72

MECHANICAL AND METALLOGRAPHIC CHARACTERIZATION OF LIGA FABRICATED NICKEL AND 80%Ni-20%Fe PERMALLOY

T.R. CHRISTENSON,* T.E. BUCHHEIT,* D.T. SCHMALE,* AND R.J. BOURCIER**
*Sandia National Laboratories, Albuquerque, NM 87185-0329
**Photonic Technologies Division, Corning Inc., Corning, NY 14831

ABSTRACT

A table top servohydraulic load frame equipped with a laser displacement measurement system was constructed for the mechanical characterization of LIGA fabricated electroforms. A drop-in tensile specimen geometry which includes a pattern to identify gauge length via laser scanning has proven to provide a convenient means to monitor and characterize mechanical property variations arising during processing. In addition to tensile properties, hardness and metallurgical data were obtained for nickel deposit specimens of current density varying between 20 and 80 mA/cm^2 from a sulfamate based bath. Data from 80/20 nickel/iron deposits is also presented for comparison. As expected, substantial mechanical property differences from bulk metal properties are observed as well as a dependence of material strength on current density which is supported by grain size variation. While elastic modulus values of the nickel electrodeposit are near 160 GPa, yield stress values vary by over 60%. A strong orientation in the metal electrodeposits as well as variations in nucleating and growth morphology present a concern for anisotropic and geometry dependent mechanical properties within and between different LIGA components.

INTRODUCTION

An accurate knowledge of mechanical properties is required for the design of a large number of LIGA applications which involve components such as flexures and parts which are plastically deformed subsequent to fabrication. The LIGA fabrication process is an additive process in which structural material is typically electrodeposited into a precision mold of plexiglass (PMMA) realized through deep x-ray lithography [1,2]. Representative dimensions of components, which have a prismatic geometry, may range from a few micrometers to several millimeters or more with a thickness of several hundred micrometers and tolerances of less than 1 micrometer.

The well known fine grain structure of electrodeposits yields mechanical properties which are unique to electrodeposition [3]. Furthermore, electrodeposit mechanical properties are a function of electrodeposition technique which involves bath chemistry, bath geometry and agitation, and current density. The variation in mechanical properties that can occur for a given material is dramatic and well documented [4,5]. Thus, there exists a need for a convenient scheme to monitor the mechanical properties of LIGA fabricated materials which may be easily accommodated by the batch nature of LIGA processing. This scheme will in turn allow for tracking of electrodeposit character over time, identify drifts in electrochemistry and yield information on sensitivity of deposit properties to controlled plating variations. This information may in turn be used to predict property variations which are due to uncontrollable localized diffusion and field variation across varying component geometry.

Two disparate approaches to this problem are conceivable. One approach makes use of conventional tensile testing methods where a tensile specimen is machined from an electrodeposit and inserted into a commercially available load-displacement measurement system. Another method proposed by Ruther [6] is to integrate the entire load-displacement measurement within the LIGA process which utilizes microactuator and displacement measurement devices constructed with sacrificial LIGA processing. The approach chosen here lies between these two extremes and takes advantage of the flexible patterning available with LIGA processing as well as the ability to construct a load frame with displacement measurement suitable for miniature tensile specimens. In this sense, the technique is similar to those demonstrated by Dual and Mazza [7] as well as by Sharpe [8].

DISCLAIMER

EXPERIMENTAL

Design of Mechanical Test System

A table top servohydraulic load frame fitted with a laser displacement measuring system was built specifically for mechanical testing LIGA fabricated materials. Several features and high resolution measuring capabilities within the design of the load frame permit the ease of mounting, testing and removing small fragile specimens [9]. The load frame is currently fitted with a 100 lb (445 N) load cell which has a load resolution of approximately 10 mN. The frame also accommodates 10 lb (45 N) or 250 lb (1110 N) load cells. The laser displacement measuring system is the LS-5000 high speed laser scan micrometer produced by Keyence Corporation [10]. The system is composed of a transmitter and a receiver. The transmitter emits a 670 nm red semiconductor laser that is cycled through the displacement measuring range at a rate of 1200 scans/sec, thus permitting a maximum of 1200 measurements per second. The maximum measuring range of the receiver is 45 mm with a resolution of less than 2 μm. Resolution is improved somewhat by averaging over a number of scans, which may be scaled by the number of data points taken per second. A secondary displacement measuring system with higher measurement accuracy includes two high resolution LVDT's and was designed into the grip housings of the load frame. Signals from each LVDT are averaged to eliminate the effect of bending during testing. Displacement measurements from the load frame grips, however, contain a small amount of compliance from the fillet regions within the test specimen and possibly from the grips themselves. Fig. 1 illustrates the tabletop mechanical test system with the laser displacement measuring system. A LIGA fabricated specimen for mechanical testing is illustrated in Fig. 2. The specimen is fabricated with small measuring tabs placed 5.08 mm apart which permit measuring displacement using the laser measuring system. Finite element analyses, illustrated in Fig. 2(c) show that at 10% strain, the tabs impart a negligible influence on the uniform strain field within the gage length of the specimen and do not rotate within experimentally detectable limits.

