Application of Diamond-Like Nanocomposite Tribological Coatings on LIGA Microsystem Parts

Somuri V. Prasad, Thomas W. Scharf, Paul G. Kotula, Joseph R. Michael, and Todd R. Christenson

***Abstract*—The major focus of this study was to examine the feasibility of applying diamond-like nanocomposite (DLN) coatings on the sidewalls of Ni alloy parts fabricated using lithographie, galvanoformung and abformung (LIGA: a German acronym that means lithography, electroforming, and molding) for friction and wear control. Planar test coupons were employed to understand the friction mechanisms in regimes relevant to LIGA microsytems. Friction tests were conducted on planar test coupons as well as between LIGA-fabricated test structures in planar–sidewall and sidewall–sidewall configurations. Measurements were made in dry nitrogen and air with 50% relative humidity by enclosing the friction tester in an environmental chamber. In contrast to bare metal–metal contacts, minimal wear was exhibited for the DLN-coated LIGA NiMn alloy parts and test coupons. The low friction behavior of DLN was attributed to its ability to transfer to the rubbing counterface providing low interfacial shear at the sliding contact. The coating coverage and chemistry on the sidewalls and the substrate–coating interface integrity were examined by transmission electron microscopy, Automated eXpert Spectral Image Analysis, and electron backscatter diffraction on cross sections prepared by focused ion beam microscopy. The role of novel characterization techniques to evaluate the surface coatings for LIGA microsystems technology is highlighted. [2008-0007]**

***Index Terms*—Coatings, electron microscopy, friction.**

I. Introduction

DEEP X-RAY lithography-based techniques such as lithographie, galvanoformung and abformung (LIGA: a German acronym that means lithography, electroforming, and molding) are currently being used to fabricate net shape components for high-aspect-ratio microsystems (HARMS). Unlike other microfabrication techniques, LIGA lends itself to a broad range of materials, including metals, alloys, polymers, as well as ceramics and composites. Currently, Ni and Ni alloys (e.g., NiMn) are the materials of choice for LIGA microsystems. In LIGA, individual mechanical elements are created by deposition of material onto microfabricated molds, followed by assembly of the micromachine elements into a microelectromechanical systems (MEMS) device [1], [2]. The various steps involved in a typical LIGA process are described elsewhere [1], [3]. While Ni alloys may meet many of the structural and mechanical (yield and tensile strength) requirements, their tribological (friction and wear) behavior can be a major reliability issue. Problems arise when metal on metal contacts result in high friction (0.6–1.4), stick–slip behavior, and wear debris particle generation. Surface treatments and coatings that mitigate friction and wear (tribological) issues are therefore essential to ensure reliable operation of the device [3]–[5].

Several studies have been conducted on the beneficial effects of surface coatings applied to LIGA Ni planar and sidewall HARMS [4]–[8]. Hybrid chemical vapor deposition (CVD)/physical vapor deposition (PVD) diamond-like carbon (DLC) coatings doped with tungsten [6] and titanium [7] have been shown to reduce friction and wear in moving LIGA parts. In addition, a hard boride compound $AlMgB_{14}$ [8] exhibited low friction due to boric acid formation during sliding and, thus, may have potential use in LIGA HARMS. In addition to finding a robust tribological coating that can function in regimes relevant to microsystems operation, application of the coating at size and length scales (several micrometers to millimeters) relevant to LIGA microsystems is a major challenge. In addition, handling of individual LIGA MEMS elements is extremely difficult, and such techniques as resin bonding and burnishing, which are commonly used for coating parts in conventional devices, can be ruled out. PVD techniques (e.g., sputtering, ion beam deposition, and evaporation) are typically line-of-sight processes, whereas the coating is most needed on the sidewalls. Unlike PVD, CVD is a non-line-of-sight technique that can deposit conformal coatings. However, CVD is a high-temperature process, and subjecting parts made from Ni alloys to CVD processing temperatures will result in grain growth and Hall–Petch softening. Thus, we have opted for plasma-enhanced CVD (PECVD) technique, which has the potential for conformally coating entire LIGA parts without having to raise the substrate temperatures. PECVD is a process in which the constituents of the vapor phase react to form a solid film. In the PECVD technique, the gas molecules are mainly dissociated by electron impact generating neutral, radical, and ion species [9]. These species arrive on a surface and react with each other in the growth process. Since the gas molecules are activated by the energetic electrons instead of thermal energy, the reaction temperature can easily be reduced. Thus, coatings can be deposited at temperatures typically less than 200 °C since the

Manuscript received January 10, 2008; revised November 11, 2008 and February 5, 2009. First published April 3, 2009; current version published June 3, 2009. This work was supported by Sandia, a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under Contract DE-AC04-94AL8500. Subject Editor D.-I. Cho.

S. V. Prasad, P. G. Kotula, and J. R. Michael are with Sandia National Laboratories, Albuquerque, NM 87185-0889 USA (e-mail: svprasa@sandia.gov).

T. W. Scharf was with Sandia National Laboratories, Albuquerque, NM 87185-0889 USA. He is now with the Department of Materials Science and Engineering, University of North Texas, Denton, TX 76203-5310 USA.

T. R. Christenson was with Sandia National Laboratories, Albuquerque, NM 87185-0889 USA. He is now with HT MicroAnalytical Inc., Albuquerque, NM 87109 USA.

Color versions of one or more of the figures in this paper are available online at http://ieeexplore.ieee.org.

Digital Object Identifier 10.1109/JMEMS.2009.2016284

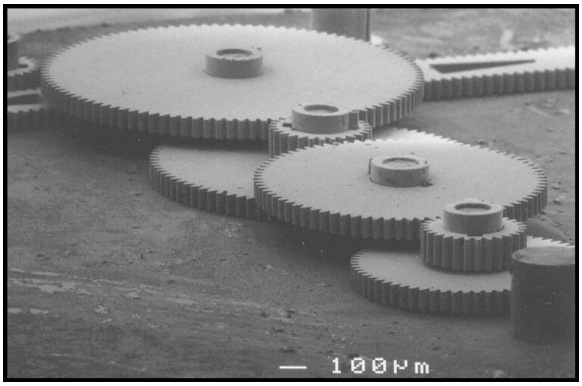

Fig. 1. Miniature gear train and rack mechanisms assembled from LIGA-fabricated parts showing several sidewall–sidewall contacts.

activation energy for the chemical reaction is provided by the glow discharge (electrons) on the biased substrate. The ability of PECVD to deposit coatings at much lower temperatures than conventional CVD prevents any potential microstructural changes and mechanical property degradation of the base NiMn alloy part.

