because crack initiation stage, essentially prior to crack growth,

Nomenclature

NC	nanocrystalline
CG	coarse grain

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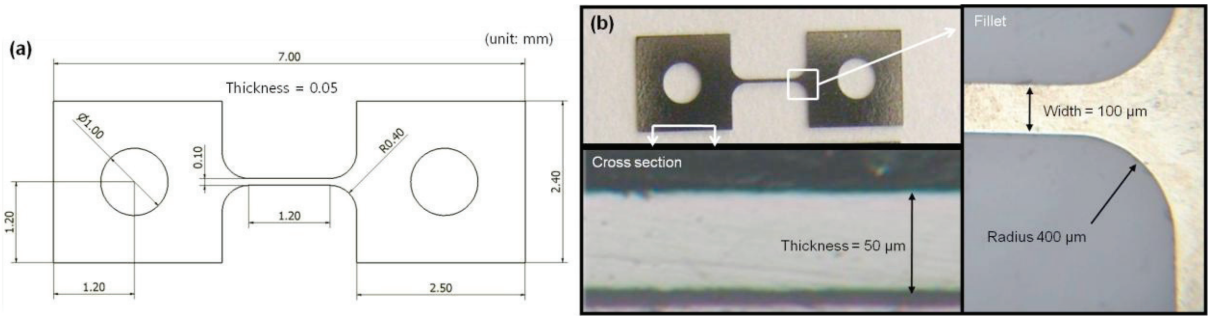

Fig. 1. (a) A drawing of the specimen design; (b) microscopic images of the electrodeposited nickel specimens

occupies most of the fatigue lifetime in high cycle fatigue region [2].

In this paper, free-standing plain nickel specimens were made by electrodeposition for studying fatigue behavior of NC metallic films. For the long term reliability assessment of micro/nano structures, a new micro fatigue testing machine was developed to obtain the high cycles fatigue behavior.

2. Experimental procedures

One of the challenges in testing thin films is inherent residual stress in a deposition process on substrate. By separating film from substrate after thick deposition, the mechanical behavior of films can be measured directly without residual stress in contrast to film on substrate [3] or cantilever type [4].

A sheet-type, 50μm in thickness, with holes at both ends was designed as shown in Fig. 1(a) in accordance with the 'pin-loaded tension test specimen' of the ASTM standard E8-08 [5]. The pin-loading type makes gripping easier and alleviates bending stress in the specimen from misaligned gripping. A nickel sulfamate bath was used to deposit nickel film onto a conductive patterned glass in the form of a patterned mold of a photoresist on a conductive seed layer for electrodeposition. Once the plated nickel was built up to the desired thickness, the electroformed part, the specimen, was stripped off the master substrate. Fig.1 (b) shows the fabricated specimen captured by optical microscopy. For a comparison with coarse grained one, wrought nickel specimens were fabricated with the same geometries by etching wrought nickel foils

For the long term reliability assessment of micro/nano structures, a new micro fatigue testing machine was developed to obtain the high cycles fatigue behavior of thin films. The force is continually monitored by loadcell and controlled in feedback by using electromagnetic coils attached to pin-typed grip while the specimen undergoes monotonic tensile loading or cyclic loading. The displacement is also monitored by measuring movement of grip with laser sensor.

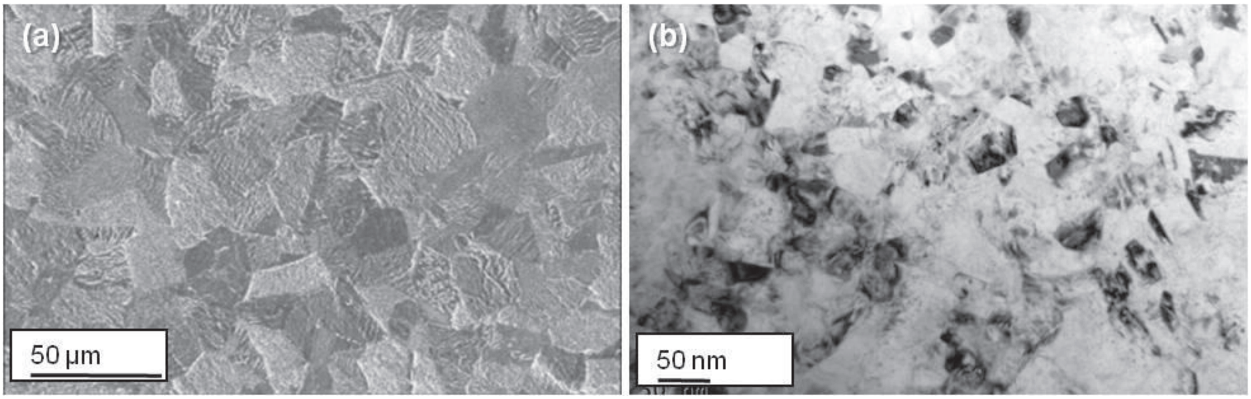

Fig. 2 Microstructures (a) SEM image of wrought nickel, and (b) TEM image of electrodeposited nickel

