

The Influence of Thermal Cycling and Compressive Force on the Resistance of Poly(3,4-ethylenedioxythiophene)/Poly(4-styrenesulfonic acid)-Coated Surfaces

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ABSTRACT: There is an increasing interest in the use of conducting polymers for a wide variety of applications. This includes the study and development of alternative contact-connector materials. The main aim is to achieve overall improvements in performance as well as cost effectiveness. Currently, extrinsic conducting polymers (ECPs) are employed as conductive coats or adhesives at contact interfaces. However, frictional abrasion within the metal-doped polymer (ECP) causes instability in the resistance. It is important to overcome this fretting effect, especially in automotive applications; hence, the possibilities of employing intrinsically conducting polymers (ICPs) are explored. Flat contact film coatings have been fabricated in-house using poly(3,4-ethylenedioxythiophene)/poly(4-styrenesulfonic

acid) (PEDOT/PSS) with dimethylformamide as the secondary solvent. Resistance is measured using the four-wire method. The conductivities of the PEDOT/PSS-coated contacts are found to be in the order of $10^{-2} \text{ S cm}^{-1}$. The change of resistance under varying compression forces has been found to be repeatable. The thermal effects on these contacts are also studied and the results are depicted as exponential negative temperature coefficients of resistance. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 101: 2445–2452, 2006

Key words: fretting; intrinsically conducting polymer; conductivity; contact connector; temperature coefficients of resistance

INTRODUCTION

All carbon-based polymers were regarded as insulators approximately three decades ago. Since then, this narrow perspective has rapidly changed, as a new class of polymer known as intrinsically conducting polymer (ICP) or electroactive polymer emerged. The potential applications of these ICPs, although currently considered in its infancy, are relatively significant.¹ One such application would be in the automotive industry, where the possibility of replacing metal terminals with conducting polymer contacts could be realized.

Polymer and polymer derivatives, when doped with a weak oxidation or reducing agent can result in a conjugated polymer. The development of conjugated polymers has led to an understanding of charge storage and charge transfer mechanisms.² This polymer morphology governs the conductivity levels of the material. For example, polyphenylene and polythiophene have shown electrical conductivities of $\sim 0.1 \text{ S}$

cm^{-1} .¹ The value is considerably low as compared with that of conventional metals such as copper, which has the conductivity of $0.596 \times 10^6 \text{ S cm}^{-1}$.³ However, there are initial indications that the electrical conducting capability can be increased further for such polymers. The major problems with conducting polymers are the processability and the stability. The development of highly conducting polymers ($>10^2 \text{ S cm}^{-1}$) with a good stability and an acceptable processability has been the focus of recent studies.^{4,5}

Conducting polymers doped with metal particles used for electrical contact applications are known as extrinsic conducting polymer (ECP) contacts; these are currently used in a range of electronic and electrical systems. The advantages include low cost, easy fabrication methods, and good thermal and mechanical properties. Employing ECPs as alternative contact materials reduces the use of precious metals and could improve reliability by reducing fretting.^{6,7} The application of ECP contacts in automotive systems, although reducing fretting, does not eliminate it. The presence of doped metal particles may cause some degrees of abrasion or fretting at the particle–surface interfaces, when subjected to vibration and temperature, normally encountered in the automotive environment. This leads to possible deformation of the contact and variations in resistance, hence affecting the reliability of the overall system.⁷ To overcome this

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drawback of ECPs for this application, ICPs possessing the elasticity capability of absorbing external forces acting upon the contact interface are exploited. The ultimate aim is to develop contact connectors using ICP, which would further reduce or eliminate the influences of fretting and still have good electrical conducting properties.

