Positron annihilation in the epitaxial superconducting thin-film $GdBa_2Cu_3O_{7-\delta}$ studied by using a pulsed positron beam

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(Received 20 February 1996)

The positron lifetime as a function of implantation energy was measured on the epitaxial superconducting thin film $GdBa_2Cu_3O_{7-\delta}$ at different temperatures. The coexistence of both shallow and deep positron trapping centers was observed in the film. The shallow trapping centers include the screw dislocations and twin boundaries. The binding energy of the shallow trapping centers was estimated to be 56 ± 12 meV. The deep trapping centers are assigned the cation vacancies, especially barium vacancies. On the surface of the sample there are macroscopic free volume holes in which positronium could be formed. [S0163-1829(96)02826-3]

I. INTRODUCTION

Current interest is focused on epitaxial superconducting thin-film $YBa_2Cu_3O_7$ and its analogs in the research for high-T_c superconducting devices. The thin films exhibit a high critical current J_c typically a factor of ~1000 higher than the high-quality single-crystal $YBa_2Cu_3O_7$.[1] Irradiation with neutrons and heavy ions can significantly improve J_c of the single crystals,[2] but J_c enhancement in thin films due to 200-keV He^+ ion irradiation is much less.[3] These differences have motivated many investigations on defect structures in the films. Using scanning tunneling microscopy (STM) it was found that a high density ($\sim10^9$ cm^{-2}) of spiral growth structures, each of which contains a screw dislocation, exists in epitaxial $YBa_2Cu_3O_{7-x}$ films.[4–8] Additionally, inclusions, twin boundaries, c-stacking faulty holes, and a number of nanometer-scale holes were also observed by using transmission electron microscopy in the films.[8]

As an atom-sensitive probe, positron annihilation spectroscopy (PAS) has been employed to research defect structures of bulk high-T_c superconducting materials and has made much progress.[9] In conventional PAS, where positrons are emitted over a broad distribution of energy, it is very difficult to use these positrons for microstructure investigation of thin films. Variable energy positron beams allow the depth-resolved measurement[10] and have been used to characterize open-volume defects in near-surface and buried interfaces, especially in semiconductors.[11] The slow positron beam experiment on the sintered material $YBa_2Cu_3O_y$ was mentioned in Ref. 12. The S parameter of the sintered $YBa_2Cu_3O_y$ as a function of positron energy was given by Anward *et al.*[13] but this was only a preliminary exploration too. Compared to Doppler broadening of annihilation radiation, the positron lifetime spectroscopy appears to have an obvious advantage. A positron lifetime spectrum can be resolved into several components and provide much more information than obtained from Doppler broadening measurements. At present there are two beams capable of measuring the positron lifetime spectroscopy with a resolution comparable to conventional lifetime spectrometers. One was discussed by Suzuki *et al.*[14] and the other by Schödlbauer *et al.*[15]

In this paper we present the results of positron annihilation study on superconducting thin film probed by using the pulsed beam used in Ref. 15. The positron lifetime spectra of the superconducting epitaxial thin-film $GdBa_2Cu_3O_{7-\delta}$ as a function of positron energy were measured in the temperature range of 90–300 K. The results show that the positron lifetime parameters of the film are sensitive to implantation energy and temperature.

TABLE I. Positron lifetime parameters at low implantation energies.

T (K)	E (keV)	τ_1 (ps)	τ_2 (ps)	τ_3 (ps)	I_2 (%)	I_3 (%)	Variance
100	1	208± 9	542±20	1866±700	9.9± 8	2.3±1	1.112
	2	199±22	338±17	1164±200	67 ±10	2.0±0.6	1.204
	3	194±13	336±13	1445±250	60 ± 7	2.0±0.2	1.083
304	1	158±18	355± 4	2455±101	82 ± 2	4.5±1	1.087
	2	124±10	326± 5	1065± 72	19 ± 1	3.5±0.4	0.981
	3	101± 7	301± 4	802± 62	78 ± 1	3.8±0.7	1.095

