

# Metal Diffusion in Polymers

J. H. Das and J. E. Morris, *Senior Member, IEEE*

**Abstract**—The diffusive properties of copper, nickel, titanium and chromium have been studied in three polyimides and a fluoropolymer using both sputter-deposited surface film and shallow ion-implant sources. No measurable diffusion was found for either chromium or titanium, but diffusion constants and their thermal activation energies are presented for copper and nickel in each polymer. All the copper and nickel systems exhibit the clustering effects previously reported, and both atomic and cluster diffusion data are cited where possible. The metallic diffusion profiles from the surface films typically display three components: an initial subsurface distribution (which is actually due to the penetration of the surface by low energy ions during sputter-deposition), a shallow cluster distribution which contains the bulk of the diffusing material, and a deeper dilute atomic distribution.

**Index Terms**—Copper, nickel, polyimide, fluoropolymer, diffusion, cluster.

## I. INTRODUCTION

POLYMERS are playing an increasingly important role in the protection and interconnection of a variety of electronic components at both the chip and packaging levels. The copper-polyimide system is especially prominent in MCM (multi-chip module) developments [1]–[6], and understanding of the long term stability of the metal/polymer interface is vital to this and other developing metal/polymer technologies. In particular, the interdiffusion mechanisms at polymer/metal interfaces and the relative significances of physical structure (e.g., polymer voids) and chemistry (e.g., oxide formation) are not yet fully understood.

With diffusion from ion implanted Gaussian profiles of copper in Kapton® polyimide [7], we have previously observed three different types of implanted copper ion activity, each dominating the observations in each of three separate temperature ranges, namely:

- 1) A low temperature diffusion (below 185°C) with an activation energy of 0.60 eV,
- 2) an intermediate range of temperatures over which self-gettering of copper atoms into clusters was observed, in

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J. H. Das was with Department of Electrical Engineering, T. J. Watson School of Engineering and Applied Science, State University of New York at Binghamton, NY 13902-6000 USA. He is now with the National Nanofabrication Facility, Knight Laboratory, Department of Electrical Engineering, Cornell University, Ithaca, NY 14853 USA.

J. E. Morris is with the Department of Electrical Engineering, T. J. Watson School of Engineering and Applied Science, State University of New York at Binghamton, NY 13902-6000 USA.

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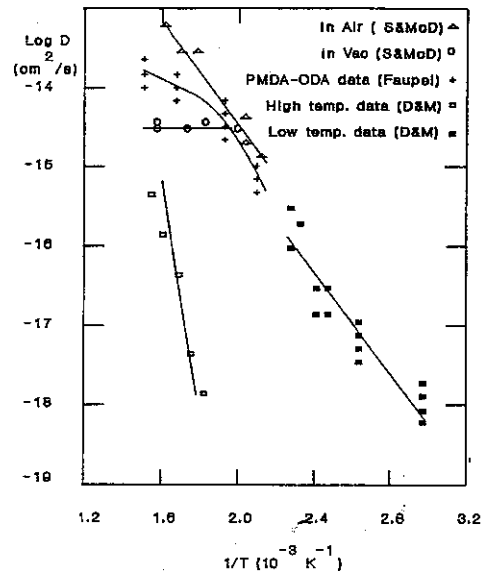


Fig. 1. Arrhenius plots of the Cu diffusion constant in polyimide. Data replotted from Das and Morris [9] (ion implants), Shanker and MacDonald [11], and Faupel *et al.* [13] (surface films).

which the half width of the profile decreased and the peak concentration increased, and

- 3) a high temperature activation energy (above 250°C) of 1.54 eV, attributed to cluster motion [8]–[10].