In this paper, we have selected a diamond-like nanocomposite (DLN) coating that can be applied by PECVD from siloxane vapors that contain Si and O in addition to C and H species [10], [11]. A commercial PECVD process was modified to get the right chemistry and thickness on the sidewalls of LIGA-fabricated parts, which is complicated due to unusual sidewall morphologies produced by LIGA processing [12]. In LIGA-fabricated devices, forces often tend to be transmitted between structural elements in the plane of the device, leading to the result that sliding interactions are predominantly between sidewalls. The gear train and rack mechanism assembled from parts fabricated by LIGA highlights this point (see Fig. 1). Therefore, the major focus of this paper was to examine the feasibility of applying DLN coatings on the sidewall LIGA-fabricated microsystems parts. Novel test structures were developed to enable friction measurements in sidewall–sidewall configurations. This paper also highlights the need to apply advanced characterization techniques such as focused ion beam (FIB) microscopy, electron backscatter diffraction (EBSD), and transmission electron microscopy (TEM) to analyze the coatings on the sidewalls of miniature parts.

II. Experimental

A. Fabrication of Test Structures

The electroformed Ni and NiMn alloys were prepared by mimicking the LIGA process, as shown schematically in Fig. 2. The metals (pure Ni and NiMn alloy) were electroplated onto an array of micromolds defined by a photoresist layer on a metallized silicon substrate. After deposition, a surface lapping procedure was used to planarize the surface. The photoresist was then dissolved, and test coupons were diced. The details of this process are described elsewhere [13]. Pure Ni was electroplated from a sulfamate bath as 10 mm $\times$ 10 mm square coupons. As reported earlier [14], the resulting microstructure of electroplated Ni exhibited a columnar grain structure with highly twinned grains of approximately 1–2 μm in width. The NiMn coupons were electroplated into 3-mm-diameter circular micromolds by utilizing a similar sulfamate bath with a small amount of manganese (0.5 g/L of Mn added as $MnCl_2$). Additionally, pulsed plating methods were used to minimize the buildup of internal stress during plating [15]. This plating procedure resulted in a much finer columnar grain structure with grains approximately 0.2–0.5 μm in width [15] for the NiMn alloy. Cylindrically tipped friction probes with an $\sim$500 μm radius of curvature were fabricated by standard LIGA processing that included the final release step from the substrate. A typical scanning electron microscope (SEM) image of a pure Ni friction probe that was used as one of the counterfaces in friction studies is shown in Fig. 3(a). In addition to the planar test coupons described above, the sidewall of a LIGA-fabricated NiMn spring in Fig. 3(b) was also used as a test specimen to generate friction data between sidewall–sidewall contacts using the friction probe tip shown in Fig. 3(a). The LIGA Ni test structure shown in Fig. 3(c) was used to evaluate the sidewall coverage and thickness of the DLN coating.

B. Application of DLN Coating

A novel coating strategy was developed to circumvent the problems associated with handling miniature parts and to ensure coverage on their sidewalls. Instead of coating the individual parts, the entire wafer was coated before releasing the parts, i.e., after electroplating, planarizing, and dissolving the mold material. The PECVD DLN coatings were supplied commercially (Bekeart Advanced Coatings Technology, Buffalo, NY). The plasma was formed from a siloxane precursor using a hot filament, and ionized species are deposited onto negatively biased substrates. The process results in a diamond-like network of a-C:H and a second network of Si:O with minimal bonding between the two networks. The targeted coating thickness on LIGA test structures was 100 nm, and a 30-nm-thick Ti interlayer was necessary for improved adhesion of DLN to Ni and NiMn [4]. A 500-nm-thick coating was applied on planar coupons and Si wafers for baseline tribological measurements. More complete descriptions of the coating processes are given elsewhere [10], [11], [18]. The coated wafer was ion etched to remove the coating from the bare substrate and the planar surfaces of the LIGA parts to make the substrate/LIGA part interface accessible to the release agent. This was performed by a commercial ion etcher processing tool (Oxford Instruments Ionfab). Since sputtering is a line-of-sight process, this step only removed the coating from the planar surfaces of the parts where it is not critical, leaving the sidewalls untouched. The coated parts were subsequently released from the wafer using the standard LIGA release step. A schematic illustration of the modified LIGA processing route incorporating the coating step between the mold removal and parts release steps is shown in Fig. 4. The LIGA part shown in Fig. 3(c) was used to characterize the DLN coating thickness and coating chemistry on the sidewalls.

C. Friction Testing

A photograph of the linear friction and wear tester is shown in Fig. 5. Normal load (L) was applied by means of

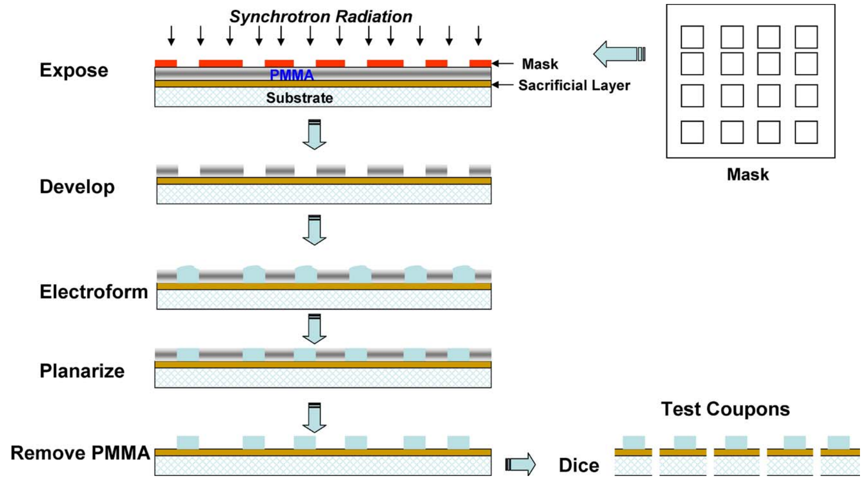

Fig. 2. LIGA processing steps for making planar tribology test coupons.

