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SAND2001-0912
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Printed April 2001

Surface Hardening by Nanoparticle Precipitation in Ni(A1,0)

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Surface Hardening By Nanoparticle Precipitation In Ni(A1,O)

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Nanostructure & Semiconductor Physics

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Abstract

Ion implantation of O and Al were used to form nanometer-size precipitates of NiO or Al_2O_3 in the near-surface of Ni. The yield strengths of the treated layers were determined by nanoindentation testing in conjunction with finite-element modeling. The strengths range up to ~5 GPa, substantially above values for hard bearing steels. These results agree quantitatively with predictions of dispersion-hardening theory based on the precipitate microstructures observed by transmission electron microscopy. Such surface hardening by ion implantation may be beneficial for Ni components in micro-electromechanical systems.

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Introduction

Shear-resistant precipitates in metals impede the glide of dislocation and thereby increase the applied stress required for plastic deformation. Continuum-models analysis indicates that the resultant yield strength is approximately proportional to the reciprocal of the average spacing between particles, so that refinement of the precipitate microstructure to the nanometer scale is predicted to result in very high strengths [1]. In fact, however, the degree to which this scaling extends into the nanometer range is an open scientific issue because of the inevitable emergence of alternative deformation processes at sufficiently high stresses and the increasing inapplicability of continuum mechanics at microstructural scales comparable to the lattice parameter. In the present investigation, we addressed this question experimentally through micromechanical testing of Ni layers containing highly refined oxide-particle dispersions introduced by ion implantation. A further motivation for the work is the potential utility of ion implantation for surface hardening of Ni components in micro-electromechanical systems.

Procedure

Nickel discs with a nominal purity of 99.99% were first vacuum-annealed at 1000ºC for 2 hours, resulting in a grain size of several tenths of a mm. Oxygen was then ion-implanted at room temperature to a concentration of about 8 at.% in the depth range 0-0.2 μm. An approximately uniform concentration was achieved through implantation at multiple energies: 3.0×10^{16} O/cm^2 at 50 keV, 3.3×10^{16} O/cm^2 at 100 keV, and 8.5×10^{16} O/cm^2 at 150 keV. In some cases an equal concentration of Al was also implanted, the sequence of treatments being 2.0×10^{16} Al/cm^2 at 50 keV, 2.0×10^{16} Al/cm^2 at 100 keV, and 10.0×10^{16} Al/cm^2 at 180 keV.

Three types of specimen with different microstructures in the implanted region were subjected to mechanical testing. First, we examined Ni implanted only with O and not annealed. Transmission electron microscopy (TEM) showed that this treatment produced a homogeneous dispersion of NiO precipitates with sizes in the range 1.5-3.5 nm. The volume fraction of the oxide phase in the layer can be calculated from the average concentration of O, 8 at.%, and the known density of NiO, giving 14 vol.%. Second, other samples implanted in the same way were subsequently vacuum annealed at 550ºC for 2 hours, which changed the range of precipitate sizes to 7-20 nm while maintaining constant volume fraction. Finally, the material implanted with both O and Al was annealed at 550ºC for 2 hours in order to drive the formation of Al_2O_3 to completion. Transmission electron microscopy confirmed this final microstructural condition, and showed also that the highly stable γ-Al_2O_3 phase underwent little ripening during the heat treatment, with the precipitate size range being 1-2 nm. The volume fraction in this case is 12 vol.%. Figure 1 shows a cross-section TEM image in which the Al_2O_3 particles appear as light spots.

The mechanical properties of the implantation-formed microstructures were determined by nanoindentation testing, wherein a diamond probe impinged progressively to a depth of 0.16 μm with force being measured as a function of depth. In order to take account of the influence of the unimplanted substrate and so extract the intrinsic yield strength of the treated region, the mechanical response of the specimen was modeled using finite-element methods discussed elsewhere [2].

Results and Discussion

The yield strengths obtained for the three microstructures are given in Table I together with other pertinent information. Included for comparison is the strength of pure Ni. The high strengths produced by the nanoparticle dispersions are noteworthy, being about twice the value for hard bearing steel and ~30 times that for pure Ni. This demonstrates a new level of dispersion hardening in Ni-Fe materials. Moreover, it reinforces the potential utility of ion implantation for hardening of Ni components in micro-electromechanical systems.