II. EXPERIMENT

The sample used in the present measurement was c-oriented epitaxial superconducting thin-film $GdBa_2Cu_3O_{7-\delta}$. The film was epitaxially grown *in situ* on (100)-oriented yttrium-stabilized zirconia single-crystal substrate by dc magnetron sputtering using a single planar target. The preparation of the films was reported in detail previously.[16] Briefly, the disklike stoichiometric target $GdBa_2Cu_3O_{7-\delta}$ of 40 mm diameter and 7 mm thickness was prepared from an appropriate mixture of Gd_2O_3, $BaCO_3$, and CuO powders by the common solid-state reaction method. A NdFeB magnet placed on the top of the target provided a plasma ring of 25 mm diameter. A mixture of 2×10^{-1} Torr oxygen and 4×10^{-1} Torr argon was used as for the sputtering gas. The discharge was run at 120 V and 0.3 A. The substrate was placed 20 mm apart from the target on a Pt stripe that could be resistively heated up to 1000 °C. The substrate was placed at an inclination of 60° to the target in order to minimize the negative-ion bombardment on the film. The deposition rate was 10 nm/min. After deposition the chamber was vented with pure oxygen and the temperature of the deposited film was cooled to 430 °C, held at this temperature for 10 min, and then allowed to cool down to room temperature.

The superconducting transition temperature of the film is 91 K with the transition width 0.8 K, which was determined by ac susceptibility measurement. The thickness of the film was estimated to be 300 nm. Grain sizes are typically several thousand angstroms.

The film structure was checked by x-ray-diffraction spectra. In addition to the peak of the substrate, there were strong (00l) peaks besides the very weak (200) peak. The rocking curve at the (005) peak displayed only broadening of 0.8° [full width at half maximum (FWHM)]. These results indicate that the sample was grown epitaxially with c axis normal to the film surface.

With the improved pulsed low-energy positron beam in Ref. 15 the positron lifetime spectra of the film were measured as a function of positron energy at different fixed temperatures. The parameters of the beam were described in detail in the previous paper by Willutzki *et al.*[17] More than 10^6 events were accumulated with time resolution of 230 ps (FWHM) for each spectrum. The spectra were analyzed with a modified version of the computer program POSITRONFIT. Variances were better than 1.35. All the results were obtained with the unconstrained fitting. During the measurement the maximum fluctuation of temperature was 1 K. The mean implantation depth $\overline{z}$ was calculated from the usual equation commonly used in semiconductor studies.[11]

III. RESULTS AND DISCUSSION

A. Implantation energy dependence of positron lifetime parameters

At a fixed temperature the variation of positron lifetime spectrum with implantation energy E was measured. For all measured temperatures, the variation obviously displays some common features: When $E\leqslant3$ keV ($\overline{z}\leqslant33$ nm) three lifetimes could be identified. The typical results are listed in Table I. In addition to a typical oxide surface lifetime in the range of 300–500 ps,[18] a long lifetime of around 1 ns could be found. Recently Hill *et al.*[19] reported the existence of three components including a long lifetime of 1.5–3.0 ns with an intensity of 1–2.5 % in the whole temperature range of 30–300 K for a sintered $YBa_2Cu_3O_{6.91}$, in which pores of diameter ~50 nm were present. In general, the positron lifetime of 1 ns or so is considered as a characteristic of the formation of ortho-positronium atoms (O-Ps) and of the annihilation in macroscopic free volume holes due to the pickoff mechanism.[10] Thus we can deduce that at the surface layer of the sample there are macroscopic free volume holes in which the positronium could be formed and annihilated. These macroholes may be similar to the nanometer holes observed in the epitaxial superconductive thin-film $YBa_2Cu_3O_{7-\delta}$ by STM.[7] Because of positron diffusion back to the surface, the annihilating signal of O-Ps can still be detected at energies up to $E=3$ keV ($\overline{z}=33$ nm).

The three-component analysis was also applied to the lifetime spectra at $E=4$ keV. The results gave an unacceptable variance (>1.4). After adopting two-component analysis, the variance improved and was better than 1.2. The long ~1-ns component was not seen. This fact tells us that the fraction of positrons with $E=4$ keV diffusing back to the surface is small and that the pickoff annihilation of O-Ps at the surface cannot be observed.

