Nonlinear I-V characteristics near the percolation threshold

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The I-V characteristics of a carbon-wax mixture slightly above the percolation threshold are found to be nonlinear. Generally for low (linear) -resistance samples, the I-V curves are smooth. The leading nonlinear term is quadratic, rather than cubic. For high-resistance samples, the onset of nonlinearity is marked by the appearance of resonance like structures in the R-V curves. The current I_c at which nonlinearity starts scales with the linear conductance Σ_1 as $I_c \sim \Sigma_1^x$, with $x \approx 1.4$ in both cases. The second-order conductance, Σ_2, in the case of smooth curves, also scales with Σ_1 as $\Sigma_2 \sim \Sigma_1^y$, with $y \approx 0.59$. Results are discussed within the framework of two previously suggested models. It is shown that none of these models can explain fully results obtained with the carbon-wax system.

I. INTRODUCTION

In recent years there have been several studies of percolation phenomena in nonlinear, inhomogeneous media.[1–10] A characteristic feature of the nonlinearity in such media is its enhancement near the percolation threshold (p_c) where a spanning conducting cluster appears for the first time and many physical quantities (e.g., electrical conductance) become critical.[11] Such nonlinearities already form the basis of several practical applications.[12] There exists a strong motivation for studying this phenomena for further understanding. In this regard it is of much interest to know how the theory of percolation for linear media is affected. The appropriate physical systems are mostly random binary mixtures of an insulator and a conductor, although there are instances of other types of mixtures, e.g., or normal metal and superconductor.

It is the macroscopic nonlinearity that is of interest here, in relation to microscopic mechanisms, including nonlinearities of conducting elements. In fact, on the basis of this relation, previous studies could be divided into two groups. The first group[2–7] involves conducting elements that have nonlinear I-V characteristics. Consequently, samples are nonlinear even at compositions far from p_c. The second group[8–10] involves conducting elements that have linear I-V characteristics (except possible at very high bias). The macroscopic I-V curves are also linear at compositions far from p_c but become increasingly nonlinear as one approaches p_c. In this case, it is useful to note that nonlinearities are reversible in some cases[8] and irreversible in other cases, as in the dielectric breakdown model[9] or random fuse model,[10] primarily due to catastrophic breakdown events.

In this paper we focus on some aspects of the reversible nonlinear behavior near percolation threshold of a random insulator-conductor mixture, namely, the collective nature of the I-V characteristics of such mixtures and its relation with microscopic features and crossover from the linear regime to the nonlinear one. Previously Kenkel and Straley[6] speculated on the I-V characteristics of a network of nonlinear conducting components with power-law characteristics near percolation threshold (p_c). Ohtsuki and Keyes[7] studied a model of a binary mixture of a normal conductor and superconductor just below p_c in which the normal elements were assumed to be nonlinear. The particular form of current-voltage characteristics was assumed to be a polynomial. The authors derived expressions for higher-order conductivities which were increasingly diverging as order increased. Recently, Gefen *et al.*[8] measured I-V characteristics of a two-dimensional (2D) system of thin gold films near a metal-insulator transition. I-V curves were initially linear and became nonlinear with increasing bias. The authors investigated essentially the linear regime of those curves and found that the current threshold, I_c scales with the linear conductance, Σ_1 as

$$I_c \sim \Sigma_1^x \quad \text{with } x = 1.47 \; . \tag{1}$$

They also considered two theoretical models for onset of nonlinearity, one of which is termed as the nonlinear-random-resistor-network (NLRRN) model. This model is essentially same as the classical random-resistor-network model[11] except that resistors are assumed to be nonlinear. The NLRRN model is also very similar to the one considered by Ohtsuki and Keyes[7] except that it is studied above percolation threshold. In this model, the component I-V characteristics were taken to be of the form ($\alpha > 1$):

$$V = rI + aI^{\alpha} \; . \tag{2}$$

The other one, the dynamic-random-resistor-network (DRRN) model, assumes conductors to be linear, but for a sufficiently strong local field, a nonconducting channel becomes conducting on the microscopic scale. The value of the exponent x, predicted by the DRRN model, was found to be compatible with the experimental results. A particular feature of the NLRRN model is that it easily lends itself to an analysis[4,13] that relates the coefficients of various powers of applied current (or voltage) in sample characteristics to the corresponding moments of linearized current (or voltage) distribution in the sample. The scaling properties of these moments have been extensive-

ly studied.[14] A similar analysis of the DRRN model is not available at present. The power-law characteristics [$r=0$ in (2)] have been studied theoretically for other properties, e.g., noise,[3] multiscaling,[5] etc. There have been few other studies of nonlinearities far from p_c.[15]