Currently, there are a number of available ICPs such as polyaniline, polypyrrole, and polythiophenes for various applications.⁸⁻¹⁰ This article presents investigations on the ICP material poly(3,4-ethylenedioxythiophene)/poly(4-styrenesulfonic acid) (PEDOT/PSS) with different doping levels of dimethylformamide (DMF) as the secondary solvent. DMF is a popular organic polar solvent having a wide range of applications, and its secondary solvent effect arises from a further stabilization of the complex compound by long-range polarization interactions with solvent molecules. Although this technique of enhancing ICP conductivity levels has previously been studied by other researchers,^{11,12} the work presented in this article adopts and applies this methodology to further investigate the various properties directly related to the design and development as well as the reliability of electrical contacts, such as the mechanical structure, processability, and conductivity. This study is also the continuation of the initial investigations made on PEDOT/PSS with DMF,¹³ where different ICP contact designs have been developed and analyzed.

In this article, the extension of the investigation addresses a number of improvements made on the ICP prototype structure and a study of thermal behavior of the ICP. The resistances of PEDOT/PSS- and PEDOT/PSS : DMF-coated surfaces are measured using the four-wire technique. They are subjected to two modules of experiments which independently study the influence of temperature and varying compressing forces in the normal direction. In addition, physical changes of the ICPs are observed using 3D surface-scanning techniques to measure surface changes. This ICP material is chosen for the study, mainly because of its easy processing capability and its level of conductivity, which presents the potential of enhancement by secondary doping.

INTRINSICALLY CONDUCTING POLYMERS

Intrinsically conducting polymers (ICPs) belong to a class of organic materials with unique electronic properties such as electric conductivity, electrochromism, and electroactivity.¹⁴ Conjugated electrons in the backbone of the macromolecules are accountable for these properties. ICPs have a number of potential applications in electronics, microelectronics, and optoelectronics applications, especially for the aerospace and automobile industries.

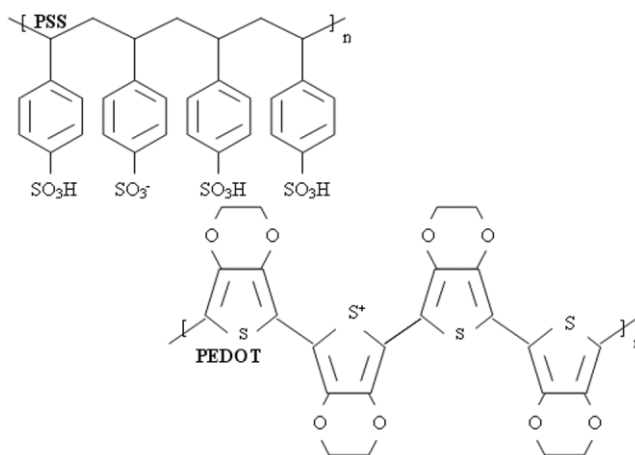


Figure 1 Chemical structure of PEDOT/PSS.

PEDOT is a relatively new member in the conducting-polymer family. It displays interesting properties, including relatively good electrochemical, ambient, and thermal stability of its electrical properties as compared with that of other polythiophenes.¹⁰ Pristine PEDOT is insoluble in many common solvents and unstable in its neutral state, as it oxidizes rapidly in air. To improve its processability, a polyelectrolyte solution (PSS) can be added, and this results in an aqueous dispersion of PEDOT/PSS, where PEDOT is in its oxidized state.

PEDOT/PSS (structure as shown in Fig. 1) was synthesized in the 1980s and had since been one of the most successful conjugated-polymers for commercial applications. Its advantages include high stability in its p-doped state, relatively high conductivity, and good film-forming properties.¹⁵ The polymer chain in the PEDOT/PSS dispersion is usually of a random coil conformation. When deposited onto a substrate, a thin coat of film is formed with grains of doped conjugated-polymer coils.^{16,17}

