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ELECTRODEPOSITION OF NI FROM A SULFAMATE ELECTROLYTE

Part I. Effect of a Stress Relief on Annealing Behavior and Film Metallurgy

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ELECTRODEPOSITION OF NI FROM A SULFAMATE ELECTROLYTE

Part I. Effect of a Stress Relief Agent on Annealing Behavior and Film Metallurgy

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ABSTRACT

Ni and Ni alloys are being developed as baseline materials for LIGA technology and prototyping at Sandia National Laboratories. A conventional, additive-free sulfamate electrolyte has been chosen for pure Ni electrodeposition due to its simplicity and ability to produce ductile, low-stress films. When depositing certain Ni alloys, saccharin is typically employed as an electrolyte bath additive. While saccharin is well known and effective as a stress reliever, it has a significant impact on the microstructure of the deposit and its annealing behavior.

The electrodeposition of pure Ni in the presence of saccharin is studied here to understand its effects in the absence of an alloying element (such as Co or Fe). The grain structure and Vickers hardness of Ni deposited with and without saccharin on a rotating disk electrode were all found to be consistent with previous studies available in the literature. The following observations were made:

1) The fine, columnar morphology obtained without saccharin became an equiaxed, nano-sized grain structure with saccharin (from ~1.5 μm to ~40 nm nominal grain size, respectively). The grain refinement resulting from saccharin is not accompanied with an increase in film stress, in contrast to the grain refinement associated with certain Ni alloys.
2) A change in the deposit texture from weak (210) to (111) along the film growth direction with the addition of saccharin.
3) An increase in Vickers hardness by a factor of ~2 (from ~170 to ~320) upon the addition of saccharin.
4) A rapid decrease in hardness with annealing from the high, as-deposited values for films deposited with saccharin to a value lower than that of annealed Ni from an additive-free bath.
5) Accelerated grain growth during annealing for films deposited with saccharin; this has not been observed previously in the literature to the authors' best knowledge.

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CONTENTS

INTRODUCTION

Studies of Ni electrodeposited from sulfamate electrolytes are not uncommon in the literature (in fact, several exist from this laboratory).[1-9] The sulfamate chemistry produces Ni having the lowest stress, highest ductility, and highest purity of all the available Ni electrodeposition chemistries; physical property data are available in the literature.[1-10] For example, it is possible to deposit low stress Ni from a sulfamate bath with <10 ppm incorporated S, 25-30% elongation to failure, and 313-470 MPa (46-68 ksi) yield strength.[5,10] Although such data exist from other laboratories, it is necessary to validate the Ni electrodeposition process currently employed at Sandia Livermore. Since we plan to characterize the effects of parameters unique to through-mask electrodeposition (topics not well-addressed in the literature, such as fluid flow effects, mold geometry, and pulsed deposition parameters, for example), the need for a thorough initial "baseline" study seems more essential. This report represents the initial process study.

Electrodeposited Ni films are contrasted with those produced under similar conditions but with the electrolyte bath additive saccharin. Saccharin is a stress relief agent that is often used during Ni alloy deposition (necessary for Ni-Fe and occasionally used for Ni-Co)[10] and to brighten electrodeposited Ni.[11-14] Although in practice its use is avoided in ductile Ni plating (since it contributes ~100 ppm S to the deposit), the comparison is fundamentally important. Studying saccharin with an elemental system (Ni) instead of an alloy permits one to decouple effects due to the additive and those due to the alloying element. As the use of S-containing stress relievers (such as saccharin) is problematic when the deposit is annealed,[15] the insight gained from studying its effect on pure Ni deposition may be useful in minimizing stress for pure Ni and Ni-alloy deposition.

EXPERIMENTAL

Materials Fabrication Procedure

All Ni films deposited for cross sectioning were produced at 50°C with unsparged solutions of 1.54 M $Ni(SO_3NH_2)_2 \bullet 4\ H_2O$ (497 g/L) and 0.73 M (45 g/L) boric acid; 0.2 g/L of sodium dodecyl sulfate (SDS) was added as an anti-pitting agent. This electrolyte composition is typical for a Ni sulfamate bath,[16] in accord with manufacturer specifications,[5] and summarized in Table 1. All chemicals were certified ACS grade. As commonly recommended by the manufacturer, S-depolarized Ni was used as a counterelectrode in a two-electrode arrangement. Where noted, certain deposits were made with the same electrolyte outlined above but with the addition of 2 g/L of the sodium salt of saccharin. The use of sulfur-containing additives such as saccharin typically introduces ~100 ppm of S to the deposit.[10] It should be noted that, under typical operating conditions (used here), (*i*) the S in the counterelectrodes and in the SDS is stable and does not contribute S to the deposit, and (*ii*) the additive saccharin is not typically used for plating pure, ductile Ni.[1-10] The pH of the electrolyte was always between 3.5 and 4.0, and the deposited film thickness for cross sectioning was ~75 μm.

Constituent	Specification
$Ni(SO_3NH_2)_2 \bullet 4\ H_2O$	1.54 M (497 g/L)
H_3BO_3 (boric acid)	0.73 M (45 g/L)
sodium dodecyl sulfate (SDS)	0.2 g/L
saccharin (added as sodium salt, where noted)*	2 g/L
Operating temperature	50°C
pH (@ 50°C)	3.5-4.0
Conductivity (@ 50°C)	0.080 S/cm

*Saccharin is not normally added to Ni sulfamate baths for ductile Ni plating.

Table 1. Electrolyte composition and properties

Cu disks polished to a 400-grit finish (3.8 cm^2 in area, 99.9 %, Johnson Matthey) were used as substrates. It was verified that the surface preparation of the Cu substrate had no significant effect on the experimental results. The Cu disks fit into holders that were attached to standard electrode rotators (Pine). A rotation speed of 100 rpm was used for all experiments to approximate fluid flow conditions for the process currently in use for Ni

deposition. Current densities of 15 and 40 mA/cm^2 were employed for deposition; it was verified that in this range, the deposition current density effect on the observed film structure is negligible as compared to that of saccharin (see Figure 1). All micrographs shown and hardness values are at 15 mA/cm^2, except for TEM micrographs of saccharin-free Ni; these were deposited at 40 mA/cm^2. The effects of larger changes in processing parameters such as fluid flow, electrolyte composition, and deposition current density on film microstructure will be considered in another study. A PAR 273A potentiostat was used to obtain polarization curves with a Pt rotating disk electrode (Pine). The area of the Pt electrode was 0.126 cm^2. Heat treatments on certain samples were carried out in a vacuum of 1×10^{-5} torr. The samples were introduced into the chamber at room temperature and ramped up to temperature in 30 minutes. They were left for one hour, and then cooled to room temperature in 30 minutes.