Here we present some results of our investigation of the reversible nonlinearity present in I-V characteristics of a three-dimensional system of carbon-wax mixture of low conductance (i.e., slightly above percolation threshold p_c). We have extended the analysis of I-V characteristics beyond the linear regime. An exponent for second-order conductance has been determined. Our measurements also indicate a yet unexplained phenomenon in that the onset of nonlinearity in generally high-resistance samples is accompanied by the appearance of resonancelike structures in R-V curves. The carbon-wax mixture has been previously used as a percolative system for studying $1/f$ noise,[16a] dielectric constant,[16b] and other properties.[16c] Carbon-polymer mixtures[17] are similar to the carbon-wax system. Carbon mixed with yet another insulator, Teflon, has been used for ac measurements.[18] Carbon, with low electrical conductivity, is a suitable conducting material to use for obtaining sufficient control over composition range near p_c.[18] Below we describe sample preparation and characterization in Sec. II and present our data and its analysis in Sec. III. Finally, we conclude our discussion in Sec. IV.

II. EXPERIMENT

The carbon power used in this study was quite structured in that it was made up of aggregrates (size $\sim 0.1\mu$) of smaller particles (size ~ 300 Å). Electron microscope photographs of carbon particles were similar to ones reported in literatures.[19] The preweighed paraffin wax (mp 61 °C) and carbon-black powder, corresponding to a desired volume fraction p of carbon, were mixed vigorously with a Teflon-coated magnetic stirrer at a temperature slightly above the melting point of wax. The molten mixture was then cast in a die to obtain disc-shaped samples 2 mm in thickness and 10 mm in diameter. Samples were then annealed at 50 °C for 48 h.[16a] Normally samples were again annealed if they were used for measurements several months after they were first prepared. The carbon powder used was such that, for more than 6–7 % of carbon by volume, the mixtures were too viscous to be stirred with the magnetic stirrer.

For dc resistance measurements, samples were held between two circular brass electrodes slightly larger in diameter than those of samples. The two-point probe method used as contact resistance was found insignificant compared to sample resistances. Carbon-wax samples showed a time of electrification[20] effect in that, from the start of a measurement, the resistance decreased, at first rapidly and then slowed down to reach a steady value. The typical time for attaining steady values in case of high-resistance ($1/\Sigma_1$) samples was about 20 min. All measurements, reported here were taken in steady-state dc conductances (Σ_1) in the ohmic (linear) region and were fitted (Fig. 1) to the relation $\Sigma_1 \sim (p-p_c)^t$, where t is

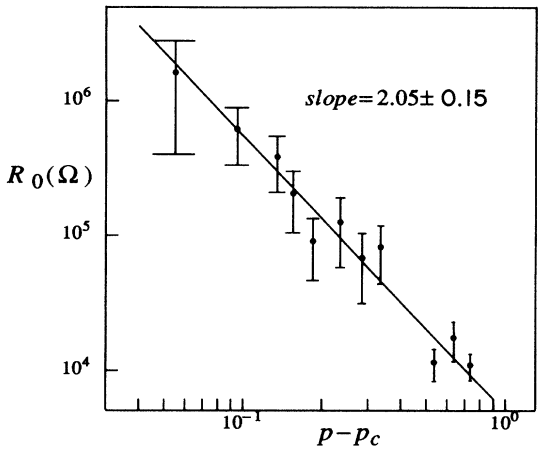

FIG. 1. Plot of linear dc resistance as a functionn of $(p-p_c)$. The solid line is a least-squares fit to $R_0 \sim (p-p_c)^{-t}$. Vertical lines indicate errors in the measurement of R_0.

conductivity exponent and p_c is the percolation threshold in the classical percolation theory.[11] Each point in Fig. 1 represents an average over about 30 samples from the same batch, i.e., with same nominal p.