Continuous studies have been carried out to improve the conductivity levels of conducting polymers via secondary doping.¹⁸ Ghosh and Inganas¹⁹ blended PEDOT/PSS with polyethylene oxide (PEO). The overall conductivity levels increased primarily because of the contribution from the intrinsic ionic conductivity of PEO. In addition, PEO swelled in a liquid electrolyte and this created space for ionic movement. Another successful method was by doping the PEDOT/PSS dispersion with a mixture of sorbitol, *N*-methylpyrrolidone, and isopropanol.²⁰ The electronic, chemical, and morphological changes induced by the solvents were then investigated by Jönsson et al.¹⁸ A substantial amount of work has also been invested in PEDOT/PSS blend with dimethyl sulfoxide²¹ and DMF,¹³ where conductivity levels can be increased by a factor of up to 100. This phenomenon is mainly due to the screening effect of the polar solvent. By defini-

tion, the screening effect is a reduction in effective nuclear charge on an electron that is caused by the repulsive forces of other electrons between it and the nucleus. In this case, polar solvents with higher dielectric constant induce the stronger screening effect between counterions and charge carriers. This effectively reduces the Coulomb interaction between positively charged PEDOT and negatively charged PSS dopants, thus enhancing the hopping rate and conductivity in the PEDOT/PSS systems. The screening effect due to the polar solvent between the dopant and the polymer main chain plays an important role for charge transport properties such as conductivity and its temperature dependence.²¹

Conduction principle

To understand the conductivity of polymers, the way in which charges are stored along the polymer chain is important. The polymer usually stores charges in two ways. During the oxidation process, it could either lose an electron from one of the bands or it could localize the charge over a small section of the chain. A local distortion—an effective change in geometry—occurs when the charge is localized and this reduces the overall energy of the polymer. The generation of the local geometry decreases the ionization energy of the polymer chain and increases its electron affinity, hence increasing its ability to accommodate newly formed charges. A similar situation is experienced in the reduction process.

In typical conducting polymers, the combination of a charge site and a free radical is known as a polaron. When subjected to further oxidation, the free radical of the polaron is removed, creating a new spinless defect called a bipolaron. For a very heavily doped polymer, the upper and the lower bipolaron bands will merge with the conduction and the valence bands respectively, to produce partially filled bands and metallike conductivity. Charge transfer between the conducting domains takes place by thermally activated hopping or tunnelling.²¹

Polymer stability

Extrinsic stability is reflected by the vulnerability towards external environmental factors such as oxygen, water, and peroxides. This is determined by the polymer's susceptibility of charged sites to attacks by nucleophiles, electrophiles, and free radicals. A protective layer can be applied to minimize this effect. It is common that many conducting polymers degrade over time, even in dry and oxygen-free environments. This leads to intrinsic instability and the likely cause is attributed to the irreversible chemical reaction between charged sites of polymer and either the dopant counterion or the p-system of an adjacent neutral

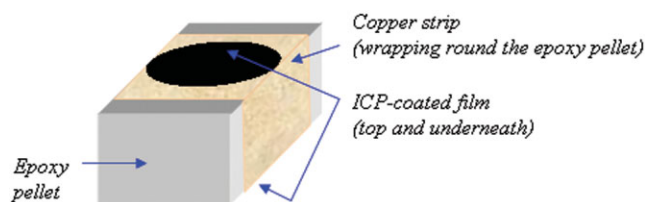


Figure 2 ICP-coated contact pellet structure. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

chain, which produces an sp^3 carbon by breaking the conjugation. In other words, the charge sites become unstable, because of conformational changes in the polymer backbone.

The stability of various conducting polymers is accentuated by the effect of adverse temperature values and thermal variations.²² For the intended purpose of the automotive application, the operational temperature of the ICPs is kept within the range of 5–90°C, based on the thermal shock and thermal cycling procedures of the USCAR standards.^{23,24} In this study, the stability of the ICPs is monitored accordingly and ascertained by performing repeated measurements. The type of unblended ICP films employed in the investigations have shown to have thermal stability of up to 280°C²⁵ and this material has also been used in other applications.^{26,27}