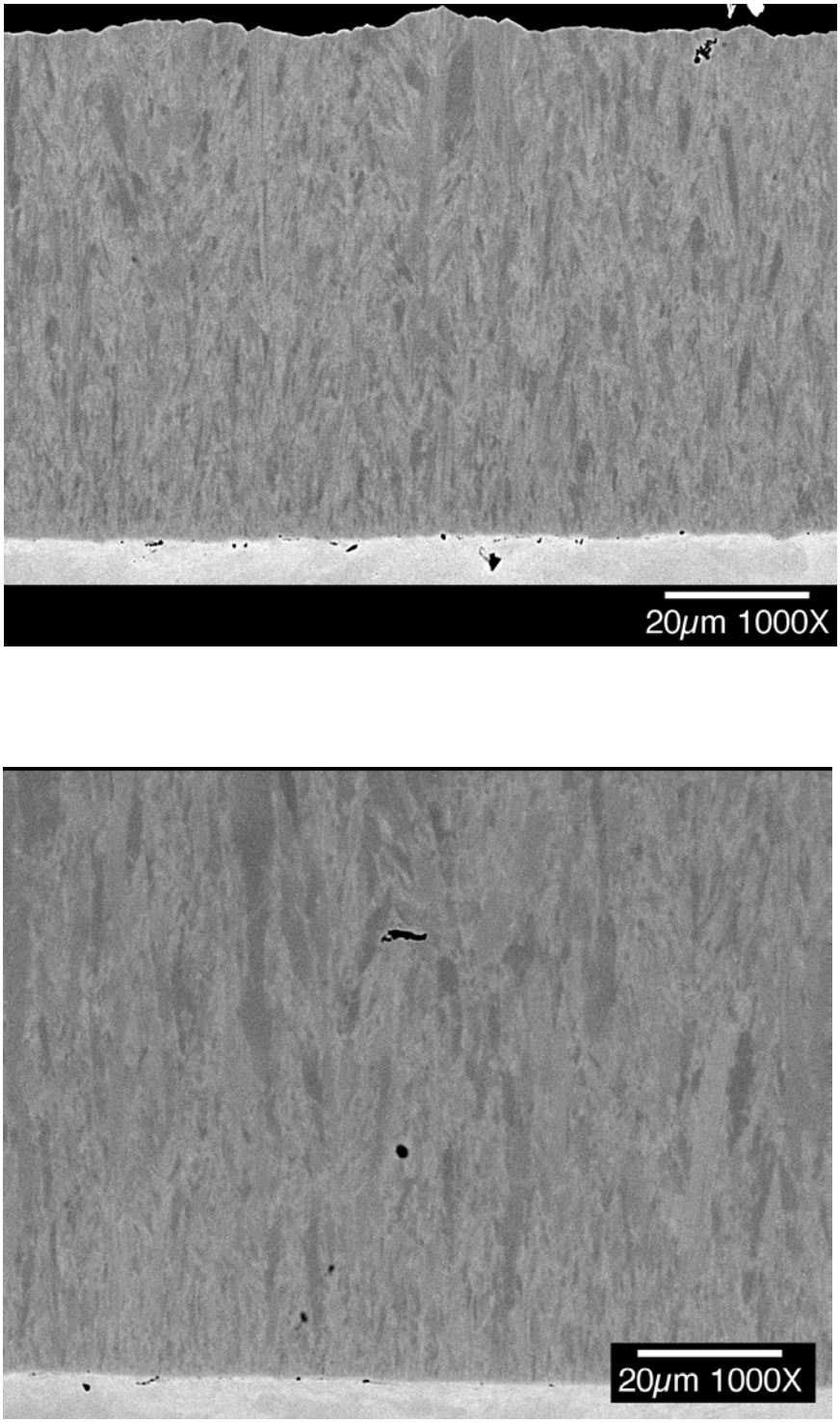

Figure 1. Comparison 15 (top) and 40 (bottom) mA/cm^2 current density morphology. Both films were produced without saccharin, cross-sectioned, and imaged using SEM-BEI.

Metallurgical Examination

Observations of the deposits were conducted on sample longitudinal cross sections that were prepared by conventional metallographical mounting and polishing. Scanning electron microscopy/backscattered electron images (SEM/BEI) were obtained on JEOL model 840 and 6400F (field emission) scanning electron microscopes. All transmission electron microscope (TEM) micrographs presented are bright field images from a Philips CM 30 TEM. The intercept method was used to determine nominal grain diameters as described in ref. 17, except for the as-deposited Ni films made with saccharin, where the grain size was estimated from TEM micrographs. The sample microhardness was measured using a LECO M400 microhardness tester. A 25 g load and a Vickers indenter was used directly into the cross section of the deposited film. A minimum of four measurements were made. Reported hardness values are the averages of all measurements, while the error bars are their standard deviation. Film texture was examined using grain orientation maps and pole figures generated from the JEOL 6400F SEM fitted with an electron backscatter diffraction (EBSD) system (Oxford Instruments ISIS-OPAL).

RESULTS AND DISCUSSION

Electrode Kinetics

Figure 2 shows steady state polarization curves for the Ni deposition reaction. Curves were taken potentiostatically and corrected for ohmic drop.[18] The rotation rate has little effect on the observed current; this is consistent with the fact that the sulfamate chemistry exhibits a high limiting current for Ni^{2+} reduction.[5] The effect of the deposition current, mixing conditions, and other operating parameters on film morphology will be considered in another study. Their effects on film structure relative to those of additives are expected to be small.[19]