When implantation energy was raised gradually, one or two components could only be resolved. [Figs. 1(a) and 1(b)]. In the range of 4–10 keV (52–200 nm), the long-lived component τ_2 monotonically decreased with positron energy increasing while its intensity I_2 was almost constant. At energies above 10 keV, I_2 sharply dropped. Although the value of τ_2 is the same order as that of a typical oxide surface, we cannot attribute it to the surface because positrons with these energies should enter into the interior of the film body. Reasonably τ_2 is assumed to correspond to the lifetime of positrons trapped in open volume defects in the film.

The variation of positron lifetime parameters can be well understood qualitatively.[20] As implantation energy rises, the fraction of positrons in near surface or on surface decreases

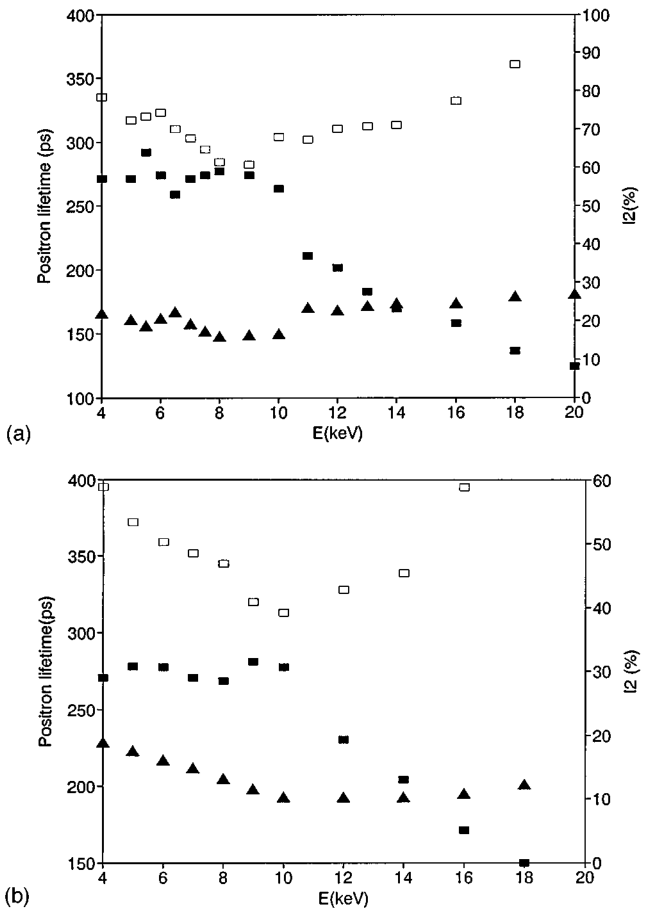

FIG. 1. Positron lifetimes as a function of implantation energy for the epitaxial superconducting thin-film $GdBa_2Cu_3O_{7-\delta}$ at (a) $T=304$ K and (b) $T=100$ K. ▲, τ_1; □, τ_2; ■, I_2.

largely because of the backdiffusion and implantation profile and positron lifetimes decrease gradually. At higher energies the fraction of positrons trapped in the interface between the film and the substrate becomes significant and results in increasing the lifetimes. As energy increases further the annihilating characteristics are those of bulk annihilation in the substrate. At intermediate energies the defects in the film body can trap positrons effectively and the information about the open volume defects in the film could be obtained. In the following sections we concentrate on the positron trapping in the film body, corresponding to incident positron energy between 4 and 10 keV.

B. Identification of τ_2 in the film body

Up to now, almost all researchers show that the members of the family of $LnBa_2Cu_3O_{7-\delta}$ (Ln=Y and rare-earth elements, except Pr) materials possess the same structure and very similar physical properties. The calculation of positron wave function in $YBa_2Cu_3O_7$ revealed that positrons are almost completely absent in the *Y* plane.[21] Thus the positron behavior in both $YBa_2Cu_3O_{7-\delta}$ and $GdBa_2Cu_3O_{7-\delta}$ should be the same. More than 95% of the positrons are confined between basal and Cu-O planes. The barium sites seem to play an important role.