Fig. 3. LIGA-fabricated test structures. (a) Ni friction probe with a 500-μm radius of curvature cylindrical tip. (b) NiMn alloy spring (friction measurement was made on its sidewall). (c) Test structure for characterizing the DLN coating on its sidewall (inset box shows the location at which the FIB section was taken).

deadweights. A force transducer (Sensotec) in the load arm measured the tangential load (W) over a track distance of 1.6 mm; the load cell has sensitivity to measure the tangential load down to $\sim$500 μN. The sliding speed was 3.7 mm/s. The ratio of tangential to normal load is the coefficient of friction (COF). The tester (tribometer) was housed in an environmental chamber with precise control of dew point and oxygen content. Measurements were made in either dry nitrogen (< 10 ppm O_2 and < 100 ppm H_2O) or ambient air with 50% relative humidity (RH) environments. The first series of measurements was made using a 3.175-mm-diameter Si_3N_4 ball as a counterface material on planar test coupons at normal loads ranging from 98 to 980 mN, corresponding to initial maximum Hertzian contact stresses from 316 to 682 MPa. Test coupons included DLN-coated Si wafer for control measurements and bare and DLN-coated Ni and NiMn alloys. In the second set of experiments, a LIGA-fabricated Ni probe tip shown in Fig. 3(a) was used as the counterface in place of the standard Si_3N_4 ball. The friction probe with a tip radius of 500 and 200 μm thickness generated an initial maximum Hertzian contact stress of 130 MPa at a normal load of 49 mN. Friction and wear tests were run for at least 1000 cycles of unidirectional sliding, and at least three measurements were made for each condition.

D. FIB Sections for EBSD and TEM

Cross sections within the wear scars were prepared using an FEI Company DB 235 system containing both a high-resolution SEM and FIB. This dual-beam configuration allows for sample imaging using the nondestructive electron beam without the damage associated with ion imaging. It has been previously shown how a FIB could be used to prepare cross

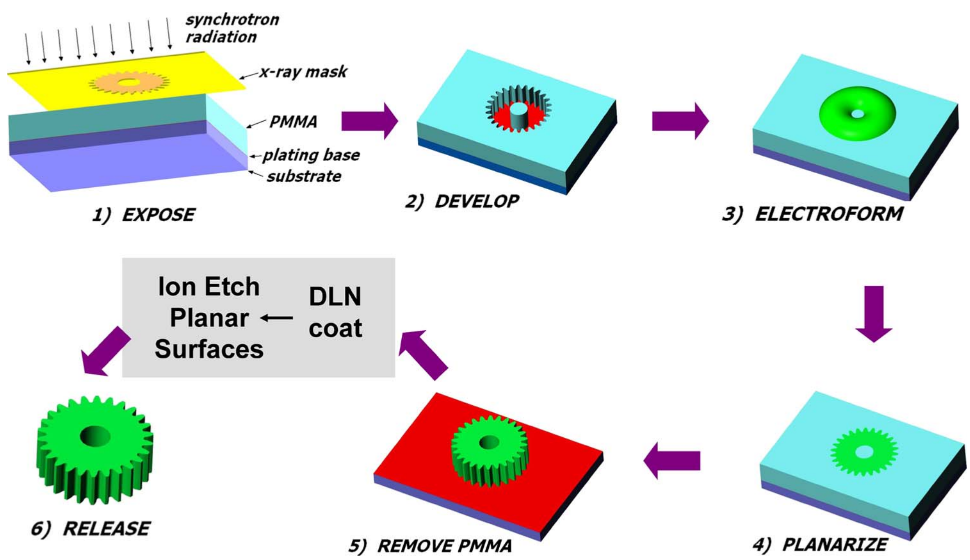

Fig. 4. Modified LIGA processing route incorporating the coating step between mold (PMMA) removal and parts release.

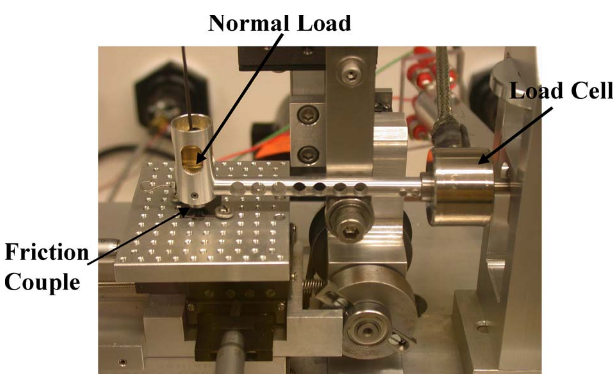

Fig. 5. Friction and wear tester.

sections for microstructural analysis at specific locations on a wear scar [13]. Since the FIB combines accurate positioning for sample milling and does not alter the microstructure (which is the way that more aggressive methods like core drilling or diamond sawing do), sample preparation-induced changes to the wear-deformed microstructure are minimized. Platinum was selectively deposited over the wear surface prior to FIB micromachining to minimize damage to the wear surface from the ion beam. The cross sections were milled using 30 kV gallium ions. For subsequent EBSD analysis, the cross section samples were removed using a micromanipulator and were placed on a carbon-coated transmission electron microscope support grid. EBSD was carried out using an HKL Channel 5 EBSD system and a Zeiss Supra 55 VP SEM operated at 20 kV. FIB sections suitable for TEM were also milled on the sidewalls of the DLN-coated LIGA structure shown in Fig. 3(c); the inset box shows the location at which the FIB section was taken.

TEM and scanning TEM (STEM) were performed on an FEI Company Tecnai F30-ST TEM/STEM operated at 300 kV and equipped with a field-emission electron source and an energy-dispersive X-ray spectrometer. Images were acquired both in TEM mode as well as in STEM mode. In STEM mode, X-ray spectral images, consisting of a 2-D array of complete X-ray spectra, were acquired from the surface region of the cross section specimen. The spectral image data was then analyzed with the Sandia National Laboratories' Automated eXpert Spectral Image Analysis (AXSIA) software [16], [17]. AXSIA analyzes the entire spectral image and reduces it to a more compact form, containing just the chemical information as a series of several chemical component images and spectral area maps with no loss of chemical information [17].

III. Results

A. Friction and Wear Behavior of LIGA NiMn and DLN-Coated LIGA NiMn

The first series of tribological measurements were made on DLN-coated silicon wafers and DLN-coated NiMn test coupons with a S_3N_4 ball. Silicon substrates were chosen to minimize the effects of surface topography and substrate–coating adhesion on friction. Second, there is prior published data on the tribology of DLN coatings on silicon wafers for comparison with current measurements [18]–[21]. Finally, initial measurements on Si ensured that the tribological behavior of the coatings was reproducible before they were applied on real LIGA NiMn parts. Table I summarizes the average steady-state COF values under varying contact stresses and testing environments. Application of DLN coatings clearly resulted in significant reductions in the COF. The following two main trends emerge from Table I: 1) Environment plays a crucial role in governing the tribological behavior of DLN coatings; under otherwise identical conditions, COF in dry nitrogen is significantly lower than the COF in humid air, and 2) COF decreases with an increase in contact stress. The mechanisms responsible for this observed behavior will be discussed in a later section. Typical friction coefficients as a function of test cycles are shown in Fig. 6. The data in Fig. 6 represent the following two sets of measurements: 1) on the sidewall of an uncoated LIGA NiMn spring with a LIGA Ni probe tip (red) and 2) on a polished Ni surface with a Si_3N_4 ball (blue). The former case represents sidewall–sidewall contacts. As can be seen in Fig. 6, the self-mated NiMn-Ni sidewall–sidewall contacts produced