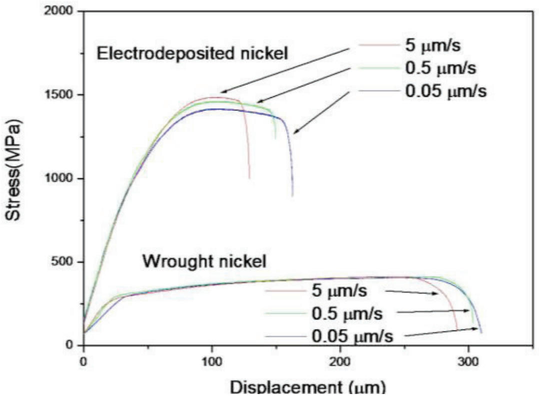

Fig. 3 Load displacement curves under different loading rate for coarse grain nickel and nanocrystalline nickel

For both CG and NC nickels, monotonic axial tensile loadings with three different displacement-controlled rates were perfomed. Displacement control at a rate of 0.5μm/s corresponds to a stress rate of 10MPa/s until the necking occurs and strain rates of 1.0 10^{-4} s^{-1} in the elastic region and 1.2 10^{-3} s^{-1} from yielding to necking. For fatigue behavior, repeated tension-tension tests with 0.2 load ratio (the ratio of the minimum stress to maximum stress in the fatigue loading) were applied to both CG and NC nickel specimen, then fracture surfaces were investigated by SEM(scanning electron microscopy). For NC nickel, two different load ratios and cyclic loading frequencies were performed additionally to investigate the loading rate dependancy.

3. Results

The surface grain structure after polishing is shown Fig. 2(a) for CG nickel and Fig. 2(b) for NC nickel. The mean grain size of CG nickel is approximately 30 μm with narrow distribution. For NC nickel, mean grain size of 40nm was estimated with unimodal distribution from quantitative image analysis, thus the grain size is uniform through the whole specimen for both CG and NC.

Fig. 3 shows load-displacement curve for CG and NC with three different displacement stroke rates. NC nickel showed much higher ultimate tensile strength, higher yield strength and lower elongation compare to CG nickel by Hall-Petch relationship, grain boundary strengthening mechanism. NC nickel, however, shows positive strain rate dependency significantly within strain rates of just two orders of magnitude even in room temperature, while wrought nickel does not show the rate dependency as typical in CG material. With increasing loading rate of NC nickel, a higher stress is required and the ultimate tensile strength increases. Molecular dynamic simulations of mechanical deformation of NC metal suggest that grain-boundary atoms are involved in plastic deformation significantly for more volume fraction and easier to deform that the associated deformation mechanisms are likely to be rate-sensitivity [6].

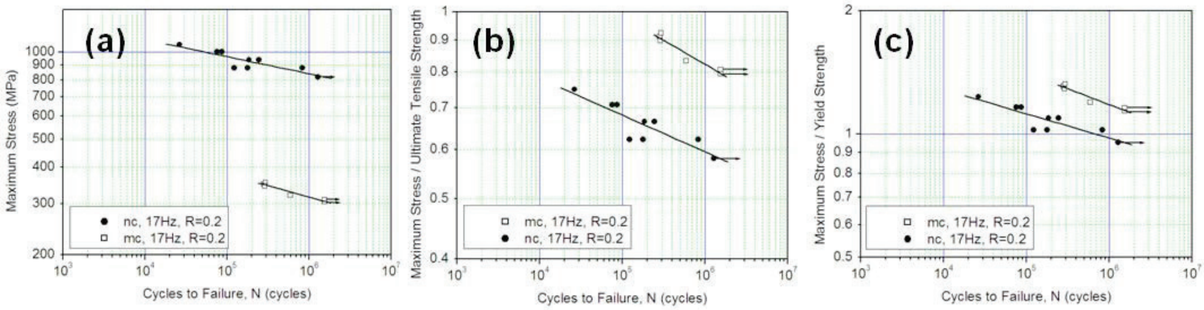

Fig. 4 Stresses-versus cycles to failure of nanocrystalline nickel and coarse grain nickel under 17Hz and, R=0.2; (a)maximum stress, (b) normalized by ultimate tensile strength, and (c) normalized by yield strength

