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Bias dependent crossover from variable range hopping to power law characteristics in the resistivity of polymer nanowires

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Abstract

The electronic transport properties of ultra-low doped conducting polymer nanowires exhibit characteristics of a pinned one-dimensional Wigner crystal (1D WC) due to the long range electron–electron interaction at low temperature (<30 K). These wires also show characteristics of three-dimensional variable range hopping (3D VRH) at higher temperature. Here we report a resistivity study of these nanowires as a function of the bias around and above 30 K, to show that a crossover takes place from 3D VRH to power law behavior as the bias voltage or current is increased from a low to a relatively high value. The experimental results for this temperature range show several similarities to the theoretically predicted properties of disordered Lüttinger liquid, though at lower temperature the characteristics of the 1D WC are obtained for these nanowires.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Electronic transport properties of quasi-one-dimensional (1D) systems attract much interest as they provide a unique test bed for studying the interplay between interactions and disorder. In 1D electronic systems, Fermi liquid theory breaks down in the presence of electron–electron interaction (EEI), which is manifested by the absence of single-electron quasiparticles. In the absence of disorder the low energy excitations of interacting 1D electronic systems are spin and charge collective modes with bosonic characteristics and the tunneling density of states shows a power law dependence on energy; these features are successfully described using the Lüttinger liquid (LL) model [1–3]. Experimentally, power law behaviors of current–voltage (I–V), $I \propto V^{1+\beta}$, and resistance–temperature (R–T), $R \propto T^{-\alpha}$, characteristics have been observed in various quasi-1D systems like carbon nanotubes [4, 5] and inorganic nanowires [6–8]. Explanations of the observed power law characteristics have been given, based on the LL or environmental Coulomb blockade (ECB) [9] theory.

Conducting polymer nanowires constitute another interesting and rich quasi-1D model system to study the effects of disorder and interactions. We have shown earlier [10–13] that low temperature electronic transport properties of nearly monodispersed, chemically synthesized, low doped polypyrrole nanowires (figure 1) exhibit several interesting features such as the existence of a 'gap' (V_G) at low bias, a switching transition to a highly conducting state at relatively large bias (V_{Th}), power law behavior in between these two bias regimes (figure 2(a)), negative differential resistance (NDR) in the switched state (figure 2(b)), noise enhancement, the presence of negative capacitance etc. All of these experimental observations could be explained by assuming the formation of 1D Wigner crystal (1D WC) in these nanowires [11]. With increasing temperature the characteristic features of 1D WC gradually diminish and above a certain temperature (depending on the nanowire diameter and synthesis details) the features vanish. It was shown earlier that these nanowires exhibit conventional three-dimensional variable range hopping (3D

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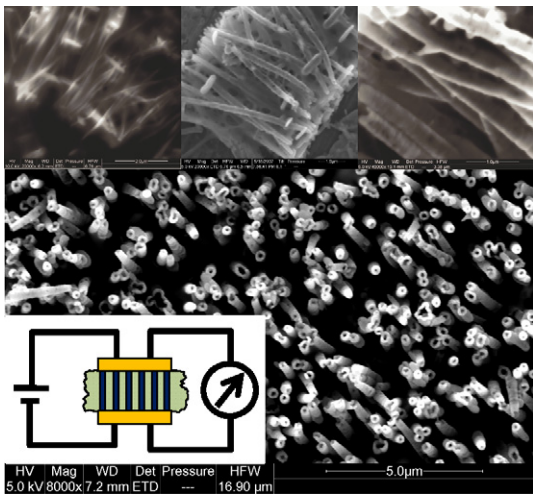

Figure 1. SEM image showing homogeneous growth over the entire membrane of nearly monodispersed nanowires having average diameter 450 nm. The tubular nature of the nanowires is evident from the picture. Images of nearly monodispersed nanowires of 50, 110 and 350 nm diameter (from left to right) have been shown in the upper inset. The pseudo-four-probe connection geometry for the electrical measurements is shown in the lower inset.

