

Electrical properties of inorganic nanowire–polymer composites†

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Composites of nanowires of ZnO, RuO₂ and Ag with polyaniline (PANI) as well as polypyrrole (PPy) have been prepared, for the first time, by an *in-situ* process, in order to investigate their electrical properties. Characterization by electron microscopy and IR spectroscopy indicates that there is considerable interaction between the oxide nanowires and the polymer. The room-temperature resistivity of the composites prepared *in-situ* varies in the 0.01–400 Ω cm range depending on the composition. While the resistivities of the PANI–ZnONW and PPy–ZnONW composites prepared by the *in-situ* process are generally higher than that of PANI/PPy, those of PANI–RuO₂NW and PANI–AgNW are lower. Composites of ZnONW with polyaniline prepared by an *ex-situ* process exhibit a resistivity close to that of polyaniline.

Introduction

There has been a surge of interest in nanostructured materials as part of the effort towards device miniaturization and understanding of novel quantum phenomena at the nanometer scale. Some of the properties have been exploited by incorporating the nanomaterials into some form of matrix such as polymers. Following the first report of the preparation of carbon nanotube–polymer composites by Ajayan *et al.*,¹ there have been efforts to combine carbon nanotubes and polymers to produce functional composite materials with desirable electrical and mechanical properties.^{2–5} These composites can have potential applications in photovoltaic devices and electrostatic as well as conductive coatings in optical devices. Carbon nanotubes have been incorporated in insulating as well as conducting polymers such as polyaniline (PANI) and polypyrrole (PPy). PANI and PPy are attractive polymers for preparing such composites in view of their low-cost, environmental stability and easy processability. Aniline has been polymerized on multi-walled carbon nanotube (MWNTs) electrodes by Downs *et al.*⁶ to obtain PANI films with novel surface characteristics and high current densities. Composites of PANI with MWNTs, with an order of magnitude lower resistivity than that of pure PANI at room temperature, have been prepared by Cochet *et al.*⁷ by *in-situ* polymerization. Blanchet *et al.*⁸ have shown that the high aspect ratio of the single-walled carbon nanotubes (SWNTs) enables percolation into the PANI network at low concentrations and that the PANI–SWNT composites can be used as printable conductors for organic electronics devices. Zengin *et al.*⁹ prepared films of PANI–MWNT composite by *in-situ* as well as *ex-situ* methods

with electrical conductivities higher than that of the pristine nanotubes. Composites of PANI with pristine MWNTs and SWNTs as well as nanotubes subjected to acid treatment and subsequent treatment with thionyl chloride were prepared by Vivekchand *et al.*¹⁰ The resistivity of these composites was generally between that of the nanotubes and of PANI. Well-aligned PANI–MWNTs composite films with electrical resistivities in between those of PANI and MWNTs have also been prepared by Feng *et al.*¹¹ The change in the sign of the magnetoresistance at low temperatures has been used by Long *et al.*¹² as the basis to reveal the strong coupling between the carbon nanotubes and polyaniline in the composites. PPy–MWNT nanocables prepared by *in-situ* oxidation polymerization in the presence of different surfactants and exhibiting a negative temperature coefficient of resistance are shown to have negative magnetoresistance.¹³ Jurewicz *et al.*¹⁴ have shown that PPy–MWNT composites exhibit a higher specific capacitance than that of pristine MWNT. Incorporation of MWNTs into semiconducting polymers such as poly(*m*-phenylenevinylene-*co*-2,5-dioctoxy-*p*-phenylenevinylene) enhances the electrical conductivity without any degradation in electroluminescent properties.¹⁵

Although the composites of carbon nanotubes with polymers have been investigated adequately for their electrical properties partly because the carbon nanotubes can be semiconducting or metallic depending on the structure, there are problems related to the chirality, purity, dispersability and other aspects of carbon nanotubes. The presence of metal catalyst particles in the nanotubes also affects the conductivity of the composites. On the other hand, a wide range of nanowires of functional inorganic materials is now available,¹⁶ opening up the possibility of a new class of nanocomposites with polymers. This prompted us to investigate the composites of inorganic nanowires with conducting polymers for their electrical properties. For this purpose, we have chosen nanowires of zinc oxide (ZnONW), ruthenium dioxide (RuO₂NW) and silver (AgNW). ZnO is a wide bandgap semiconductor with a bandgap of 3.37 eV while RuO₂ and silver are metallic. RuO₂ also exhibits a large specific capacitance and is used as

