Anomalous Resistivity Drops in YBCO above 230K

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ABSTRACT

We have prepared nonsuperconducting Y-Ba-Cu-O samples that show anomalous drops in resistivity at temperatures above 200K. We believe that these transitions are related to the granular structure of the samples and to the effect of current on the weak links inside the bulk samples.

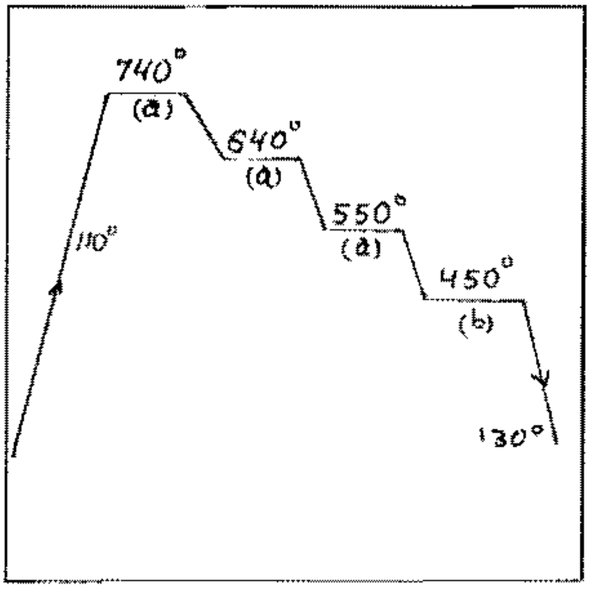
INTRODUCTION

Since the discovery of superconductivity in the ceramic compound $YBa_2Cu_3O_7$ with Tc above 90K [1] many unconfirmed reports kept coming out which claimed the observation of resistive drops at temperatures much higher than 90K. Even though measurements of this type have been usually dismissed as erroneous, something exacerbated by the non-repeatability of these measurements, many people are quick to announce the discovery of "very high" Tc superconductivity based solely on the conductivity measurement.[2]

Recently, we have been seeing such anomalies in YBCO samples prepared by standard "solid state reaction" methods but subjected to varying heat treatments. Depending on the thermal processing history of these samples, sharp resistive transitions have been seen at temperatures from 200K to 250K. We are convinced that these anomalies are not signatures of superconductivity, but rather are resulting from a current-dependent change in the microstructure of the samples as a function of temperature.

EXPERIMENT

Bulk materials were prepared by mixing Y_2O_3 (99.999%), BaO (99.999%) and CuO (99.999 %) in exact ratio 1:2:3, dissolved in concentrated nitric acid , and the mixture was evaporated to dryness to a blue material. The blue solid material was divided into two batches: one batch, sample II, was kept in a dessicator. The other sample, I, was fired in a furnace tube under air at 940° C for 50 hours with intermediate grinding. Finally the sample changed to black. The black solid was cooled to room temperature, ground into a fine powder, pressed into pellets 10 mm in diameter and 1.5 mm thick then annealed by heating under oxygen for one hour at 740° C, then cooled slowly over 6 hours to room temperature. Sample II was obtained by re-annealing sample I at 740° C in an oven as follows: It was heated for 12 hours, then the temperature was reduced to 25° C as is schematically shown in figure 1:



Samples for conductivity measurement were cut into 2x2x10 mm^3 rectangles and contacts for "4-probe measurement" were indium soldered to the samples using thin copper wire. They were then placed on the cold finger of a closed-cycle helium cryostat and cooled down to 14K. The data was collected using and IEEE-488 based data acquisition system which relied on a Keithley 6 1/2 digit model 196 programmable multimeter and where temperature was controlled using a silicon diode with +/- 0.1 K accuracy as a sensing element.

RESULTS

Figure 2 shows a typical resistance vs temperature curve for an YBCO sample that was cut from sample I above. The curve shows a dramatic drop in resistance near 250K when the current passing through the sample is about 1.7mA. For smaller currents , 50 μA for example, the resistance continued to increase to the lowest temperature achievable by our closed-cycle helium cryostat (14 K without a radiation shield) as shown in figure 3. To test the I-V characteristic of this sample at 14 K, a plot of I vs V is shown in figure 4. This curve is a stark reminder of single electron tunneling behaviour and suggests the following model for explaining the anomalous behaviour of these materials:

Depending on their thermal history, the YBCO samples have a varying degree of granularity. The SEM picture of one of our samples (figure 5), shows the existence of a distribution of granules of various sizes separated by micro-bridges and weak links. We suggest the following explanation: At high current as the temperature decreases, these bridges break down allowing for the dramatic drop in resistance. To show that this is the case, we plot in figure 6 the resistance versus current for the sample whose SEM image is shown in figure 5. Instead of increasing with current (or at least showing a current independent resistance), the sample's resistance dramatically decreases with increasing current!

