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Low-temperature transport properties of granular Pb films below the percolation threshold

A Gerber
Laboratoire Louis Néel, CNRS, 166X, 38042 Grenoble Cédex, France

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Abstract. We study the transport properties of disconnected Pb films from 77 down to 0.5 K, at zero and high applied magnetic fields. The conductivity between separated metal clusters is found to be dominated by voltage injection and not by thermal excitation of electrons at low temperatures. This explains temperature-independent resistance tails in both the superconductivity and the normal state of the films, observed at low temperatures in our and in other experiments. I–V characteristics were found to follow a power law $I \propto V^n$, $n > 2$, at low temperatures, consistent with a space-charge-limited current model for the conductivity.

1. Introduction

The onset of superconductivity in thin films has regained much interest since the recent experiments [1–3] on granular systems. It was found that the phase coherence associated with superconducting order can only extend over the entire two-dimensional film if the normal state sheet resistance R_n is less than a threshold value R_Q of about 6.5 kΩ. For $R_n > R_Q$, the sheet resistance below the bulk superconducting temperature T_c remained finite as $T \to 0$ and in some cases showed a so-called re-entrant behaviour with $R(T)$ increasing after an initial rapid decrease near T_c. It was speculated that these films show local superconductivity. On the other hand, all the samples with $R_n < R_Q$ seemed to undergo a true superconducting transition. Moreover, in the locally superconducting samples with $R_n > R_Q$, it was found that $R(T)$ exhibits temperature-independent flat tails at low temperatures and the associated residual resistance R_0 seemed to vanish exponentially quickly with $R_n - R_Q$. Various theoretical explanations have been suggested to explain this behaviour in terms of, for example, an array of Josephson junctions (see, e.g., [4]), vortex flow [5] or localisation theory [6]. However, the temperature-independent resistance tails at low temperatures have also been observed in high-resistivity samples which did not show local or global superconductivity [3]. Experiments on a range of three-dimensional granular aluminium samples [7] have shown a similar saturation of the resistance at the lowest temperatures persisting even in magnetic fields as high as 20 T.

The temperature dependence of the resistivity of granular disconnected metallic films is still another open and controversial problem. In general, the resistivity temperature dependence of insulating samples is expected to follow the relation $R(T) \propto \exp[(A/T)^\alpha]$. In amorphous superconductors [8] and granular mixtures Al–Al_2O_3 [9] the value

$\alpha = \frac{1}{4}$ is measured. It corresponds to the case where the conductivity follows the variable-range hopping process, first derived by Mott [10]. There is a large amount of experimental data on different materials [11, 12] which supports the exponent $\alpha = \frac{1}{2}$, for which an explanation has been given by Abeles *et al* [12] and also by Efros and Shklovskii [13], who introduced the correlation gap related to Coulomb interaction effects. The possibility that granular samples will show a crossover between the behaviour predicted by Efros and Shklovskii and the Mott variable-range hopping law has been discussed by Entin-Wohlman *et al* [14] and by Sheng and Klafter [15]. The predictions are quite different. Sheng and Klafter predicted the value $\alpha = \frac{1}{2}$ at high temperatures and a crossover to $\alpha = \frac{1}{4}$ as $T \rightarrow 0$. The theoretical model of Entin-Wohlman *et al* predicts an opposite transition from $\alpha = \frac{1}{4}$ to $\alpha = \frac{1}{2}$ as $T \rightarrow 0$. Both theoretical models have been supported by experiments. Glukhov *et al* [16] have reported a transition from $\alpha = \frac{1}{4}$ to $\alpha = \frac{1}{2}$ as $T \rightarrow 0$ in Sn–Ge films and Gilabert *et al* [17] have observed an opposite transition from $\alpha = \frac{1}{2}$ to $\alpha = \frac{1}{4}$ as $T \rightarrow 0$ in Pt–Al_2O_3 cermets.

A significant part of the existing experimental data on the resistivity temperature dependence of disconnected thin metallic films has been obtained using superconducting materials, such as Sn–Ge [16], $(SN)_x$ and In [18], Al–Al_2O_3 [9]. We have found that the resistance of films containing grains of superconducting materials can be enhanced significantly when finite clusters become superconducting. By applying a strong magnetic field we were able to distinguish between the transport properties of disconnected films with clusters in the superconducting and the normal state, and to give simple explanations for some of the existing discrepancies.