The low temperature (atomic) diffusion results were in fair agreement with other results obtained from constant source diffusions [11], i.e., from deposited surface films of copper. In those constant source diffusions, the activation energy was about 0.65 eV over the temperature range of 100–375°C, compared to the 0.68 eV which could be reached from our data over a range from ambient to 350°C by the inclusion of the high temperature point (Fig. 2, [8] or Fig. 2.7, [12]). At 160°C the diffusion constant,  $D$ , was in the  $10^{15}$  cm<sup>2</sup>/s range in both cases [8], [11]. At this temperature it would take approximately four months for diffusing copper atoms to pass through 1 μm of Kapton film. The high temperature (cluster) diffusion coefficient drops dramatically above the aggregation temperature range.

Fig. 1 shows a montage of these ion implant results in the chemically cured Kapton® [8] and earlier constant source diffusion data for copper in thermally cured PMDA-ODA polyimide [11], [13], suggesting that the earlier values may have incorporated both atomic and cluster processes. Note that the Kapton® data was obtained from four variant types of film, (types H, HN, V, and VN), with no discrimination possible between the four data sets [8].

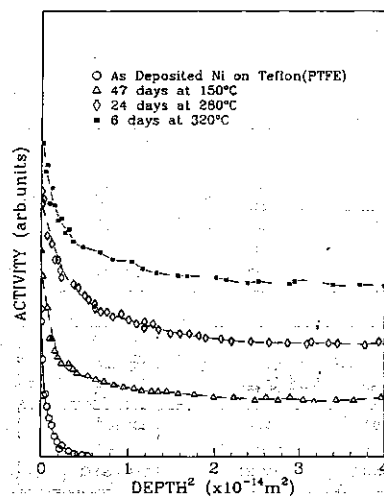


Fig. 2. Penetration profiles of Nickel from a surface film into Teflon® (PTFE), showing two parts: an initial steep drop and a Fickian tail. (Activities are offset vertically for clarity, approx. zero at 2  $\mu\text{m}$ .)

The thermal process was also computer simulated by incorporating the experimentally observed dual activation energies into conventional diffusion models [14], [15]. The self-gettering of atoms near the peak region of a Gaussian distribution was found to be due to a favorable initial free atomic distribution with a "dip" at the single atom distribution peak, due to initial clustering of metal atoms in this region. The degree of gettering was found to possess a complex dependence upon the as-implanted distribution profile. High temperature diffusion simulations also demonstrated the initial increase in total copper peak height (single and clustered atoms) due to self gettering of atoms, and the subsequent diffusion of clusters, in accordance with experiment [8], [10].

The work has been extended [16] to diffusion from ion implants of other metals—nickel, chromium and titanium—and to other polymers of future significance to electronics packaging—Teflon® and Upilex® [17]. Results from Upilex® films were found to be very similar to the Kapton Case, while significant damage to the Teflon® surface was observed and attributed to melting due to the impact of energetic ions. The primary objective was to investigate the clustering and diffusion mechanisms previously observed for copper. Nickel was found to behave in a similar fashion, i.e., gettering was observed with similar types of cluster and free atomic diffusions. Work with chromium and titanium revealed no observable diffusion within the experimental treatment durations for the selected temperature ranges.

The studies have also been extended to include comparisons with diffusion coefficients determined for diffusion from surface films of all four metals deposited on all the polymers in question by low energy RF planar magnetron sputtering. The copper diffusion profiles have previously been shown to be resolvable into two components corresponding to the diffusion of clusters and to free atomic diffusion [12], [16], [18]–[21]; there is also a shallow implant below the surface of as-deposited films. (Prior interpretations of surface film diffusion data have been based on assumptions of single

diffusant species profiles [11], [13].) Nickel from the thin film behaves in much the same fashion as copper, but with smaller diffusion rates. Work with chromium and titanium again demonstrated no observable diffusion for the experimental treatment durations and temperature ranges.