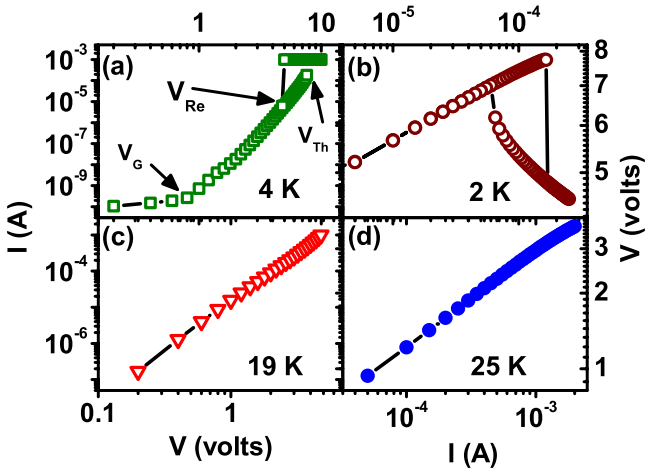

Figure 2. I–V characteristics of 350 nm diameter polypyrrole nanowires have been shown on a log–log scale. (a) Voltage biased measurements show the existence of a 'gap' (V_G), switching threshold (V_{Th}) and return voltage (V_{Re}) at low temperature. (b) Current biased measurements show the presence of NDR. At high temperature the (c) switching transition and (d) NDR vanish.

VRH) characteristics at higher temperatures [14]. Here we show that above 30 K temperature, a crossover in the R–T characteristics from 3D variable range hopping ($R \propto \exp(T_0/T)^{1/4}$) to a power law dependence ($R \propto T^{-\alpha}$) takes place as a function of the bias voltage or current. Although the high bias I–V characteristics show a power law dependence ($I \propto V^{1+\beta}$) and the value of the exponent (β) decreases with increasing temperature, its temperature dependence cannot be accounted for by the observed power law dependence of the resistivity. Above 30 K, a disordered LL model can explain the results to a certain extent; the emergence of this indicates the existence of short range electron–electron interactions at high temperature.

2. Sample preparation and measurements

Polypyrrole nanowires were synthesized using a membrane based synthesis technique by a chemical method. We have used polycarbonate membranes (Whatman) of various pore diameters (ranging from 15 to 200 nm), which determine the diameter of the nanowires. Before the polymerization reaction, pyrrole monomer (Merck, Germany) was vacuum distilled under reduced pressure. We have taken the monomer (pyrrole) and oxidizing agent ($FeCl_3$) in a 1:5 ratio and allowed them to react for 2 h inside the pores. As the diffusion of ions (here Cl^-) is hindered by the narrow pore diameter, the average doping concentration (determined by the Cl–N ratio) decreases with decreasing nanowire diameter. Fully doped polypyrrole corresponds to 0.33 doping (one dopant per three monomer units); with decreasing nanowire diameter the average doping decreases and can be as low as less than ∼0.003 [14, 15]. A few samples were prepared with 0.1 M HCl solution instead of aqueous solution. After the reaction the surface deposited polymer layer was removed by mechanically polishing with tissue paper and washed several times with water to remove the excess monomer and unreacted reagent. The polymer nanowire containing membrane was then vacuum dried for a few days. The sample quality was inspected using a Quanta 200 FEG scanning electron microscope (SEM) in low vacuum mode without depositing any metal coating. Before imaging, the supporting polycarbonate membrane was dissolved partially in chloroform and care was taken to avoid damage of the nanowires.

For electrical measurements, circular gold pads of 100 nm thickness and 2 mm diameter were deposited on both sides of the membrane containing nanowires. In this connection geometry nearly 10^5 monodisperse nanowires were connected in parallel and the insulating polycarbonate matrix isolates them from each other. We have shown earlier that gold serves as a very good contact material for polypyrrole nanowires and provides low contact resistance [14]. We found that at room temperature the two-probe resistance of the larger diameter nanowires can be as low as a few ohms, so the maximum value of the contact resistance cannot be greater than a few ohms. As the resistances of the nanowires at low temperature are larger than several kiloohms, so we can safely neglect the contribution of contact resistance in our analysis. Electrical measurements of relatively low resistivity samples were done in a pseudo-four-probe geometry (refer to the lower inset of figure 1) by driving the current using a Keithley 2400 source meter and measuring the voltage across the sample using an Agilent 34420A nanovoltmeter. For highly resistive samples (like nanowires synthesized using a 15 nm pore diameter membrane) two-probe measurements were done by measuring the current for a certain voltage bias using a Keithley 6517A electrometer. Details of the synthesis and electrical measurements were discussed in our earlier publications [10, 11, 14, 15].

