Pinning Centers in YBa₂Cu₃O₇₋δ Films

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ABSTRACT: Pinning centers in epitaxial YBa₂Cu₃O₇₋δ films have been investigated by scanning tunneling microscopy and transport studies. An astonishing surface morphology has been found which includes a high density \( \approx 10^9 \) cm\(^{-2} \) of screw dislocations and nanometer-sized holes. The density of screw dislocations can be controlled by varying the growth conditions of the YBa₂Cu₃O₇₋δ films, allowing correlations between critical currents and screw dislocation density to be investigated. Films with higher screw dislocation densities are observed to have higher critical current densities and a slower drop of \( J_c(H) \).

1. INTRODUCTION

The high critical current densities of epitaxial films of high-\( T_c \) superconductors are caused by strong pinning with a spectrum of pinning energies [1]. To date, however, no comprehensive picture of the physical nature of the pinning sources has emerged. By imaging sputtered YBa₂Cu₃O₇₋δ films using scanning tunneling microscopy (STM) we have observed high densities of defects such as screw dislocations and small holes in the epitaxial films, as well as a substantial surface roughness [2,3]. In this paper, correlations of the screw dislocation density with the critical current densities of the films are reported and the role of pinning at the screw dislocations and at other defects is discussed.

2. SAMPLE PREPARATION AND MEASUREMENT TECHNIQUES

The \( c \)-axis oriented YBa₂Cu₃O₇₋δ films were grown by dc-hollow cathode magnetron sputtering on nominally (100) oriented SrTiO₃ substrates. As described in detail elsewhere [3], the sputtering parameters used were a sputtering pressure of 650 mTorr (Ar/O₂ \( \approx 2/1 \)), a plasma discharge of 150-180 V and 260-500 mA and a heater block temperature of 750-780 °C. The films have a typical thickness of 100-150 nm, a \( T_{c0} \) of \( \approx 87-88 \) K and a critical current density of \( J_c \approx 1.5-7 \times 10^7 \) A/cm\(^2 \) at 4.2 K in self-field.

The as-grown films were investigated with a homemade STM at room temperature in air using mechanically prepared Pt₀.₈Ir₀.₂₀ tips with a tunneling current of 10-20 pA [2,3]. Many images of each sample were taken at different locations and the screw dislocation density \( n \) of a film was derived by averaging over all images obtained from the sample.

Critical current densities were measured using 8 \( \mu \)m \( \times \) 100 \( \mu \)m bridges, patterned by conventional photolithography and wet etching. Each sample for \( J_c \) measurement was cut from the same polished substrate as the corresponding samples for microscopy and mounted for deposition on the heater block close to the samples for microscopy. During the \( J_c(H) \) measurements, done by a 4-point technique with a criterion of 1 \( \mu \)V, magnetic fields of \( \mu_0H \leq 8^T \) were applied perpendicular to the transport current and to the film surface (\( H/c \)). Data were taken starting at high fields, before measuring any data points the transport current was reduced to zero. On several samples the \( J_c \) measurements were repeated after cycling to 300 K. The scatter in the resulting data give a lower bound on the precision of the \( J_c \) values of \( \approx 10\% \).

3. RESULTS

The STM studies revealed several features of interest for pinning in these films: First, the films show an unexpectedly high density of dislocations (Fig. 1): the density of screw dislocations penetrating the film surface is typically around 10⁶ cm\(^{-2} \). Second, small features, about 5 nm \( \times \) 5 nm in size, which seem to be holes more than 20 Å deep (Fig. 2) are observed with an even higher density. Third, the films have a typical surface roughness (maximum height difference within one image, corresponding to 1/4 \( \mu \)m\(^2 \)) of 100-200 Å (Fig. 1). It is noteworthy that the same features have also been reported by other groups, even for films grown by laser ablation or for films grown on other substrates such as MgO [4,5].
Ig. 1. STM images of sputtered YBa$_2$Cu$_3$O$_{7-\delta}$ films grown at various temperatures (after [3]). (a) 750 °C; (b) 760 °C; (c) 770 °C; (d) 780 °C.

As described in detail elsewhere [3] we have found that the density of screw dislocations can be controlled by adjusting growth rate, growth temperature and substrate tilt from (100). As shown in Fig. 1, by varying those parameters, films with screw dislocation densities between $5 \times 10^7$ cm$^{-2}$ and $1.5 \times 10^9$ cm$^{-2}$ have been grown. Figure 3a shows the critical current density $J_c$ at 4.2 K in self-field for all films measured as a function of screw dislocation density together with a linear least squares fit. The scatter in the data arises from inaccuracies in the measurements of $J_c$ and $n$, but may also be caused by the wide span of deposition parameters used, which are expected to affect not only the screw dislocation density, but also other film properties such as the point defect concentration, which have some influence on $J_c$. 

