

Scanning tunneling spectroscopy of a microcrystal of the high-temperature superconductor $\text{YBa}_2\text{Cu}_3\text{O}_{9-y}$

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A technique for producing a vacuum tunnel junction between the needle of a scanning tunneling microscope and the superconducting crystal $\text{YBa}_2\text{Cu}_3\text{O}_{9-y}$ is described. The distribution of the energy gap has been measured along surface areas $\sim 1 \mu\text{m}^2$ of microcrystals at 4 K.

The scanning tunneling microscope, which offers a resolution at the atomic level,¹ has made it possible to carry out several remarkable studies of the topography of conducting samples and of their various electronic characteristics.² The latter field of application of the scanning tunneling microscope is known as "scanning tunneling spectroscopy." Scanning tunneling microscopes are described briefly in Ref. 3. Scanning tunneling spectroscopy is obviously of major importance for research on superconductivity.

de Lozanne *et al.*⁴ have obtained profiles of electronic properties on a Nb_3Sn film, including a transition from a superconducting region to a normal region, at 4 K. They also obtained current-voltage characteristics of a tunnel junction between the needle of a scanning tunneling microscope and a superconducting sample at selected points. It did not prove possible to use the scanning tunneling microscope directly to study high-temperature ceramic superconductors; a surface layer 100–200 Å thick of the ceramic easily loses oxygen and goes nonconducting. As a result, when the needle is brought in contact with the sample to obtain a tunnel current, it touches the nonconducting layer.

In experiments^{5–7} in which a scanning tunneling microscope was used to study ceramic superconductors, current-voltage characteristics were obtained at various points on samples. These characteristics revealed significant differences in the energy gap from point to point. The tunnel current apparently flowed across a thin nonconducting layer and was accompanied by a parallel leakage current (more on this below). Scanning was not carried out for the reason stated above and also because the particular scanning tunneling microscopes which were used had a scanning amplitude of only ~ 1000 Å at liquid-helium temperature, or roughly equal to the thickness of the needle tip.

The scanning tunneling microscope used in the present experiments has a scanning amplitude $> 1 \mu\text{m}$ at a measurement temperature of 4 K.

The sample was a ceramic $\text{YBa}_2\text{Cu}_3\text{O}_{9-y}$, containing single crystals with sizes 20–60 μm , with a natural shiny surface and having the value $T_c = 92 \pm 2$ K. Before being immersed in the helium, part of the sample was cleaved in order to produce a surface as fresh as possible. Crystal faces suitable for study were sought by recording

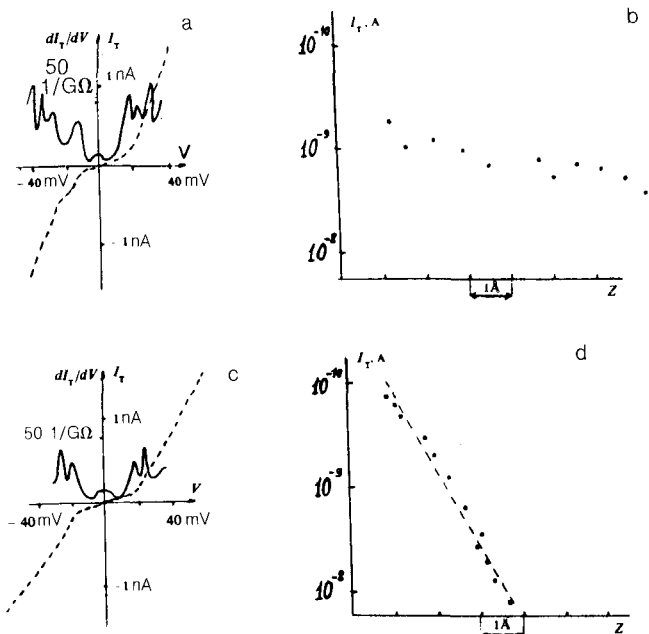


FIG. 1. a—Current-voltage characteristic of the first contact of the needle with the sample (dashed line) and its derivative (solid line); c—current-voltage characteristic and its derivative during a repeated contact; b—current versus the distance from the needle to the sample during the first contact; d—the same, during the repeated contact.

“coarse” topograms of the sample with a voltage $V \sim 6 \text{ V}$ on the needle, with a noise level $\sim 200 \text{ \AA}$, expressed in terms of the relief height.

The tunnel current from the needle to the point of the sample under study, I_T , was measured by the following procedure. At $V \leq 100 \text{ mV}$ the needle was brought close to the sample (at a point which was not touched) and immersed to a depth $z \gtrsim 200 \text{ \AA}$ (determined from the piezoelectric drive) into the nonconducting layer, until a current $I_T \sim 1 \text{ nA}$ was reached. We concluded that the needle made contact with the solid surface when the characteristic vibrational noise in I_T disappeared. The current-voltage characteristics obtained with the needle in this position were asymmetric; furthermore, they contain a significant parabolic component (a leakage current; Fig. 1a). (Similar structural features were observed on the I-V characteristics in Refs. 5–7.) The $I_T(z)$ profile recorded upon the first contact of the needle with the sample at $V = 200 \text{ mV}$ has the shape shown in Fig. 1b. The shape of this curve is quite different from the steep exponential curve characteristic of a tunnel current.