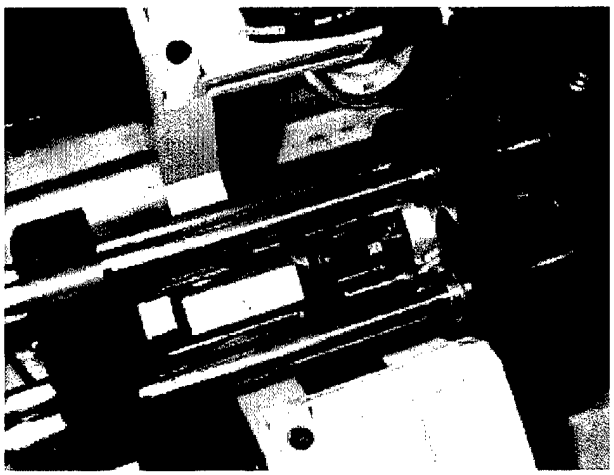
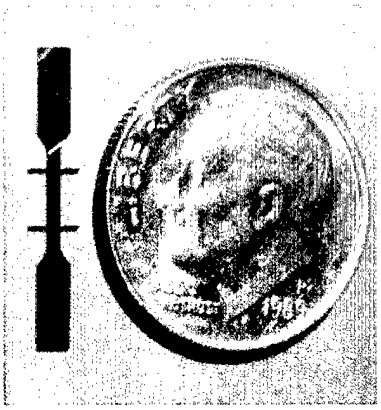
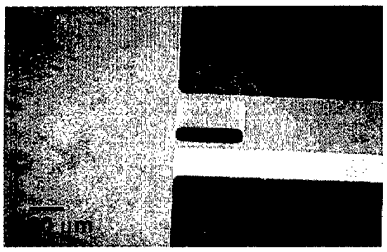


Fig. 1 Working section of tabletop servohydaulic load frame with laser displacement measuring system.



(a)



(b)

(c)

Fig. 2 (a) LIGA tension test specimen compared with a US Dime. (b) SEM micrograph illustrating details of measuring tab. (c) FE analysis results of tab region on LIGA tension test specimen.

Electrodeposition and Sample Preparation

All nickel electroplating was carried out in a nickel sulfamate bath with the following properties:

$Ni(NH_2SO_3)_2 \bullet 4H_2O$	440.1 g/l
Ni (as metal)	80 g/l
Boric Acid	48 g/l
Wetting Agent	0.2 %/vol
Temperature	50 degrees C
pH	3.8 - 4.0
Anode	sulfur depolarized nickel in Ti basket with PTFE bag

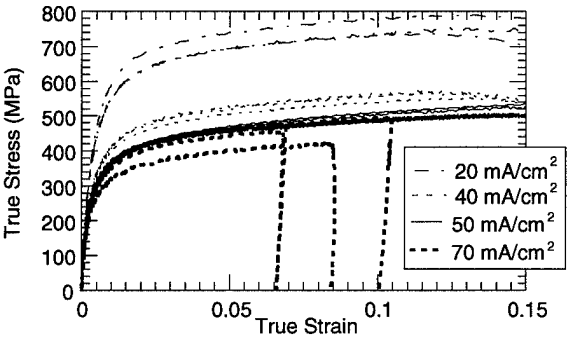
This bath formulation was chosen for its low deposit stress character which is of particular importance in precision LIGA electroforming. The bath was also continuously filtered through a 1.0 μm PTFE filter and was continuously stirred to maintain constant bath temperature. After preparing the bath a low current density treatment was performed onto a corrugated cathode at 1-5 mA/cm^2 for a 72 hour period. The pH was maintained with sulfamic acid. The nickel/iron bath was sulfate/citrate based as described by Venkatasetty [11] used for depositing 80 Permalloy.