TABLE I
STEADY-STATE COF VALUES OF DLN-COATED Si WAFER AND DLN-COATED NiMn COUPONS UNDER VARYING HERTZIAN MAXIMUM CONTACT STRESSES (P_m) AND TESTING ENVIRONMENTS (DRY NITROGEN AND 50% RH). THE COUNTERFACE WAS A 3.125-mm-DIAMETER Si_3N_4 BALL

Material	Environment	*L*, mN and (P_m, MPa)	COF
DLN coated Si	50% RH air	98 (316)	0.22 ± 0.02
		490 (541)	0.14 ± 0.02
		980 (682)	0.11 ± 0.02
	Dry nitrogen	98 (316)	0.02
		490 (541)	0.013
		980 (682)	0.009
DLN coated NiMn	50% RH air	490 (541)	0.14 ± 0.02
		980 (682)	0.09 ± 0.02
NiMn (uncoated)	50% RH air	98 (316)	0.55 ± 0.06
		490 (541)	0.76 ± 0.14

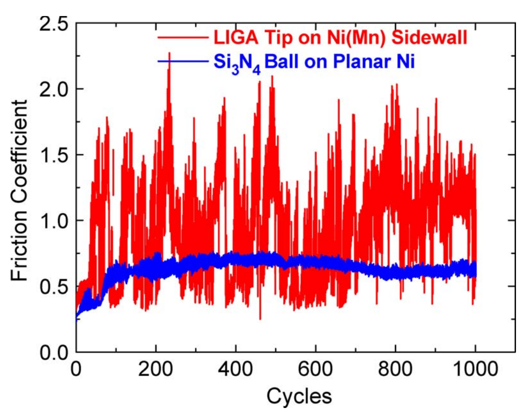

Fig. 6. Typical coefficients of friction as a function of test cycles for the case of (blue) a Si_3N_4 ball sliding on a polished Ni surface and (red) a LIGA Ni tip sliding on the sidewall of a NiMn spring in dry nitrogen test environment. Contact stress: 130 MPa.

significantly higher friction with larger fluctuations in the COF, compared with standard ball-on-flat tribological measurements with Si_3N_4 balls, highlighting the need to devise experiments that are more representative of the microsystems operating regimes.

Typical friction coefficients of uncoated and DLN-coated NiMn coupons against a LIGA Ni probe tip (i.e., in planar–sidewall configuration) as a function of test cycles are shown in Fig. 7. The data were generated at a contact stress of 130 MPa in dry nitrogen [Fig. 7(a)] and in air with 50% RH [Fig. 7(b)]. The COF for uncoated NiMn begins at a moderate value (0.2–0.4) and soon increases to a very high steady-state value with large fluctuations in the COF that are typical of metallic friction. In contrast, the average COF and the standard deviations values on DLN-coated NiMn are significantly lower, i.e., $\sim 0.08 \pm 0.002$ in dry nitrogen and $\sim 0.21 \pm 0.02$ in air with 50% RH, respectively.

B. TEM and Microanalysis

FIB microscopy was used to prepare cross sections of samples from the sidewalls of DLN-coated LIGA test structures. Fig. 8 is a bright-field TEM image of one such cross section of the sidewall of the DLN-LIGA Ni test structure. The topmost layer corresponds to an Au–Pd layer sputtered on the structure prior to FIB preparation to make the specimen electrically conductive. This conductive layer also served to help protect the coating from direct Ga ion implantation during FIB preparation. The DLN coating is continuous and dense with a thickness of 80 nm (approximately 20% less than the thickness on the planar surfaces from the same coatings batch). Fig. 9 shows the results of the AXSIA analysis of an X-ray spectral image acquired from the region denoted by the box in Fig. 8. AXSIA found four chemically significant components, of which three are shown in Fig. 9. The fourth component, which is not shown, corresponded to the Au–Pd conductive coating. Fig. 9(a) shows the DLN coating, consisting of Si, O, and C. Fig. 9(b) shows a 12–16-nm-thick Ti adhesion layer that appears to have oxidized, as evident by the strong O signal correlating with the Ti, and Fig. 9(c) shows the substrate component Ni. Ni is also seen in the Ti adhesion layer [Fig. 9(b)], but only the Ni–K line is visible without a corresponding L X-ray line. This indicated that the apparent Ni in the adhesion layer is most likely due to secondary fluorescence, given its proximity to the substrate. Measurements made at different locations on the sidewalls of various other LIGA parts also showed similar results.

C. EBSD Analyses

FIB microscopy was used to prepare cross sections of wear scars from ball-on-flat tests to understand the friction-induced microstructural evolution in the metallic substrates underneath the DLN coating. One set of images of FIB-prepared cross sections is shown in Fig. 10. In these images, the contrast in the substrate arises from the crystallographic grain orientation, also known as channeling contrast. The orientation changes between grains as well as orientation changes within grains due to deformation results in contrast differences. Fig. 10(a) shows the cross section through a wear scar on the uncoated substrate. The contrast change near the surface is caused by the deformation resulting from the friction test. Fig. 10(b) is a cross section through a wear scar on the DLN-coated sample. Note

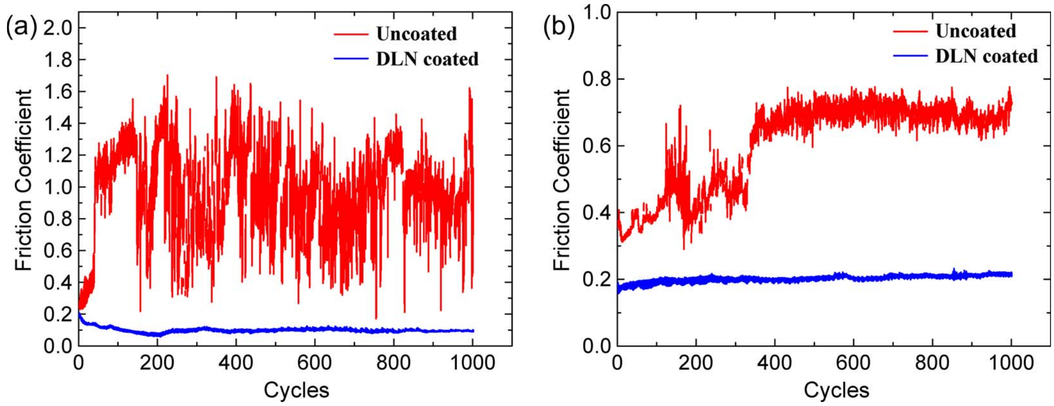

Fig. 7. Friction coefficients of uncoated and DLN-coated NiMn planar surfaces against a LIGA Ni probe tip (planar–sidewall contacts). (a) Dry nitrogen. (b) Air with 50% RH. Contact stress: 130 MPa.

