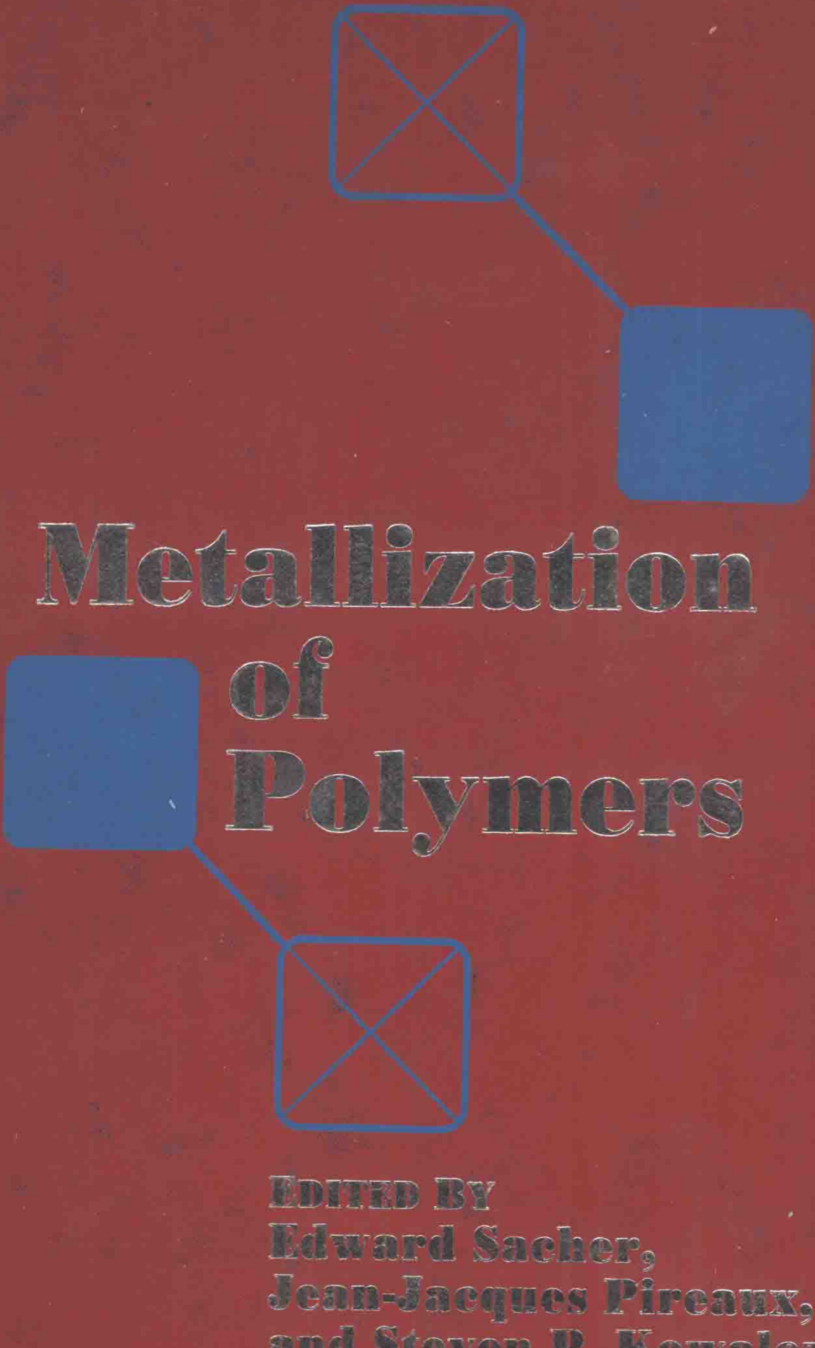

Metallization of Polymers

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ACS Symposium Series 440

ACS SYMPOSIUM SERIES **440**

Metallization of Polymers

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Developed from a symposium sponsored
by the Division of Polymer Chemistry, Inc.,
of the American Chemical Society
at the International Symposium
on the Metallization of Polymers,
Montreal, Quebec, September 24–28, 1989

American Chemical Society, Washington, DC 1990

Library of Congress Cataloging-in-Publication Data

Metallization of polymers / Edward Sacher, editor, Jean-Jacques Pireaux, editor, Steven P. Kowalczyk, editor.
p. cm.—(ACS Symposium Series ; 440).

"Developed from a symposium sponsored by the Division of Polymer Chemistry, Inc., of the American Chemical Society at the International Symposium on the Metallization of Polymers, Montreal, Quebec, September 24–28, 1989."

Includes bibliographic references and index.