Samples were prepared using deep x-ray lithography and electroplating with an unpatterned sacrificial layer. Silicon substrates were prepared with a sputtered Ti/Cu plating base at 30/100 nm thickness. Deep x-ray lithography follows with PMMA as the x-ray photoresist and electroform mold material. The copper plating base layer is used to seed the nickel and nickel/iron deposits which are electrodeposited proud of the LIGA mold and diamond lapped back flat and parallel to the substrate surface. The specimens are released from the substrate by first dissolving the PMMA and subsequently selectively etching the underlying copper with an ammonium hydroxide based etchant [12].

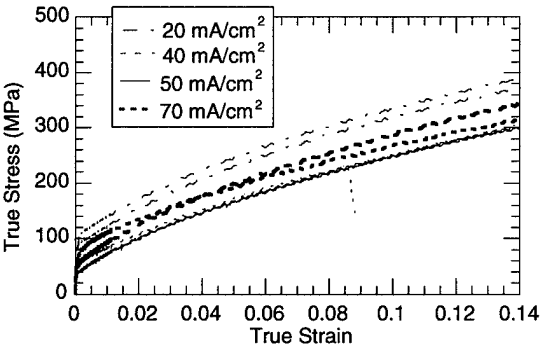
Mechanical and Microscopic Analyses

Tension tests, microhardness tests and optical metallographic analyses were performed on LIGA fabricated Ni samples at 20, 40, 50, and 70 mA/cm^2. Metallography and microhardness tests where also performed on LIGA fabricated Ni samples deposited at 80 mA/cm^2. High quality tension test samples could not be fabricated at this current density due to a large compressive strain in the deposit. Elemental and grain orientation distribution (microtexture) analyses were performed on LIGA fabricated Ni samples at 50 mA/cm^2. The elemental analysis was performed by measuring characteristic X-rays from the initial deposition surface of the sample excited with a 15 keV electron beam. The microtexture analysis was performed by measuring scanning electron microscope (SEM) electron backscatter patterns generated from the lapped side of the sample.

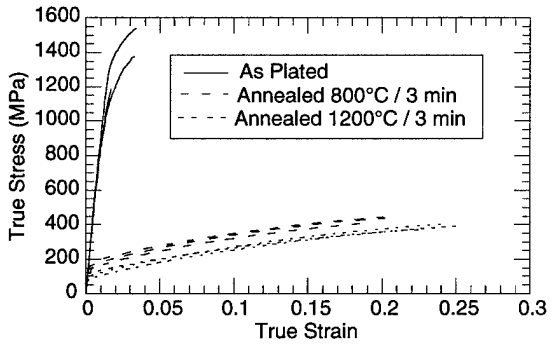
A second set of tension tests were performed on LIGA fabricated Ni samples annealed at 600°C for 1 hour. Again, samples fabricated at 20, 40, 50, and 70 mA/cm^2 were annealed and tested. A final set of tension tests were performed on a LIGA fabricated 80wt%Ni-20wt%Fe alloy, referred to as Permalloy, in the as-plated condition and after 3 minute anneals at 800°C and 1200°C.



(a)



(b)



(c)

Fig. 3 (a) Tension test results of LIGA fabricated Ni plated from a Nickel Sulfamate bath at 4 different current densities. (b) Tension test results of LIGA fabricated Ni after a 1 hr. 600°C anneal. (c) Tension test results of LIGA fabricated Permalloy.

RESULTS

Elemental Analysis on LIGA fabricate Ni at 50mA/cm^2

The electron microprobe study revealed a 100% dense nickel deposit with trace concentrations of sulfur (66 ± 8.4 ppm) and copper. The copper is believed to be residual from the plating base or the copper based plate used for diamond lapping and the sulfur is a common impurity found with sulfur based nickel electrodeposition.

Mechanical Test Results

Tension test results for electrodeposited nickel at the various current densities are shown in Fig. 3(a) and summarized in Table I. All test specimens fractured within the gage length between the measuring tabs except for the 70 mA/cm^2 samples, which fractured near one of the fillets in each tested sample. Two apparent results from the LIGA fabricated Ni mechanical test data are a dependence of yield and ultimate strength with current density and the reduced strain to fracture of the 70 mA/cm^2 samples. Microhardness data from LIGA Ni samples deposited at several different current densities is given in Table II. The data shows an overall increase in hardness with a decrease in current density and consistently higher hardness values on the nulceating side relative to the lapped, or top, side of specimens tested from each current density. Data from the 50 mA/cm^2 samples in Table II also show that hardness tests on an unlapped specimen gave results consistent with the lapped specimens.