We find that p_c is 0.76% by volume and t is 2.1 ± 0.2. The later is in good agreement with the universal exponent in 3D.[21] The value of p_c is quite low compared to the value (about 15% by volume[22]) usually expected in 3D random mixtures but is of same order in magnitude as found in many carbon-polymer systems.[23] It should also be compared with 10.8 and 7 % obtained by Chen *et al.*[16a] and Buesch,[16c] respectively, in the same carbon-wax system. Such disparities in values of p_c in seemingly the same system are due to the fact that p_c unlike critical exponents, is very sensitive to the physical details of components, preparation conditions, etc. Carbon-black powders are known to vary a great deal in their structural, mechanical, and electrical properties.[19,10]

Samples used in measurements of I-V curves had linear resistances ranging from 10 kΩ to more than 1 MΩ and nominal carbon concentration (p) varying from 0.86% to 1.26 % by volume. It was extremely difficult to obtain data with samples of resistance more than 1 MΩ due to highly increased fluctuation. I-V curves were obtained at room temperatures by sending a known amount of current from a constant current source (Keithley model 224) through samples held between electrodes and measuring voltages across them. For several samples, especially those having high resistances, data were acquired and processed through a microprocessor-based system by averaging over 1024 readings, taken at intervals of 1 sec, at a particular value of current. The maximum value of a current through a sample was chosen such that the heat dissipated ($\sim\mu$W) was too small to cause any irreversible thermal effect. It was observed that voltage fluctuations tend to increase considerably as current was increased beyond linear regimes.

III. RESULTS AND DISCUSSION

The *I-V* curves are voltage limited within the range of measurements. At sufficiently low voltages (V), *I-V* curves are linear. With increasing bias, the curves deviate reversibly from linearity and bend towards the current (I) axis. Generally, with low-resistance samples, the curves are smoothly varying within the range of measurements (type *A*, Fig. 2). But, for high-resistance samples (i.e., closer to percolation threshold), curves (type *B*, Fig. 3) contain at least one (possibly more) "step" that corresponds to a sharp minimum in the sample dynamical resistance, $R(=dV/dI)$. We will first discuss smooth curves.

Figure 2 shows a typical example of a smooth *I-V* characteristic. It is found that the *I-V* characteristic data (plus symbols) are well fitted by a linear plus a quadratic term: $I=\Sigma_1 V+\Sigma_2 V^2$. Here Σ_1 is the linear conductance and Σ_2 is the second-order conductance. The fit (solid line) to the data (plus) is clearly excellent. Values of the fitting parameters are 34.6 $\mu\Omega^{-1}$ for Σ_1, and 3.4 $\mu\Omega^{-1}V^{-1}$ for Σ_2. These correspond to a linear resistance of 29 kΩ for the sample in the figure. Notice that the maximum current used in measurements is as large as about five times the critical current at which the curve seems to deviate from linearity. Currents for forward and reverse biases were related within the accuracy of present measurement as follows:

$$I(V)=-I(-V)\ . \tag{3}$$

This means that the *I-V* relation should be written in the following manner:

$$I=\Sigma_1 V+\Sigma_2 V^2\mathrm{sgn}(V)\ . \tag{4}$$

Equation (4) should be compared with that found experimentally in the 2D gold film[8] or considered theoretically in a model calculation[2,13] where the leading nonlinear term was cubic rather than quadratic. Considering that crossover to nonlinearity takes place when two terms on right-hand side of (4) become comparable in magnitude,

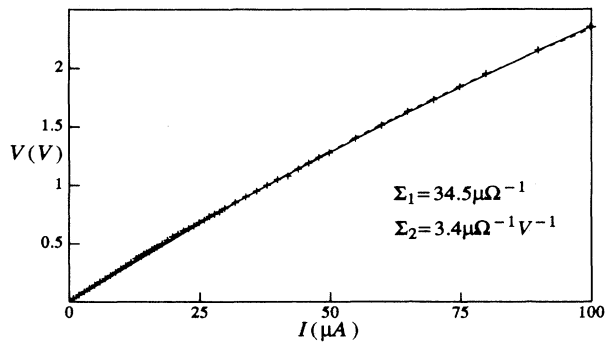

FIG. 2. A smooth, nonlinear *I-V* characteristic (type *A*) of a sample (data, plus points) of linear resistance ($1/\Sigma_1$) 29 kΩ. The solid line is a fit to the data with $I=\Sigma_1 V+\Sigma_2 V^2$. Σ_1 and Σ_2 are fitting parameters. The error in voltage measurement is less than the symbol size.