2. Experimental techniques

The samples were prepared by a conventional vapour deposition technique at a pressure of about 1×10^{-6} Torr. Lead was evaporated by an electron beam gun at a rate of about 20 A s^{-1} on a pre-evaporated layer of germanium 400 Å thick, the substrate being kept at room temperature. Lead films prepared under these conditions become continuous at an average thickness of about 110 Å. They have been shown [19] to have a two-dimensional percolating structure. An additional layer of Ge was evaporated immediately after the lead deposition in order to prevent any change in film characteristic due to reaction with gases. Nine samples, 12 mm long and 4 mm wide, were prepared simultaneously on the same substrate, their lead coverage and normal resistivity gradually varying owing to a different geometrical location. Measurements were carried out by a four-probe method when the samples were mounted in an exchange gas chamber or, alternatively, immersed in liquid helium. The magnetic fields used in the experiments were up to 6 T. *I–V* characteristics were measured using a Keithley 224 current source with DC currents above 5 nA, the voltage being measured with a Keithley 617 electrometer. A low-frequency cut-off filter was used in a part of the measurements to reduce a noise level in the circuit.

3. Experimental results

3.1. *R(T)* measurements

We have measured the resistivity temperature dependence of percolating lead films with various metal coverage. All samples were evaporated under similar conditions, their

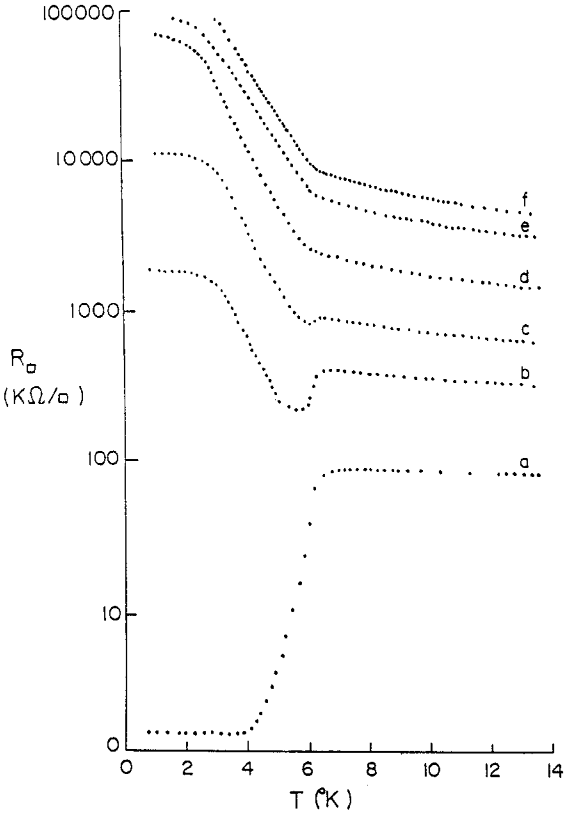

Figure 1. Some of the characteristic resistivity temperature dependences observed in lead films below the percolation threshold at low temperatures and zero magnetic field ($I = 1\ \mu$A).

room temperature resistivity varying from about 10 Ω/□ up to about 100 kΩ/□, which corresponds to about 1 Ω/□ up to a few gigaohms per square slightly above the critical temperature. When the metal coverage is high enough (above 50%), an infinite conducting cluster is developed across the sample. The resistivity temperature ratio is positive ($dR/dT > 0$, metal-like), and the sample becomes fully superconducting below T_c. Samples very close to the percolation threshold have weak links in the infinite cluster. These samples also reach zero resistivity below T_c but their behaviour above T_c is semiconductor like ($dR/dT < 0$). The maximum normal resistivity above T_c for which we have observed a zero resistivity in the superconducting state was 6.25 kΩ/□. The sheet resistivity of a two-dimensional square resistor network is equal to the resistance of a single bond. Assuming an isotropic distribution of junctions in our samples, this value confirms again the predicted value [20, 21], and previously observed in quenched condensed tin [2, 3] and other thin films [22–24] as the universal value of the critical resistance R_Q of a single Josephson junction, above which the junction remains resistive down to temperatures $T \ll T_c$.