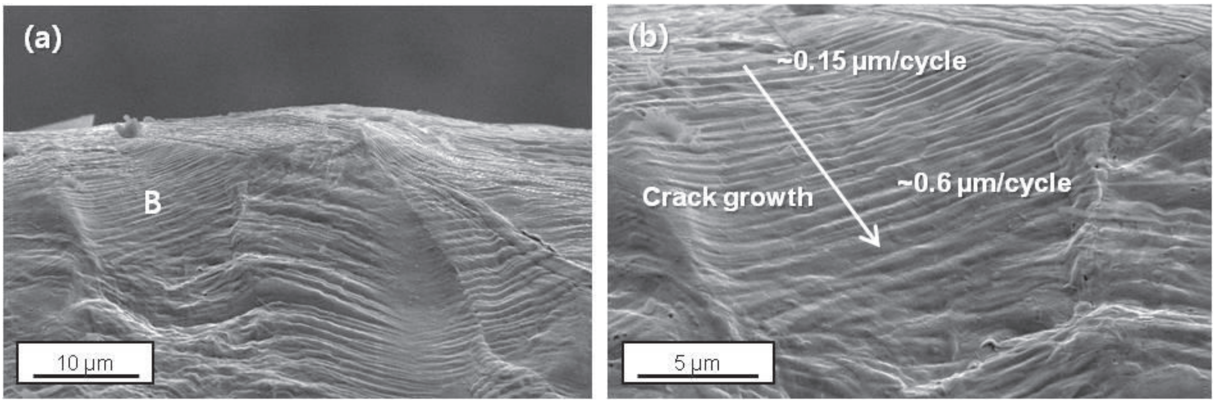

Fig.5 Fractured surface of coarse grain nickel after 2.8×10^5 cycles under 17Hz, max. stress of 350MPa and load ratio, R=0.2. (a) crack initiation at top and growth; (b) magnification of B mark in Fig. 5(a)

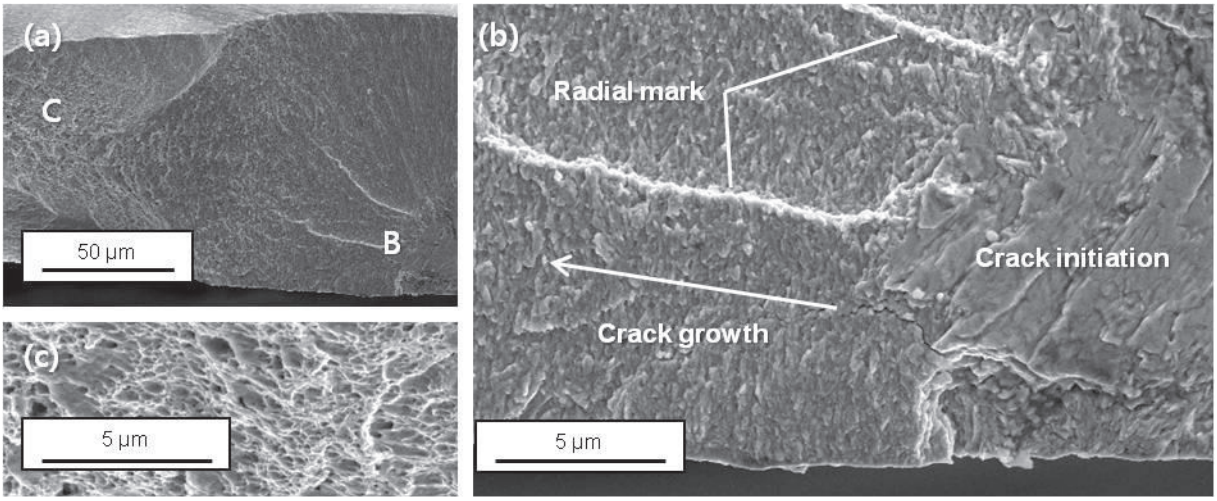

Fig. 6 Fractured surface of nanocrystalline nickel after 3.0×10^5 cycles under 131Hz, max. stress of 940MPa and load ratio, R=0.2. (a) Total fractured surface; (b) magnification of B mark and (c) magnification of C mark in Fig. 6(a) respectively.