ISBN 0–8412–1868–4
1. Metal coating. 2. Polymers—Surfaces. I. Sacher, Edward, 1934– . II. Pireaux, Jean-Jacques, 1950– . III. Kowalczyk, Steven P., 1948– . IV. American Chemical Society. Division of Polymer Chemistry. V. Series

TA491.M46 1990
668.9—dc20 90–47471
CIP

The paper used in this publication meets the minimum requirements of American National Standard for Information Sciences—Permanence of Paper for Printed Library Materials, ANSI Z39.48–1984. ∞

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PRINTED IN THE UNITED STATES OF AMERICA

Preface

THE IDEA FOR A MEETING ON THE METALLIZATION of polymers originated at the American Chemical Society National Meeting held in New Orleans in the summer of 1987. Several attendees recognized the need for a forum to discuss ancillary topics important to polymer chemists but not normally considered at national meetings. One such topic was the adhesion of metallization to polymers, particularly as used in thin film multilayer microelectronic devices. It was known to be relatively easy to get a polymer coating to adhere to a metal substrate, but far more difficult to get a metal coating to adhere to a polymer substrate. Because of the industrial importance of this topic, Dick Ikeda, then chairman of the Division of Polymer Chemistry, Inc., proposed that the division sponsor such a meeting, with Ed Sacher involved in its organization.

To gauge interest in a meeting on the metallization of polymers, some hundred questionnaires were sent worldwide to people involved in this area. The questionnaire gave the scope of the meeting, suggested session topics, and asked for preferred times and locations. Response was surprisingly positive; many respondents offered their help. The meeting was finally set for September 24–28, 1989, in Montreal.

Coeditors Jean-Jacques Pireaux and Steven P. Kowalczyk helped to organize the meeting. From the outset we worked together, sharing ideas and labor alike. Our discussions confirmed in our minds the need for major session topics on:

- analytical techniques—What do we wish to know about the unmetallized surface, the metallization process, and the interface? Which techniques are available? What can they tell us? We felt it important to include discussion of trace element detection techniques because some elements in trace amounts can severely limit the life of some multilayer microelectronic devices.
- surface morphology—What does the polymer surface look like? Is it different from the bulk? If so, why? How is the surface affected by the deposition process? Can the surface be modified to advantage?

- interfacial interactions—Under which circumstances does the metal adhere? Is there any chemical reaction? Can something be learned from model systems?
- adhesion—How does the metallization adhere? How can such adhesion be improved? Can it be quantified?

These contributions and the conference attendees attest to the academic and industrial importance of this subject. Although there were as many attendees from academe as from industry, the contributions even from industry were largely fundamental. This attention to fundamentals is interpreted as both an effort to understand the intricate question of adhesion and an effort to protect proprietary processes.

The chapters in this volume present a concise overview of surface analytical techniques from the specific viewpoint of surface morphology and its modification at the polymer–metal interface. A consistent picture begins to emerge of the chemical reactions occurring on metal deposition and why this leads to metal adhesion. The coeditors hope this information will be timely and useful.

The organizers of the International Symposium on the Metallization of Polymers wish to thank the following industrial sponsors: AKZO, BASF, Exxon, Rhône Poulenc, and Sabena.

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July 27, 1990

Contents

SURFACE ANALYSIS TECHNIQUES

SURFACE MORPHOLOGY AND MODIFICATION

INTERFACIAL INTERACTIONS

ADHESION

INDEXES

Chapter 1

The Present State of Metallized Polyimide

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Due to the incorporation of mechanical stresses during polymer/metal multilayer device fabrication, strong interfacial adhesion must be maintained in order to sustain mechanical integrity. Here we consider the adhesion of metal deposited onto polymer. In order to assure chemical bonding of the sort leading to strong adhesion, one must consider the structure (and modification) of the polymer surface, the energy released by the depositing metal and how it is taken up in reaction, and the aging effect of contaminant ions on the fabricated device.

Polymer/metal multilayer devices are used in the microelectronics industry. These devices are composed of alternating layers of polymer and metal, the metal is etched into lines and, except where via holes permit the contact of different metal layers, the polymer serves as an insulator. Because the polymer must withstand rather hostile environments during fabrication, the choice is narrowed to those which are stable to chemical treatment, high temperature (for short periods of time) and humidity. The polymers of choice here are the polyimides, although others are certainly used.

Each of the components, polymer and metal, has both thermal and hygroscopic expansion coefficients which may differ between components by orders of magnitude. Thus, the very act of fabrication introduces internal stresses into the device. If permitted to act, these stresses are capable of ripping apart the device, destroying it Such destruction is prevented by strong interfacial adhesion, maintained at every step during manufacture, as well as throughout the use life of the device.