We show in figure 1 some of the characteristic resistivity temperature dependences observed in lead films below the percolation threshold when measured at low temperatures and zero magnetic field. Samples slightly below the percolation threshold (curve a in figure 1) show an incomplete superconducting transition; their residual resistivity was found to be final and temperature independent down to the lowest temperatures that we measured. Samples further below the percolation threshold

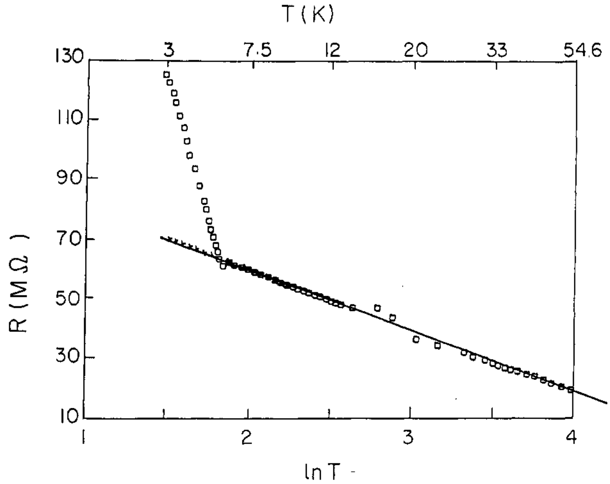

Figure 2. The resistance of a sample of the 're-entrant' group measured at zero magnetic field (□) and a 40 kG magnetic field (+), plotted against the temperature on a logarithmic scale.

(curves b and c in figure 1) show the so-called 're-entrant' behaviour [1, 25, 26]. The resistance of these films increases as the temperature decreases (insulator like) above the superconducting critical temperature; there is a resistance drop or local resistance minima near T_c, followed by a rapid increase upon further temperature reduction. Samples with an even lower metal coverage show an insulator-like resistance temperature dependence $dR/dT < 0$, with or without a 'kink' at T_c in the entire temperature range below room temperature (curves d–f in figure 1). At the lowest temperatures the resistance of all the samples that did not become fully superconducting was found to show a flattening-off towards the temperature-independent value.

At temperatures above 3–4 K, the resistivity of lead films in which 're-entrant' behaviour has been observed was found to follow a logarithmic behaviour of the form

$$R \propto -R_0 \ln T. \tag{1}$$

We show in figure 2 the resistance of a sample of this group measured at zero magnetic field and a high applied magnetic field plotted against temperature on a logarithmic scale. Note the very good fit to the $\ln T$ dependence and the very large change in resistance. In this case the resistance varies by about 350% over a decade change in temperature. Systematic measurements were performed at temperatures below 77 K; the logarithmic dependence can be seen from this high temperature down to temperatures below T_c if measured at a high magnetic field. The similar huge logarithmic change in the resistance was observed in granular niobium nitride cermet films [27]. The origin of this logarithmic behaviour is unclear. There have been many observations of logarithmic variations of the resistance in disordered and reduced dimensionality materials over the years, particularly in systems exhibiting weak localisation and/or electron–electron interaction effects. Such behaviour has been observed in some granular films [11, 28, 29]. A common feature of these systems is that the logarithmic temperature dependence is very weak and occurs over a limited temperature range. In weak-localisation theory, the total change in resistance over a decade of temperature is

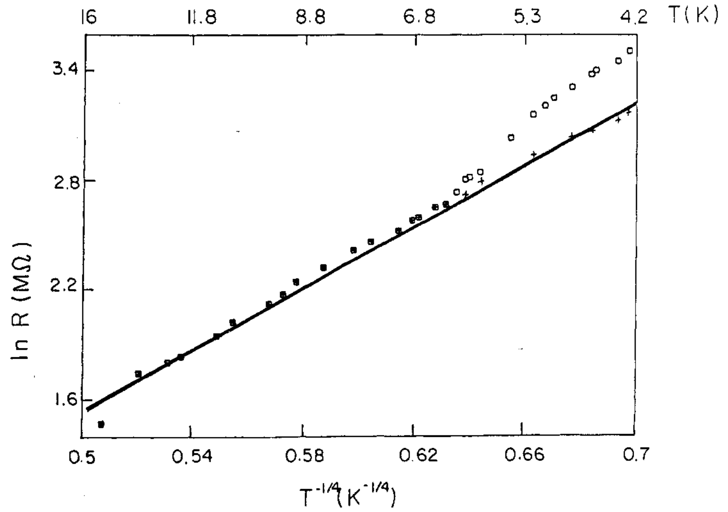

Figure 3. The logarithm of the resistance plotted against $T^{-1/4}$ of a lead film with room-temperature resistance of 23 kΩ/□, measured at zero magnetic field (□) and a high magnetic field (+).

typically of the order of a few per cent or less. The lead samples studied here show resistance variations two orders of magnitude larger than that.