EXPERIMENTAL

A commercially available form of PEDOT/PSS, known as BAYTRON P[®] from Bayer AG²⁸ (Leverkusen, Germany) has been used. The weight ratio of PEDOT to PSS is 1 : 1.6, with the mixture diluted in H₂O. BAYTRON P[®] is easily manipulated in terms of fabricating conductive coats and films. In this study, the main issue focuses on the influence of adding different weight ratios of DMF into PEDOT/PSS in order to increase the conductivity of the overall polymer, based on the in-house fabrication methods used to generate ICP contact surfaces. Figure 2 illustrates the pellet structure on which a layer of ICP has been coated. This type of pellet is the first attempt as a viable ICP connector contact prototype. The original PEDOT/PSS solution and four different ICP blends, namely PEDOT/PSS : DMF with weight ratios of 5 : 1, 4 : 1, 3 : 1, and 2 : 1 are studied in the experiments. ICP blends are coated by “dripping” the ICP solutions onto the individual cleaned copper strip surfaces and dried in ambient temperature conditions for 24 h, resulting in an approximate ICP coat thickness of 200 μm .

To carry out four-wire resistance measurements, the ICP-coated contact pellet is “sandwiched” across two copper terminals that are attached to a Keithley 2000

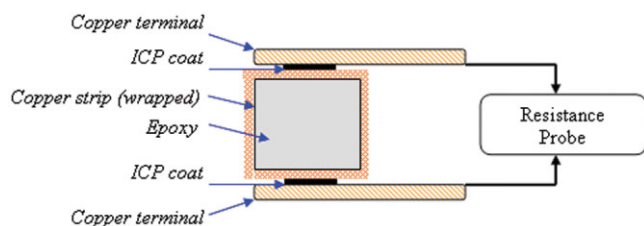


Figure 3 Cross-sectional view of experimental assembly. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

multimeter (resistance probe), as shown by the cross-sectional view in Figure 3 (not drawn to scale).

Investigation of contact force

The experimental assembly for the samples with different ICP blends (as shown in Fig. 3) is placed between the clamping devices as seen in Figure 4. A force sensor (model: FSG15N1A from Honeywell Micro Switch Sensing and Control) is placed in position to record the force exerted by the clamp.

The adjustable screw of the clamping device is set to exert a fixed amount of force across the sandwiched configuration. For the force experiment, the resistance of the sample is measured for the range 7–10.5 N. Lower forces are not investigated here, since it is noted that below 7 N, the resistance increases significantly to the mega-ohm range, suggesting poor or no contact.

Thermal effects

With reference to Figure 3, the copper terminals are pressed across the ICP-coated pellet with an applied force of 8 N. The whole assembly is placed and tested in a temperature chamber (Climatic testing cabinets, series SB, System Weiss), where the temperature profiles could be programmed. Measurements are acquired by a data-logging system consisting of a computer using RS-232 data acquisition and the Keithley

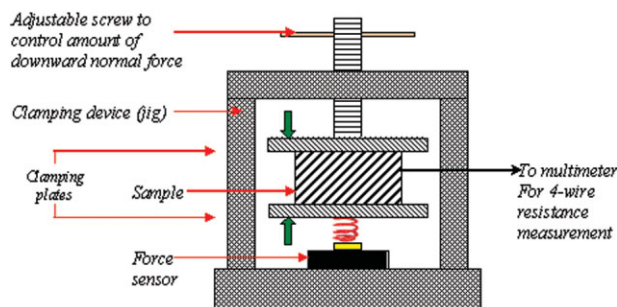


Figure 4 Clamping setup for experiments using force exertion. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

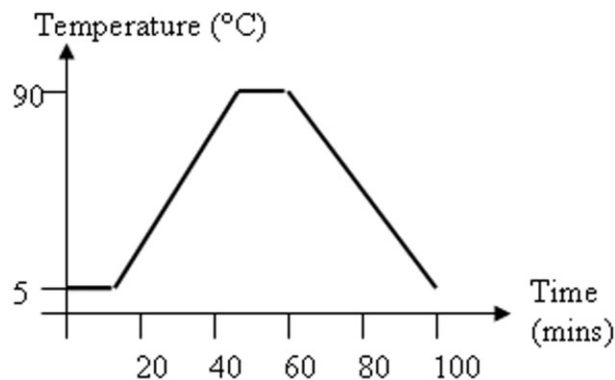


Figure 5 Temperature profile for climate chamber.

multimeter. This acquisition system accommodates five sets of analogue input channels that allow four-wire resistance signals to be sampled simultaneously. A LABVIEW software program has been written to automate the procedure.