POLYMER ADHESION TO METAL

Now, the adhesion of polymer to metal presents no difficulty: poor adhesion of the polymer to the bare metal substrate is improved through the use of silane adhesion promotors(1-7). These are

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orthoesters of silane, having the general formula Z-R-Si(OR')$_3$, where Z is a functional group specifically chosen to react with the polymer, and R and R' are alkyl groups. When added to water, the orthoester hydrolyzes (sometimes slowly) to the silanetriol, -Si(OH)$_3$, whose hydroxyl groups are extremely reactive. These groups react with each other as well as with hydroxyl groups on clean metal surfaces, to deposit a thin polysiloxane layer strongly bound to the metal. Among the groups lying at the new surface are the Z groups capable of reacting with the polymer to chemically bond it to the polysiloxane. This chemical bonding is accomplished with a minimal increase in thickness (~100Å), through the introduction of a surface-reactive polysiloxane rubber layer capable of dissipating some of the internal stresses.

Sadly, such adhesion promotors are not available for the adhesion of metal being deposited onto a polymer surface. Here, one must rely entirely on reactions provoked by the deposition process to provide the adhesion. This requires a thorough understanding of both the polymer surface and the deposition process.

POLYMER SURFACE STRUCTURE

There is an excess energy at a surface, measurable as a surface tension(8). This causes the surface layer of a curing polymer to react farther and faster than the bulk. In the case of polyimide, it also promotes some surface layer structural rearrangement (~8%) to isoimide(9), causing changes in both mechanical(10) and electrical(11) properties. Further, a recent IR study(12) demonstrated that the polyimide structure obtained on curing depended on the cure schedule.

Indeed, the whole question of polyimide structure has yet to be properly addressed. As normally written, the precursor polyamic acid cyclizes to a 5-member polyimide ring, splitting out water; however, objections(13) have been raised to this cyclization. In particular, reactions carried out on monomers show(14-16) that imide formation requires prolonged heating in the presence of dehydrating agents and that such imides revert quickly to the amic acid reactant in the presence of atmospheric moisture. This makes it highly unlikely that a polyimide film formed through the cyclization of the polyamic acid would be as stable as such films are found to be. Yet, IR evidence(17) leaves no doubt that the polyamic acid has imidized.

Several lines of investigation have been brought together (13) to suggest that what in fact happens to the polyamic acid is that it transimidizes. That is, imidization takes place between adjacent chains. This explains many facts, such as the loss of solubility on imidization, the compatibility of the data with second order kinetics and the lack of XPS data indicating increased electron delocalization on imide formation. It would appear that the polymer structure is largely a crosslinked transimide and that, at its surface, it contains ~8% isoimide. This is the structure upon which the metal deposits.

Burkstrand has shown(18-25) that evaporated metals react only with oxidizing polymers. In the case of polystyrene, for example, reaction takes place only at surface sites oxidized during polymer fabrication (e.g., the melt extrusion of polymer films). Thus, to assure good adhesion to hydrocarbon polymers, the surface must be treated in such a way as to introduce oxidizing groups. While several types of surface oxidation treatment are available, the use of plasmas seems to be the treatment of choice(26-34).

METAL DEPOSITION

Because the adhesion of a deposited metal depends on the formation of chemical bonds across the interface, one must determine if the metal deposition process is capable of releasing the energy necessary to cause reaction between the depositing metal atoms and the polymer surface. For metal deposition by sputtering, the answer is an unqualified yes: for both Cu (35,36) and Ni(35), for example, there is an impact energy maximum near 950 kJ/mole and an average energy near 3500 kJ/mole. These values should be compared with covalent bond energies, which are in the 340-400 kJ/mole range.

The situation for metal deposition by evaporation is somewhat different: the average impact energies of such atoms are of the order of 10 kJ/mole(37). However, at least for the metals one would normally use, their condensation energies, the negative of the heat of sublimation(38), are of the same order of magnitude as covalent bond energies. One should note that this heat of condensation is for metal condensing on metal. That is, after several monolayers are deposited, assuming no reaction of the initial monolayers, enough heat may be released to break covalent bonds.

One should more properly consider the condensation energy of the first monolayer of metal on polymer. No experimental values exist, making it necessary to infer reaction from other data. Here, there is a great divergence of opinion, not only as to whether reaction occurs but, if so, where the structure is attacked. A particular example of this is found in the deposition of Cr onto polyimide, where all agree that reaction occurs: one group maintains that the Cr forms a π-complex with the aromatic ring of the dianhydride moiety(39,40) while another insists that reaction occurs with the carbonyl oxygens(41,42).