SEM images reveal near monodispersed nature of the polymer nanowires (refer to figure 1). Hence it can be assumed

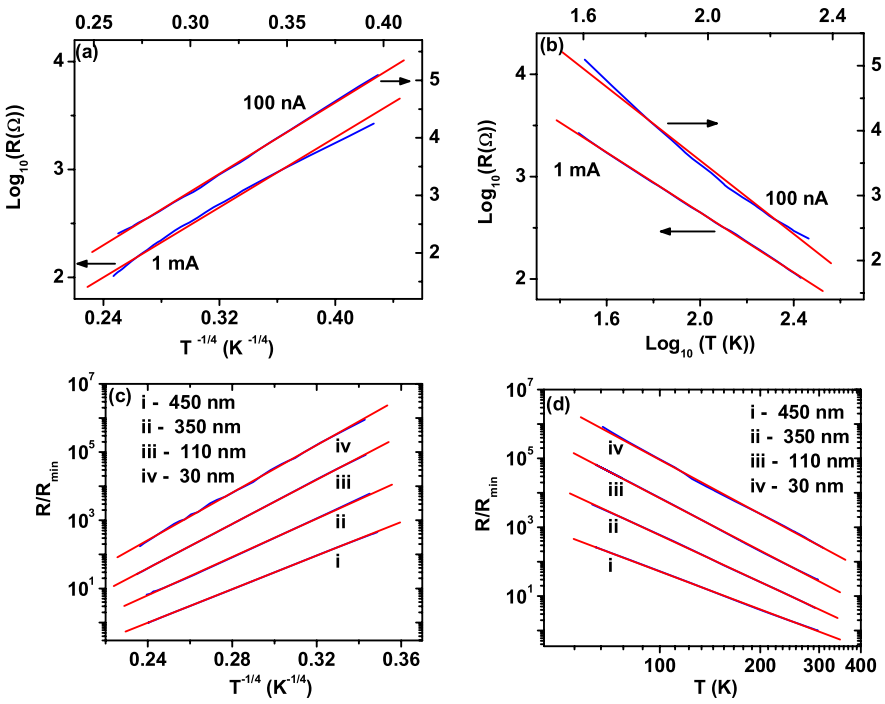

Figure 3. (a) $\log_{10}(R)$ versus $T^{-1/4}$ data of 70 nm diameter nanowire are plotted for 100 nA and 1 mA bias current. The linear fit shows that low bias data give a better fit to 3D VRH behavior. The same data are plotted on a log–log scale (b) to show that a power law gives a better fit with $\alpha = 1.5$ for high bias data. $R/R_{\min}$ as a function of temperature (graphs other than the 450 nm diameter one have been shifted for visual clarity) for various diameter nanowires also shows that (c) VRH gives a better fit to low bias data and (d) a power law gives a better fit to high bias data (see the text for details).

that the electrical properties of the collection of nanowires connected in parallel geometry will not differ much from their individual properties and finer features will not get averaged out. The tubular nature and homogeneous growth of the polypyrrole nanowires over the entire membrane is also evident from the images.

3. Results and discussion

3.1. Low temperature (<30 K) properties

Before the high temperature (>30 K) measurements, we confirmed the sample quality from their low temperature I–V characteristics. We observed a 'gap' at low voltage bias (V_G), a sharp switching transition to a highly conducting state above V_{Th}, power law behavior in between V_G and V_{Th} (refer to figure 2(a)) and the presence of NDR in the switched state (refer to figure 2(b)). These nanowires also exhibit negative capacitance, noise enhancement and other characteristic features of Wigner crystals similar to those observed earlier in these systems [10–13]. With increasing temperature these features gradually vanish; in figures 2(c) and (d) we have shown representative data showing the absence of a switching transition and NDR at high temperature respectively.