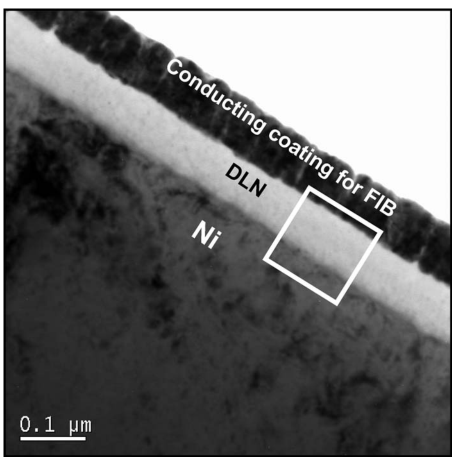

Fig. 8. Bright-field TEM image of the FIB cross section taken from the sidewall of the DLN-coated LIGA test structure shown in Fig. 3(c). Inset box denotes the region from which an X-ray spectral image was acquired.

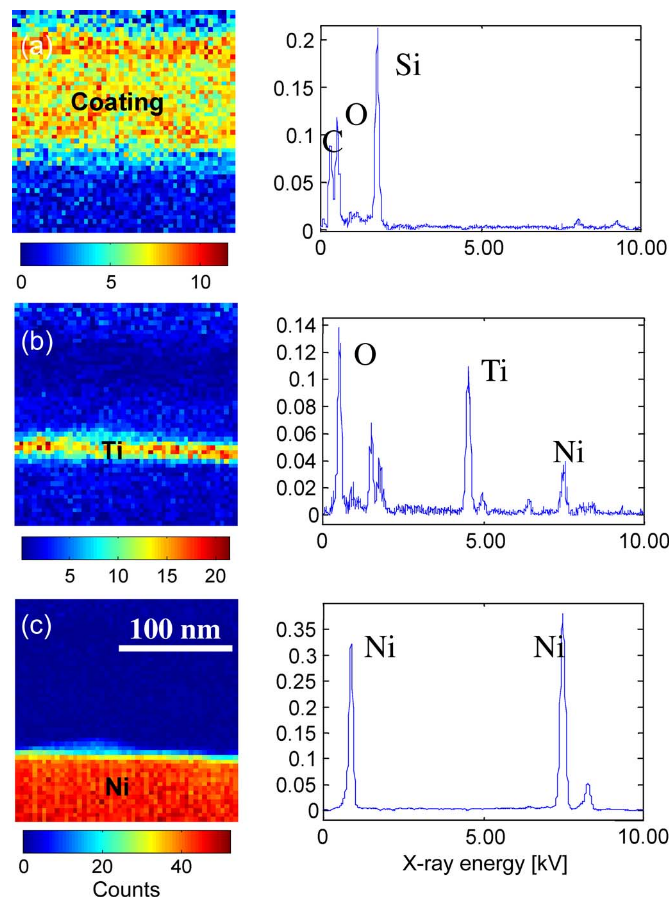

Fig. 9. Results of the AXSIA analysis of the X-ray spectral image acquired from the region denoted in Fig. 8 showing three chemical components. Component image and spectrum pair from (a) the DLN coating, consisting of C, O, and Si, (b) the Ti adhesion layer, and (c) the Ni substrate. The spectral scale is in normalized counts.

that there is little or no contrast change near the surface of the sample and that there is no significant deformation of the grains. It is readily apparent that there is a significantly reduced amount of deformation in the substrate of the DLN-coated sample. The uncoated sample shows many grains near the surface that are deformed in the direction of sliding contact. The DLN-coated sample shows little or none of the deformation, even though both samples had been friction tested under identical conditions.

EBSD was used to further demonstrate the lack of deformation in the substrate of the friction-tested DLN-coated sample. EBSD results are shown in Fig. 11. In this figure, the orientation map is colored with respect to the growth direction of the electrodeposited substrate. The red regions are grains with the $\langle 001 \rangle$ direction parallel to the deposition direction. EBSD is sensitive to small orientation changes (on the order of 0.1°), and there is no detectable orientation change in the substrate under the wear scar. These results demonstrate the improvement in subsurface friction-induced damage as a result of DLN coating.

IV. Discussion

Most engineering metallic surfaces, such as those presented in this paper, have native oxides and adsorbents on them. Once the adsorbed surface layers and native oxide films are removed due to wear, the softer metallic materials (e.g., NiMn and Ni) will begin to deform plastically and, in extreme cases, form metallic junctions at the asperity level. It is worth noting that though the total applied load may be low, the contact stress at the asperity level can be significantly higher; in fact, asperity stresses are typically near the flow stress of the softer material. The large fluctuations in the COF on bare NiMn coupons seen in Fig. 7 are a result of the making and breaking of these metallic junctions. It should be noted that both the average COF