## II. EXPERIMENTAL

### A. Ion Implantation

225 keV metal ion implantations were performed at the National Nanofabrication Facility at Cornell University. Copper ions were produced by thermal evaporation, with a copper wire wrapped around a tungsten filament, prior to extraction from the source and acceleration towards the polymer target. The implant current was in the  $\mu\text{A}$  range, and the process was reproducible. It was not possible to follow this method for the other metals due to their much higher melting temperatures. Another technique implemented with limited success for nickel, chromium and titanium required the selection of a suitable metal compound which was then heated in an oven source to about 500°C with metal ions extracted into the acceleration field utilizing their sublimation properties. The beam currents are very low, requiring several (4–20) hour implantations to obtain dosages in the range of  $5 \times 10^{15}$  to  $1 \times 10^{16}$  ions/cm<sup>2</sup>. These ion implanted samples were subsequently thermally treated in air for varying times (4 h to 14 days) in the temperature range of 75–375°C.

### B. Sputter Deposition

Very thin films of these metals were deposited by Ar<sup>+</sup> sputtering, with surface coverages ranging from  $6 \times 10^{16}$  to  $1.8 \times 10^{17}$  atoms/cm<sup>2</sup> (equivalent to 7.2–21.5-nm-thick continuous films of copper). These films were subsequently thermally treated in air for varying times (3 to 48 days) in the temperature range of 100–375°C. (Magnetron sputter deposition was used for nickel, despite exceptionally slow rates, to maintain consistency with the other systems studied).

*Distribution Profiles:* A typical set of diffusion profiles is presented in Fig. 2. The initial distribution below the substrate surface has the appearance of a diffusion profile characteristic of  $Dt \sim 4.2 \times 10^{-11}$  cm<sup>2</sup>. This factor can be neglected for high temperature diffusion measurements if  $D$  is sufficiently large, but at low temperature very large diffusion times are required to achieve the same effect (48 days at room temperature).

During thermal treatments, two distinct types of activity in the distribution were observed. A slow moving front (confined to the steep drop in concentration) near the surface, and a fast diffusing front (deeper into the substrate) with a very dilute concentration.

*Cluster Distribution:* Most of the diffusants inside the substrate are contained in a slow moving front with much less penetration than the Fickian distribution tail extending deep in the polymers. The diffusion mechanism for this slow moving front has been attributed to cluster diffusion between polymer voids [22], which is consistent with the higher activation energy requirements of relatively large sized clusters.  $D_{\text{cluster}}$  values were obtained by approximating this steep edge by an

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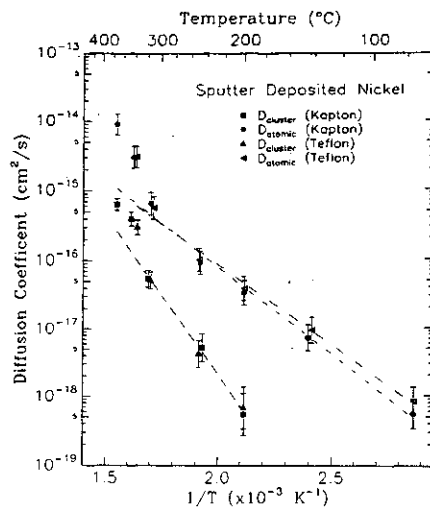


Fig. 3. Arrhenius plots of the diffusion coefficients  $D$  of atoms and clusters of Nickel in Kapton and Teflon (PTFE). (Surface film data).

erfc distribution and monitoring its movements, i.e., fitting the profile at varied times for each test temperature.

**Atomic Distribution:** The more rapid motions of the Fickian tails extending further into the polymers correspond to the diffusion characteristics of free atoms, as they penetrate deeper into the substrate. The diffusion characteristics from sputtered films were found to be a function of the metal concentrations, particularly at low metal concentrations of the interfaces under investigation here. For thick surface films, the source of diffusants is the thin film interface itself, from which individual atoms are released into the substrate; clustering by these atoms may follow. By contrast, a very thin quasi-continuous film presents a reduced interface area from which atoms may enter the substrate, and will also contain discrete surface islands which may diffuse into the substrate directly.