We have not yet performed any magnetic measurements on the samples to prove the existence or lack thereof a superconducting phase , but simple levitation experiments indicate that if a superconducting phase exists, then it should be very small indeed! This is indeed an oxide material which has a current-induced transition in its resistivity as a function of temperature. The resistive transition itself is completely reversible and there is no evidence of any hysteretic behaviour other than that accounted for the positions of the sample and temperature sensor relative to cryostat heater!

CONCLUSION

We suggest therefore that reports of superconductivity at very high temperatures (much higher than 125K) can actually be verified by testing the current dependence of the resistivity. We conclude that changes in the microstructure of these samples are causing these anomalies rather than a superconducting phase change.

We wish to thank M. Seely for a lot of technical assistance, and also Y. Salamin, A. Shawabkeh, M. Seely, and E. Sader for many helpful discussions. This work was supported by the Birzeit university research fund and a generous grant from OPEC.

1. M.K.Wu, J. Ashburn, C.J. Torng, P.H. Hor, R.L. Meng, L. Gao, Z.J. Huang, Y.Q. Wang, and C.W. Chu, **Phys. Rev. Lett.**, **58**, 908(1987).

2. Shahnaz Malik, M. Mohammad, and A. Y. Khan, "Superconducting Ceramics with zero resistance at and Near Room Temperature", High T_c Update, **6**(11), June 1992.

FIGURE CAPTIONS

Figure 1: Typical thermal history of an YBCO sample that shows anomalous resistive drops at temperatures above 250 K.

Figure 2: Resistance versus temperature for the sample whose thermal history is displayed in figure 1.

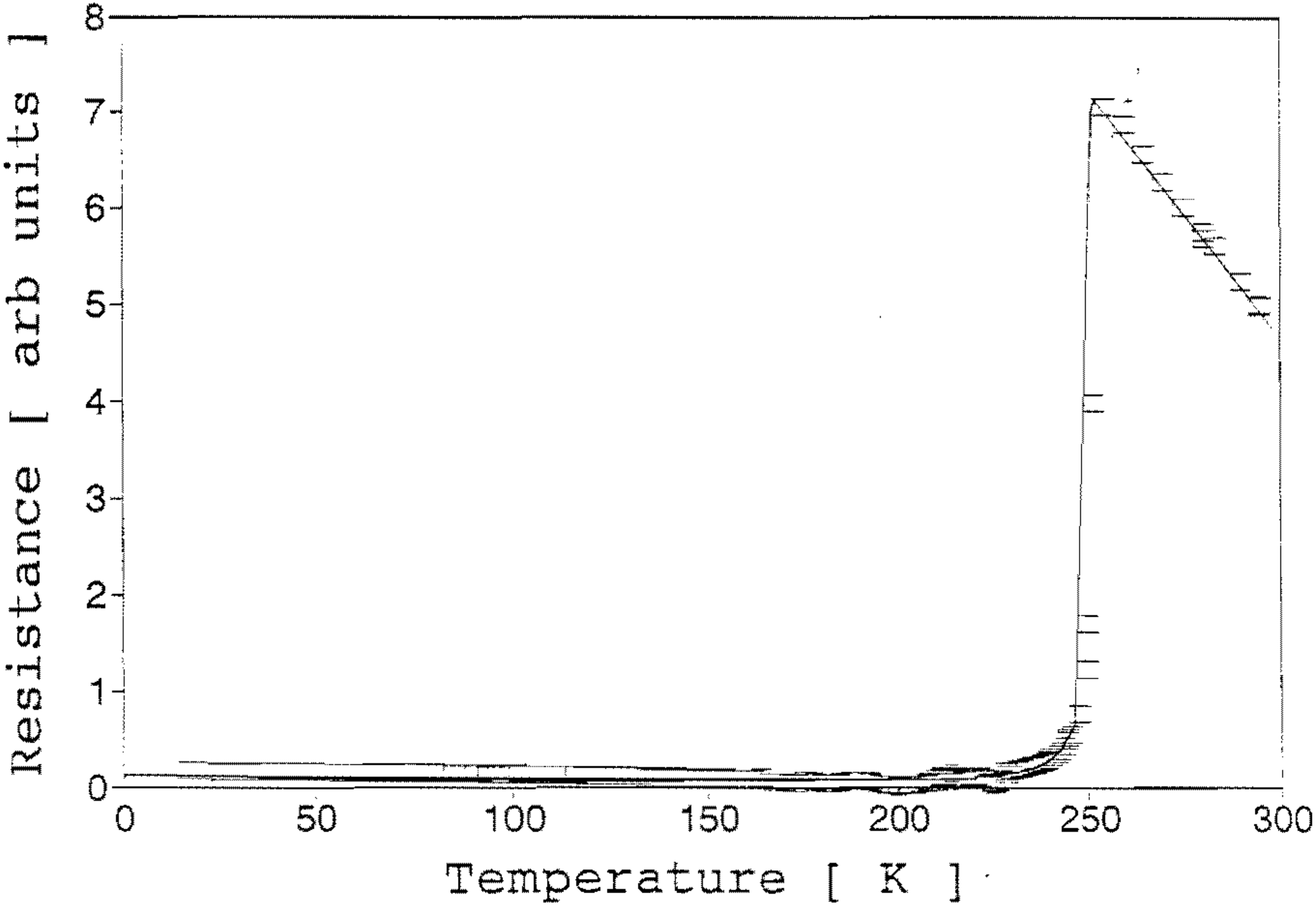
Figure 3: Current dependence of R(T) for the YBCO samples. Curve b shows that at low currents the anomalous resistivity drops are absent and they only appear when there is enough current to allow for micro-bridges to break down.