3.2. The bias dependence of R–T above 30 K

For low (current or voltage) bias the nanowires show Mott's 3D VRH behavior [14] and at relatively large bias the resistance follows a power law behavior at temperature above 30 K. In figures 3(a) and (b) we have shown R–T data for 70 nm diameter nanowires measured using 100 nA and 1 mA bias currents, and their fit with 3D VRH and a power law. It is clear from the figures that for low bias (100 nA) R–T data, 3D VRH gives a better fit than power law behavior, but for high bias (1 mA) data, a power law gives a better fit. Similar behavior has been observed for other diameter nanowires; in figures 3(c) and (d) we have shown $R/R_{\min}$ versus T data (where $R_{\min}$ is the minimum value of the resistance which corresponds to the maximum temperature data) for (i) 450, (ii) 350, (iii) 110 and (iv) 30 nm diameter nanowires. It is clear from the data that, in the linear regime, i.e., at low bias (typically 1 μA, 1 μA, 200 nA and 10 mV bias applied to 450, 350, 110 and 30 nm diameter nanowires respectively), R–T data are better fitted with a 3D VRH law (refer to figure 3(c)). At high bias (typically 100 μA, 200 μA, 5 μA and 1 V bias applied to 450, 350, 110 and 30 nm diameter nanowires respectively), a power law gives a better fit to the measured R–T data over a wide range of temperature (shown in figure 3(d)). It is evident from the data and the fits that voltage biased measurements give similar results (see data (iv) in figures 3(c) and (d)) to current biased ones. So a bias (voltage or current) dependent crossover from 3D VRH to power law behavior in the R–T data has been observed for these nanowires.

3.3. The effect of nonlinearity on the resistance

We have observed that the I–V characteristics of all the nanowires show a power law behavior, $I \propto V^{1+\beta}$, at high

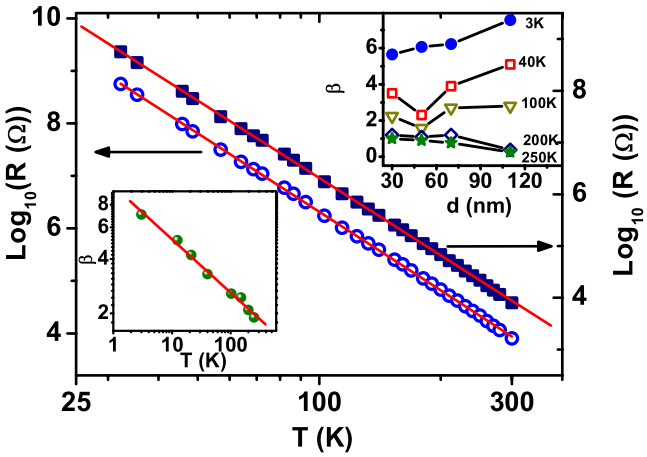

Figure 4. Fits of the R–T data with equations (2), taking $\beta_0 = 10$ (left side scale) and $\beta_0 = 0$ (right side scale) for 50 nm diameter nanowire, have been shown (see the text for details). The variation of β as a function of diameter, for various temperatures, has been shown in the upper inset. The temperature dependence of β and its fit with equation (1) have been shown in the lower inset.

bias [10, 11, 15]. With increasing temperature the exponent β decreases and at relatively high temperature ohmic I–V characteristics ($\beta = 0$) were obtained [15]. At high bias the measured R–T characteristics can get affected by the temperature dependence of β, which can be expressed as

$$\beta(T) = \beta_0\, T^{-\gamma} \tag{1}$$

where the exponent γ depends on the detailed nature of the wires (diameter, synthesis condition etc) and was found to have values between 0.2 to 0.5. Including the above temperature dependence of β, the power law behavior of the R–T characteristics can be written in a general form as