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an electrode material in supercapacitors.¹⁷ We have prepared composites with different molar ratios of the monomer (aniline or pyrrole) and the inorganic nanowires and studied their electrical properties. While most of the composites were prepared by the *in-situ* polymerization of aniline or pyrrole in the presence of the nanowires, we also prepared a few PANI–ZnONW composites by an *ex-situ* process, for purpose of comparison. The ZnONW were also functionalized with thionyl chloride in an effort to enhance the interfacial bonding between the nanowires and the polymer in the composites prepared by the *in-situ* process. In this article, we report the results of the first study of the electrical properties of the inorganic nanowire–polymer composites.

Experimental

Zinc oxide nanowires were prepared by the carbon-assisted route using zinc oxalate and MWNTs in the absence of any catalyst.¹⁸ The ZnONWs are crystalline and show the characteristic hexagonal structure (JCPDS File No.: 36-1451). In Fig. 1, we show electron micrographs of the as-synthesized nanowires used for the preparation of the composites. Fig. 1(a) shows a SEM image of the ZnO nanowires with diameters ranging between 100 and 300 nm and lengths extending to several microns. A TEM image of the ZnONWs is shown in Fig. 1(b) and its corresponding selected area electron diffraction (SAED) pattern (shown in the inset) confirms the single-crystalline nature of the nanowires. ZnONWs were functionalized by treating them with SOCl_2 followed by thorough washing with distilled water. We designate these nanowires as Cl–ZnONW. Nanowires of silver were obtained using the solution-based approach as reported by Xia and co-workers.¹⁹ Fig. 1(c) shows a SEM image of silver nanowires

with diameters ranging between 100 to 200 nm and lengths extending to several microns. RuO_2 NWs were obtained by the method reported in the literature.²⁰ The RuO_2 NWs had diameters in the 50–100 nm range with lengths of a few hundred nanometers as shown in Fig. 1(d). The RuO_2 NWs are crystalline and show the characteristic rutile structure (JCPDS File No.: 43–1027). We have prepared composites of inorganic nanowires with polyaniline as well as polypyrrole by *in-situ* and *ex-situ* methods.

In the *in-situ* method of preparation of the PANI–inorganic nanowire composites, the nanowires were dispersed in distilled aniline, followed by polymerization. In a typical synthesis of the PANI–ZnONW (2 : 1) composite, an ammonium persulfate solution (0.6867 g dissolved in 13.7 ml of 1 M aqueous HCl solution) was slowly added to a mixture of aniline (0.67 g, 7.3 mmol) and ZnONWs (0.303 g, 3.65 mmol) in a round bottom flask containing 13.7 ml of 1 M aqueous HCl solution under sonication at 0 °C. Sonication was carried out for another two hours after the addition of the ammonium persulfate solution. The product was filtered and washed thoroughly with distilled water (to remove the unreacted ammonium persulfate and excess of HCl) and then with methanol (to remove the oligomers). The filtered sample was dried under a dynamic vacuum at room temperature for 24 h to ensure the absence of moisture, which affects the conductivity of polyaniline.²¹ The various compositions of the PANI–ZnONW composites prepared by us are: 6 : 1, 2 : 1 and 1 : 2 (molar ratios). The 6 : 1 and 2 : 1 PANI– RuO_2 NW composites and 6 : 1 PANI–AgNW composites were prepared by a procedure similar to that described above.

The polypyrrole–nanowire composites were prepared by a method similar to that outlined in the literature using camphorsulfonic acid (CSA) as the dopant.²² For the synthesis of PPy–ZnONW (2 : 1), 0.5 ml distilled pyrrole was added to 0.303 g ZnONW with 26 ml water containing 0.587 g CSA. $(\text{NH}_4)_2\text{S}_2\text{O}_8$ (1.7 g) dissolved in 26 ml water was later added dropwise to the previous solution while under sonication. All the solutions are precooled to 0 °C before the two solutions were added together. The composite thus prepared was washed thoroughly with distilled water and methanol and dried in vacuum for 24 h. The compositions of the PPy–ZnONW composites prepared by us are: 6 : 1 and 2 : 1 (molar ratio of pyrrole and ZnONW).