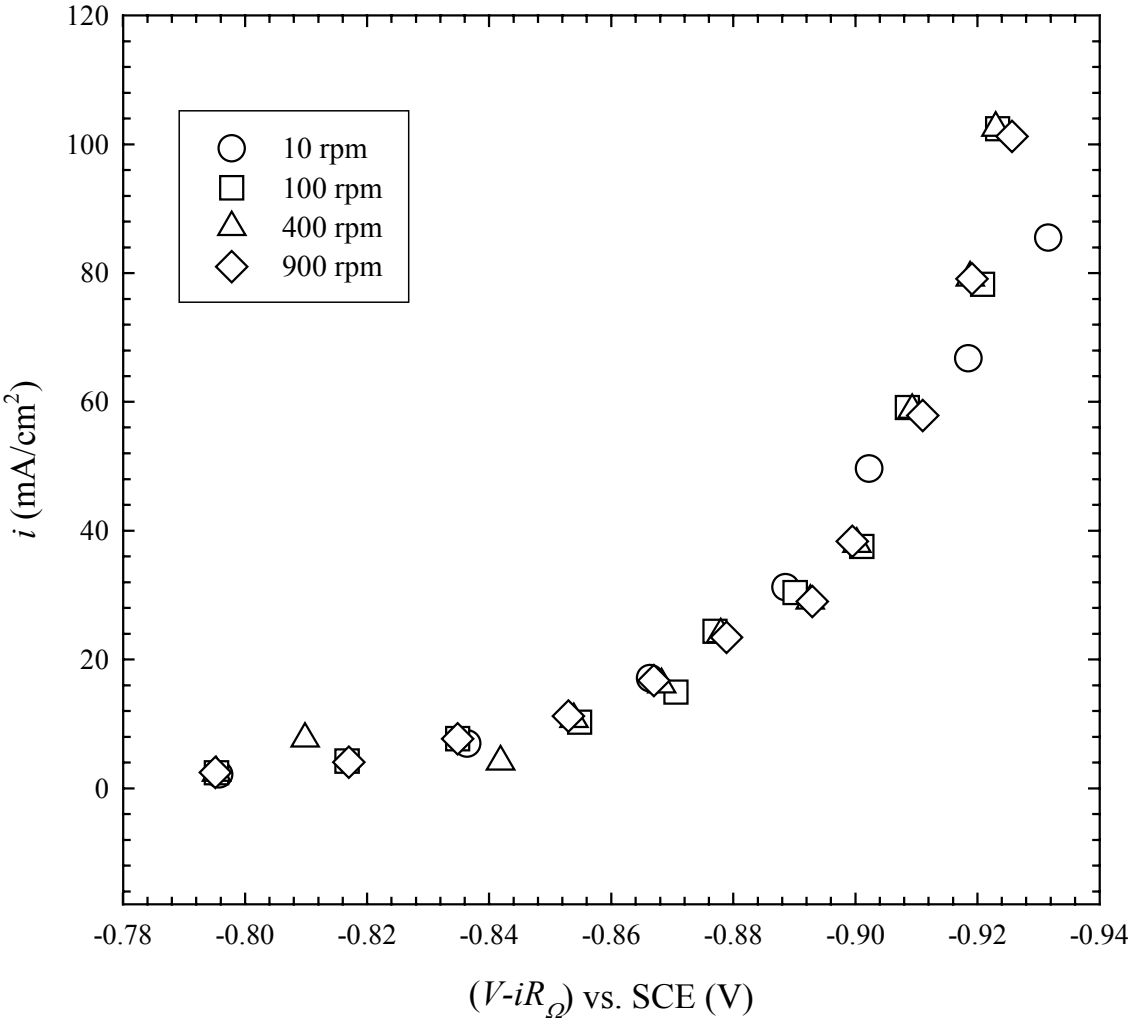

Figure 2. Steady state polarization curves for the saccharin free Ni sulfamate electrolyte. Potentials are plotted versus saturated calomel electrode and are corrected for ohmic drop. The deposition current densities of interest (10-40 mA/cm^2) are well below the limiting current density since the rotation rate does not have a great effect in this range.

Grain Structure

SEM-BEI images in Figure 3 shows the effect of saccharin on the grain structure of the as-deposited film cross section. For the film deposited without saccharin, fiber-like (or columnar) grains perpendicular to the substrate are evident and consistent with the literature,[4,6,10] whereas for the film deposited with saccharin, no structure is apparent. The free surface of the film deposited without saccharin is less smooth than that of the film deposited with saccharin, contributing to the brightening of the deposited surface. Saccharin is well known as a grain-refining agent that reduces stress in the deposited films, in some cases eliminating the columnar morphology associated with the electrodeposited iron group metals altogether.[20] The effect in Figure 3 is consistent with previous work in that the grain structure of the Ni film deposited with saccharin is refined to the point where it is not easily observable *via* SEM.

Figure 4a is a cross-sectional TEM micrograph of a Ni film deposited without saccharin. The central portion of the micrograph consists of a single grain (confirmed by dark field imaging and selected area diffraction); the dark features are subgrains that are defined by high concentrations of lattice defects, most probably dislocation tangles.[21] Arrows in the micrograph show the clear grain boundary. The micrograph in Figure 4b shows a cross section of a film deposited with saccharin; the grain structure is very fine, the average grain diameter being about 40 nm. Although there is no clear columnar morphology, the grains appear to be elongated in the film growth direction. Both samples were made from near the top of the film, where the grains tend to be larger for the Ni deposited without saccharin (see Figure 3).

Figures 5a and b are TEM micrographs showing the as-deposited film-substrate interface (note the difference in scale). For the film deposited without saccharin, the grains next to the interface are relatively fine and evolve into larger, columnar ones about 2 μm away from the interface. Much twinning is apparent within the columns. This twinned, columnar structure is very similar to a fine, “field texture” oriented film as classified by Fischer.[22-23] The film deposited with saccharin exhibits a fine grain structure similar to that in Figure 4b, but at this magnification the high degree of twinning is apparent. This twinning is on a much finer scale than that for the non-saccharin film and could contribute, along with the fine grain structure, to its differing mechanical properties (discussed below).