$$R(T) = R_0 T^{-\alpha} V^{-\beta_0 T^{-\gamma}}. \tag{2}$$

R–T characteristics in the ohmic region can be obtained by putting $\beta_0 = 0$. In the lower inset of figure 4, we have shown the β–T characteristics of 50 nm diameter nanowire (symbols). From the power law fit (continuous line) we obtained $\beta_0 \simeq 10$ and $\gamma = 0.3$. Using these values we fitted the R–T characteristics using equation (2) (refer to figure 4) and found $\alpha = 5$. If we put $\beta_0 = 0$ then the fit gives $\alpha = 4.97$ (refer to figure 4). This nearly equal value of α suggests that the temperature dependence of β does not have a considerable effect on the observed power law R–T characteristics. This is because the value of γ is much smaller than that of α. The diameter dependences of β (measured on a different batch of samples) for various temperatures have been shown in the upper inset of figure 4. At low temperature (<30 K) β shows a gradual increase with increasing diameter, which is consistent with our earlier results [11]. It is interesting to note that, with increasing temperature, β shows some randomness, but at significantly higher temperature, β decreases with increasing diameter of the nanowires. It is clear from the data that, for any diameter nanowire, β decreases with increasing temperature.

3.4. Similarities with the Lüttinger liquid above 30 K

In our earlier studies we have shown that conducting polymer nanowires show properties of a weakly pinned Wigner crystal state at low temperature [11]. It has been predicted theoretically that quasi-1D systems will form Wigner crystals in the presence of long range EEI [16–18]. At low temperature and low bias, an electron crystal remained pinned by the impurities present in the system. If the impurities act as strong pinning centers, then a quasi-1D Wigner crystal shows VRH conductivity with various exponents which depend on the impurity concentration [19, 20]. At large impurity concentration, the conductivity follows the Efros–Shklovskii law [21] and at very low impurity concentration Mott's 3D VRH [22] type behavior is predicted. On the other hand, for weakly pinned Wigner crystal, the conductivity shows a power law temperature dependence [23–26]. The impurities destroy the long range order; however, for weak impurity strength quasi-long range order may exist [27]. For quasi-1D systems, like nanowires, the observation of 3D VRH behavior at low bias can be justified by considering the hopping movement of pinned Wigner crystal at low bias. At large bias the relative strength of the impurity becomes weak and the motion of weakly pinned Wigner crystals gives rise to power law dependent I–V and R–T characteristics.

With increasing temperature, the kinetic energy of the charge carriers increases; as a result the Wigner crystal state is destroyed. In these polymer nanowires, the maximum temperature above which the characteristic features of the Wigner crystal (such as the existence of a gap, switching, NDR, noise enhancement etc) vanish is found to be ∼30 K; hence this can be considered as the 'melting' temperature. Above the 'melting' temperature (above which the kinetic energy dominates over the EEI and the long range nature of the EEI becomes insignificant) of Wigner crystal, the possible interaction left is the short range EEI and the system can show characteristics of a LL [24]. The power law behavior of the I–V and R–T characteristics reported here above about 30 K is consistent with previous results on polymer nanofibers [28] and can be attributed to the presence of a LL state in these systems. According to LL theory, the I–V curves for different temperatures can be scaled [29] to a master curve by plotting $I/T^{1+\alpha}$ versus $eV/(k_{\mathrm{B}}T)$. The power law behavior of the I–V characteristics has been observed over a wide range of bias and temperature for the smaller diameter nanowires; one such representative data set has been shown in figure 5(a) for 30 nm diameter nanowires. We also observed that lower diameter nanowires show better scaling to the master curve and that the scaling holds up to relatively high temperature (see figure 5(b)). With increasing diameter of the nanowires the temperature span over which power law behavior is observed decreases (refer to figures 5(c)–(f)). These observations indicate that LL like characteristics may be present for the lowest diameter nanowires at relatively high bias and over a wide temperature range. This may arise due to the fact that, in the lower diameter nanowires, the polymer chains are better aligned, so the signature of quasi-1D behavior is more prominently observed and sustained up to higher temperature. For other (higher) diameter nanowires, the scaling to the master curve