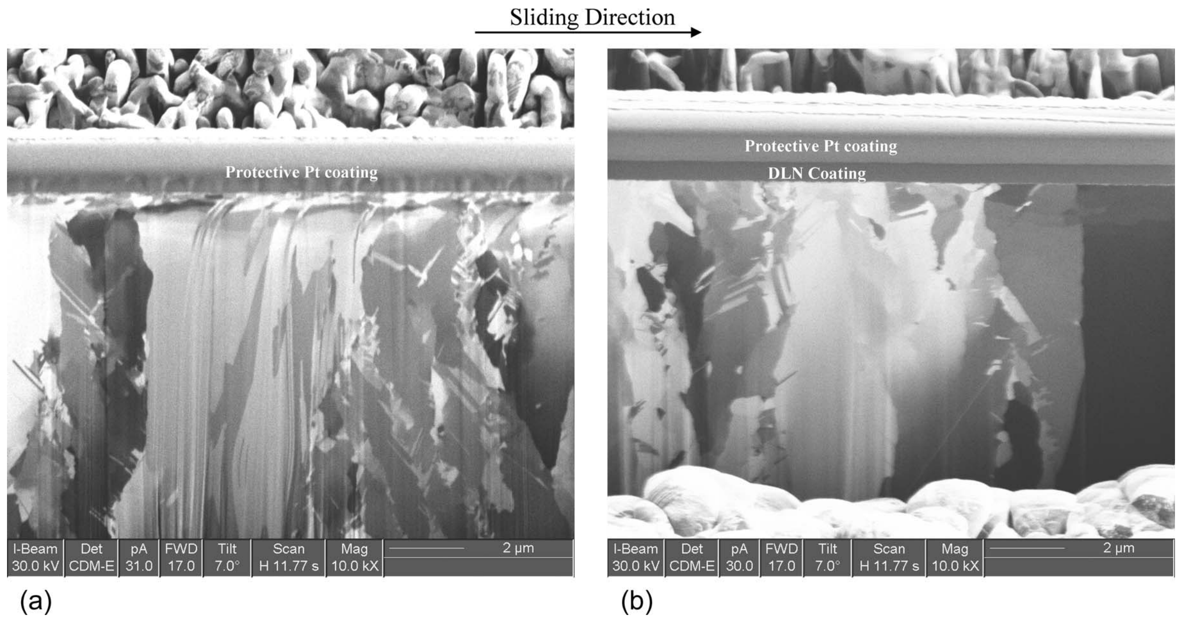

Fig. 10. Ion-induced electron images of cross sections of wear scars on planar Ni surfaces. (a) Uncoated Ni. (b) DLN-coated Ni. Friction tests were conducted with a Si_3N_4 ball at a normal load of 98 mN for 1000 sliding cycles in dry nitrogen.

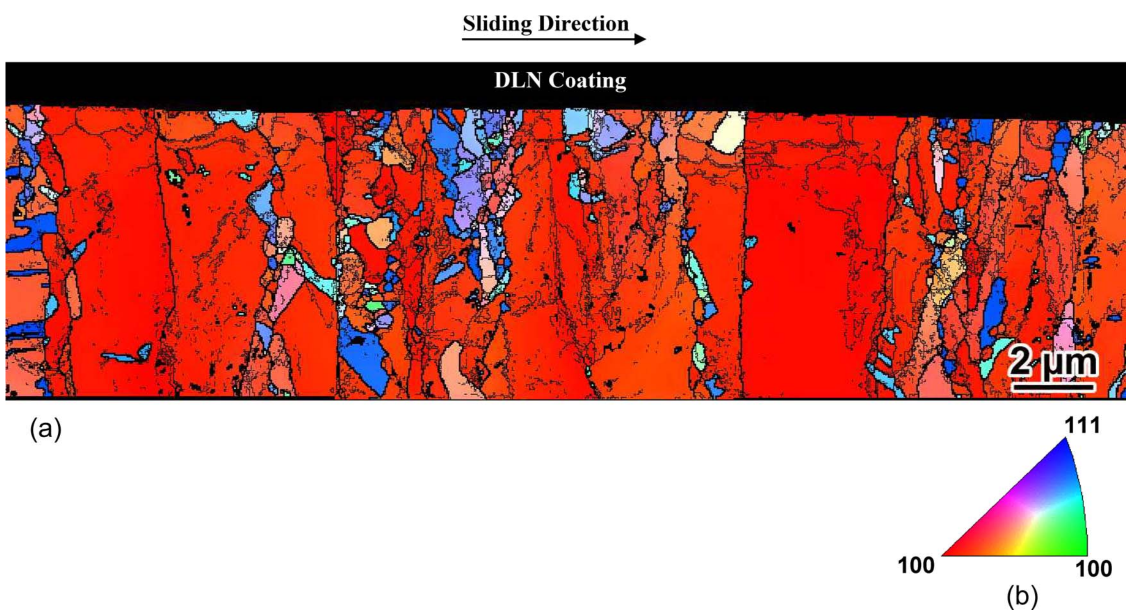

Fig. 11. (a) EBSD image corresponding to Fig 10(b), showing practically no friction-induced plastic deformation in Ni substrate underneath the DLN coating. (b) Stereographic triangle with color key for EBSD images.

and the standard deviation of measurements on bare NiMn in dry nitrogen (0.93 $\pm$ 0.32) are higher than the corresponding values in air with 50% RH (0.68 $\pm$ 0.06) and that the transition to steady-state regime occurred much earlier in dry nitrogen (Fig. 7). The lack of oxygen and water vapor in the environment perhaps reduced the wear life of native oxide films as well as the *in situ* reformation of native oxides. These complications in the tribological behavior of electroformed metallic materials could potentially interfere with the performance and reliability of LIGA microsystems. Since the relative maturity of Ni and Ni alloy processing make them the materials of choice for LIGA microsystems, coatings to mitigate this type of wear behavior are therefore essential.

This paper has demonstrated that the COF as well as fluctuations in friction measurements can be significantly reduced by the application of DLN coatings. The DLN coatings, like most other solid lubricating films, function by transferring a thin film onto the counterface [19]–[22]. The transfer film mechanism implies that there is no need to coat both surfaces in order to achieve low friction and wear. Although not shown, friction measurements between DLN-coated NiMn coupons and DLN-coated Ni probe tips also confirmed that no further reduction can be achieved by coating both surfaces.

The low interfacial shear stress between the DLN coating and the friction-induced transfer film on the Ni friction probe tip is responsible for the low friction. The experimentally observed

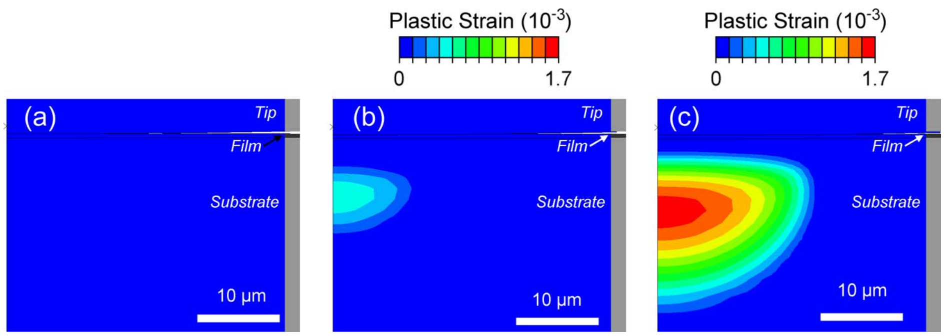

Fig. 12. FEM simulations of accumulated plastic strain on DLN-coated Ni under a Si_3N_4 ball at three peak contact stresses: (a) 500 MPa; (b) 850 MPa; and (c) 1 GPa.