Figure 4: SEM picture ....

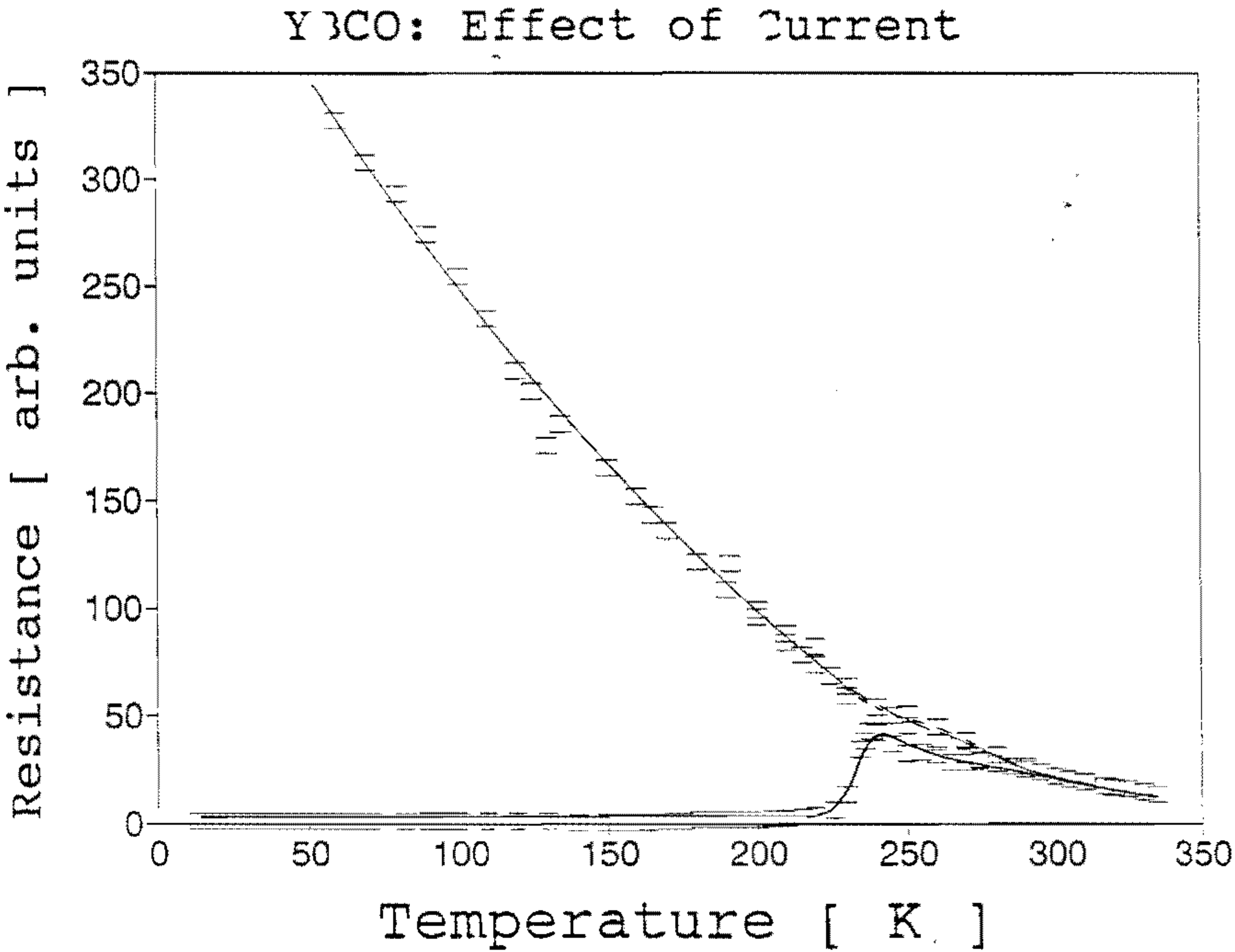
Figure 5: I-V characteristic of the sample showing typical N-I-N behaviour.