Samples with a lower metal coverage and a higher room temperature resistance were found to follow a resistivity temperature dependence of the form

$$R \propto \exp(T_0/T)^{1/4}. \qquad (2)$$

This corresponds to the case where the conductivity follows the variable-range hopping process [10]. Figure 3 shows the logarithm of the resistance against $T^{-1/4}$ of a sample with a room-temperature resistance of 23 kΩ/□, measured at zero magnetic field (open squares) and a high magnetic field (crosses). The relation remains correct down to temperatures below T_c if measured at a high magnetic field.

No good fit to a single simple function was found for samples further below the percolation threshold with a room-temperature resistance above about 32 kΩ/□.

In spite of the different temperature dependences at high temperatures, one common feature was observed in all the lead samples studied: flattening-off of the resistance at low temperatures (figure 1). Similar temperature-independent resistance tails have been observed [3, 7] in quenched tin, lead and gallium films.

Investigation of the samples below the percolation threshold in a transmission electron microscope confirms a fractal morphology with finite clusters only. The grain size is approximately 200 Å and clusters sizes range up to a few micrometres slightly below the threshold. Intercluster distances can be as small as a few tens of ångströms. The conductivity in this case is dominated by quasiparticle tunnelling. In the zero-temperature limit no quasiparticle current can pass the superconductor–insulator–superconductor (s–i–s) junction if the voltage V applied across the junction is less than $2\Delta/e$ (Δ is the superconducting energy gap). At $V = 2\Delta/e$ the current increases sharply, since the electrons on the left side of the junction can tunnel into the empty states above the gap on the right side of the junction. If the metal clusters across the junction are in the normal state (n–i–n junction), the junction resistivity is ohmic at low values of the applied voltage V, i.e. the current is linearly proportional to the applied voltage. Therefore, for $V < 2\Delta/e$ the effective resistance of the quasiparticle tunnelling across a single junction is higher when the metal clusters are in the superconducting state than in their normal state. The superconducting energy gap reduces the probability that a single electron tunnels between the finite clusters and increases significantly the total sample's resistivity.

We present in figures 2 and 3 the typical DC resistance temperature dependence of a lead film below the percolation threshold measured in a zero magnetic field (open squares) and 40 kG magnetic field (crosses), respectively (at which superconductivity is quenched). The zero-field and the high-field curves coincide at high temperatures above 7 K and deviate strongly below the superconducting transition temperature (essentially that of bulk lead). One can see that the resistance of the disconnected films measured at zero magnetic field (superconducting clusters) is significantly higher than that measured at high magnetic field (normal clusters).

The magnetoresistance of the films measured at temperatures above T_c was found to be field independent. If measured at temperatures below T_c, the resistance of the samples was found to be maximal at $H = 0$, then gradually reduced with the magnetic field increase and saturated to the magnetic field independent value, when the sample became normal [30]. While the return to the normal state of the infinite cluster above the percolation threshold is related to the loss of the superconducting coherence length, we relate the opposite behaviour of the magnetoresistance below the threshold to the progressive reduction of the order parameter in the finite clusters. Below threshold, the conductivity of the sample is dominated by intercluster quasiparticle tunnelling. The conductivity in the normal state (n–i–n junction) is then larger than when the clusters are superconducting (s–i–s junction), owing to the opening of the superconducting energy gap. The reduction in this gap by the applied magnetic field leads to the progressive reduction in the resistance to its normal state value.

We show in figure 4 the resistance of the sample with a room-temperature resistance of 6.75 kΩ/□ measured at a zero magnetic field (open squares) and a high applied magnetic field (crosses) as a function of temperature, plotted on a logarithmic scale. When the resistivity was measured at a high magnetic field no 're-entrant' or 'kink' behaviour was observed, and the curve was smooth (see inset). However, the resistances measured at both zero and high magnetic field are found to become temperature independent at low enough temperatures, which indicates that this behaviour is caused by the normal rather than by the superconducting properties of the films.