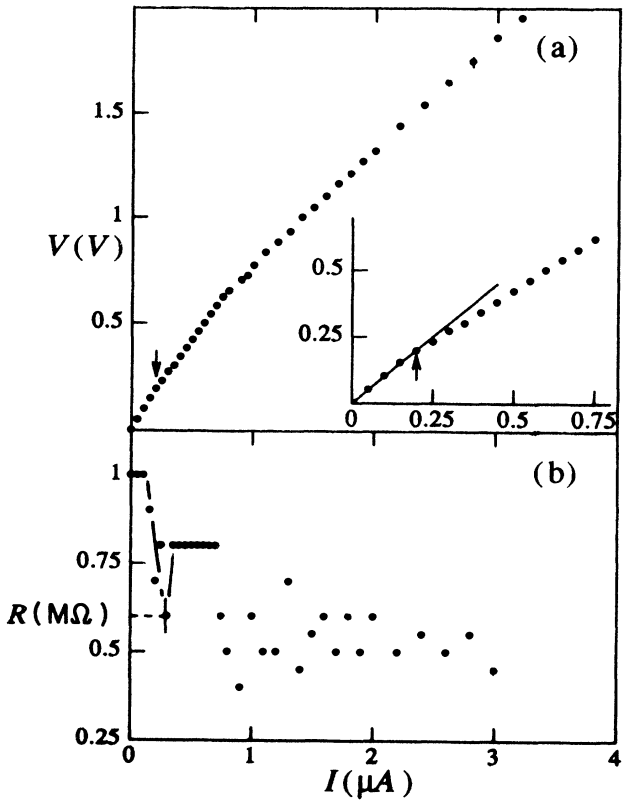

FIG. 3. (a) An *I-V* characteristic (type *B*) with a step. The linear resistance of the sample is 1 MΩ. The onset of nonlinearity is shown in the inset for greater clarity ; (b) plot of dynamical resistance ($R=dV/dI$) vs current (I). The dashed line is a guide to the eye. The vertical lines indicate typical errors in the region of measurement as shown.

it follows that the threshold current I_c scales as $I_c\sim\Sigma_1^2/\Sigma_2$. From (1), $I_c\sim\Sigma_1^x$ and assuming that $\Sigma_2\sim\Sigma_1^y$, we obtain

$$x+y=2\ . \tag{5}$$

Equation (5) significantly relates the critical behavior of two quantities, one characterizing the linear response and another characterizing the nonlinear response. A similar connection, though somewhat different in nature, between linear and nonlinear quantities has been found previously by Aharony[4] and Stroud and Hui.[2] Actually, Eq. (5) is valid only for the case $b=2$ where the first nonlinear term in the macroscopic *I-V* characteristics is proportional to V^b. The generalized form of (5) is given by

$$(b-1)x+y=b\ . \tag{6}$$

For $y=0$ i.e., if the scaling of Σ_2 is ignored, Eq. (6) reduces to the expression for x as given by Gefen *et al.*[8b] Equation (5) could not be verified in this work because both exponents were found from the same measurements. I_c (within an arbitrary factor) and Σ_2 as a function of Σ_1 are shown in Figs. 4(a) and 4(b), respectively. The range of Σ_1 for samples with smooth curves is less than two de-