Fig. 2. STM image of a sputtered YBa$_2$Cu$_3$O$_{7-\delta}$ film. The small dark spots are holes or insulating regions. The steps between the growth terraces are about one unit cell high.
Figure 3a indicates that for the samples investigated, a higher screw dislocation density accompanies a higher critical current density at 4.2 K in self-field. A high screw dislocation density also leads to a slower drop of $J_c$ with increasing magnetic field as shown in Fig. 3b. The magnetic field dependence of the critical current density and the volume pinning force are plotted in Figs. 4 and 5 for two samples selected on the basis of least squares fit calculations as being representative samples with small and large $n$. As these figures demonstrate, films with high screw dislocation densities may have a volume pinning force of more than a factor of two higher than those with a smaller $n$. Further transport data can be found in [3].

Fig. 3. (a) Critical current density at 4.2 K in self-field vs screw dislocation density for all films investigated. (b) Critical current density at 4.2 K and 0.5 T // c normalized to the self-field value vs screw dislocation density for all films investigated. In (a) and (b), the straight lines represent least squares fits; the data points numbered 1 and 2 refer to the samples presented in Figs. 4 and 5.

Fig. 4. $J_c(H)$ at 4.2 K of two characteristic samples with screw dislocation densities of 0.5 x $10^8$ cm$^{-2}$ and of 13.5 x $10^8$ cm$^{-2}$. The magnetic field was applied parallel to the c-axis, perpendicular to the transport current and to the sample surface.

Fig. 5. Magnetic field dependence of the volume pinning force for the two samples in Fig. 4.

4. DISCUSSION

There is general agreement in the literature that in epitaxial YBa$_2$Cu$_3$O$_{7-\delta}$ films pinning is characterized by a spectrum of pinning sites of different strengths. Pinning by the film surface, by the interface to the substrate and by the film edges has been reported [6]. These pinning centers do not seem to be strong enough to account for the $J_c$ values observed. The same applies to pinning at twin planes. Pinning by the layered structure of YBa$_2$Cu$_3$O$_{7-\delta}$ is not applicable to the configuration investigated. There will be some pinning due to the surface roughness of the films, as the areas between the growth spirals are low energy positions for the flux lines. But surface roughness is not expected to be the main source of pinning either, because the resulting pinning force will be small for
two reasons: First, due to the small slopes of the growth spirals, the potential recovers only gradually (see Figs. 1 and 2), and second, the flux lines may wander with ease in the valleys between the growth spirals. Possible strong pinning sites include dislocations and point defects. Pinning at dislocations may be strong if the flux lines are oriented parallel to the dislocation cores, which is the dominant orientation in our studies [3]. It has been suggested that dislocations provide strong elementary pinning [7, 8], but they were considered unlikely to be a major source because simple estimations show that dislocation densities of the order of the flux line densities are required, equaling 10^8-10^9 cm^{-2} in self-field, which seemed excessively high. Surprisingly, the screw dislocations observed in the STM studies have this density, indicating that they may be pinning sites. This idea is corroborated by the correlation between the screw dislocation density and the critical current density as shown in Figs. 3-5, but it is pointed out that the correlations give no proof for a casual relationship. Above \( \approx 0.1 \) T the flux line density greatly exceeds the screw dislocation density, and the screw dislocations cannot account for all the pinning required in that field range even if collective pinning is taken into account. Nevertheless, as shown in Fig. 5, the volume pinning force also increases with screw dislocation density in the high field regime, providing evidence that in this field regime defects correlated in growth to the screw dislocations are a main source of pinning. Defect candidates are point defects or edge dislocations. The latter may form at precipitates, or during the coalescence of misoriented growth spirals. For example, 10^{11} edge dislocations per cm^2 were observed in the coalescence of YBa\(_2\)Cu\(_3\)O\(_{7-\delta}\) nuclei on MgO substrates in the early stages of sputtered film growth [9]. Also, if the nanometer holes are of sufficient depth, they will be strong pinning sites.

5. SUMMARY

Using STM, screw dislocations with densities in the range of 10^9 cm^{-2} have been found in epitaxially grown, c-axis oriented YBa\(_2\)Cu\(_3\)O\(_7\) films. The density of screw dislocations is sufficient to account for the observed \( J_c \) at small magnetic fields, provided that they are effective pinning sites. Their density can be controlled by adjusting growth parameters such as temperature, growth rate or substrate misorientation. Transport measurements indicate that the critical current densities of samples with high screw dislocation densities are higher than current densities of samples with fewer dislocations.

The authors gratefully acknowledge helpful discussions with E.H. Brandt, M. Ferrari, T. Frey, P. Martinoli and S. Shinde as well as support from R.F. Broom, A. Catana and the Laser Science and Technology department at the IBM Zurich Research Laboratory.

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