Tension test results obtained from annealed LIGA Ni and Permalloy specimens are illustrated in Fig. 3(b) and Fig. 3(c). A considerable decrease in strength is noted after a 1 hour 600 °C inert annealing treatment on the LIGA Ni specimens. Even after annealing, however, a dependence on current density still remains. The as-plated Permalloy specimens have a higher yield strength than any of the LIGA Ni specimens. Since the Permalloy samples were not measured using the laser extensometer but rather using an uncalibrated LVDT extensometer, elastic modulus values extracted from tests illustrated in Fig. 3(c) may not be accurate. Similar to LIGA Ni samples, high temperature annealing reduces yield strength considerably.

Table I Summary of Mechanical Properties - LIGA fabricated Ni

Current Density (mA/cm^2)	No. of Tests	Elastic Modulus (GPa)	0.2% Proof Stress (MPa)	Max. Stress (MPa)	Strain @ Max. Stress
20	3	156 ± 9.3	441 ± 27	758 ± 28	0.126 ± 0.008
40	3	155 ±11	305 ± 12	562 ± 9.2	0.124 ± 0.005
50	3	160 ± 20	277 ± 7.6	521 ± 19	0.151 ± 0.012
70	4	131 ± 13	275 ± 18	460 ± 31	0.080 ± 0.017

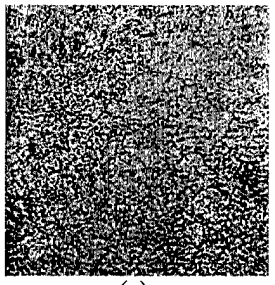
Metallography and Texture

Table II Microhardness results of LIGA fabricated Ni

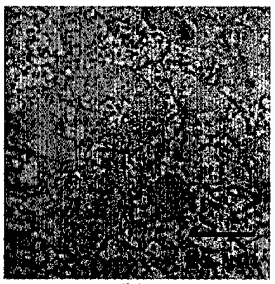
Current Density (mA/cm^2)	No. of Tests	Vickers Hardness 100 g / 15 sec		
		Deposited Surface	Side	Lapped Surface
20	10	257 ± 3.5	230 ± 11	242 ± 9.1
40	5 -10	195 ± 9.5	177 ± 1.5	163 ± 3.8
50	5 -10	191 ± 4.5	175 ± 2.4	187 ± 5.5
70	10	188 ± 5.5	165 ± 5.7	162 ± 5.5
80	7-9	172 ± 6.8	167 ± 5.1	*164*
50 unlapped	10	179 ± 6.1	191 ± 11	167 ± 7.4

Fig. 4 illustrates micrographs of the deposited and lapped side from a 50 mA/cm^2 specimen. They reveal a very fine grain structure on the deposited side compared with a significantly coarser grain structure on the lapped side of the sample. Fig. 5 illustrates the edge view micrographs of LIGA fabricated Ni samples at several different current densities. As suggested by the micrographs in Fig. 4, a very fine grain structure nucleates on the sputter deposited surface at the beginning of the electrodeposition. This fine grain structure gradually coarsens, over a range of 10-25 μm into a larger columnar structure, which persists during the rest of the deposition. The micrographs in Fig. 5 also suggest an increase in grain size with increasing current density.

The microtexture analysis revealed a strong ⟨100⟩ texture relative to the growth direction in the 50 mA/cm^2 sample. Fig. 6 illustrates an inverse pole figure representation of the microtexture results.