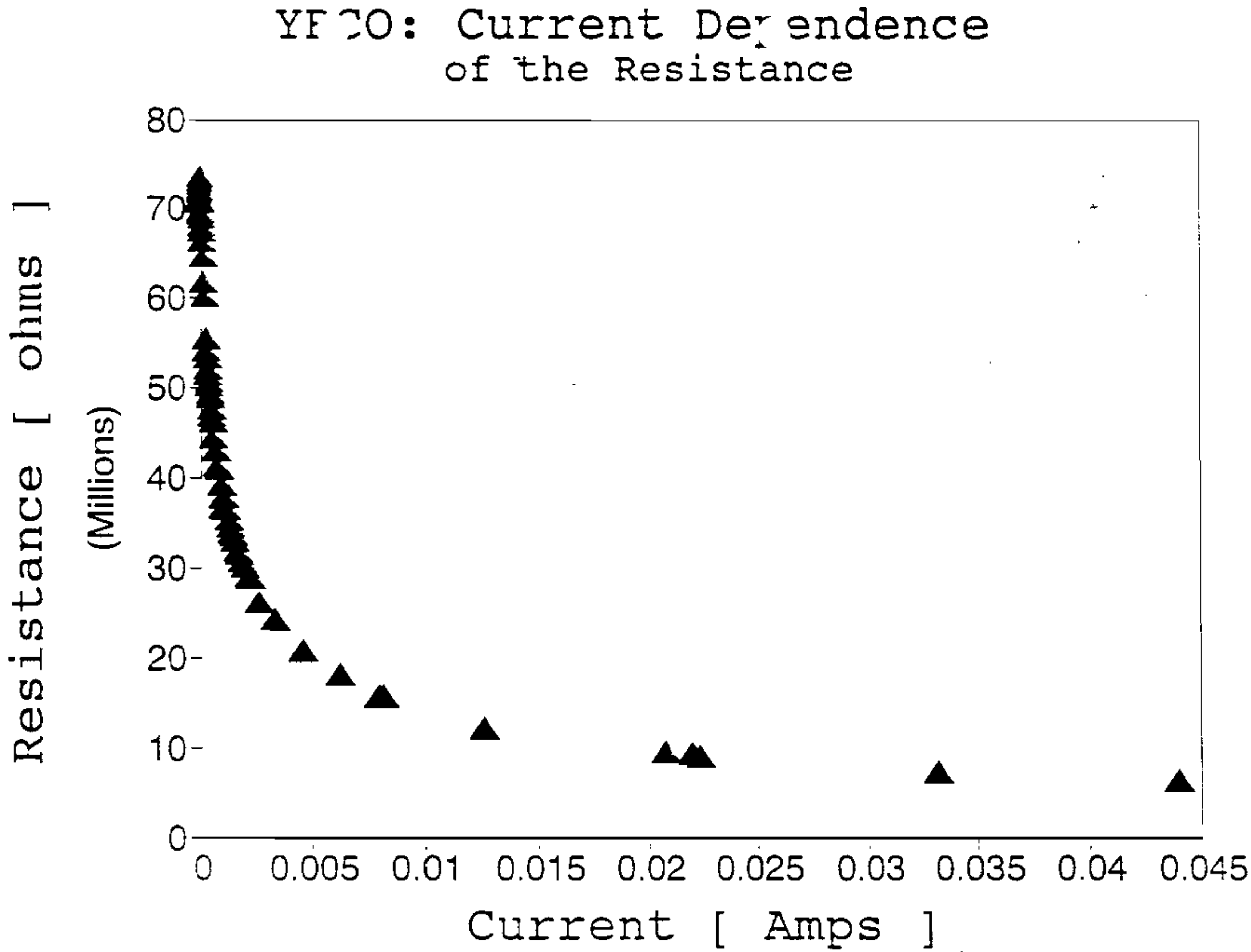
Figure 6: R-I curve showing a current-induced drop in resistance.