3.2. I–V characteristics measurements

The I–V characteristics of the films have been measured, and the first conclusion drawn is that using the term 'resistance' is meaningless at low temperatures. The resistance at low temperatures becomes strongly non-ohmic. A typical dynamic resistance measured at 4.2 K as a function of the voltage applied across the sample is shown in figure 5. The ratio of the zero-bias to the high-voltage resistance can be as large as an order of magnitude and increases when the temperature is lowered. The I–V curve can be described by

$$V \propto I \qquad \text{for low currents} \qquad (3a)$$

$$V \propto I^m \qquad \text{for high currents} \qquad (3b)$$

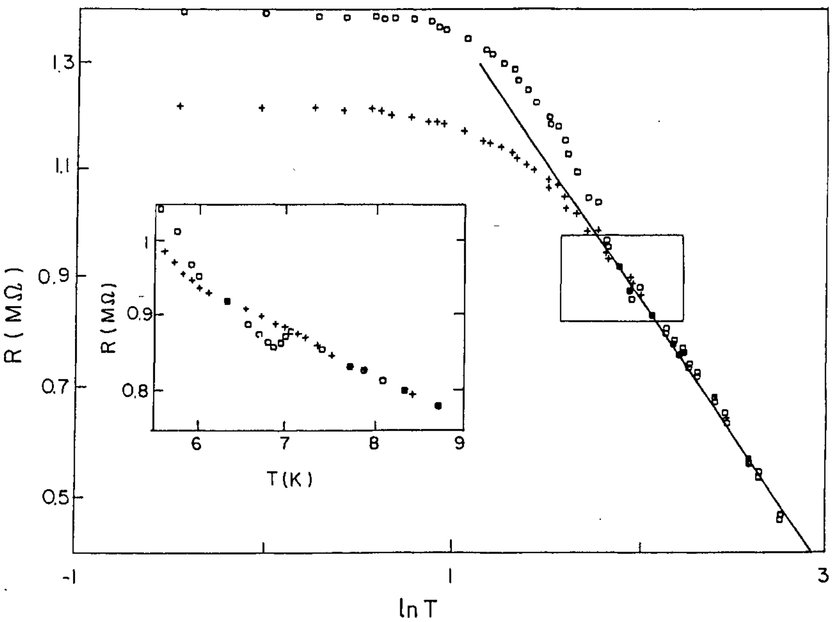

Figure 4. The resistance of a sample with room-temperature resistance of 6.75 kΩ/□ measured at zero magnetic field (□) and a high magnetic field (+), as a function of temperature down to $T = 0.6$ K ($I = 1\ \mu$A).

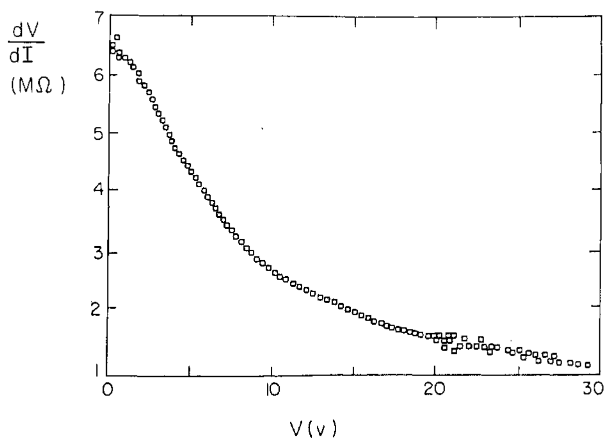

Figure 5. Typical dynamic resistance dV/dI of a disconnected lead film measured at 4.2 K as a function of the voltage applied across the sample.

where $m < 1$ is a temperature-dependent constant. Figure 6 shows a typical ln I–ln V dependence of a disconnected lead film measured at different temperatures above and below T_c. The crossover current between the ohmic and non-ohmic regions decreases quickly when the temperature is lowered and, for high-resistivity samples, is below 5 nA, the lowest current that we used for measurements, at 1.6 K. The values of m, measured