The needle was then withdrawn from the sample to $z \approx 200 \text{ \AA}$ and then brought back close to the sample, until a current $I_T = 1 \text{ nA}$ was reached. A needle displacement $z \approx 100 \text{ \AA}$ (according to the piezoelectric drive) was sufficient for this purpose. The apparent reason for the difference is a deformation of the needle and the sample during the first contact. The I-V characteristic and $I(z)$ now acquired the form char-

acteristic of a tunnel current across a vacuum gap (Fig. 1, c and d). The noise in I_T was of the usual nature. A repetition of this operation (at the same point on the sample) caused no further significant change in the characteristics of the contact.

The apparent explanation of these observations is that when the needle first makes contact, an oxygen-poor, nonconducting surface layer of the sample crumbles and is removed, exposing the superconducting part of the sample. There is no further loss of oxygen at liquid-helium temperature. When it is subsequently moved toward the sample, the needle approaches the surface of the superconductor without hindrance.

Distributions of the energy gap along the surface of a sample were measured by the following technique. The current-voltage characteristic of the tunnel junction at the given point on the sample was differentiated (Fig. 1c), and the distance between the maxima of the derivative was stored as a measure of the energy gap. The needle was then raised to $z \approx 200 \text{ \AA}$ and moved above the surface of the sample to the next measurement point. The procedure for producing a tunnel contact with a characteristic of the type in Fig. 1c was then repeated. This procedure was used over the entire surface area of the crystal under study, at 11×10 points, at steps of 1000 \AA . The steps could not be made smaller because of the resulting instabilities and scatter in the measurements, apparently because the needle tip, 1000 \AA in diameter, struck a part of the crystal surface which had been damaged in the preceding step.

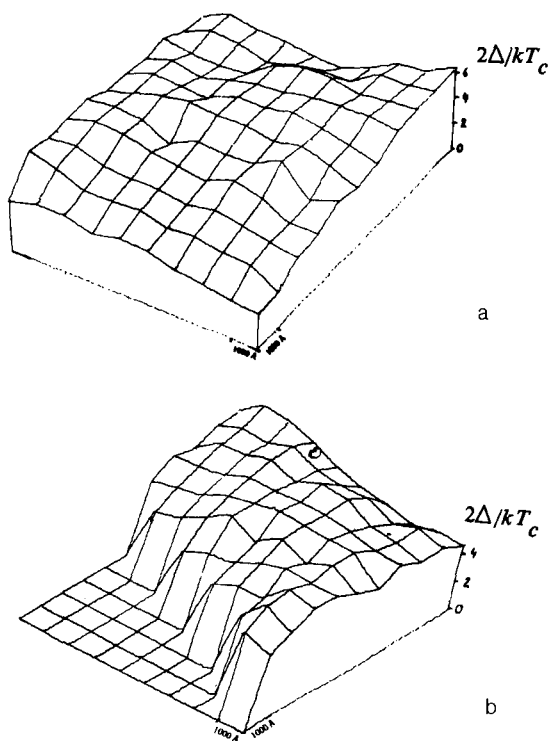


FIG. 2. Distribution of the energy gap. a—Over the surface of a superconducting microcrystal; b—over a part of a sample containing superconducting and normal regions.

Figure 2, a and b, shows results of measurements of the initial gap in two areas with sizes of $1 \mu\text{m}^2$. The size of the gap, expressed in units of $n = 2\Delta/kT_c$, is shown by the height of the points above the mesh point of the x, y coordinate system. The points are connected by straight-line segments to help depict the $n(x, y)$ surface. In Fig. 2a, the values of n range from 2.4 to 6.3; those in Fig. 2b range from 2.5 to 7; the error in the measurements is $\Delta n \approx 1$. The boundary between a superconducting crystal and a normal region of the sample falls in the field in Fig. 2b; the tunneling current-voltage characteristic here is a straight line, so we have $n = 0$.

In Fig. 2, a and b, we can clearly see valleys and ridges running along the $n(x, y)$ surfaces. This reproducibility of the results of measurements on several scan lines of the needle along the sample indicates that this measurement procedure is reliable. It also indicates a definite relationship between the size of the gap and structural features of the superconducting crystals; these features should be studied in parallel by other methods involving scanning tunneling microscopes and scanning tunneling spectroscopy. However, such studies will require developing a method for exposing the surface of the superconductor or saturating the depleted surface layer with oxygen, in order to make it possible to use the highest resolution of the scanning tunneling microscope.

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