The samples are subjected to the temperature profile as depicted in Figure 5. The profile is adopted from the USCAR standards²³ and has been previously used to study the environmental influences on commercially available automotive connectors.²⁴ Three continuous cycles are carried out to determine the repeatability of the measurements. The temperature profile is monitored using a PTFE-insulated K-type thermocouple.

RESULTS AND DISCUSSION

Contact force and relative conductivity

Experiment 1. The change of relative conductivity with ICP blend

The samples are placed in the clamp as shown in Figure 4, with an applied force of 8 N at room temperature. Using the four-wire resistance measurement probe, the averaged resistance of the unblended PEDOT/PSS-coated pellet is measured to give 5.75 k Ω . On application of the force, the assembly takes \sim 1 min for the resistance to stabilize, after which the resistance remains stable; the averaged resistance taken over a period of 3 min (60 readings) is then recorded. This measurement is repeated for the four blends. The averaged resistances are summarized in Table I.

TABLE I
Averaged Resistances (%) of Various ICP Blends

PEDOT/PSS : DMF ratios	Resistances (k Ω)
5 : 1	4.25 \pm 4.71%
4 : 1	3.14 \pm 6.34%
3 : 1	0.74 \pm 6.94%
2 : 1	17.00 \pm 6.94%
Unblended	5.75 \pm 8.82%

To determine a relative conductivity for the various ICP blends, the followings assumptions are made. Firstly, it is assumed that all the copper components in the path have negligible resistance (see Fig. 3). It is also assumed that the contact resistance between the copper support and the ICP is negligible. It is further assumed that the interface resistance between the ICP and the wrapped copper strip around the epoxy is negligible. The relative conductivity is then simply

$$\sigma_{\text{rel}} = \frac{l}{RA} \quad (1)$$

where l is the combined thickness of the two ICP layers (~ 0.4 mm), A is the apparent surface area of ICP (~ 0.1 mm²), and R is the measured resistance (given in Table I).

The apparent surface area is determined using the surface profile of each tested ICP film, where an indication of the contact area can be observed as shown by an example in Figure 6. The noncontact scanning technique carried out by the TaiCaan Xyris system has accompanying software which is able to calculate the specified area in the plot. For each surface measured, the corresponding σ_{rel} of each sample can be evaluated as summarized in Figure 7. Future studies will seek to determine the actual conductivity (σ) by taking all the assumptions into consideration and how this value would relate to σ_{rel} .

From Figure 7, the results agree with the findings from previous investigation¹³ where different structures have been used that the PEDOT/PSS : DMF mixture at 3 : 1 gives the optimum relative conductivity value of $\sim 5.41 \times 10^{-2}$ S cm⁻¹. As the amount of DMF increases beyond the 3 : 1 optimum ratio, a sudden sharp decrease in relative conductivity is observed as seen in the graph for the mixture at 2 : 1. One possible reason could be due to the saturation of the derivatives, where PEDOT/PSS no longer dominates the

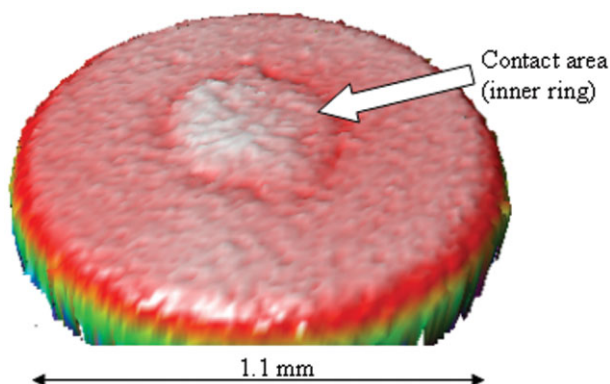


Figure 6 Surface profile of contact area. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