In the *ex-situ* synthesis of PANI–ZnONW composites, a camphorsulfonic acid (CSA)-doped polyaniline solution was prepared in *m*-cresol as described by Reghu *et al.*²³ The ZnONWs were then dispersed in different volumes of CSA-doped polyaniline solution by ultrasonication and the solvent was removed by drying at 50 °C for 24 h to yield the composite of polyaniline and ZnONW. The compositions of the PANI–ZnONWs prepared by the *ex-situ* process are: 2 : 1, 1 : 2 and 1 : 4 (molar ratios).

A polyvinyl alcohol (PVA)–AgNW composite (0.065 volume fraction of AgNWs) was prepared by adding AgNWs (50 mg) to a hot aqueous solution of PVA (0.95 g). This mixture was sonicated for 2 h and the water removed by evaporation at 60 °C.

The composites and the parent nanowires were characterized by various techniques. Powder X-ray diffraction

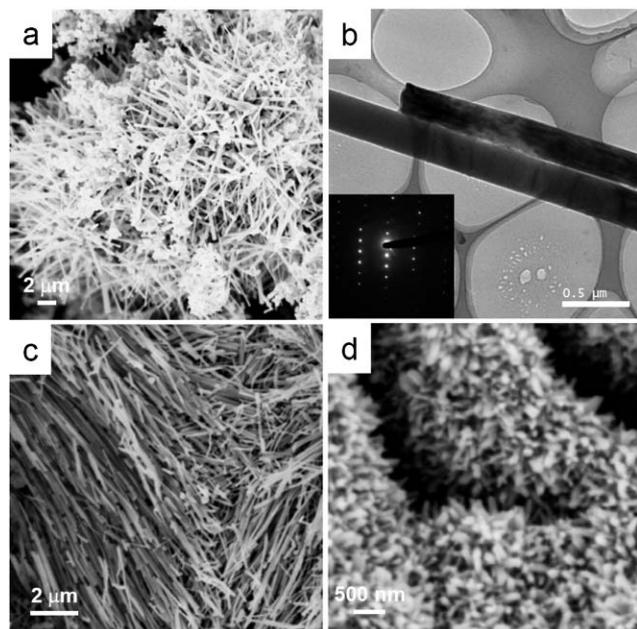


Fig. 1 (a) SEM image of as-synthesized ZnO nanowires, (b) corresponding TEM image with SAED of a ZnO nanowire as inset, (c) SEM image of silver nanowires and (d) SEM image of the RuO_2 nanowires.

(XRD) patterns were recorded using a Seifert XRD 3000-TT instrument. Scanning electron microscope (SEM) images and energy dispersive analysis of X-rays (EDAX) were obtained with a Leica S-440I microscope fitted with a Link ISIS spectrometer. Transmission electron microscope (TEM) images were obtained with a JEOL JEM 3010 instrument fitted with a Gatan CCD camera operating at an accelerating voltage of 300 kV. Infrared spectroscopy (IR) measurements were done on a Bruker FT-IR spectrometer. Electric transport properties were measured by the 4-probe technique between 300 K and 20 K.

Results and discussion

In order to verify that the nanowires were homogeneously distributed in the polymer composites, we carried out an electron microscopic examination of the composites. A SEM image of the PANI-ZnONW (2 : 1) composite prepared by the *in-situ* procedure is shown in Fig. 2(a). The nanowires are seen along the edges and are randomly dispersed. The presence of the nanowires is also confirmed by EDAX. We show a SEM image of the PANI-Cl-ZnONW (2 : 1) composite in Fig. 2(b). A SEM image of the PANI-RuO₂NW (6 : 1) composite is shown in Fig. 2(c). The nanowires are embedded in the polymer matrix as clearly evident from the TEM image of PANI-AgNW (6 : 1) shown in Fig. 2(d).