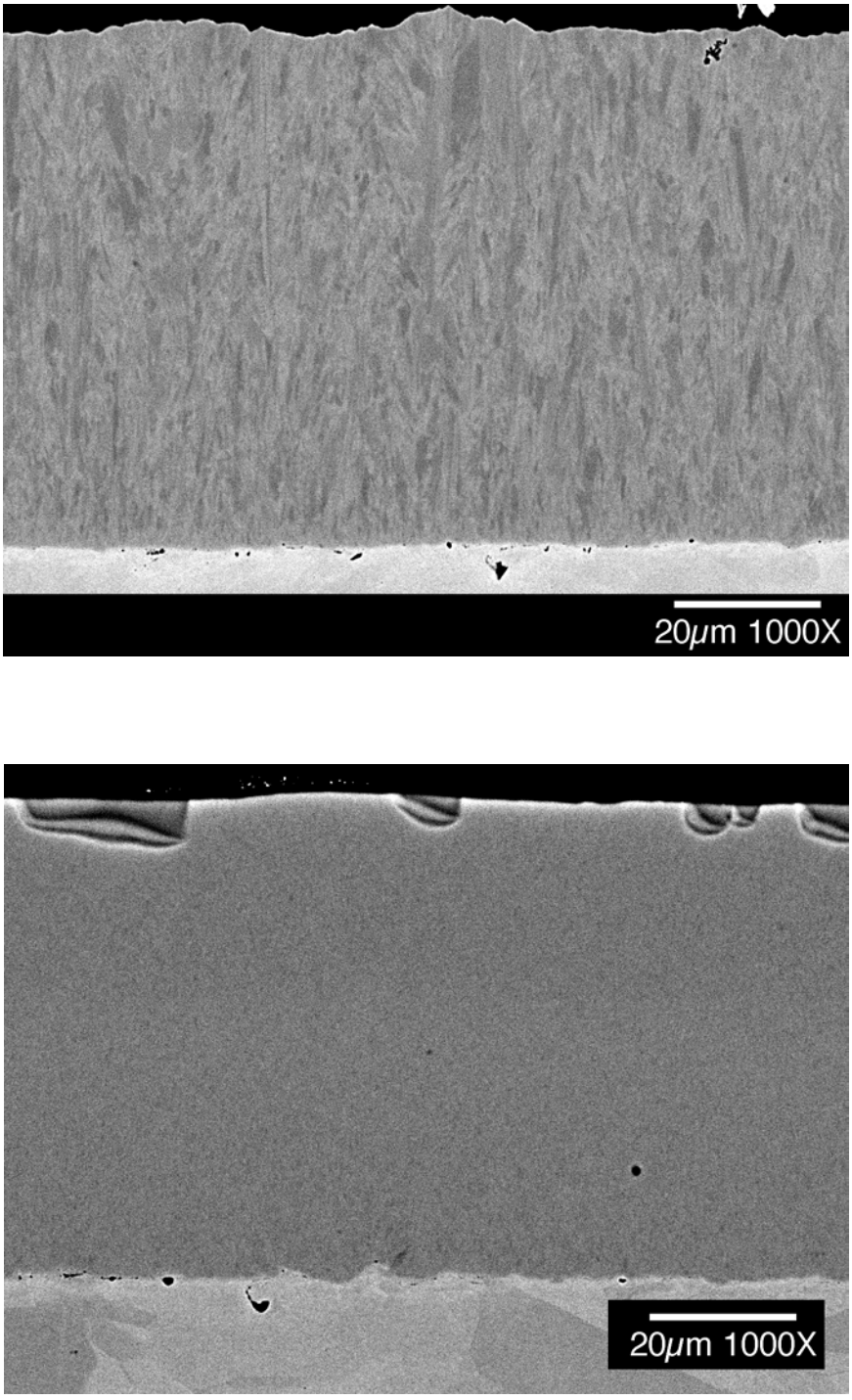

Figure 3. SEM-BEI images of Ni film with (bottom) and without (top) saccharin. Saccharin refines the columnar morphology of the Ni film obtained in its absence, reducing the nominal grain size from ~1.5 μm to ~40 nm (determined *via* TEM).

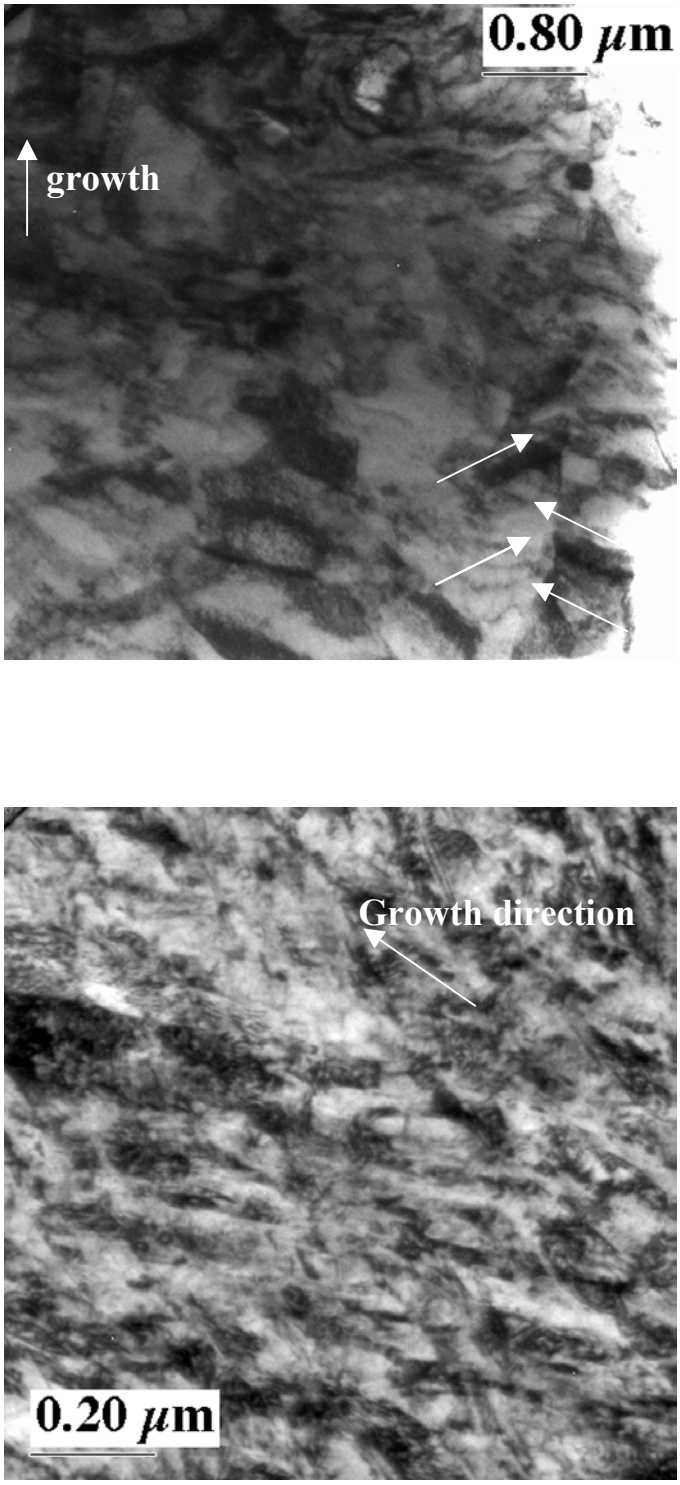

Figure 4. (a) Cross-sectional TEM micrograph of Ni from a saccharin-free electrolyte. Arrows marks the only grain boundary visible in this region; the dark features towards the center are subgrains. (b) same as (a) but with saccharin. Grains are slightly elongated in the growth direction, but the columnar morphology is absent. Both (a) and (b) are towards the top of the film surface (away from the substrate interface).