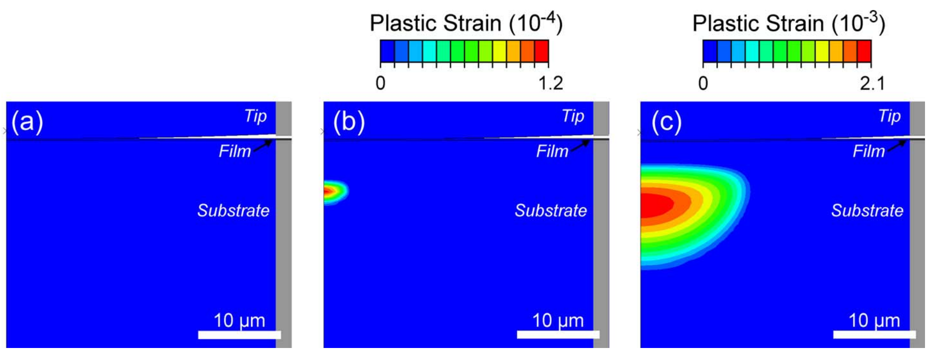

Fig. 13. FEM simulations of accumulated plastic strain on DLN-coated NiMn alloy under a Si_3N_4 ball at three peak contact stresses: (a) 1 GPa; (b) 1.4 GPa; and (c) 1.7 GPa.

decrease in the COF with increasing contact stress is consistent with the behavior observed in other solid lubricating systems that was explained based on the Hertzian contact model, i.e., COF $\sim S_o/p$, where S_o is an interfacial shear strength, and p is the mean elastic contact stress [23]. It should be pointed out that COF values of DLN coatings in air reported by Kester *et al.* [18] are lower than the present measurements $(\mu \sim 0.2)$ made in 50% RH air. The major difference is the initial mean Hertzian contact stress. Previous researchers used higher contact stresses (1.1 GPa). According to the Hertzian contact model described above, increasing contact stresses should result in decreasing COF for lubricating material like DLN [19]. Although the RH levels between measurements from different groups were identical, the role of other minor species (other than water vapor) in the environment on friction may not be completely ruled out.

The Hertzian contact model is valid as long as elastic contact conditions are satisfied, i.e., the underlying substrate does not plastically deform during sliding contact, as shown in Figs. 10(b) and 11. Finite-element simulations [24] showed that contact stresses higher than a certain critical value (750 MPa for Ni and 1350 MPa for NiMn) could result in the initiation of substrate plasticity underneath the DLN coating, resulting in coating fracture/delamination and loss of lubrication. Two examples of FEM simulations from our previous study [24] are shown in Figs. 12 and 13 to illustrate this point. Note that these contact stresses are much higher than the ones used in this paper and in microsystems operation, which were typically around 400 MPa [25]. The FEM simulations also revealed that varying the coating thickness from 150 to 1000 nm had little impact on these critical contact stress values required to initiate plastic deformation in the underlying substrate [24].

Another critical issue is the application of the DLN coating conformally to the entire LIGA-fabricated part, particularly on the sidewalls. Previous studies [3], [4] revealed that some of the standard commercial processes may be tailored to conformally coat and provide the same DLC coverage, structure, and chemistry on the more complicated sidewall. The process incorporates planetary substrate rotation, in which samples are rotated individually on a platen, which also rotates at an angle to the source, thereby exposing all surfaces for coating to obtain the DLC structure/chemistry on the planar and sidewalls parts. Finally, the presence of a thin titanium bond layer was found to be necessary for the coating to remain adhered on Ni alloy parts during sliding contact.

V. Summary and Conclusion

The feasibility of applying DLN coatings on the sidewalls of LIGA-fabricated microsystem parts by commercial PECVD techniques has been demonstrated. A novel strategy was devised to overcome the problems associated with handling miniature LIGA parts during deposition. The friction coefficients were very low in both dry nitrogen and ambient air environments, i.e., 0.08 and 0.2, respectively, in comparison to the high values for uncoated Ni alloys. In addition to improving the tribological performance, DLN coatings were very effective in suppressing the plastic deformation in the underlying substrates. The critical role of novel test structures and characterization techniques in evaluating the tribological behavior of surface coatings in regimes relevant to LIGA microsystems was illustrated.

ACKNOWLEDGMENT

The authors would like to thank C. Venkatraman and C. Brodbeck of Bekaert Advanced Coating Technologies for supplying the DLN coatings and Dr. J. Jungk for performing the FEM simulations.

REFERENCES

[1] T. R. Christenson, "X-ray based fabrication," in *MEMS Handbook*, G.-E.-H. Mohammed, Ed. Boca Raton, FL: CRC Press, 2001.

[2] J. Hruby, "LIGA technologies and applications," *MRS Bull.*, vol. 26, pp. 337–340, 2001.

[3] S. V. Prasad, M. T. Dugger, T. R. Christenson, and D. R. Tallant, "LIGA microsystems: Surface interactions, tribology, and coatings," *J. Manuf. Process.*, vol. 6, no. 1, pp. 107–116, 2004.

[4] T. W. Scharf and S. V. Prasad, "Diamond-like nanocomposite coatings for LIGA nickel alloy parts," Sandia Nat. Lab., Albuquerque, NM, p. 1004, Rep. SAND2005-1004, 2005.

[5] W. J. Meng, L. S. Stephens, and K. W. Kelly, "LIGA-based micro-mechanical systems and ceramic nanocomposite surface coatings," in *Nano-Tribology: Critical Assessment and Research Needs*, S. M. Hsu and Z. C. Ying, Eds. Norwell, MA: Kluwer, 2003.

[6] D. Kim, D. Cao, M. D. Bryant, W. Meng, and F. F. Ling, "Tribological study of microbearings for MEMS applications," *J. Tribol.*, vol. 127, no. 3, pp. 537–547, Jul. 2005.

[7] D. M. Cao, W. J. Meng, S. J. Simke, G. L. Doll, T. Wang, and K. W. Kellog, "Conformal deposition of Ti-C:H coatings over high-aspect ratio microscale structures and tribological characteristics," *Thin Solid Films*, vol. 429, no. 1/2, pp. 46–54, Apr. 2003.

[8] Y. Tian, A. F. Bastwros, C. C. H. Lo, and A. P. Constant, "Superhard self-lubricating $AlMgB_{14}$ films for microelectromechanical devices," *Appl. Phys. Lett.*, vol. 83, no. 14, pp. 2781–2783, Oct. 2003.

[9] R. F. Bunshah, *Handbook of Hard Coatings: Deposition Technologies, Properties and Applications*, R.F. Bunshah, Ed. Park Ridge, NJ: Noyes, 2001, pp. 66–69.

[10] V. F. Dorfman, "Diamond-like nanocomposites (DLN)," *Thin Solid Films*, vol. 212, no. 1/2, pp. 267–273, May 1992.