Fig. 4(a) shows high cycle fatigue life results for CG and NC nickels under 17 Hz. Repeated tension-tension loads were applied to specimens with load ratio R of 0.2 to prevent film buckling, thus mean stress causes ratchetting in cyclic behavior, i.e. continuous mean strain increments. NC nickel's fatigue strength in terms of maximum stress in high cycles region, $10^4 \sim 10^6$ cycles, is much higher than CG nickel. The maximum stress normalized by the untimate tensile strength shows lower value than the CG nickel as shown in Fig. 4(b). Even in high cycle fatigue where loading conditions are nominally elastic in overall, plastic deformation occurs locally and makes crack initiation in CG metals due to the formation of surface extrusion and intrusion caused by persistent slip bands, thus yield strength is correlated to crack initiation. And crack initiation process occupies almost lifetimes in case of high cycled fatigue region because specimen has no cracks or voids, thus there is relationship between high cycles fatigue lifetime and yield strength. Fig.4(c) shows the maximum stress normalized by the yield strength versus fatigue life for NC nickel and CG nickel, which shows that yield strength correlates with fatigue life better than ultimate tensile strength shown in Fig. 4(b). Fig. 5(a) shows fatigue fracture surfaced of CG nickel. The crack initiated from top of specimen and then grew downward while making striation caused by each cycle as it seen in typical wrought nickels. Radial marks between striation groups indicate multiple crack initiation origins as a result of local stress concentration. The number of cycles occurred by crack growth was estimated as hundreds, relatively few cycles to total lifetime 3.0 10^5 as shown in Fig. 5(b).

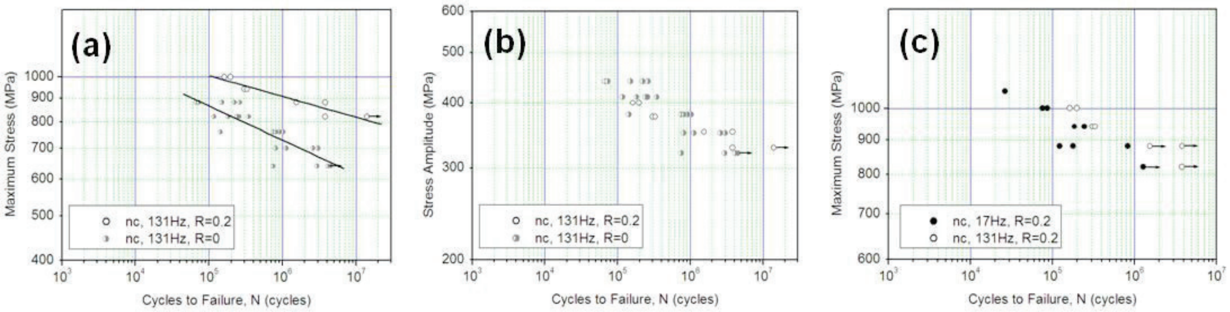

Fig.7 Stress amplitude versus cycles to failure of nanocrystalline nickel

For NC nickel, crack initiation, growth and final rupture was seen also as shown in Fig. 6(a). One noticeable thing is localized grain growth in crack initiation zone, as shown in Fig. 6(a). The grain size of crack initiation zone is large as well as CG so that enable to form surface extrusion and intrusions caused by persistent slip bands which were suppressed due to nano sized grains. Parallel lines in crack initiation zone look like slip bands formed by dislocation activity as typical in CG metals. For crack growth, striation was not seen in NC but brittle-like facture surface along the grain boundary, as shown in Fig 6(b). The fraction of cycles by crack growth could not be estimated directly, however, the fraction of crack growth to total lifetime would be still small because the crack growth rate of NC is faster than CG due to small-crack effects [7]. As grain size is refined, the tortuosity of fatigue cracks is reduced so that shielding and crack closure associated with crack deflection may be minimized. Moreover, with limited traditional dislocation-mediated plasticity, it is unlikely that plasticity-induced crack closure remains a substantial mechanism in NC [1]. It seems that crack growth occurred along the grain boundary without substantial deformation. And finally then, ductile fracture occurred catastrophically while making dimples, as shown in Fig. 6(c).

For NC nickel, high cycle fatigue tests with two different load ratios were performed to assess the mean stress effects. Fig. 7(a) shows that maximum stress is not a proper parameter for fatigue life of NC nickel, while Fig. 7(b) shows that stress amplitude correlates fatigue life well with insignificant mean stress effect. Frequency effects on high cycle fatigue life of NC nickel were also investigated with 131Hz and 17 Hz under same load ratio of R=0.2. Significant frequency effects were observed at 17Hz and 131Hz such that longer fatigue life was obtained at higher frequency as shown in Fig.7(c). In high cycle fatigue region crack growth life is negligible compared to the crack initiation life, and the grain growth in crack initiation stage of NC nickel is affected by the strain rate, which induces the longer fatigue life at the higher frequency test.

In conclusion, NC nickel undergoes fatigue assisted grain growth to CG level simultaneously or prior to crack initiation which enables traditional crack initiation mechanism, dislocation–mediated slip, and then crack grows along the NC grain boundaries without crack tip blunting mechanism by limited dislocation activity. Positive strain rate sensitivity exists significantly in NC material even in room temperature so that affects tensile behavior after yielding and fatigue behavior by affecting grain growth at crack initiation stage. For improving fatigue strength of NC film, suppressing fatigue assisted grain growth of NC at crack initiation stage is the key.

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