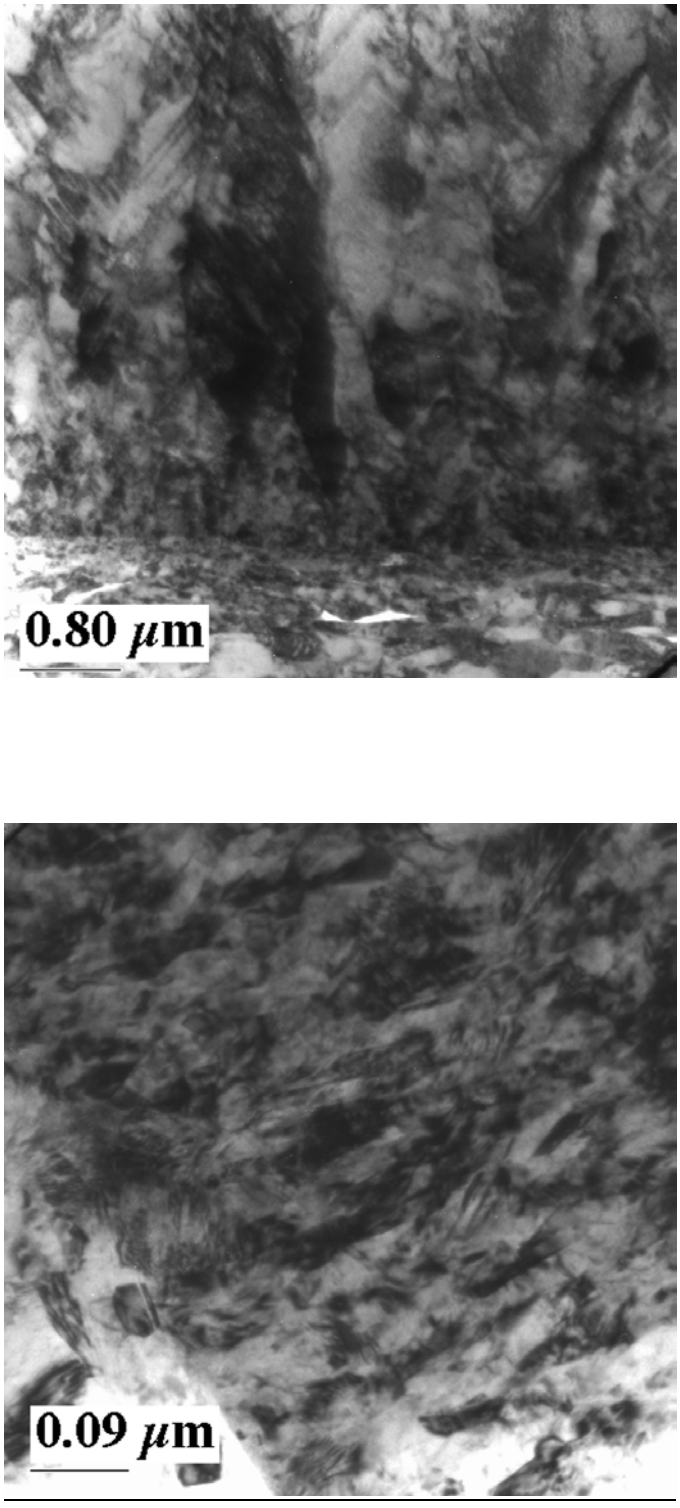

Figure 5. (a) Cross-sectional TEM micrograph of Ni/Cu substrate interface for a Ni film from a saccharin-free electrolyte. Columns are very fine close to the substrate and evolve into broader features. Twinning is extensive within the columns. (b) same as (a) but with saccharin. The grain size is similar to that seen in Fig. 4b, but at this magnification the twinning within the nano-sized grains is evident.

Annealing Effects

Figure 6 shows SEM micrographs of the effect of a 550°C heat treatment. For the sample deposited without saccharin, some grain growth occurs, primarily towards the top of the film. The small dark spots on the Cu substrate that appear after annealing are perhaps attributable to polishing damage during surface preparation; they were not present to the same extent when Ni was deposited and annealed (with and without saccharin) on a few microns of electrodeposited Cu in experiments on Si wafers. For the sample produced with saccharin, significant grain growth occurs, yielding some grains than span half the film thickness in height and a film thickness in width. It is possible that impurities or "dopants" incorporated into the film from the saccharin segregate to grain boundaries and/or generate heavy twinning and defects, providing a driving force to promote grain growth or recrystallization during annealing. Such an effect has been observed during Cu electrodeposition for on-chip interconnects.[24-25]

Also shown are the EBSD pole figures for each film. These indicate that saccharin changes the film texture from a weak (210) to an appreciable (111) preferred orientation (*i.e.*, the close-packed plane perpendicular to the growth direction). These results are consistent with the literature; Ni deposited from an additive-free bath may exhibit (110), (100), or (210) texture, while Ni plated with additives has exhibited (100) and (111) textures.[6,10] In fact, the weak (210) orientation exhibited by the Ni considered in this report has been correlated with good corrosion resistance in a previous study.[26] Since the as-deposited grain size of the Ni is very small, it was not possible to perform this analysis for the unannealed films. Nonetheless, other work with saccharin-free sulfamate electrolytes indicates that annealing does not change or intensify the as-deposited structure for the temperature ranges in the current investigation.[7]

Figures 7 and 8 show backscattered electron images and corresponding energy dispersive x-ray spectroscopy sulfur maps for Ni films produced without and with saccharin, respectively. For all as-deposited films, no sulfur concentrations were apparent. Upon annealing, the Ni films produced with saccharin exhibited locally high sulfur signals along the Cu substrate/Ni interface. It is possible that any sulfur incorporated in the Ni has a preference for the Cu substrate, leading to diffusion and penetration into the Cu during annealing.