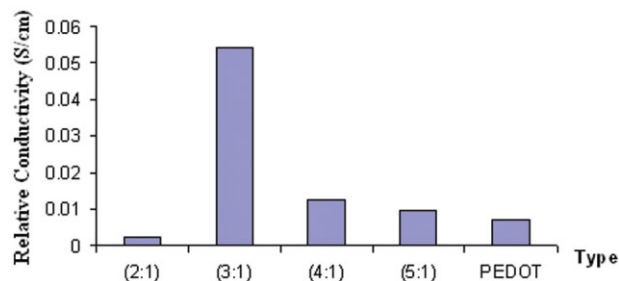


Figure 7 Relative conductivity levels for different ICP blends. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

electrical characteristics of the overall conducting polymer. This would subsequently lead to polymer degradation. Furthermore, at high DMF doping levels of 33% or greater, relaxation in the material may take place, which in turn could affect the energy band gap within the polymer, resulting in lower conductivity levels. The trend relating the performance of various ICP blends, which is the key focus of this initial investigation, has been presented. To verify the chemical formulation of the optimum blend, intermediary levels of doping between 3 : 1 and 2 : 1 will be thoroughly analyzed in future studies.

It is also observed that the differential increase in relative conductivity from the unblended ICP to the 4 : 1 mixture is relatively smaller compared with the exponential rise in conductivity experienced by the 3 : 1 mixture. The exact reason is not entirely known; hence it will not be further discussed in this article. Nonetheless, further work will be carried out to study this phenomenon.

Experiment 2. The ICP resistance with variation of applied force

At a constant force, the resistance measurements are stable; however, as the force is varied, the resistance show an instability, where a time (~ 1 min) should be allowed for the resistance value to stabilize, as discussed above. This partially results from the variation in compliance of the clamping device.

The poly(3,4-ethylenedioxythiophene)/poly(styrene-sulfonate) samples are subjected to an increasing force from 7–10.5 N. The resistance values are shown in Figure 8.

Figure 8 shows relative resistance stability over the selected range of applied force, with the average resistance shown to decrease slightly at a gradual rate. The higher variation in resistance for the 2 : 1 ratio may be the result of the constitution of the material. Although not included in the graph, the relationship between resistance and decreasing applied force is found to follow similar trend as compared with that for the increasing force, despite having some hystere-

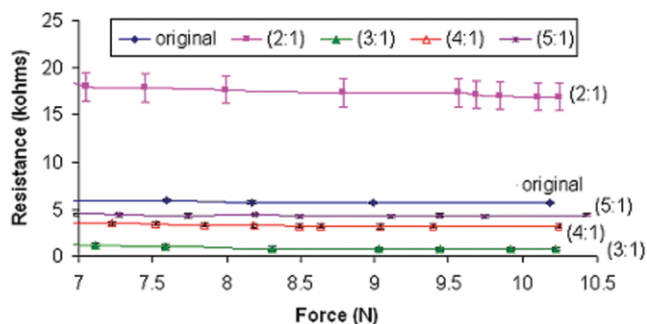


Figure 8 ICP resistances against applied force for selected blends. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

ses (similar to those observed from the results obtained in the The influence of thermal effects on the ICP resistance section; Fig. 9). The resistance measured at the minimum applied force of 7 N at the start of the test subjected to an increasing force is the same as that at the end of test employing a decreasing force and this indicates reversibility in the material.

The measured resistance can be collectively represented by the sum of the bulk resistance, the film resistance at the interface, and the constriction resistance. The

contact pellet has a complex structure; hence it is difficult to study each of these resistances independently. Nonetheless, the approximated constriction resistance (R_c) is found to be relatively small at 0.927Ω by using Holm's formula,²⁹ as given in eq. (2).

$$R_c = \frac{\rho}{2a} \quad (2)$$

where ρ is the resistivity of the ICP at $0.033 \Omega \text{ cm}^{11}$ and a is the contact radius of 0.0178 cm . Therefore, it can be considered negligible in the analysis of the results.