Y Ba Cu O

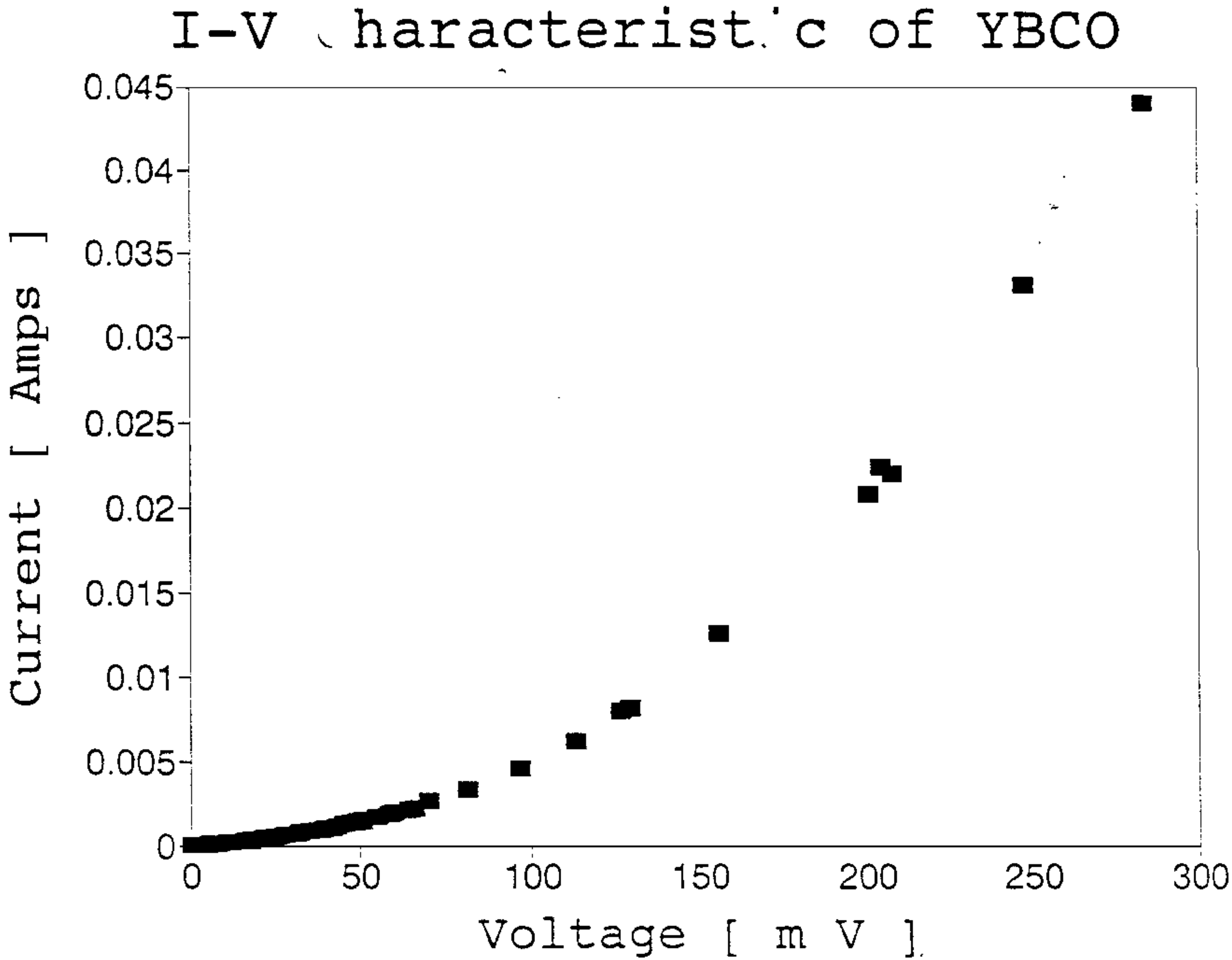


Resistance [arb units]
0
1
2
3
4
5
6
7
8
0
50
100
150
200
250
300
Temperature [K]


YBCO: Effect of Current
Resistance [arb. units]
0
50
100
150
200
250
300
350
Temperature [K]



YBCO: Current Dependence
of the Resistance
Resistance [ohms]
(Millions)
80
70
60
50
40
30
20
10
0
0
0.005
0.01
0.015
0.02
0.025
0.03
0.035
0.04
0.045
Current [Amps]


I-V Characteristic of YBCO
Current [Amps]
Voltage [m V]
0.045
0.04
0.035
0.03
0.025
0.02
0.015
0.01
0.005
0
0
50
100
150
200
250
300