Kögel[20] proposed a method of quantitative analysis for a variable energy positron lifetime spectrum, but a practical program is still to be developed. The defect profiling cannot be ruled out at present. However, qualitative analysis for τ_2 can deepen the understanding to the defects in the superconducting epitaxial film $GaBa_2Cu_3O_{7-\delta}$. The magnitude of τ_2 ranging from 350 to 283 ps at 304 K shows the existence of open-volume defects in the film body. The intensity of τ_2 is 53% at 304 K and 30% at 100 K, respectively [Figs. 1(a) and 1(b)]. However, it is now accepted that I_2 should rise with decreasing temperature and the defects concerning τ_2 consist of shallow positron trapping centers in high-quality sintered $YBa_2Cu_3O_{7-\delta}$. The decrease of the I_2 with temperature decreasing illustrates that the defects concerning τ_2 in the epitaxial thin-film $GdBa_2Cu_3O_{7-\delta}$ are deep trapping centers for positrons. The calculations of positron lifetime in $YBa_2Cu_3O_7$ were performed by several groups.[9] In general, anional vacancies (i.e., oxygen vacancies) are shallow trapping centers, of which the binding energy is only the order of 0.1 eV or less. Cational vacancies have a strong ability to trap positrons. Among them the barium vacancy has the maximum binding energy of 3.5 eV and the longest positron lifetime of 263 ps. The observed τ_2 in the film body is larger than the theoretical value.[9]

As yet, not much information has been obtained about the cational vacancies in high-T_c superconductors. An atomistic calculation of point defect energies found the barium has the smallest formation energy in $YBa_2Cu_3O_7$.[22] The author also showed significant lattice relaxation and neighboring ion polarization accompanying cational vacancies. For doped $YBa_2Cu_3O_{7-\delta}$ bulk materials a positron lifetime in the range of 240–380 ps has been reported.[23–25] Jean *et al.* attributed them to positron annihilation in the vacancies in the materials.[23] Groznov *et al.*[26] found a long lifetime of 350–550 ps in the superconducting oxide $BaPb_{1-x}Bi_xO_3$, which possesses a perovskite structure similar to $YBa_2Cu_3O_{7-\delta}$. They considered the long lifetime from positron annihilation in the monovacancies of barium. Consequently, we suspect that the observed τ_2 is mainly from positrons trapped in barium vacancies, although positron trapping in other cational vacancies could not be discounted.

More interestingly, an obvious temperature dependence of τ_2 was observed too, especially at low temperatures (Fig. 2). The increase of τ_2 with temperature decreasing can be explained in terms of aggravation of the relaxation and the polarization at low temperature. We notice that the positron lifetime of 240–380 ps is temperature independent in bulk doped $YBa_2Cu_3O_{7-\delta}$.[23,25] This fact tells us the effect of the lattice relaxation and the polarization in the film is more remarkable than in the bulk materials at low temperature. Lu *et al.*[24] found a long lifetime of 385 ps in the single-crystal $YBa_2Cu_3O_7$. After fast neutron irradiation, it was increased to 465 ps. At the highest irradiation doses (9×10^{17} ncm^{-2}) its intensity increased to 8%. The result showed that the defects with positron lifetime of 465 ps were effective pinning centers of $YBa_2Cu_3O_{7-\delta}$. Because of the existence of a remarkable lattice relaxation and neighboring ion polarization at low temperature around cational vacancies, the cational vacancies, especially barium vacancies, may be extended to larger defects, similar to those in fast neutron-irradiated single-crystal $YBa_2Cu_3O_7$, and hence become one of the effective pinning centers.