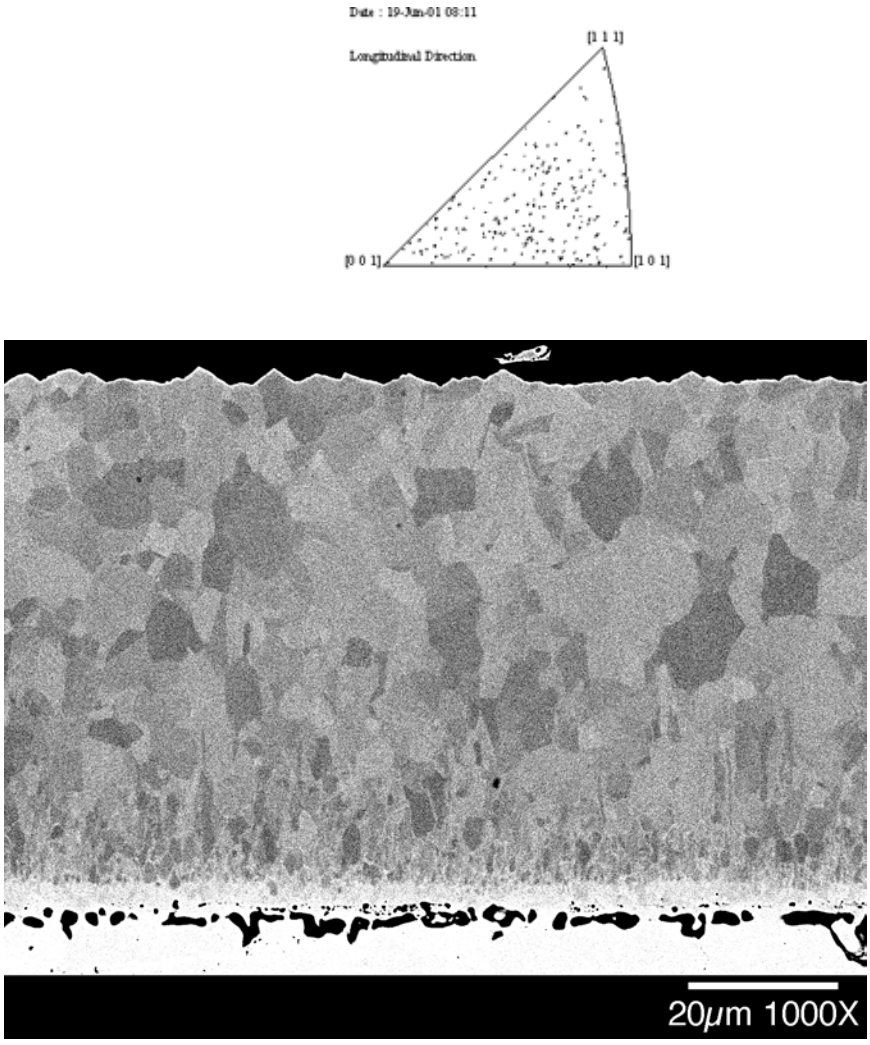

Figure 6a. SEM/BEI micrograph of Ni from a saccharin-free electrolyte annealed at 550°C for one hour. Texture is weak (210)

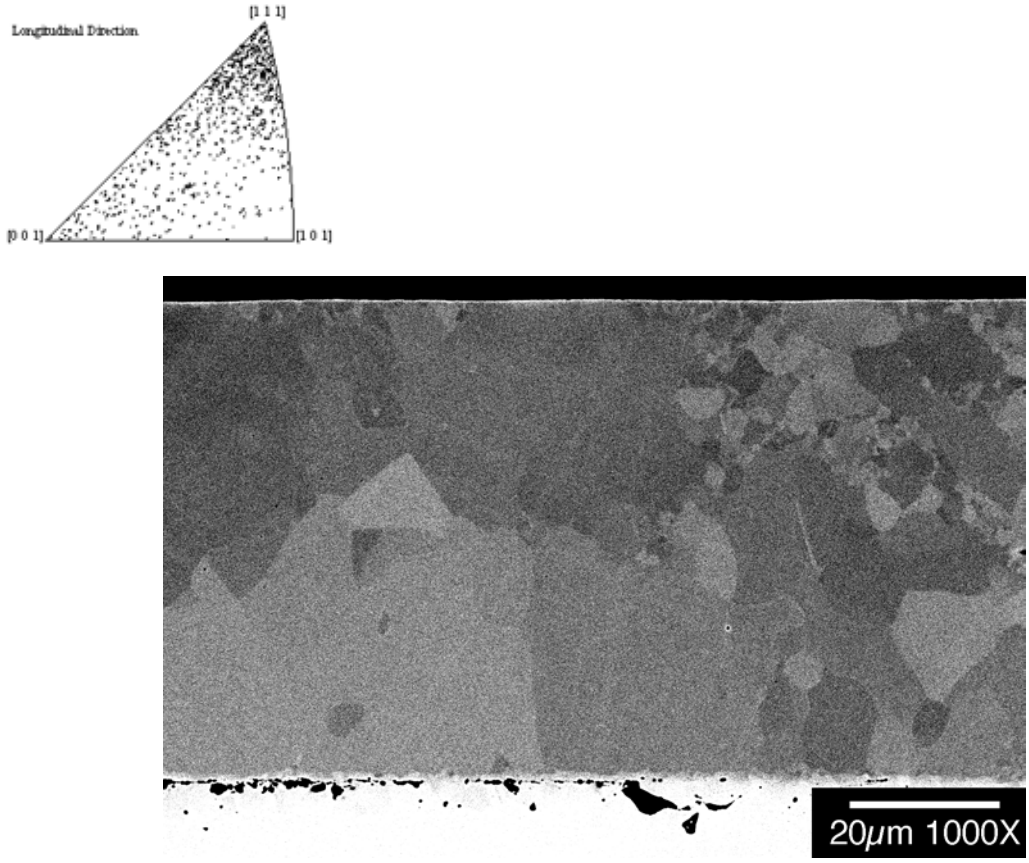

Figure 6b. Same as (a) but with saccharin. Significant grain growth has occurred as compared to (a). Texture is (111)