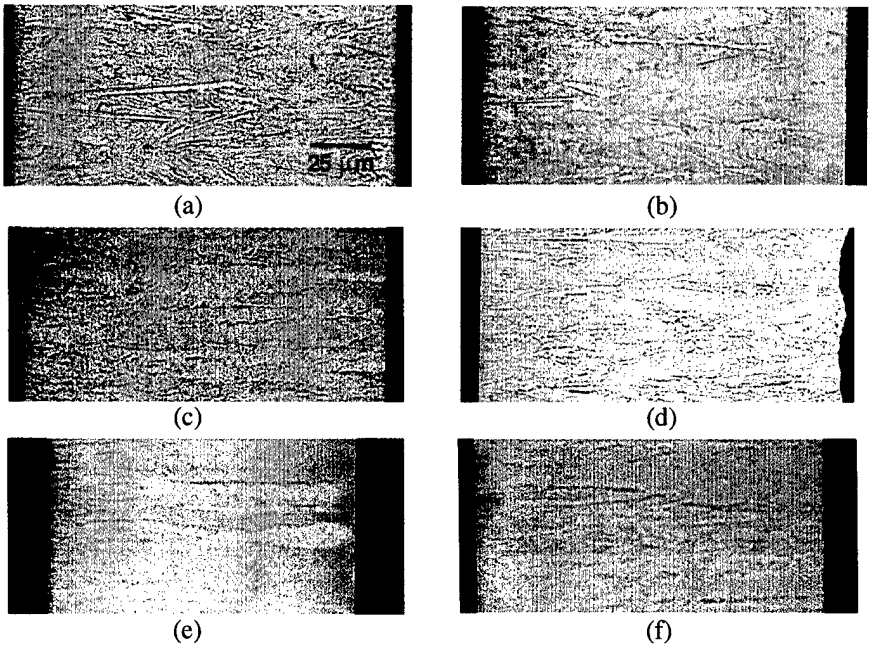


(a)



(b)

Fig. 4 Optical micrographs of (a) deposited and (b) lapped surfaces of LIGA fabricated Ni at 50 mA/cm^2.



(a) (b) (c) (d) (e) (f)

Fig. 5 Edge view optical micrographs of LIGA fabricated Ni at (a) 20 mA/cm^2, (b) 40 mA/cm^2, (c) 50 mA/cm^2, (d) 50 mA/cm^2 unlapped, (e) 70 mA/cm^2, and (f) 80 mA/cm^2. Growth direction is from left to right on all micrographs.

This representation plots the axis normal to the deposition substrate relative to the crystal lattice coordinate system. Fig. 6 shows the results for 12 grains and for each grain, the direction normal to the deposition substrate is always oriented near a crystal lattice ⟨100⟩ direction.

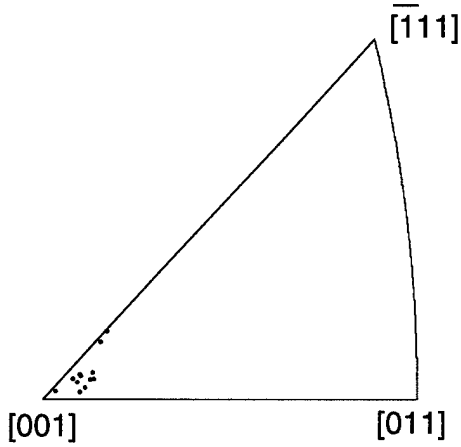


Fig. 6 Inverse pole figure representation of microtexture results from a LIGA fabricated Ni at 50 mA/cm^2 sample.

DISCUSSION

The table top mechanical test system presented in this paper has been found to offer a convenient means to characterize and monitor LIGA electrodeposit properties. The pull test specimens with a size of roughly 2x17mm may be easily accommodated on a particular device mask and released jointly with other components, and convienently possess *in situ* gauge length measurement identifiers.

Correlating metallurgy results with mechanical properties suggests the well-known observation of an increase in yield strength and hardness with decrease in grain size. A decrease in grain size, in turn, is obtained in lower current density deposits. The apparently smaller grains and increased microhardness found at the plating base surface is likely due to nucleation of a very fine microstructure on the small grains of the sputter deposited copper plating base. These observations suggest a strength variation in the direction of growth within the first 50 µm of deposit thickness, as previously noted by Johnson[13]. Results in Table II indicate this variation may cause an increase in material strength of up to 15% near the deposited surface. The ⟨100⟩ texture relative to the growth direction observed in the 50 mA/cm^2 LIGA Ni fabricated material is consistent with previously observed data by Evans[14], who identified that ⟨100⟩ oriented deposits possess lower internal stress, a property of sulfamate based nickel baths. Further, the rapid growth direction in Ni and all other FCC metals is ⟨111⟩. Averaging or adding the four ⟨111⟩ directions together gives a ⟨100⟩ orientation. Although not confirmed in this work, results indicate that small grains nucleate

with random texture on the deposited face and the grains with other "slower growth" orientations are rapidly consumed by the fast growing ⟨100⟩ grains during growth. If nucleation and growth does indeed occur in this fashion, then the variation in mechanical properties through the thickness of the deposit would be explained by the changing morphology and texture of the grains, from small randomly oriented grains on the deposited surface to larger columnar ⟨100⟩ oriented grains on the lapped surface. The marked softening and weakening of nickel sulfamate electrodeposits as compared with bulk wrought nickel upon annealing is also well established [15] and is attributed to the presence of sulfur which is free to migrate without the presence of metallic desulfurizers such as manganese, magnesium or calcium that are present in bulk commercial nickel.