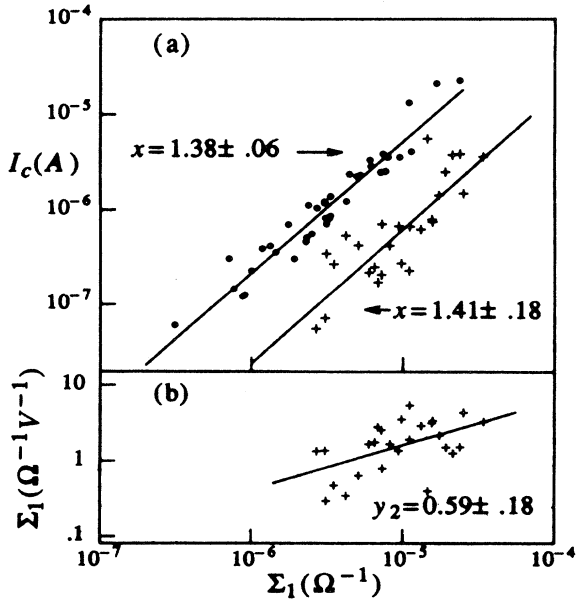

FIG. 4. (a) Plot of current threshold, I_c vs linear conductance. Σ_1 for smooth curves (+ symbols) and for curves having steps (solid points); (b) plots of second-order conductance, Σ_2 vs Σ_1 for smooth curves. The slopes are indicated in the figure.

cades. Nevertheless, in view of Eq. (5), we estimate the exponents from the slope in the Fig. 4(b):

$$x=1.41\pm0.18,\quad y=0.59\pm0.18\ . \tag{7}$$

This is the first determination of the exponents x and y in a 3D system. It is interesting to note that the value of x in 3D is same as that in 2D [Ref. 8a] within experimental errors.

Figure 3(a) shows the I-V characteristics (type B) of a sample of linear resistance 1 MΩ. As in a previous case, the characteristic is linear at low bias. However, unlike a previous case, the onset of nonlinearity is typically marked by the appearance of a small reversible step in the I-V curve that corresponds to a sharp minimum in the resistance R [Fig. 3(b)]. The threshold current I_c is 0.2 μA. R was obtained by point-by-point derivation of the I-V curve.[24] It is to be noted that, in this method, for given uncertainties in the measurements of V, the closer two successive values of currents are, the more uncertain R tends to be. An estimate of error in R is also shown in the Fig. 3(b). R-I curves obtained in this manner were, in general, noisy. However, considering the fact that the points in the I-V curve after a step shift as a whole parallel to the I-axis to higher values of current, it is believed that the structure in R-I is genuine and beyond uncertainties in data. The first such step is always preceded by a linear regime and thus marks the onset of nonlinearity, indicated by arrows [inset in Figs. 3(a) and 3(b)]. Since data became more noisy with increased current through a sample, it was difficult to draw definite conclusions about higher steps. There is a second step at about $i\sim0.8$ μA in Fig. 3(a) characterized by sharp drop in dV/dI. But the subsequent behavior of R showed wide fluctuations and the existence of minima is not clear. Steps are observations near percolation threshold. The sharpness of the minimum in R can be gauged from the fact that the width in current between the onset and the minimum is only about 150 nA. To see whether the structure of carbon particles plays any particular role in giving rise to steps, we repeated the experiment with a sample made of wax and graphite powder. The I-V characteristic was found to exhibit the same kind of structure as that of carbon powder. Therefore, we conclude that at least morphological structure of conducting particles is not responsible for such steps.

The current threshold I_c for characteristics of these types was found out quite easily from either the I-V or R-I curves. The plot of I_c versus Σ_1 is shown in Fig. 4(a). It is seen that I_c scales with Σ_1 as $I_c\sim\Sigma_1^x$, where x is given by

$$x=1.38\pm0.06\ . \tag{8}$$

Note that the value of x in this case is same as that in the previous case of smooth curves. It is seen from Fig. 4 that samples with high linear resistances generally have structures in their I-V curves. Samples with low resistances possess smooth I-V curves. The boundary line, though somewhat fuzzy, appears to be roughly near 120 kΩ. The sample in Fig. 3 has the resistance minimum at about 0.6 MΩ. We found that, if a quantity $\Delta\Sigma$, defined as the difference between Σ_1 and the maximum conductance just after the onset was plotted as a function of Σ_1, the data appear not to be incompatible with a power-law behavior. If $\Delta\Sigma\sim\Sigma_1^z$ then z is estimated to be about 1.3.