**Analysis:** RBS (Rutherford Back Scattering) methods were used to monitor distribution profiles in both the ion implantation and sputter deposition cases. RBS data were taken using a 2.2 MeV  $\text{He}^{2+}$  beam at Cornell University. The raster scanned beam current was restricted to  $<12 \text{ nA/cm}^2$  for Kapton® and Upilex®, and to  $5 \text{ nA/cm}^2$  for Teflon, with current integration a low  $5 \mu\text{C}$  to minimize beam damage. The relatively low RBS energy of 2.2 MeV gave good resolution of the spectrum, but was high enough to provide the depth range needed. The measured distributions were matched to theoretical profiles to extract diffusion constants [23]–[25].

### III. RESULTS

#### A. Diffusion Results

**Cu/Polymer:** The low temperature diffusion results from ion implantation experiments [8]–[10] are in fair agreement with others' results obtained at higher temperatures from constant source diffusions [11], [13], i.e., from a deposited surface film of copper. The results were also comparable with the later sputter-deposited cases already reported [12], [19]. Results with the various types of polymers were also

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TABLE I  
COPPER DIFFUSION DATA IN VARIOUS POLYMERS ( $D_0 \text{ cm}^2/\text{s}$ ,  $E_a \text{ eV}$ )

	Upilex® R	Upilex® S	Kapton®	Teflon®(PTFE)
Sputter:				
Atomic $D_0$	$8.2 \times 10^{-11}$	$3.5 \times 10^{-12}$	$2.1 \times 10^{-11}$	$7.9 \times 10^{-13}$
Atomic $E_a$	$0.52 \pm 0.16$	$0.42 \pm 0.13$	$0.49 \pm 0.17$	$0.38 \pm 0.15$
Temp.	25–250°C	25–250°C	25–250°C	25–250°C
Cluster $D_0$	$5.0 \times 10^{-9}$	$1.3 \times 10^{-9}$	$4.3 \times 10^{-8}$	$1.0 \times 10^{-7}$
Cluster $E_a$	$1.66 \pm 0.46$	$2.25 \pm 0.29$	$1.05 \pm 0.13$	$1.07 \pm 0.33$
Temp.	25–300°C	26–375°C	25–350°C	25–300°C
Ion Implant:				
Atomic $D_0$	$1.4 \times 10^{-11}$	$1.1 \times 10^{-11}$	$5.0 \times 10^{-10}$	N/A
Atomic $E_a$	$0.46 \pm 0.16$	$0.46 \pm 0.15$	$0.60 \pm 0.16$	
Temp.	25–150°C	25–150°C	25–170°C	
Cluster $D_0$	$1.5 \times 10^{-9}$	$2.2 \times 10^{-12}$	$6.2 \times 10^{-4}$	N/A
Cluster $E_a$	$0.83 \pm 0.16$	$0.57 \pm 0.11$	$1.54 \pm 0.32$	
Temp.	250–300°C	250–330°C	250–350°C	

comparable with each other and the variations between the various polymers were small.

All copper/polymer interfaces exhibited similar cluster diffusion behaviors from an initially gettered distribution, although it was not possible to measure the diffusion coefficients for ion-implanted Teflon due to excessive irradiation damage to the samples. There was some variation in the activation energies, attributed mostly to RBS data scatter.

**Ni/Polymer:** Nickel behaved in a similar fashion to copper, but with lower magnitudes of atomic and cluster diffusion constants. Ion implanted results were comparable to the sputter deposited cases. Results with the various types of polymers were also comparable to each other and variations between the various polymers were small.

**Cr/Polymer and Ti/Polymer:** Extended periods of very high temperature thermal treatments (3 days at 375°C) of the chromium and titanium profiles showed no measurable diffusion of free atoms or clusters. The results are in agreement with the high stability expected of both the chromium/polymer and titanium/polymer interfaces. Results from the sputter deposited chromium/polymer and titanium/polymer interface studies also revealed similar superior thermal stabilities, attributed to stronger chemical bonding. Reports of observable chromium diffusion in polyimide have been disputed in the literature [18].