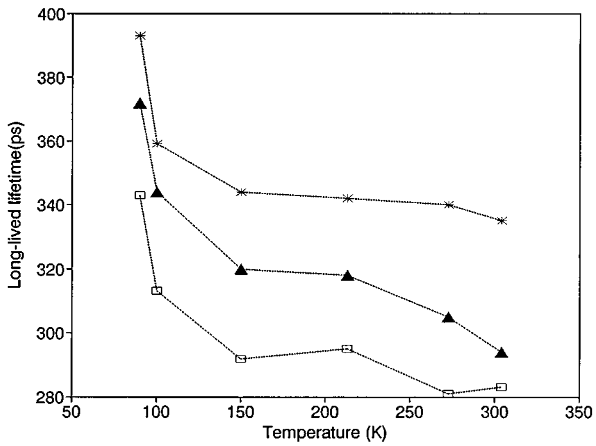

FIG. 2. Long-lived lifetime τ_2 as a function of temperature in the film body. The dashed lines are a guide for the eye. *, 6 keV; ▲, 8 keV; □, 10 keV.

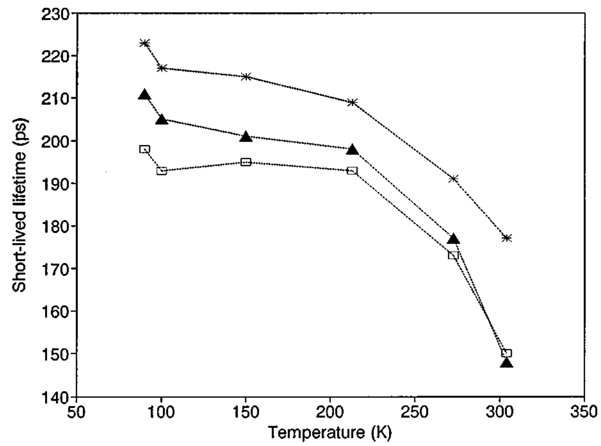

FIG. 4. Short-lived lifetime τ_1 as a function of temperature in the film body. The dashed lines are a guide for the eye. *, 6 keV; ▲, 8 keV; □, 10 keV.

C. Shallow trapping

Figure 3 shows the intensity I_2 of the long-lived lifetime τ_2 vs temperature. It can be seen that I_2 decreases as temperature is lowered. Almost all known experiments in metals and alloys indicate that this case is possible only when positrons are trapped in voids and larger clusters, in which the characteristic lifetime is 500 ps or so and independent of temperature.[27–29] In our case, no lifetime of ~500 ps could be found and τ_2 is temperature dependent. The observed positive temperature dependence of I_2 means that two trapping state model cannot be applicable and certainly there is another trapping center in the epitaxial superconducting thin-film $GdBa_2Cu_3O_{7-\delta}$, although we only see two components in the positron lifetime spectra.[30,31] The other center should have a positron lifetime so close to τ_1 that we cannot separate it from τ_1. The center should also have a low binding energy of positron and could trap positrons at low temperature. As temperature decreases, more and more positrons are trapped in the shallow trapping centers. Hence I_2 decreases and τ_1 increases at low temperatures. These illustrate the coexistence of both deep and shallow trapping centers in the sample. This phenomenon was observed in the carefully prepared metallic samples with a suitable deformation. Only two of the previous observations were reported: one is nickel[30] and the other is for silver.[31] According to Eq. (2) in Ref. 31 and from the data Fig. 3, we can estimate the positron binding energy of the shallow trapping centers to be (56 ± 12) meV in our sample. This value is similar to the reported by Usmar *et al.*,[32] but smaller than that reported by Hentrich, Kluin, and Hehenkamp[33] in the sintered $YBa_2Cu_3O_{7-\delta}$ McMullen *et al.*[34] obtained theoretically the binding energy of $O(1)$ vacancy to be 60 meV and that of twins 10 meV in $YBa_2Cu_3O_7$ crystals. Besides $O(1)$ vacancy and twin boundaries there is a high density of screw dislocations, usually $10^9\ \mathrm{cm}^{-2}$, in epitaxial superconducting thin films[4–7]. A preliminary observation of STM showed the density of the screw dislocations to be $4\times10^9\ \mathrm{cm}^{-2}$ in the sample used in this work. Dislocations are also electron-deficient defects and generally considered to be one of positron trapping centers with a small binding energy. Hence the screw dislocations should be one of the shallow trapping centers in the epitaxial superconducting thin-film $GdBa_2Cu_3O_{7-\delta}$.