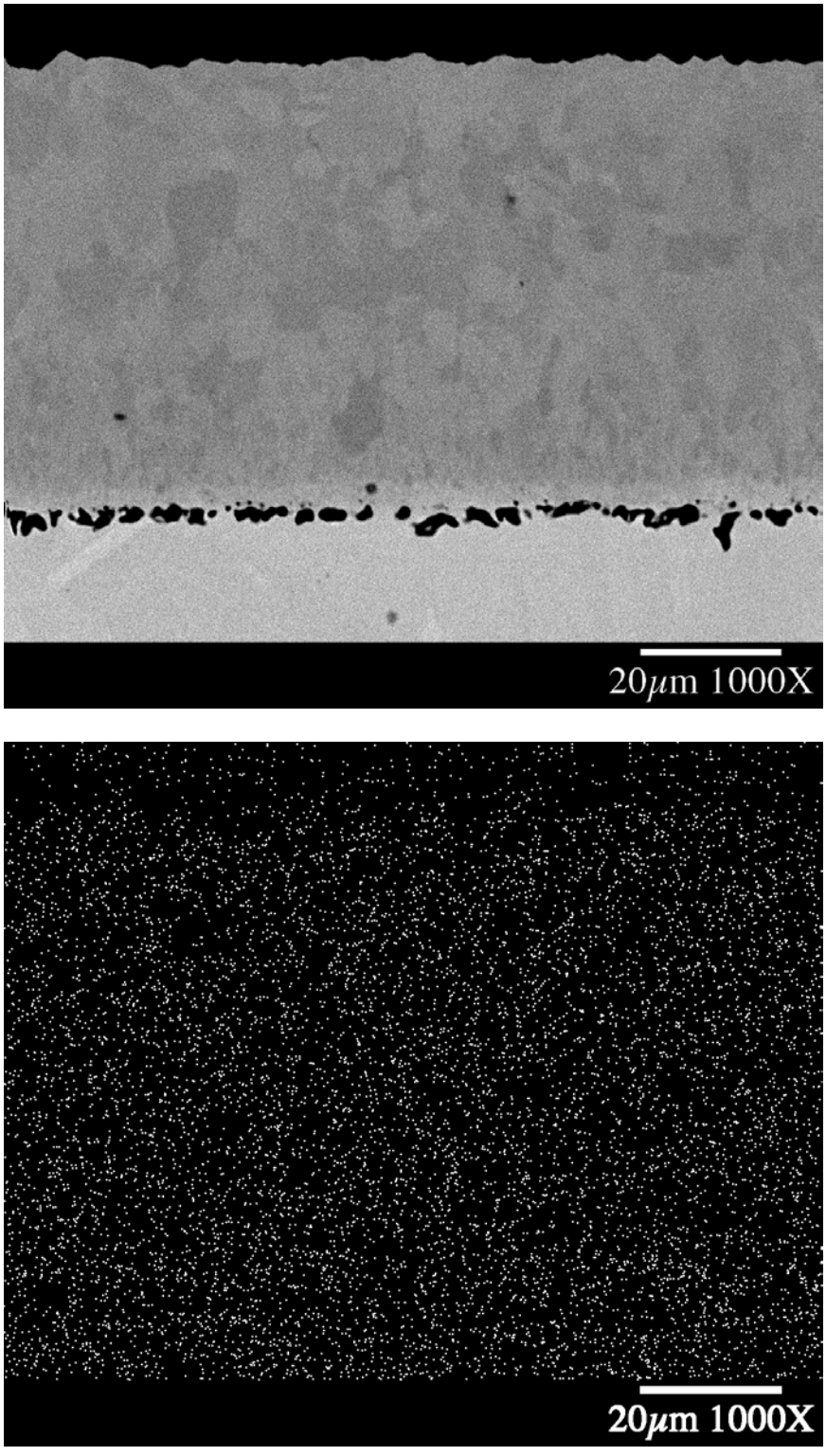

Figure 7. Same as Fig. 6(a) with energy dispersive spectroscopy sulfur map. There is no apparent S segregation.

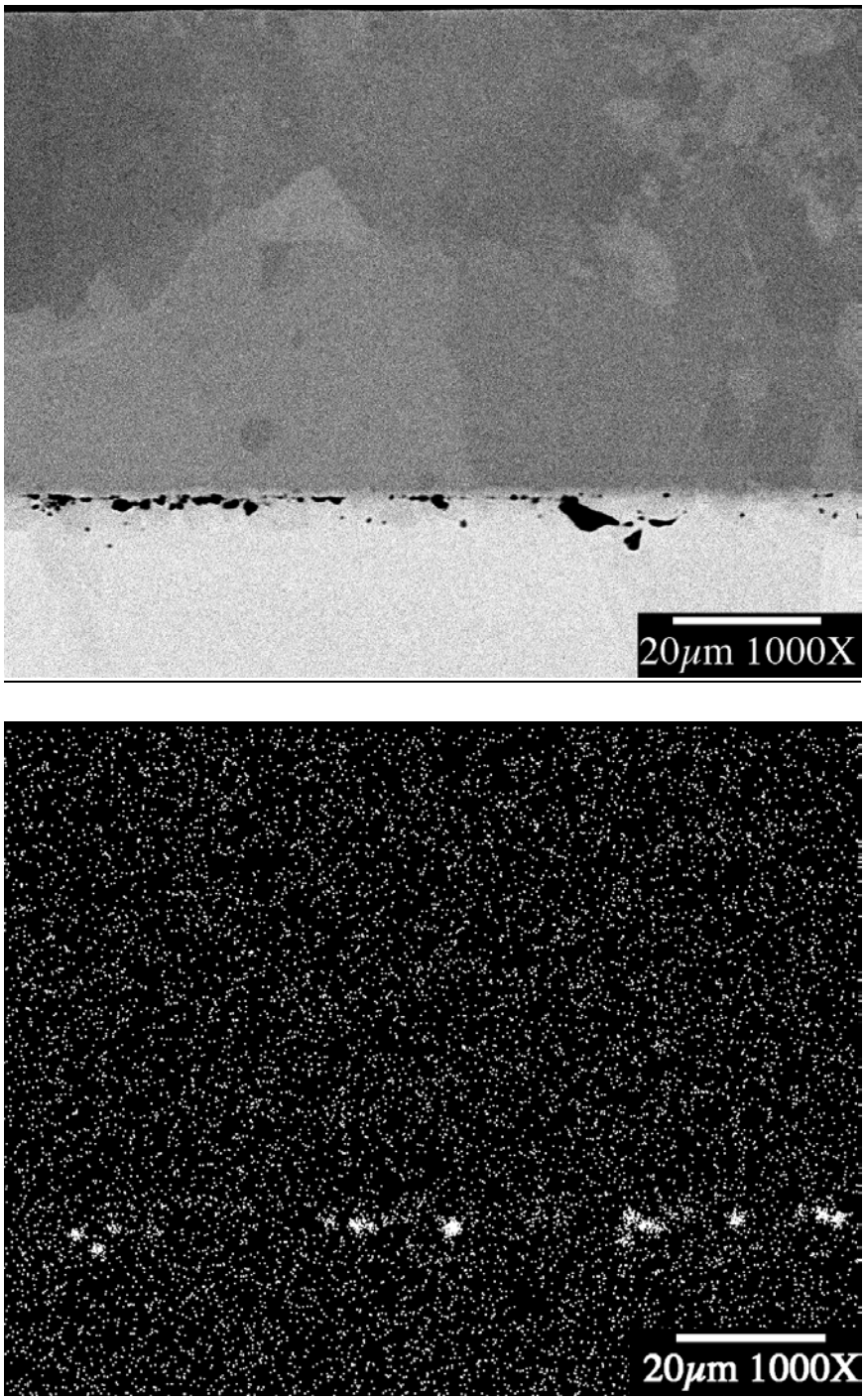

Figure 8. Same as Fig. 7 but with saccharin. There appears to be some S segregation towards the Ni/Cu interface.