#### B. Activation Energies

An example of the Arrhenius plots of diffusion data from sputtered films is presented in Fig. 3. Note the high temperature curvature which appeared to correspond to substrate softening above about 300°C. The results presented below are determined from the lower temperature linear regions, except where otherwise indicated (by the temperature range cited) when there was insufficient low temperature data to establish a reliable straight line.

**Cu/Polymer:** A summary of the diffusion results for copper obtained from the two techniques is tabulated in Table I, which includes details of the temperature range employed for each value presented.

There is good agreement between the atomic diffusion  $E_a$  and  $D_0$  values for the two techniques. For atomic diffusion,

TABLE II  
NICKEL DIFFUSION DATA IN VARIOUS POLYMERS ( $D_0$  cm<sup>2</sup>/s,  $E_a$  eV)

	Uplex® R	Uplex® S	Kapton®	Teflon®(PTFE)
Sputter:				
Atomic $D_0$	$1.75 \times 10^{-11}$	$3.3 \times 10^{-12}$	$6.1 \times 10^{-12}$	$2.0 \times 10^{-12}$
Atomic $E_a$	0.51 +/- 0.13	0.46 +/- 0.12	0.49 +/- 0.16	0.44 +/- 0.12
Temp.	25-250°C	25-250°C	25-250°C	25-250°C
Cluster $D_0$	$4.05 \times 10^{-9}$	$2.15 \times 10^{-8}$	$4.2 \times 10^{-9}$	$3.1 \times 10^{-9}$
Cluster $E_a$	0.91 +/- 0.28	1.00 +/- 0.20	0.93 +/- 0.20	0.91 +/- 0.28
Temp.	25-300°C	25-375°C	25-300°C	25-300°C
Implant:				
Cluster $D_0$	$1.5 \times 10^{-13}$	$1.1 \times 10^{-12}$	$4.8 \times 10^{-13}$	N/A
Cluster $E_a$	0.49 +/- 0.17	0.60 +/- 0.11	0.55 +/- 0.21	
Temp.	250-300°C	250-330°C	250-350°C	

$E_a$  was calculated from the least squares error fitted lines of the Arrhenius plots. The error bars on the  $D$  values reflect the accuracy of the RBS analysis during the computer profile simulations [20]–[22]. The fitted lines were restricted to the safe operating temperature ranges for the polymers, yielding  $E_a$ 's consistent for both sputtered and ion implanted cases.

In all cases, the cluster  $E_a$  was found to be higher than the corresponding atomic  $E_a$  value, with large variations in cluster  $E_a$  and  $D_0$  values observed for the different types of polymers.

In contrast to the atomic case, cluster diffusion was only observable at higher temperatures, where significant polymer degradation seems to have occurred (near  $T_g$ ), affecting both  $D_{\text{cluster}}$  and  $D_{\text{atomic}}$  values. (We postulate a reduced  $T_g$  due to implantation damage; oxidation effects are also possible, but the purpose of the implantation technique was to avoid surface oxidation, so penetration depth becomes an issue here.) Cluster activity diminishes rapidly as temperature is decreased, and at the lower temperature range (200–250°C) experimental  $D_{\text{cluster}}$  values are necessarily less accurate for finite observation periods due to very small measurable changes in the distribution profiles. Thus the accuracies of  $D_{\text{cluster}}$  measurements are degraded:

- 1) At higher temperatures by polymer degradation, and
- 2) at lower temperatures by diminished cluster activity.