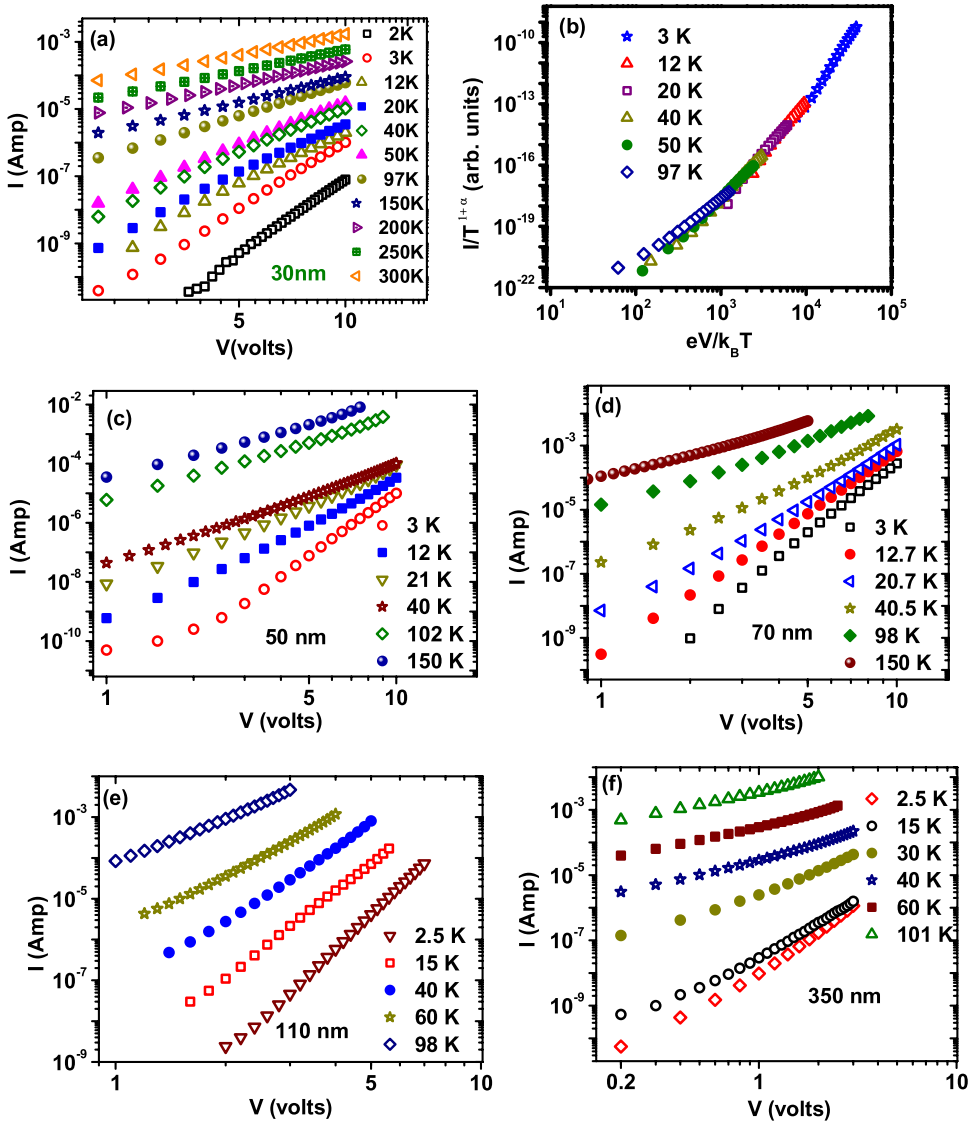

Figure 5. (a) I–V characteristics of 30 nm diameter nanowire for various temperatures have been plotted on a log–log scale to show that power law dependence holds over a wide temperature range. (b) Scaling to the master curve is shown for 30 nm diameter nanowire. I–V characteristics of (c) 50 nm, (d) 70 nm, (e) 110 nm and (f) 350 nm diameter nanowires have been plotted on a log–log scale.

over a wide temperature range has not been observed, but the bias dependent crossover has been observed. This can be explained by considering disordered LL state formation in quasi-1D systems.