Film Hardness

Figure 9 shows the Ni hardness values as a function of annealing temperature. Also shown is the nominal grain size of the film (note that the scale is logarithmic). The accelerated grain growth in the films made with saccharin leads to a faster decrease in hardness with annealing. The as-deposited hardness values are in agreement with ranges found in the literature,[4,6,10,15] and suggest ultimate tensile strengths (based upon a correlation developed in ref. 10 from literature data) of approximately 490-588 MPa (about 72-86 ksi) for the as-deposited material.[10] Table 2 lists additive-free Ni hardness values for this study and the literature. The as-deposited hardness values obtained for the films from the saccharin-free bath are on the low end of the range, suggesting that the deposits are largely impurity-free, as foreign matter (organics or other metal ions) typically augments film hardness.[4,6,10,15]

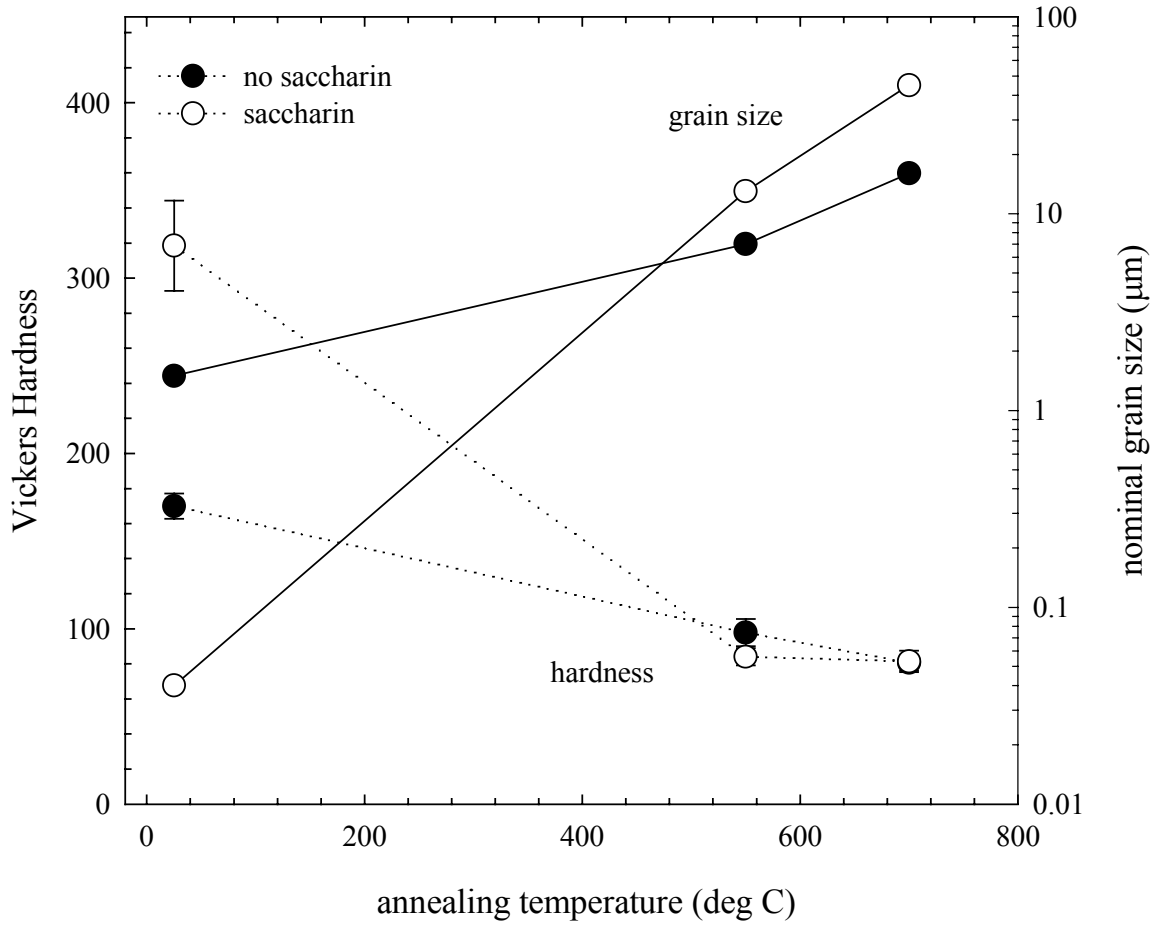

Figure 9. Hardness and nominal grain size as a function of annealing temperature. Note scale on right is logarithmic. The faster rate of grain growth for the film with saccharin results is a rapid loss of as-deposited hardness.

Reference	VHN	notes
This study	169.9 ± 7.2	25 g load
(1)	260	500 g load Knoop indenter; values converted to Vickers scale
(4)	230	100 g load
(6)	270	50 g load
(15)	175	500 g load Knoop indenter; values converted to Vickers scale

Table 2. Comparison of Vickers hardness values for this study and the literature.

The observed annealing behavior has important implications since heating electroformed metal parts to fuse them together is a possible assembly process alternative for components having more complex geometries. Besides possible embrittlement complications (depending on the heating temperature),[15] the data in Fig. 9 suggest that, despite their small as-deposited grain size, metal parts from an electrolyte containing saccharin would undergo significant grain growth and experience a concomitant loss in hardness upon heating. According to Fig. 9, the gain in hardness from the use of saccharin is lost upon exposure to temperatures > ~550°C. Decreases in the tensile strengths of Ni and Ni-Co alloys (made without saccharin) observed in the literature are consistent with reductions in hardness in this study for the annealed, saccharin-free Ni films.[10] To the authors' best knowledge, the grain growth and hardness loss of Ni deposited in the presence of saccharin has not been dealt with in the literature before.

Summary

The additive saccharin was shown to have a major effect on metallurgical characteristics, *i.e.* the film texture, grain structure, and hardness of Ni electrodeposited from a sulfamate electrolyte. Its main effects are an appreciable reduction of the as-deposited grain size and a reorientation of the film texture. Appreciable grain growth occurs with annealing, resulting in a concurrent loss of hardness. These results suggest that stress relief associated with the use of saccharin is correlated to grain refinement. In fact, pulse plating and alternative additives have been considered in previous attempts to reduce the stress in

electrodeposited Ni-Fe alloys.[1,27-28] Such studies are few in number, and the likelihood of success using such an approach is not clear.

CONCLUSIONS

Nickel was electrodeposited from a conventional sulfamate bath with and without the stress relief agent saccharin on Cu rotating disk electrodes. In cross section, films produced without saccharin have a fine, fibrous-like morphology and a weak (210) texture, while adding saccharin yields a fine-grained film with a less pronounced columnar nature but distinct (111) texture. The as-deposited Ni exhibits nominal grain sizes of about 1.5 and 0.040 μm with and without saccharin, respectively. Using saccharin increases the as-deposited Ni hardness. Annealing at temperatures of 550 to 700°C caused more grain growth (and hardness loss) in the films deposited with saccharin as compared to those produced without. The microstructure, texture, and hardness of the electrodeposited Ni with and without saccharin were all found to be consistent with literature values.

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