Due to the resultant narrow range of valid temperatures here, there is a shortage of reliable data points over which to establish an effective least squares error fit, and the cluster  $E_a$  values presented in Table I consequently display significant random error. Nevertheless, the values  $D_0 = 4.3 \times 10^{-8}$  cm<sup>2</sup>/s and  $E_a = 1.05 \pm 0.13$  eV correspond rather well to the independent results  $D_0 = 5.1 \times 10^{-8}$  cm<sup>2</sup>/s and  $E_a = 1.2$  eV obtained for the 320–460°C range [23], [24].

*Ni/Polymer:* A summary of the diffusion results obtained for nickel from the two techniques is tabulated in Table II. Nickel's values of atomic  $D$  are lower than copper's, and consequently surface loss during the initial stages of heat treatment is sufficient to distort the Gaussian atomic ion implants noticeably, and hence make any reliable determination of  $D_{\text{atomic}}$ , which relies upon the continued integrity of the Gaussian profile, impossible for the ion implantation case. (Loss of volatile C/N/O components from the damaged polymer surfaces was monitored by RBS for all samples [16]).

TABLE III  
EXTRAPOLATED ROOM TEMPERATURE  $D_{\text{atomic}}$  FOR  
Cu AND Ni IN VARIOUS TYPES OF POLYMERS

$D_{\text{atomic}}$ (cm <sup>2</sup> /s)	Uplex® R	Uplex® S	Kapton®	Teflon®(PTFE)
Cu/Polymer				
Ion Implantation	$4.2 \times 10^{-19}$	$3.5 \times 10^{-19}$	$1.2 \times 10^{-19}$	N/A
Sputtered Film	$5.0 \times 10^{-19}$	$4.0 \times 10^{-19}$	$2.2 \times 10^{-19}$	$2.5 \times 10^{-19}$
Ni/Polymer				
Ion Implantation	N/A	N/A	N/A	N/A
Sputtered Film	$3.0 \times 10^{-20}$	$3.6 \times 10^{-20}$	$2.5 \times 10^{-20}$	$5 \times 10^{-20}$

As for copper, there are also similarities in the diffusion parameters for atomic diffusion from sputtered films in the various polymers. The  $D_0$  and  $E_a$  values were calculated from least squares error fitted lines as in the copper case.

In contrast to the copper case, the sputtered film experiments here were designed in such a fashion that the higher temperature  $D_{\text{cluster}}$  values could also be least squares error fitted. The activation energies are tabulated in Table II. In comparison with copper's case, the cluster  $E_a$ 's here are seen to be more consistent between the various polymers, but with a similarly consistent discrepancy evident between values for the sputter deposited and ion implanted cases.

#### C. Room Temperature Diffusion Coefficients

The Arrhenius plots were extended to predict the room temperature diffusion coefficients (Table III) for copper and nickel. Diffusion coefficients for copper all fall in the  $10^{-19}$  cm<sup>2</sup>/s range, with the expected accuracy within an order of magnitude. The variations between the ion implant and sputtered film values provide a means of assessment of the consistency and accuracy of the RBS methods and the experimental setups. Although it was not possible to obtain atomic diffusion coefficients of nickel from the ion implants, diffusion coefficients of nickel from the sputtered films are all seen to be in the  $10^{-20}$  cm<sup>2</sup>/s range, consistently an order of magnitude lower than copper's. Previous attempts to measure nickel diffusion have apparently been unsuccessful [11], [18].

#### IV. DISCUSSION

Recent work on the thermal stability of metal/polymer interfaces has been concentrated on the copper/polyimide systems, due to their significance to current MCM technologies. The thermal treatments were restricted to high temperatures (above 250°C) of brief durations (ranging from several minutes to a few hours) [11], [13]. Although it was not reported explicitly, such short temperature treatments were selected to minimize polymer degradation. Hence the contribution of polymer degradation to the overall degradation of the interfaces was not clear. We have found polymer degradation to play a significant role in the stability of these interfaces in these temperature ranges, and these results (which are not central to the current study's objectives) can be found in greater detail elsewhere [16]. One question raised by earlier research results was of the role of oxygen or air in the diffusion process from surface films [11]. It was this issue which drove our first effort to study diffusion from ion-implant sources, i.e., remote from