A continuum-matrix analysis of the interaction of dislocations with a dispersion of spherical, shear-resistant precipitates gives for the resultant increment in yield strength

$$\Delta Y = \left(\frac{0.84}{2\pi(1-\nu)^{1/2}} \right) \frac{\mu b}{\lambda} \ln\left(\frac{d}{b}\right) \qquad (1)$$

where ν is Poisson's ratio, μ is the shear modulus of the host metal, b is the length of Burger's vector, λ is the separation between precipitates, and d is the precipitate diameter [1]. Applying this equation to the results of the present experiments, we take ν = 78 GPa, μ = 0.312, and b = 0.28 nm from the literature [3], substitute d from Table 1, and estimate λ by using the known values of d and precipitate volume fraction and assuming that the particles occupy a close-packed fcc array. A comparison between theory and experiment is made in Fig. 2 for the three investigated microstructures, where the horizontal coordinate gives the values of ΔY from Eq. (1) and the vertical coordinate is the yield strength obtained from nanoindentation. Exact agreement corresponds to the dashed line.

The agreement between theory and experiment in Fig. 2 is seen to be good, both in absolute magnitude and in the variation among microstructures. It thus appears that the continuum analysis of hard-particle dispersion strengthening remains applicable for particle sizes and separations approaching 1 nm.

References

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Table I. Properties of implanted Ni from TEM and nanoindentation testing.

Ppt. phase	Ppt. vol. fract.	Avg. ppt. size	Ppt. spacing	Yield strength
None				0.15 GPa
NiO	14%	2.5 nm	1.9 nm	4.6±0.5 GPa
NiO	14%	13.5 nm	15.8 nm	1.15±0.12 GPa
Al_2O_3	12%	1.5 nm	1.3 nm	4.2±0.3 GPa

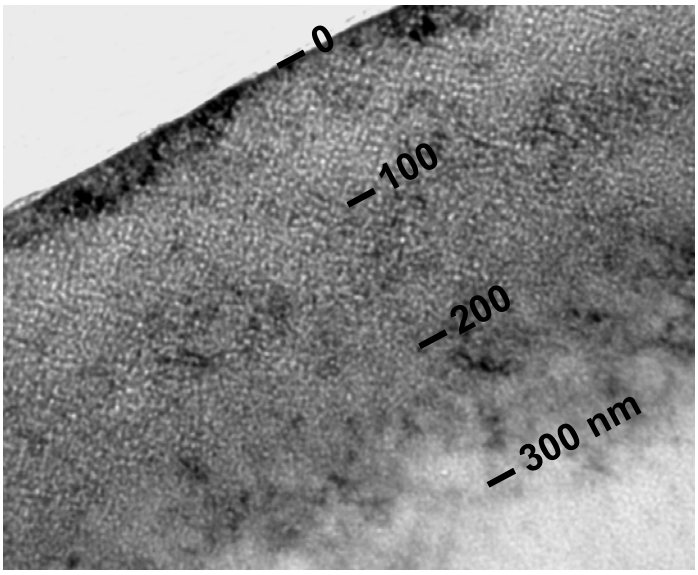

Figure 1. Cross-section TEM of Al_2O_3 precipitates in Ni implanted with O and Al and then annealed at 550ºC. The bright-field image was obtained in slight underfocus, causing the oxide particles to appear as light spots.

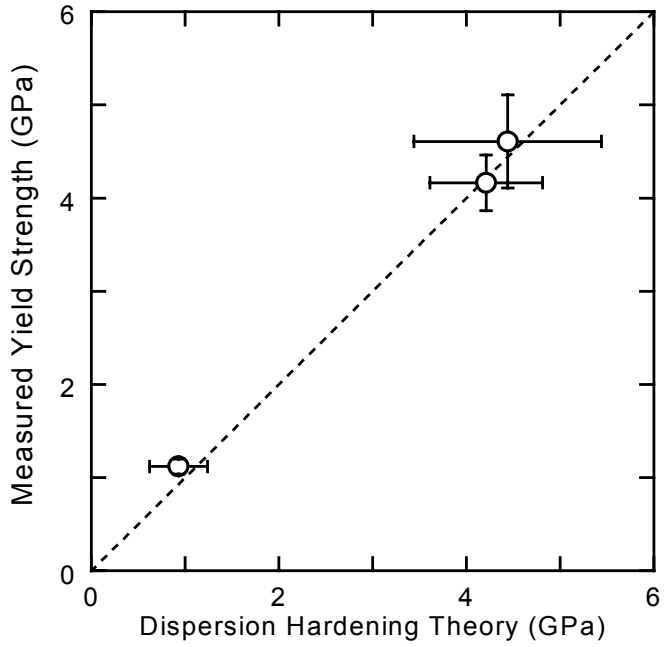

Figure 2. Comparison between measured and theoretically predicted yield strengths of Ni containing oxide precipitates. The dashed line corresponds to exact agreement.

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