The influence of thermal effects on the ICP resistance

For this investigation, the resistance is measured at 45-s intervals over the cycling period shown in Figure 5. All experiments are conducted at a constant force of 8 N, which is also typically used for the ECP-contact system. Figure 9(a–e) show the changes of resistances with temperature for the respective PEDOT/PSS:DMF-coated surfaces, over a single temperature cycle.

The primary observation is that the ICP-coated pellets have a negative temperature coefficient of resis-

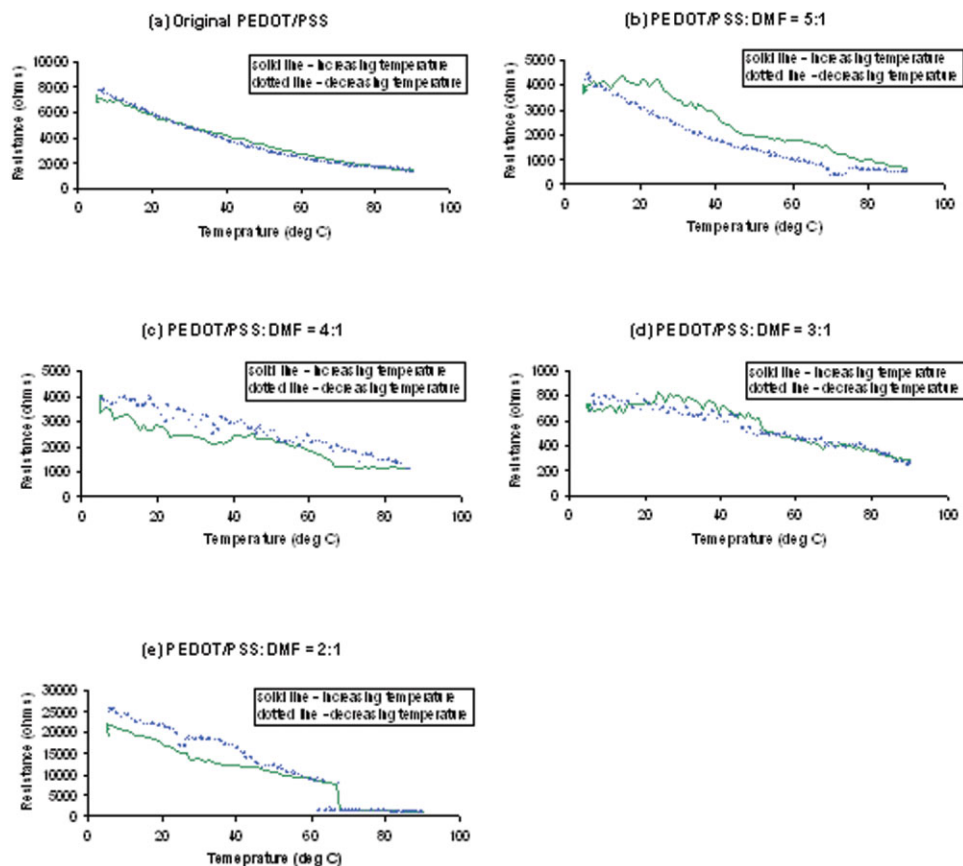


Figure 9 Effects of temperature on resistances. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

tance. With a constant force, the length and contact area has been assumed to remain unchanged within the operational temperature of 5–90°C. This implies that the relative conductivity levels increase with temperature.

At this point, it is also important to note that the epoxy employed in the pellet structure has not been affected by the thermal effects. Changes in the physical structure of the contact pellet do not occur as the operating temperatures of the epoxy employed are between –50°C and +250°C. Therefore, it would be appropriate to assume that variations in resistance measured would be mainly attributed to the change in relative conductivity levels of the ICP blends.