The XRD patterns of the PANI-ZnONW composites prepared by the *in-situ* procedure were characteristic of ZnO with the hexagonal structure, showing that the crystallinity of ZnONW is not affected in the composite. PANI exhibits the characteristic stretching bands of the quinoid and benzenoid rings²⁴ at 1560 cm⁻¹ and 1475 cm⁻¹ respectively in the infrared spectrum (Fig. 3(a)), indicating the oxidation state of

emeraldine salt. The quinoid band is less intense than the benzenoid band in PANI. The C-N stretching bands of the quinoid and benzenoid rings are seen at 1291 cm⁻¹ and 1236 cm⁻¹ and the in-plane C-H stretching band at 1110 cm⁻¹. The Zn-O stretching mode of the ZnONW appears as a broad band around 450 cm⁻¹ (Fig. 3(e)).²⁵ The PANI-ZnONW composites prepared by the *in-situ* method show a significant shift of the quinoid band to 1563 cm⁻¹ in the PANI-Cl-ZnONW (2 : 1) composite and to 1580 cm⁻¹ in the PANI-ZnONW (1 : 2) composite (Fig. 3). The benzenoid band is also shifted to higher wavenumbers with the increasing proportion of ZnONW, 1479 cm⁻¹ in the PANI-Cl-ZnONW (2 : 1) composite and 1489 cm⁻¹ in the PANI-ZnONW (1 : 2) composite. There is also an increase in the intensity of the quinoid band with the addition of ZnONW, suggesting that the nanowires promote and/or stabilize the quinoid form to a greater extent. Similar observations are reported in the case of PANI-MWNT composites.⁹ The intensity of the Zn-O stretching band in the composites increases with the increase in the proportion of ZnONWs as expected. PPy shows characteristic C=C stretching, C=N stretching, C-N stretching and C-H stretching at 1551, 1477, 1300 and 1045 cm⁻¹ respectively in the infrared spectrum.²⁶ In the PPy-ZnONW composites, shifts in the C=C, C=N, C-N and C-H stretching bands to 1564, 1485, 1285 and 1046 cm⁻¹ are observed. The increases in the C=C, C=N and C-H stretching frequencies are attributed to a decrease in the doping level of the polymer, which in turn can lead to a higher resistivity.

In Fig. 4(a), we show the electrical resistivity data of the PANI-ZnONW composites. The ZnO nanowires exhibit a room-temperature resistivity of 27600 Ω cm, which is higher than that of single crystals.²⁷ Polyaniline exhibits semiconducting behavior with a room temperature resistivity of 0.03 Ω cm, in accordance with the value reported in the literature.^{20,21} The PANI-ZnONW composites are also semiconducting and

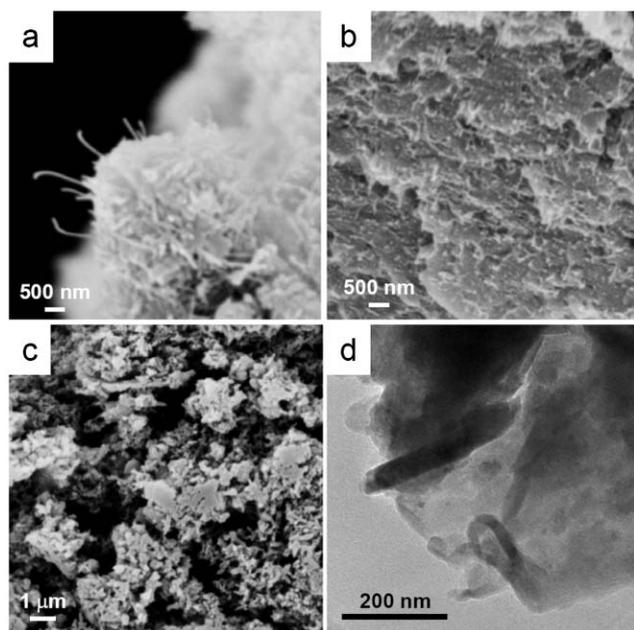


Fig. 2 SEM images of (a) PANI-ZnONW (2 : 1), (b) PANI-Cl-ZnONW (2 : 1), (c) PANI-RuO₂NWs (6 : 1) and (d) TEM image of PANI-AgNWs (6 : 1). All the composites were prepared by the *in-situ* polymerization of aniline.