Two major concerns have been raised from this study. The presence of a highly textured columnar structure of the nickel electodeposits results in anisotropic mechanical properties. A comparison of elastic moduli observed for LIGA fabricated Ni in Table I, about 160 GPa with the accepted value for bulk randomly textured Ni, 200 GPa, indicates the degree to which mechanical anisotropy may be present in the LIGA fabricated samples. Mechanical properties parallel to the deposit growth direction have yet to be measured, however, and are important for many components including those which comprise microactuators. Secondly, the variation in mechanical properties across a given sample is also not well accounted for by this technique even presupposing accurate control of bath agitation, temperature, chemistry, and current density across the sample area. This is already evident in the variation of mechanical properties within a sample due to differences in nucleation and growth microstructure. Investigations to prove that texture is the major cause of the lowered elastic modulus in the tensile test direction and development of techniques to ascertain the mechanical properties of all types of LIGA fabricated components are therefore needed.

ACKNOWLEDGEMENTS

The authors gratefully acknowledge Charlie Carter, Joe Micheal and Paul Hlava from the Materials Characterization dept. at SNL for performing the metallographic and microscopic analyses. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under contract DE-AC04-94AL8500.

REFERENCES

1. E.W. Becker, W. Ehrfeld, P. Hagmann, A. Maner, and D. Munchmeyer, Microelectronic Eng. **4**, pp. 35-56 (1986).
2. T.R. Christenson, H. Guckel in Micromachining and Microfabrication Process Technology, (SPIE Proceedings, **2639**, Austin, TX, 1995) pp. 134-145.
3. B.E. Jacobson and J.W. Sliwa, Plating and Surface Finishing, pp. 42-47, (Sept. 1979).
4. J.W. Dini, Electrodeposition - The Materials Science of Coatings and Substrates, (Noyes Publications, Park Ridge, NJ, 1993).
5. W.H. Safranek, The Properties of Electrodeposited Metals and Alloys, (American Electroplaters and Surface Finishers Society, Orlando, FL, 1986).
6. P. Ruther, W. Bacher, K. Feit, and W. Menz, in Proceedings IEEE Tenth Annual International Workshop on Micro Electro Mechanical Systems, (Nagoya, Japan, 1997) pp. 541-545.
7. J. Dual, E. Mazza, G. Schiltges, and D. Schlums, in Microlithography and Metrology in Micromachining III, (SPIE Proceedings, **3225**, Austin, TX, 1995), pp. 12-22.
8. W.N. Sharpe, Jr., D.A. LaVan, and R.L. Edwards, in Technical Digest of 1997 IEEE International Conference on Solid-State Sensors and Actuators - Transducers '97 (Chicago, IL 1997) pp. 607-610.
9. D.T. Schmale, R.J. Bourcier, and T.E. Buchheit, Description of a Micro-Mechanical Testing System, SAND97-1608, (Sandia National Laboratories, Albuquerque, NM 1997).
10. Keyence Corporation, http://www.keyence.com.
11. H. V. Venkatasetty, J. Electrochem. Soc., **117**, 403-407 (1970).
12. P. Walker and W. H. Tarn, CRC Handbook of Metal Etchants (CRC Press, Boca Raton, FL, 1991).
13. H.R. Johnson, J.W. Dini, and R.E. Stoltz, Plating and Surf. Finishing, **66**, (3), 57-62 (1979).
14. D.J. Evans, Trans. Of the Faraday Soc., **54**, 1086 (1958).
15. J.W. Dini, H.R. Johnson, and L.A. West, Plating and Surf. Finishing, **65**, 36-40 (Feb. 1978).

Report Number (14) SAND--98-0906C
CONF-980405--

Publ. Date (11) 19980413
Sponsor Code (18) DOE/DP, XF
UC Category (19) UC-700, DOE/ER

DOE