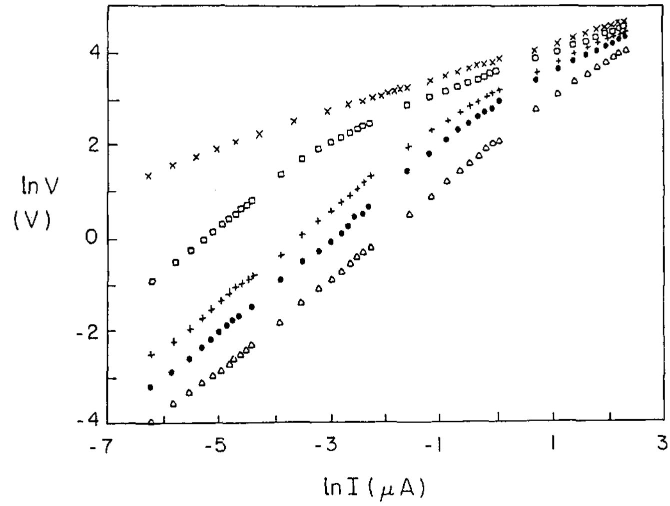

Figure 6. Typical ln I–ln V characteristics of a disconnected lead film measured at different temperatures above and below T_c ($H = 0$): △, 16.5 K; ●, 8.4 K; +, 6.5 K; □, 4.2 K; ×, 1.6 K.

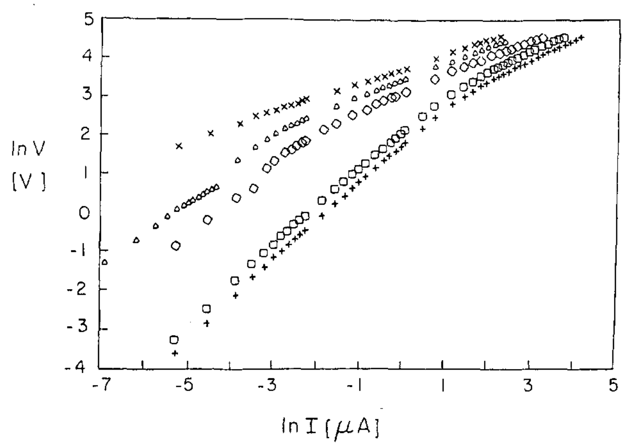

Figure 7. ln I–ln V characteristics of different disconnected lead samples measured at 4.2 K ($H = 0$).

in the non-ohmic region at temperatures of 6.5 K, 4.2 K and 1.6 K are 0.482 ± 0.003, 0.412 ± 0.002 and 0.340 ± 0.002, respectively. A similar power-law behaviour was observed when the resistivity was measured at a given temperature below T_c under different applied magnetic fields. The resistance measured at a given current through the sample reduces with the field increase, but a power-law dependence was observed at any given field both below and above the critical one. Figure 7 shows the ln I–ln V dependences of different samples at 4.2 K. The values of m, measured in the non-ohmic region, decrease with increase in the sample's normal resistivity and are 0.487 ± 0.003, 0.449± 0.003, 0.393 ± 0.002, 0.379 ± 0.002 and 0.331 ± 0.001, respectively. The value of the ohmic–non-ohmic crossover current depends on the sample and is higher for the samples with higher metal coverage and lower normal (room-temperature) resistivity. The fit to the ln–ln dependence is extremely good in all the samples measured.

4. Discussion

Granular lead films below the percolation threshold consist of finite clusters of random shapes and sizes separated by insulator gaps, the width of which varies from tens to hundreds of ångströms. Electrons can flow from one cluster to another either by direct tunnelling from one isolated cluster to the next through the germanium layer, or by intermediate hopping via the localised trapping centres.

Electrons are injected into the insulator both by thermal excitation and by an applied electric field. The low-field regime is when the voltage difference ΔV between neighbouring metal grains is much smaller than kT/e. Thermal excitations are therefore the main mechanism responsible for charge-carrier generation. As the temperature is lowered and the magnitude of the electric field is increased, $e\,\Delta V$ becomes comparable with or greater than kT. Field-induced tunnelling replaces thermal excitation as the dominant process of charge-carrier generation. This high-field electrical resistivity is voltage dependent. Abeles *et al* [12] predicted a resistance–voltage dependence of the form $\ln R \propto 1/V$. The DC hopping current has also been shown to depend strongly on the electric field. Hopping theory predicts the general form

$$I(E) \propto \exp(BE^{a}) \tag{4}$$

where, according to Mott [31] and Hill [32], $a = 1$ and $B \propto T^{-5/4}$ but, according to Apsley and Hughes [33], $a = 2$ and $B \propto T^{-9/4}$. An increase in the hopping current with increasing field of the type (4) was also found by using ideas of percolation theory: Pollack and Riess [34] predicted $a = 1$, $B \propto T^{-1}$, Shklovskii [35] $a = 1/(1+\nu)$, $B \propto T^{-1/(1+\nu)}$ (where ν is the critical exponent of the correlation length L) and Van der Meer *et al* [36] $a = 2$, $B \propto T^{-9/4}$.