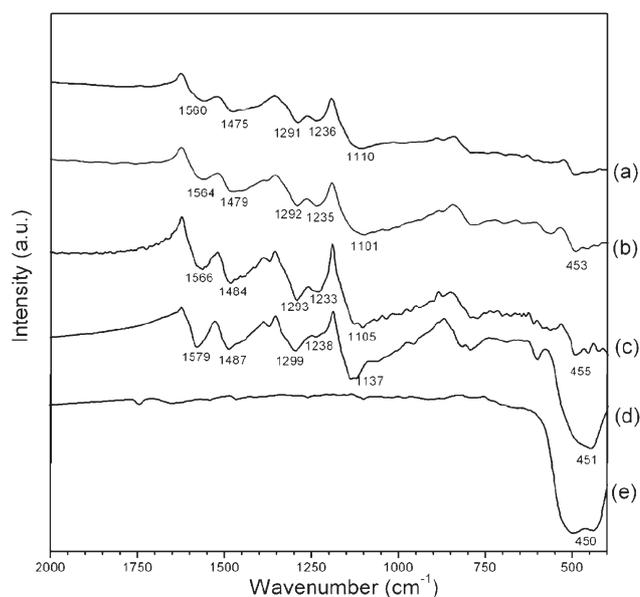


Fig. 3 Infrared spectra of (a) PANI, (b) PANI-Cl-ZnONW (2 : 1), (c) PANI-ZnONW (2 : 1), (d) PANI-ZnONW (1 : 2) composites prepared by the *in-situ* method and (e) ZnONW.

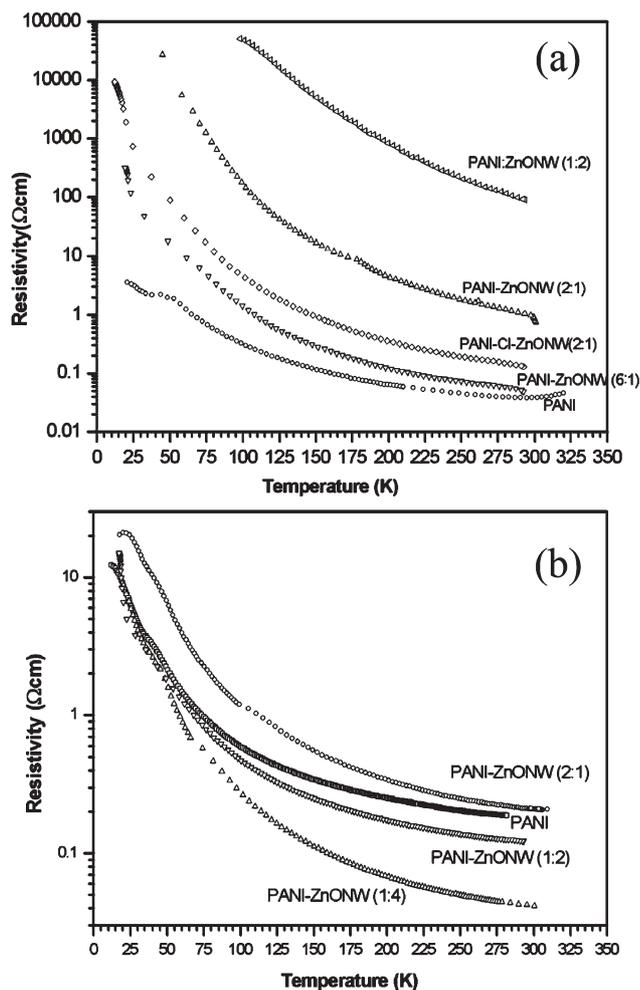


Fig. 4 (a) Electrical resistivity of PANI-ZnONW composites prepared by the *in-situ* method and (b) electrical resistivity of PANI-ZnONW composites prepared by the *ex-situ* method.

exhibit room-temperature resistivities intermediate between those of PANI and ZnONW. There is an increase in the temperature dependence of the nanowires with the increase in the proportion of ZnONWs. It is possible that an increase in the ZnONW concentration leads to an increase in the pH of the reactant mixture, resulting in a different doping level of the conducting polymer. The PANI-Cl-ZnONW (2 : 1) composite shows a slightly lower room-temperature resistivity compared to that without functionalization by an order of magnitude. It may be noted that in PANI-Cl-ZnONW (2 : 1), the NH groups of the polymer can undergo a chemical reaction with the chlorines on the nanowire surface.