3.5. The effect of impurity on Lüttinger liquids

LL theory can be used to explain the behavior of interacting electrons in purely 1D systems. But in a strictly 1D system, the power law behavior may be washed out by quantum fluctuations destroying the long range order [30]. However, in quasi-1D systems (systems composed of several parallel chains) where the different channels are statistically independent, the power law behavior may be observed clearly due to the averaging of fluctuations. In quasi-1D systems the exponents depend on the number of the 1D channels [9]. But in a quasi-1D system, if interchain electron hopping plays a significant role then the system loses its one dimensionality and β becomes equal to zero, thereby inducing a crossover to 3D behavior. This could be the case for quasi-1D systems at very low temperature, where arbitrarily weak interchain hopping can drive the system towards a 3D one [31–33]. In a disordered 1D system, impurity acts as an infinite tunneling barrier [9, 34, 35]. However the presence of disorder in 1D systems can stabilize the LL state, where the LL state is preserved between two consecutive impurities; thus a disordered quasi-1D system can be considered as a collection of LL stubs [36]. For such systems, weak interchain hopping does not destroy the LL state at low enough temperature (compared to the minimum excitation energy of the LL collective modes), and low bias conductivity shows variable range hopping behavior following Mott's 3D VRH law for short range interaction and the Efros–Shklovskii law [21] for long range interaction between electrons of different segments. At high bias, if the voltage drop over a single LL stub is nearly equal to or greater than the energy of the zero-mode

level, then one observes a power law behavior [36]. So, the observations that high bias R–T data show power law behavior above ∼30 K and low bias R–T characteristics show 3D VRH behavior in the same temperature range are consistent with the disordered LL model. With decreasing doping concentration, the average separation between the impurities (in conducting polymer the dopant also acts as an impurity) increases, and as a result the extent of the LL stub increases, and hence the minimum excitation energy decreases [36]. As the average doping concentration decreases with decreasing nanowire diameter [14], the 1D nature becomes more prominent in the thinner nanowire. We have observed power law behavior over a wide range of temperature for the lowest diameter (30 nm) nanowire (see figure 5(a)), which supports the above argument.

3.6. Bias dependent enhancement of the 1D nature

In disordered quasi-1D systems like polymer nanowires (composed of several parallel chains), increasing bias along the chain axis enhances the 1D nature. Both electric field and temperature energetically help the localized electrons to hop across the barrier. The electric field increases the probability of hopping along the field direction; on the other hand, the thermal energy is direction independent. When we apply an electric field along the nanowire axis the probability of hopping along the chain (as the chains are aligned along the nanowire axis) increases, enhancing the 1D nature. If temperature is increased, then electron hopping tends to become direction independent and the probability of interchain hopping increases, resulting in a 3D nature. The probability of hopping between two site (i, j) is defined as

$$\Gamma_{ij} = \Gamma_0 \exp(-r_{ij}/\xi) \exp(-\epsilon_{ij}/k_B T) \tag{3}$$

where ϵ_{ij} is the energy difference, r_{ij} is the spatial separation between the hopping sites and ξ is the localization length. In the presence of an electric field (E) applied along the nanowire axis, the ratio of the probabilities of hopping parallel and perpendicular to the polymer chains is

$$\frac{\Gamma_\parallel}{\Gamma_\perp} = \exp((r_\perp - r_\parallel)/\xi) \exp((\epsilon_\perp - \epsilon_\parallel + Er_\parallel)/k_B T), \tag{4}$$

were $r_\perp$ $(r_\parallel)$ and $\epsilon_\perp$ $(\epsilon_\parallel)$ are the spatial separation of the sites and the difference in site energy in the perpendicular (parallel) direction, respectively. Considering $r_\perp = r_\parallel = r$ and $\epsilon_\perp = \epsilon_\parallel = \epsilon_0$, the ratio becomes

$$\frac{\Gamma_\parallel}{\Gamma_\perp} = \exp(Er/k_B T). \tag{5}$$

It is clear from equation (5) that at low temperature and large bias, $\Gamma_\parallel \gg \Gamma_\perp$ and a 1D nature dominates. Defining $p = \Gamma_\parallel/\Gamma_\perp - 1$ and considering $r = 10$ nm we have shown, in figure 6, the calculated variation of p as a function of temperature for an applied voltage of 1 V and 1 mV across a 10 μm long nanowire. It is evident from figure 6 that p is significantly smaller for low bias over the entire temperature range, and the system can behave as a 3D one. At high bias, hopping becomes highly asymmetric and it is more