To discuss the above results, we should first consider the material picture. Apart from being a continuum, the carbon-wax system is different from the ideal random-resistor network[11] in that the system even above p_c does not appear to have a single entity (e.g., carbon) percolating by itself. In the case of carbon-polymer[15,19] and thick-film resistors,[25] it has been concluded that the conducting backbone is made of conductors (carbon chains or aggregates) broken by thin layer of insulating components (bridges). We expect the same to also be true in the present carbon-wax system. In such situations one would expect that tunneling[15,19,25] across thin insulators will constitute an additional mode of conduction mechanism, besides the usual conduction through conducting material (carbon). This is supported by the temperature variation data[19b,26,27] that sample resistances increased by more than an order of magnitude when samples were cooled from the room temperature to that of liquid nitrogen. Thus, a sample may be considered a mixture of normal conductors (e.g., carbon) forming long chains but not yet percolating in the classical sense and tunneling junctions whose resistance range from very low values (small widths) to very high values (large widths). Obviously, sample resistance would be largely determined by the distribution of the tunneling junctions. In such situations, the percolation threshold cannot be given the same geometrical meaning as that in the ideal case.[11] Hence, the quantity, p_c strictly speaking, is best treated as a fitting parameter in the present case.

We will now consider the exponents for smooth curves

as given in (7). Let us first consider the NLRRN model using the node-link-blob (NLB) picture[28] for the percolating backbone. We write the *I-V* relation of a junction in the following manner:[7]

$$I_{\rm el}=\sum_n \sigma_n V_{\rm el}^n \ , \tag{9}$$

where σ_n is the nth order conductance of an element. The current through a ξ channel (ξ is the correlation length), considering only "links," is given by (sample dimension $>\xi$)

$$I(\xi)\sim \sum_n \sigma_n \left[\frac{V(\xi)}{\zeta}\right]^n , \tag{10}$$

where $V(\xi)$ is the voltage across a length ξ and ζ is the chemical length $\sim(p-p_c)^{-t+\nu(d-2)}$. On the other hand, if I and V are sample current and voltage, respectively, we have

$$I(\xi)\sim I\xi^{d-1}, \quad V(\xi)\sim V\xi \ , \tag{11}$$

We note from Eq. (11) that current through a channel increases tremendously as the percolation threshold is being approached. This results in the enhancement of nonlinearity near p_c. Using these, we obtain following expressions for exponents:[29]

$$x\le\frac{\nu}{t}(d-1), \quad y\ge 2-\frac{\nu}{t}(d-1) \ . \tag{12}$$

In 3D, $x\le 2(\nu/t)=0.86$ and $y\ge 2[1-(\nu/t)]=1.14$. These values are clearly incompatible with those in (7). The expression for x in 2D has been previously obtained by Gefen *et al.*[8a] Within DRRN it has been shown, using general scaling arguments,[8a] that

$$x\le 1+\frac{\nu}{t} \ . \tag{13}$$

In 3D, $x\le 1.43$. This is consistent with (7). However, the prediction becomes worse when we consider the results[8a,8b] on the basis of assumptions of the general analytic properties of conductance as a function of V. If the leading nonlinear term is $\sim V^b$, then it can be shown that $x=b/(b-1)$. For $b=2$, as in the present case, x is 2. Thus, an agreement on the value of x may be fortuitous. No expression for y is available. There is another problem with the DRRN model, it cannot explain the temperature dependence of resistance of carbon-wax samples. The behavior of sample resistance as a function of temperature in the DRRN model should follow that of conducting elements. Carbon in the present case is known to have a small negative temperature coefficient of resistance. But, as mentioned earlier, the resistances of carbon-wax samples increase by more than an order of magnitude when cooled from room temperature to that of liquid nitrogen. On the other hand, the NLRRN model naturally can explain this property if some individual elements are assumed to have similar properties.

Regarding the *I-V* characteristics (type *B*, Fig. 3) of high-resistance samples, it is not clear how to account for the outstanding feature of resonancelike structures in *R-I* curves. No such structure has been reported in gold films[8] or in other binary mixtures.[17,25] Neither NLRRN nor DRRN model[8] can generate such a structure. Within the latter model, the conductance as a function of applied bias, averaged over several thousand configurations simulated on a two-dimensional lattice, was found to be monotonically increasing.[8a] Indeed, there is no mechanism in the DRRN model that can cause the conductance to decrease at any stage. Once an insulating bond breaks, it remains broken. It will be interesting to find out the effects of replacing "resistors" in the above models with devices having more complex behavior, for example, tunneling junctions with resonant centers. Such a mechanism has been discussed previously in case of thick-film resistors[25] and is potentially capable of giving rise to structures like the observed ones. However, statistical properties of an ensemble of such resonant tunneling junctions are yet to be determined. Resonant centers usually arise due to impurities in the insulating medium that have energy levels near about those of tunneling electrons.