The electrical resistivity data of the PPy-ZnONW composites are shown in Fig. 5. The room-temperature resistivity of PPy is 0.088 Ω cm. The electrical properties of these composites are similar to those with PANI, and we see an increase in the resistivity with the increasing proportion of ZnONWs. The temperature dependence of resistivity of the composites is similar to that of PANI-ZnONWs. The electrical resistivity data of the nanowires composites in Figs. 4(a) and 5 demonstrate that we can manipulate the resistivity behavior of the composites over a large range by varying the composition.

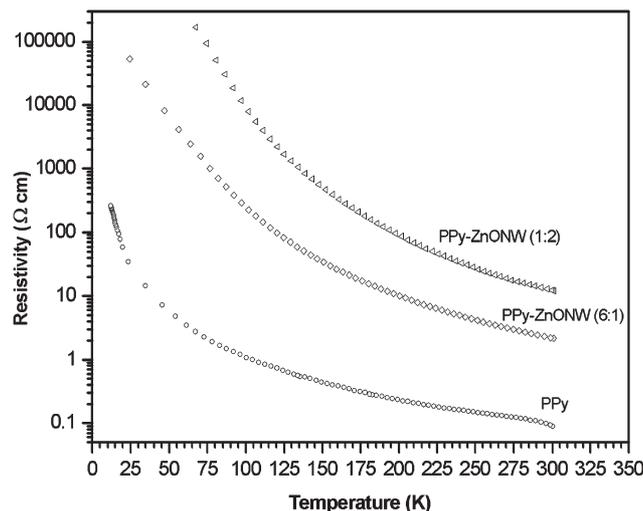


Fig. 5 Electrical resistivities of PPy-ZnONW composites prepared by the *in-situ* method.

In the PANI-ZnONW composites prepared by the *ex-situ* method, the nanowires are embedded in a matrix of polyaniline as evident from the SEM and TEM images (ESI†). The presence of ZnONWs is also confirmed by the EDAX spectrum. The crystallinity of the ZnONWs is preserved in the composite as revealed by the XRD patterns. In Fig. 4(b), we show the electrical resistivity data of PANI-ZnONW composites prepared by the *ex-situ* method. The resistivity of the PANI sample used here is different from that in Fig. 4(a) because of the different method employed for the synthesis. There is a decrease in the room temperature resistivity and an increase in the temperature dependence of resistivity after the incorporation of a large proportion of ZnONW in PANI. The slightly lower resistivity of the PANI-ZnONW *ex-situ* composites in comparison with the *in-situ* composites may be due to the chemical modification of the nanowire surfaces that may occur in the *in-situ* process and due to the chemical interaction between the NH groups of the PANI and surface Zn²⁺ of the nanowire.²⁸ Infrared spectroscopic evidence for the interaction of the oxide nanowires with the polymer matrix was provided earlier.

Electrical resistivities of the composites of PANI with RuO₂NWs and AgNWs are shown in Fig. 6. RuO₂NW is metallic and the resistivity increases with increasing temperature. The electrical resistivity of the PANI-RuO₂NW composites lies in between those of PANI and RuO₂, the resistivity decreasing with increasing the proportion of RuO₂NWs. The temperature dependence of these composites is similar to that of PANI. The resistivity of the 6 : 1 PANI-AgNW composite is lower than that of pristine PANI. It is noteworthy that the resistivity of 6 : 1 PANI-AgNW is comparable to that of 6 : 1 PANI-RuO₂NWs.