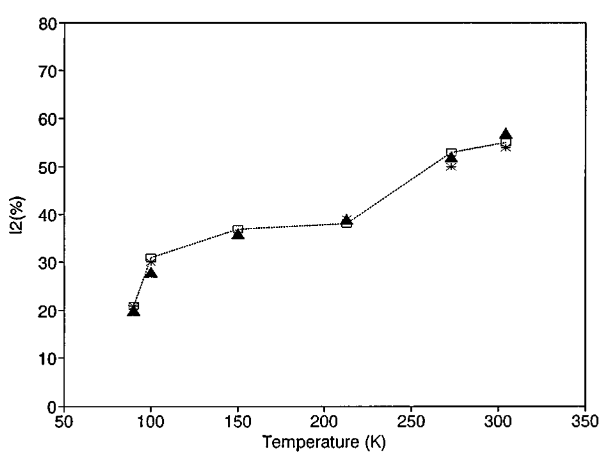

FIG. 3. Intensity of the long-lived component I_2 as a function of temperature in the film body. The dashed lines are a guide for the eye. *, 6 keV; ▲, 8 keV; □, 10 keV.

The variation of τ_1 with temperature is shown in Fig. 4. Strictly, we should use the three-state trapping model, in which a shallow trapping center is also included, to fit the experimental results and extract the physical parameters. In fact, many unknown parameters and limited data points make it difficult to obtain a unique set of solutions. As an approximation, we take τ_1 as the average lifetime of the shallow trapping positrons and free state positrons and apply the two-state trapping model to analyze the experiment. The trend of τ_1 coincides with the relationship between mean lifetime and temperature predicated by using two-state trapping model with the prefactor $D_0=1$.[34] The shallow trapping lifetime could be evaluated by using Eq. (6) in Ref. 34. The theoretical study of the positron states in $YBa_2Cu_3O_7$ found that positrons are almost completely absent in Y plane.[21] Experimental observation also confirmed the existence of the same delocalized-state lifetime, 164 ps, independent of x in

$Y_{1-x}Pr_xBa_2Cu_3O_{7-\delta}$.[35] So we take τ_b of $GdBa_2Cu_3O_{7-\delta}$ to be 164 ps and get the shallow trapping lifetime $\tau_S \sim 176$ ps, close to the bulk lifetime and less than the shallow trapping lifetime in the bulk material $YBa_2Cu_3O_{7-\delta}$.[32–34] Combined with the I_1 as a constant in the film body we deduce that the screw dislocations are the main shallow trapping centers in the epitaxial superconducting thin-film $GdBa_2Cu_3O_{7-\delta}$. Twin boundaries and perhaps $O(1)$ vacancies should be included in the centers.

IV. CONCLUSION

In high-quality sintered material and single-crystal $YBa_2Cu_3O_{7-\delta}$, only shallow trapping was observed.[32–35] In the F-doped Bi(Pb)-2223 bulk materials only deep trapping centers exist.[36] With the pulsed low-energy positron beam, we found the coexistence of both shallow and deep trapping centers in the epitaxial superconducting thin-film $GdBa_2Cu_3O_{7-\delta}$. The shallow trapping centers include the screw dislocations, twin boundaries, and $O(1)$ vacancies. The binding energy and the lifetime of the shallow trapping centers are estimated to be 56 ± 12 meV and 176 ps, respectively. The deep trapping centers are assigned to the cational vacancies, especially barium vacancies, in the film body. They extend to larger defects and become one of effective pinning centers at low temperature. In addition, positronium could be formed on the surface layer of the film.

The present work shows that the positron lifetime parameters are sensitive to the implantation energy of positrons and temperature for epitaxial superconducting thin films and the pulsed low-energy positron beam is an effective nondestructive method to study microstructures of superconducting thin films. This has been shown clearly for bulk $YBa_2Cu_3O_{7-\delta}$ by using a continuous beam.[37]

ACKNOWLEDGMENTS

We are grateful to C. R. Wang for his measurements of STM and to J.A. Duffy, P. Sperr, P. Willutzki, G. Triftshäuser, and M. Falter for their help in this work. One of the authors (X.Y.Z.) would like to thank the Alexander von Humboldt Foundation for its support during his stay in Germany.

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