Our results do not fit any of these predictions over a sufficiently wide range of voltages. However, at low temperatures, the data fit extremely well a relation of the form $I \propto V^{n}$ with $n \equiv 1/m$ larger than 2 and depending on sample and temperature. Similar behaviour has been observed by Milgram and Lu [37] in discontinuous chromium films. They proposed a model of conductivity in discontinuous thin metal films based on the concept of space-charge-limited currents [38].

5. Space-charge-limited model of conductivity

The principal charge-transfer mechanism is hopping via the localised trapping centres between metal particles. The existence of trapping centres between the particles has been suggested by Hill [39] and Herman and Rhodin [40]. The origin of the trapping states is believed to be associated with the surface of the substrate. Isolated metal atoms and defects on the substrate may also act as trapping centres. The number of field-induced charge carriers depends on the voltage while the number of excited charge carriers is independent of applied voltage. At very low temperatures, the number of thermally excited charge carriers will approach zero. At high fields, the field-induced charge carriers will dominate the total current at any temperature.

At zero temperature and near-zero field, there will be no conduction current, since all the localised states below the Fermi level are filled and no charge can transfer from one state to another by tunnelling.

If the applied field is high enough, charges in the metal particles can be injected into the localised states in the insulator (substrate). The space charge Q which can be forced into the insulator is given by

$$Q = CV \tag{5}$$

where C is the capacitance and V is the voltage difference between the two metal particles. If charges can move in the insulator by a hopping process via the localised states, the space-charge-limited current I_s is given by

$$I_s = Q/t = CV/t \tag{6}$$

where t is the charge transition time between two localised states. In a trap-free insulator the transit time of the charge Q between two electrodes is given by

$$t = d/E\mu \tag{7}$$

where E is the electric field in the insulator and μ is the drift mobility. The space-charge-limited current is then $I \propto V^2$. If only shallow traps, i.e. traps lying close enough to the conduction band, are considered, the same proportionality will be obtained, but the drift mobility now becomes the product of the drift mobility for free electrons and the fraction of the total space charge which is free. The drift mobility must be reduced by the same fraction. The value of this fraction is determined by the number and depth of traps and is independent on the applied voltage. In the case of trapping states which are exponentially distributed, i.e. if the density n of trapping states per unit energy, near the Fermi level can be defined [38] by

$$n = N \exp(E/kT_0) \tag{8}$$

where N is a constant, E is the energy with respect to the bottom of the conduction band of insulator and T_0 is a characteristic temperature which describes the trap distribution, the voltage dependence of space-charge-limited current is given, for $T < T_0$, by

$$I_s \propto V^{T_0/T+1}. \tag{9}$$

For $T_0 < T$ this reduces to the case of shallow traps where the exponent of V is 2. Small values of T_0 lead to a trap distribution that varies rapidly with energy, while large values of T_0 approximate a slowly varying trap distribution.

Space-charge-limited currents at $T < T_0$ increase as the square or some higher power of the voltage. Ohmic currents increase linearly with voltage. One would expect, therefore, that for any finite conductivity there would be a range of currents and/or voltages near zero for which the ohmic currents would predominate. For currents and/or voltages higher than some critical value, space-charge-limited currents would predominate. The critical value at which this transition from linear to power-law I–V dependence takes place should increase as the normal conductivity of the film increases. This prediction is also clearly observed in our experiments and is shown in figure 7.

6. Conclusions

Two parameters have been found to play an important role in the transport properties of granular disconnected metallic films. The resistance of films composed of the grains of superconducting materials can be enhanced significantly below the superconducting transition temperature owing to the sensitivity of quasiparticle tunnelling to the state of finite clusters. This is especially pronounced in films with large grains in which the charging effects are small. Application of magnetic fields suppresses superconductivity and recovers the intrinsic normal state resistivity.

The second important conclusion is that for any small measuring current there is a finite temperature at which voltage-induced conductivity becomes comparable with and then dominates the thermally excited conductivity. In this regime the resistance becomes temperature independent and the *I–V* characteristic becomes non-ohmic. In our experiments with lead films below the percolation threshold, the crossover to voltage-dominated conductivity occurs at temperatures of a few kelvins for the lowest measuring currents we used. The *I–V* dependence was found to follow a power law, which is consistent with a space-charge-limited current model.

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