In order to understand the electrical behavior of the polymer-nanowire composites, it is important to know whether the nanowires provide a percolation path. We have therefore estimated the minimum molar ratio of PANI to metallic nanowires to form a connected network. In the case of metallic RuO₂ and AgNWs, the required molar ratios are 3 : 1

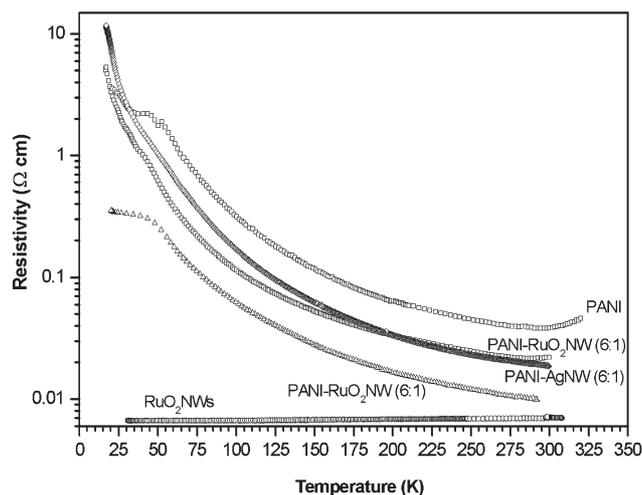


Fig. 6 Electrical resistivity of PANI composites with RuO₂NW and AgNW prepared by the *in-situ* method.

and 2.4 : 1 respectively.^{29–31} These values are much larger than the ratios reported for polymer–SWNTs composites.^{30,32} This is due to the larger diameter, smaller aspect ratio and higher density of the metallic nanowires in comparison with the SWNTs. It therefore becomes clear that there is no percolation network in the polymer–metal nanowire composites giving rise to the metallic behavior. In order to convince ourselves of this conclusion, we prepared a PVA–AgNWs composite with a composition close to that required for percolation, but the composite failed to show high conductivity.

A comment on the conduction process in the polymer–inorganic nanowire composite would be in order: The electrical conductivity of a conducting polymer arises due to the introduction of charge carriers by doping, the doped form being more conducting than the undoped form. In the inorganic nanowire composites, the nanowires are embedded in a conducting matrix and the conductivity of the composite would be a function of both the matrix and the nanowires. In the case of ZnONW composites, the electrical conductivity is mainly due to the polymer matrix as the conductivity of the ZnONWs is far lower than that of the polymer. The nanowires and the polymer contribute to the electrical conductivity in the case of the RuO₂NW and AgNW composites. Since the nanowires do not form a connected network, the obtained increase in the conductivity can arise due to the participation of the nanowires in the conduction process in some way. The resistivity of the PANI composite therefore lies in between those of PANI and metallic nanowires.

Conclusions

It has been possible to prepare composites of ZnO, RuO₂ and Ag nanowires with polyaniline and polypyrrole by an *in-situ* procedure. The ZnONW composites show a wide range of resistivities between those of the nanowire and the polymer. Polyaniline composites with chlorinated ZnONWs show lower resistivity than the unfunctionalized nanowires. Composites of ZnONWs with PANI prepared by the *ex-situ* process show slightly lower resistivities than that of the polymer. The PANI

composites with RuO₂NWs and AgNWs exhibit lower resistivities than that of PANI. The range of composites prepared by us with room temperature resistivity (0.01–400 Ω cm) indicates that the composites could be useful in some applications. For example, the PANI–RuO₂NW composites may be used as electrode materials in supercapacitors. This work constitutes the first study of the electrical properties of inorganic nanowire–polymer composites. There is clearly great room for further studies on these materials for potential applications.

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- 31 From reference 29: The minimum volume fraction of rods needed to form a connected network is given by $\phi = 3D_{\text{eff}}^2/2L^2$, where D_{eff} and L are the effective diameter and length of the rod respectively. In the case of RuO₂NWs $D_{\text{eff}} = 80$ nm and $L = 300$ nm and for AgNWs, $D_{\text{eff}} = 150$ nm and $L = 1$ μm . The diameters of the nanowires were estimated from transmission electron micrographs while the lengths of the nanowires were determined by SEM. Due to the intense sonication involved during the preparation of the polymer composites, the average length of the AgNWs decreases to 1 μm . In terms of volume fractions, the volume fraction of RuO₂NWs and AgNWs needed are 0.0711 and 0.0459 respectively.
- 32 Molar ratio for polymer-SWNTs composites cannot be defined. The composites can be described in terms of volume fractions. The following are the minimum volume fractions of the filler (nanowire/nanotube) needed for the formation of a connected network: SWNTs 5.2×10^{-5} (from reference 30), RuO₂NW 0.711 and AgNWs 0.0459.



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