[11] C. Venkatraman, D. J. Kester, A. Goel, and D. J. Bray, "Diamond like nanocomposite coatings—A new class of materials," in *Surface Modifications Technologies IX*, T. Sudarshan, W. Reitz, and J. J. Stiglich, Eds. Warrendale, PA: The Minerals, Metals and Materials Society, 1996.

[12] A. Hall, M. T. Dugger, S. V. Prasad, and T. Christenson, "Sidewall morphology of electroformed LIGA parts—Implications for friction, adhesion and wear control," *J. Microelectromech. Syst.*, vol. 14, no. 2, pp. 326–334, Apr. 2005.

[13] S. V. Prasad, J. R. Michael, and T. R. Christenson, "EBSD studies on wear-induced subsurface regions in LIGA nickel," *Scr. Mater.*, vol. 48, no. 3, pp. 255–260, Feb. 2003.

[14] T. E. Buchheit, D. A. LaVan, J. R. Michael, T. R. Christenson, and S. D. Leith, "Microstructural and mechanical properties investigation of electrode posited and annealed LIGA nickel structures," *Metall. Mater. Trans. A*, vol. 33, no. 3, pp. 539–554, 2002.

[15] S. J. Goods, J. J. Kelly, and N. Yang, *Microsyst. Technol.*, vol. 10, p. 498, 2004.

[16] M. R. Keenan and P. G. Kotula, "Apparatus and system for multivariate spectral analysis," U.S. Patent 6 584 413, Jun. 24, 2003.

[17] P. G. Kotula, M. R. Keenan, and J. R. Michael, "Automated analysis of EDS spectral images in the SEM: A powerful new microanalysis technique," *Microsc. Microanal.*, vol. 9, pp. 1–17, 2003.

[18] D. J. Kester, C. L. Brodbeck, I. L. Singer, and A. Kyriakopoulos, "Sliding wear behavior of diamond-like nanocomposite coatings," *Surf. Coat. Technol.*, vol. 113, no. 3, pp. 268–273, Mar. 1999.

[19] T. W. Scharf, J. A. Ohlausen, D. R. Tallant, and S. V. Prasad, "Mechanisms of friction in diamond-like nanocomposite coatings," *J. Appl. Phys.*, vol. 101, no. 6, pp. 063 521-1–063 521-11, Mar. 2007.

[20] T. W. Scharf and I. L. Singer, "Role of third bodies in friction behavior of diamond-like nanocomposite coatings studied by in situ tribometry," *Tribol. Trans.*, vol. 45, no. 3, pp. 363–371, 2002.

[21] T. W. Scharf and I. L. Singer, "Monitoring transfer films and friction instabilities with in situ raman tribometry," *Tribol. Lett.*, vol. 14, no. 1, pp. 3–8, Jan. 2003.

[22] I. L. Singer, "Solid lubrication processes," in *Fundamentals of Friction: Macroscopic and Microscopic Processes*. Dordrecht, The Netherlands: Kluwer, 1992, pp. 237–261.

[23] D. Bowden and F. P. Tabor, *The Friction and Lubrication of Solids*. Oxford, U.K.: Clarendon, 1986.

[24] J. J. Jungk, J. R. Michael, and S. V. Prasad, "The role of substrate plasticity on the tribological behavior of diamond-like nanocomposite coatings," *Acta Mater.*, vol. 56, no. 9, pp. 1956–1966, May 2008.

[25] A. D. Romig, M. T. Dugger, and P. J. McWhorter, "Materials issues in microelectromechanical devices: Science, engineering, manufacturability and reliability," *Acta Mater.*, vol. 51, no. 19, pp. 5837–5866, Nov. 2003.

Somuri V. Prasad received the Ph.D. degree in materials science from the University of Sussex, Brighton, U.K., in 1977.

He was with the Air Force Research laboratory, Wright Patterson Air Force Base, Dayton, OH, and with the Council of Scientific and Industrial Research, India. In 1999, he joined Sandia National Laboratories, Albuquerque, NM, where he is currently a Principal Member of Technical Staff. He is also an Adjunct Professor of mechanical engineering with the University of Colorado, Boulder. For the past 25 years, his research has focused on the fundamental mechanisms of friction, and the synthesis of novel materials and coatings for tribological applications.

Dr. Prasad is a Fellow of ASM International and the Society of Tribologists and Lubrication Engineers.

Thomas W. Scharf received the B.S. degree in ceramic science and engineering from Pennsylvania State University, University Park, in 1995, and the Ph.D. degree in metallurgical and materials engineering from the University of Alabama, Tuscaloosa, in 2000.

From 2000 to 2003, he was a Postdoctoral Fellow with the Naval Research Laboratory, Washington, DC. From 2003 to 2005, he was a Limited-Term Member of Technical Staff with Sandia National Laboratories, Albuquerque, NM. He is currently an Assistant Professor with the Department of Materials Science and Engineering, University of North Texas, Denton, performing research on hard and solid lubricant coatings and studying the fundamental mechanisms of friction, wear, and lubrication of sliding and rolling interfaces by electron microscopy, vibrational spectroscopy, and atom probe tomography techniques.

Paul G. Kotula received the Ph.D. degree in materials science and engineering from the University of Minnesota, Minneapolis, in 1995.

He is currently a Principal Member of Technical Staff with Sandia National Laboratories, Albuquerque, NM, and an Adjunct Professor with North Carolina State University, Raleigh. He is the holder of two patents. For the past ten years, his research has focused on the development of improved microanalysis methods, including spectral imaging, multivariate statistical analysis, 3-D microanalysis, and advanced X-ray detectors.

Dr. Kotula is a Past President of the Microbeam Analysis Society. He received an R&D 100 Award in 2002 for the development of the AXSIA software, which, to date, can be found as a commercial product in over 500 laboratories worldwide.

Joseph R. Michael received the Ph.D. degree in materials science from Lehigh University, Bethlehem, PA, in 1984.

He is a Distinguished Member of Technical Staff with Sandia National Laboratories, Albuquerque, NM. His research has focused on the application of electron and ion beams to the characterization of materials.

Dr. Michael is a member of the Microscopy Society of America and the Microbeam Analysis Society.

Todd R. Christenson received the Ph.D. degree in electrical engineering from the University of Wisconsin, Madison, in 1995.

He was with Sandia National Laboratories, Albuquerque, NM. He is currently the Chief Technical Officer with HT MicroAnalytical, Inc., Albuquerque, a company he cofounded to manufacture precision metal-based micromechanisms. For the past 23 years, his research has centered on the design and microfabrication of integrated physical sensors and actuators. He is the author of the book chapter "X-Ray Based Fabrication" in the *CRC MEMS Handbook*.