Let us, for a moment, consider the reaction of evaporated metal atoms with monomers. While here, too, there appear to be no data on condensation energies, there is a large body of experimental data(43-47) which demonstrate compound formation under conditions less apt to sustain chemical reactions than those during polymer metallization. This strongly suggests that, with the exception of purely aliphatic, non-oxidizing substrates, reaction probably does take place. To demonstrate this, let us consider several recent XPS studies of metallized polyimide; these metals include Al(48-50), Au(51), Ce(52), Co(53), Cr(42), Ge(54) and Ti(55). Whether the authors posit reaction between metal and polymer or not, the metal XPS spectra at very low coverages all show either a chemical shift to higher binding energy or a shoulder at the higher binding energy side of the peak, neither of which is seen at greater depositions. Such behavior indicates the formation of ionic species. Clearly, then, the question is not whether reaction occurs but where it occurs and how this conveys adhesion.

IONIC CONTAMINATION

The question of trace ionic contaminants is often overlooked. There is no doubt, however, that trace ions initially present in the polymer or subsequently introduced during the manufacturing process can wreak havoc with the device as well as with device-mounted components as the device ages and the ions migrate. Thus, not only can Na ions migrate to and destroy an FET by lowering the threshold resistance in the gate, halide ions can attack some metals, which may lead to a loss of interfacial adhesion and will certainly lead to a loss of conduction.

While it is true that, with the exception of the proton, the trace ions found in polymers do not migrate in the absence of water(11,56-58), it is also true that microelectronic devices and components are not hermetically sealed: they are always exposed to atmospheric humidity. It is, therefore, necessary to know, both qualitatively and quantitatively, which trace ions are present. This writer has had experience(59) with four techniques which are capable of identifying contaminants in the ppm-ppb (i.e. ug/ml-ng/ml) concentration region. Each has its own advantages and disadvantages, necessitating the simultaneous use of several of the techniques found in Table I. A particular advantage of ion chromatography, for example, is its sensitivity to some organic ions. It was in this fashion that polyimide was shown to undergo a slow surface hydrolysis(59): ion chromatography showed the presence of both carboxylate and substituted ammonium ions, the hydrolysis products of polyimide, whose concentrations increased with water immersion time. It should be noted that these carboxylate ions, like halide ions, attack some metals, and could ultimately lead to a loss of adhesion between metal and polymer or to the formation of a brittle insulating layer. Internal stresses could then destroy the device.

Table I. Techniques Capable of Detecting Trace Contaminants in the ppm-ppb Concentration Range

	Ion Chromatography	Plasma Spectroscopy	Neutron Activation Analysis	Proton-Induced Xray Emission
Agueous solutions	Yes	Yes	Yes	Yes
Solid	No	Yes (slurry)	Yes	Yes
Species detected	Ions	Ions, atoms (no distinction)	Ions, atoms (no distinction)	Ions, atoms (no distinction)
Advantages	Fast; Sensitive to some organic species	Fast	Large range of elements	Fast; Large range of elements
Disadvantages	Limited number of ions	Cost per element detector	Slow; Poor deconvolution	Not sensitive to high atomic

ADHESION MECHANISM(S)

Here, the question of how the metal adheres to the substrate remains largely unanswered. A review on this subject(60), now more than a decade old, could do no more than list the then extant proposed mechanisms (mechanical/interlocking, weak boundary layer, chemical, electrostatic) and offer experimental evidence which tended to support each of them. The general conclusion was that one or more of these mechanisms gave rise to the observed adhesion.

Little has changed during the intervening years, with the exception that the chemical mechanism is presently favored (25,48-55,61-74), the reason being that adhesion is clearly observed in association with the occurrence of chemical reaction. This does not answer the question of how chemical reaction leads to adhesion or why exposure to atmospheric humidity is so detrimental(73).

CONCLUSIONS

The interface of metallized polymers has been considered from the points of view of the polymer surface, reaction during metal deposition and the effect of contaminant ions. Each is discussed in terms of the critical factors which maintain the mechanical integrity of polymer/metal multilayer devices, in light of the present view that interfacial chemical reactions are involved. Thus, the true structure of the polyimide surface is described, followed by a discussion of why metal deposition provokes a chemical reaction with the substrate polymer. Trace ions are then considered, from the point of view that subsequent reaction with polymer and/or metal can lead to the failure of interfacial adhesion.

ACKNOWLEDGMENTS

The writer wishes to thank the Natural Sciences and Engineering Research Council of Canada and the Fonds pour la Formation de Chercheurs et l'Aide à la Recherche du Québec for financial support.

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RECEIVED May 16, 1990