the surface. The behavior of the implant profiles with time led to the separation of diffusion data for the atomic and clustered species. (Note that the existence of subsurface clustering had been previously directly observed for the surface film case [13], [26], [27].) The next step in the sequence was to relate diffusion data obtained from deposited copper films on the polyimide surface [19] to the implant profile data [8]–[10]. The re-interpretation of thin film diffusion profiles in terms of multiple diffusing species led to the identification of not only the two expected overlapping atomic and cluster diffusion profiles, but also to the discovery of a shallow implant from the initial sputter-deposition process which had to be taken into account. Atomic diffusion data was found in Table I and II to be consistent between the two systems (implants and surface films), but not the cluster results. At the same time, the experiments were extended to include other polymers regarded as having future significance to the electronics packaging field. The agreement between atomic diffusion data from implant and thin film copper sources extended across all the polymers tested, as did the inconsistency in the cluster results.

The purpose of the work reported here was to expand the range of systems studied to include metals of interest to future metal/polymer systems under study for electronics packaging applications, and specifically nickel, chromium and titanium with each of the polymers used earlier. The full range of experiments was performed for both chromium and titanium, but the null result of zero observable diffusion was the expected one. In general terms, the results for nickel parallel those of copper, but with lower diffusivities—in fact an order of magnitude lower at room temperatures.

Despite the error sources discussed above, there is a general trend evident in both Tables I and II in the comparison of cluster diffusion data from the two types of source; with one exception (copper/Kapton) cluster  $D_0$  and  $E_a$  values are larger when determined at the surface (from the thin films) than when determined in the bulk (from the implants). Returning to the original question, it appears that air might indeed have an effect on the diffusivities of clusters, probably by oxidation of the copper clusters at the surface. Indeed, others have concluded that the diffusing clusters are actually oxides [22]. An alternative or supplementary viewpoint could be based on a model of cluster diffusion through polymer voids; it would not be unreasonable to assume different void structures at the polymer surface and in the interior. And similarly one might expect greater differences between the void structures, and hence the cluster diffusivities, of the different polymers, than at the molecular level, and hence of atomic diffusivities. This model is consistent with the results above, and with others' observations of similar atomic diffusivities for different polymers [25].

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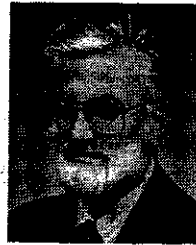
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**John H. Das** received the B.S. degree with highest honors in applied physics and electronics from the University of Dhaka, Bangladesh, in 1979, the M.S.E.E. degree from the South Dakota School of Mines and Technology, Rapid City, in 1983, and the Ph.D. degree from the State University of New York at Binghamton in 1991. His doctoral dissertation was on the stability of metal-polymer interfaces, with work conducted at the State University, the National Nanofabrication Facility and the Materials Science and Engineering Department of Cornell

University.

After graduation, he joined the Electrical Engineering Department and the NNF at Cornell University as a Postdoctoral Associate, working on the development, design, fabrication and testing of low work function silicon field emission devices. Recently his research has expanded to incorporate the field emission devices into suspended silicon microstages and microstructures.



**James E. Morris (S'69-M'70-SM'82)** received the B.Sc. and M.Sc. with first class honors in radio-physics from the University of Auckland, New Zealand, in 1965 and 1967, respectively. He received the Ph.D. in electrical engineering from the University of Saskatchewan, Canada, in 1971, with research in discontinuous thin metal films, a continuing interest.

He is Professor and Chair of Electrical Engineering at the State University of New York at Binghamton. He was the first Director of the University's

Institute for Research in Electronics Packaging. Current research interests also include the electrical properties of conductive adhesives, resonant tunnel diode modeling, thin films, and sensors.

Prof. Morris has served as the Components, Packaging and Manufacturing Technology Society Treasurer since 1991.