Particularly keeping *I-V* curves (type *B*) in mind, it may be pertinent to ask whether the onset of nonlinearities could be interpreted as some sort of breakdown phenomenon or not. Breakdown phenomena are basically random processes and its signatures (e.g., threshold current in present case) are often discrete in nature. We present in Fig. 5 some data in this respect in the form of histograms for samples with resistance within small bands. The ordinate is a number of samples and the abscissa is a normalized deviation in threshold current, i.e., $(I_c-I_m)/I_m$, where I_c is the threshold current of a

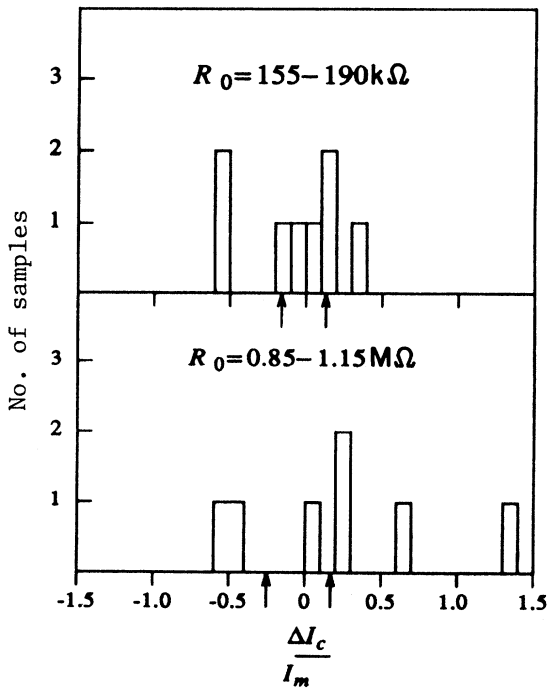

FIG. 5. Histrograms of threshold currents of samples in two resistance bands. See text for details.

sample and I_m is the threshold current for the median band resistance (173 kΩ and 1 MΩ) according to the fit in Fig. 4(a). The calculated values of threshold currents at band edges are also shown with errors. The total number of samples is small as it is difficult to prepare samples with predetermined resistance. Nevertheless, we see that fluctuations in values of I_c are quite large (note that a value of 1 in the ordinate means 100% deviation). It may be indicative of the random nature of the onset of nonlinearity. One implication of this is that averaging over large numbers of sample configurations in a simulation may obscure any structure that may be present.[30] One wonders whether the absence of an "avalanche effect" in the DRRN model[8a] is precisely due to averaging or not. It must be said, however, that, as far as our observation is concerned, an avalanche effect is not adequate. This will not produce any minimum in the R-I curve.

IV. CONCLUSIONS

In summary, we have studied the nonlinear I-V characteristics of carbon-wax samples near the percolation threshold. Two types of curves were observed. Some curves were smooth and others showed outstanding features of reversible resonancelike structures in R-I curves that are yet to be explained. The exponents for current threshold in the case of both types of curves and the exponent for second-order conductance in the case of smooth curves have been found in the present system. Results for smooth curves were analyzed within the frameworks of the two models suggested earlier. We showed that none of the models can fully account the experimental results obtained with the carbon-wax system. There is need to explain newly observed structures in some I-V curves. A successful model must be applicable for all samples with both types of I-V characteristics and have elements that will allow one to explain naturally why structures are present in certain I-V curves and absent in others. It may be significant that the exponent x is the same for both types of I-V curves. We believe that such a model has to take into account the underlying microscopic processes that may explain the absence of structures in I-V characteristics in gold films. Similar measurements in other systems (e.g., carbon polymers) could give further insight into electrical properties of such binary mixtures. Currently, efforts are underway to measure temperature dependence of resistivity and $1/f$ noise.

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