A theory proposes that the influence of temperature on the conductivity is a result of increased tunnelling transport through the insulating barriers between grains.³⁰ It also describes the charge transport in systems with a phase segregation of polymer colloidal suspensions or blends resulting from film coating processes, which results in exponential temperature dependence for conductivity as shown in eq. (3)

$$\sigma \approx \sigma_0 e^{\left(\frac{T_1}{-T_2+T}\right)} \quad (3)$$

where σ_0 is the initial conductivity and T_1 and T_2 are temperatures depending on the energy barrier and width of the insulating layer.

With suitable known values for the ICP blends used in this study, the theoretical model [eq. (3)] corresponding to an exponential response can easily represent and fit to the experimental results obtained for the temperature range 5–90°C (refer to Fig. 9). In addition, the experimental results also agree with the general trend and thermal characteristics of other PEDOT/PSS blends investigated in a previous study,³¹ which focuses on the annealing processes of ICP films as well as the influence of moisture and temperature on conductivity.

The results in Figure 9 show a typical hysteresis cycle with blend. For the unblended ICP, there are little changes across the temperature cycle, where as for the four blended ICPs, the resistance–temperature cycle varies with the blend. The thermal hystereses are evident in the samples with presence of DMF levels in the polymer blend and this could suggest possible thermal instability of the PEDOT/PSS : DMF material. Another interesting point emerges with the sample at weight ratio of 2 : 1 as shown in Figure 9(e). Within the range of 62–68°C, the resistance appears to encounter an abrupt decrease of an estimated factor up to 7 times. In this case, the material may have undergone possible changes in the charge transport mechanism that resulted in the relatively higher conductivity at elevated temperatures. This sample will be further investigated, as it displays characteristics which do not comply with that of the other ICP blends, although this response is repeatable.

CONCLUSIONS

In this article, the results have shown that the ICP material PEDOT/PSS with different doping levels of DMF as the secondary solvent indicates the possibility of conductivity enhancement. This improvement is determined by the intrinsic conductivity levels of the materials as well as the process and fabrication techniques of the film layers.

The investigations involving the influences of compressing forces revealed that the averaged resistances for all samples are decreasing slightly at a gradual rate as the compressing force changed from 7 to 10.5 N. With forces beyond 10.5 N, the resistances appear to remain relative unchanged as long as the materials have not been physically damaged. With excessively large amount of forces exerted across the pellet structure, the ICP surfaces would eventually suffer cracks under further stress. By regressing the collected data, it is noted that the change in resistance is larger for the polymer blend with a higher level of DMF. This could indicate the possibility of having a more elastic material or a material that could be experiencing a higher degree of deformation or instability with the presence of DMF.

At a fixed, constant compressing force, the increasing amount of DMF in the polymer blend gives rise to reducing levels of the resistances. It is found that the PEDOT/PSS : DMF mixture at 3 : 1 provides the optimum relative conductivity value of $\sim 5.41 \times 10^{-2} \text{ S cm}^{-1}$. When the amount of DMF is increased beyond this ratio, for example in the mixture of 2 : 1, a sudden rapid decrease in relative conductivity is observed.

The results for thermal influence have shown that the ICP materials studied in this context have exponential negative thermal coefficients of temperature. This phenomenon is supported by previous literature, attributing to the explanation of charge transport mechanism. Furthermore, the samples subjected to several cycles of thermal shocks have produced repeatable measurement. Thus, acceptable levels of thermal fretting experienced by the ICP contacts due to rapid changes in temperature have been observed for this brief, short term evaluation.

The study presented in this article has shown the thermal characteristics and reliability issues related to the application of ICP materials toward the development of electrical contact, especially for automotive purposes. The ultimate aim of developing such contact connectors would be ideally to minimize or eliminate the effects of fretting, with the elasticity property of ICP. To achieve this novel development, much future work such as improving the ICP contact structure as well as performing accelerated tests involving forced fretting and long-term thermal fretting procedures would be required. Currently, the conductivity enhancement of ICP via secondary doping and process techniques is actively being addressed.

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