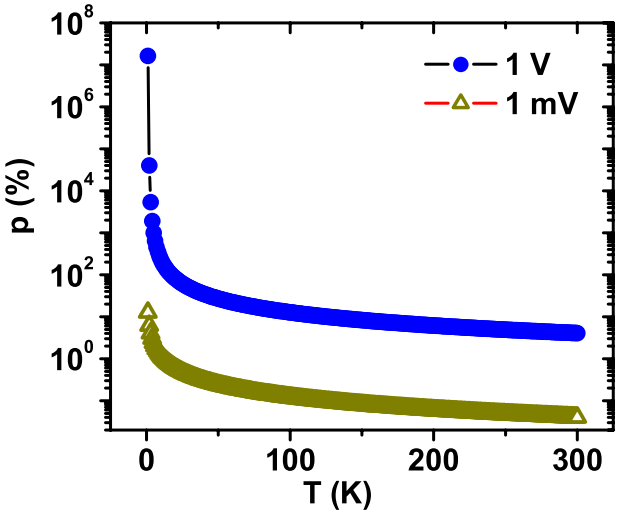

Figure 6. p is plotted as a function of temperature for 1 mV and 1 V bias. It is clear from the plot that at high bias the hopping asymmetry is significantly large even at high temperature.

favorable towards the applied field direction than any other direction, which helps to preserve the quasi-1D nature of the nanowires. For 1D hopping of localized carriers, VRH predicts $\ln R \propto T^{-1/2}$, but we do not find this type of behavior at high bias. In a quasi-1D system power law hopping can be observed due to the EEI at sufficiently low temperature (where electron–phonon coupling can be neglected). In this case, EEI causes delocalization and gives rise to power law behavior with non-universal exponents [37, 38]. However, the power law behavior was observed over a wide range of temperature and this suggests that disordered LL is more suitable for explaining the experimental results above ∼30 K. The inertness of α as regards the temperature dependence of β is also probably related to the impurity distribution and quasi-1D nature of the system. Under a certain applied voltage the voltage drop across a LL stub depends on the impurity distribution (determining the extent of the stub) but the thermal energy of the electrons depends only on the temperature of the sample.

3.7. The effect of electron–phonon interactions

Power law behavior can also arise in a 1D disordered system due to the presence of electron–phonon interaction [39], where electrons were delocalized due to the coupling with 3D phonons and show a power law hopping mechanism. In this case one gets $R(T) \sim T^{-3}$ type dependence at low temperature. The exponent α that we got from the resistance versus temperature data $(R \propto T^{-\alpha})$ is different from 3 and depends on the nanowire diameter. This suggests that electron–electron interaction is responsible for the observed power law behavior. In our previous study we have shown that electron–phonon interaction plays a significant role in determining the electronic transport properties of electrochemically synthesized polypyrrole nanowires, but it does not play a significant role for chemically synthesized nanowires [15].

In the presence of short range EEI, the interplay between interaction and disorder can lead to power law behavior in long nanowires ($\geqslant$10 μm) having random strong impurities where the mean impurity spacing $l \gg 1/n$; n is the average electron density [40]. In this case, the two exponents differ from each other ($\alpha \gg \beta$) and their value depends on the nature of the impurity distribution. Although the predictions are for long ($\sim$10 μm) disordered wire, which is very similar to our system, we observed $\beta > \alpha$ at low temperature.

4. Conclusions

In conclusion, we have observed that R–T characteristics of conducting polymer nanowires show a 3D VRH behavior at low bias and power law behavior at high bias around and above 30 K temperature. A disordered LL model can explain most of the results presented here. At lower temperature (<30 K) these nanowires show characteristics of pinned 1D WC; hence the emergence of a LL like state at high temperature (>30 K) is probably due to the increase in kinetic energy of charge carriers which reduce the range of the electron–electron interaction. The presence of disorder is expected to break the system into small LL stubs, where the LL state is preserved between two consecutive impurities. At low bias, conductivity follows Mott's 3D VRH law due to the lack of interaction between electrons of different segments. At high bias, when the voltage drop over a single LL stub is nearly equal to or greater than the energy of the zero-mode level, power law behavior is observed. LL features are more prominent in lower diameter nanowires where the minimum excitation energy is low, as the average separation between the impurities, and hence the extent of the LL stubs, is large.

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