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# SUPERCONDUCTING PROPERTIES OF AN Ag-CLAD $(\mathrm{BiPb})_2\mathrm{Sr}_2\mathrm{Ca}_2\mathrm{Cu}_3\mathrm{O}_{10+y} \text{ TAPE}$

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AC susceptibility and V-I characteristics of a well characterised Ag-clad Bi-Pb-Sr-Ca-Cu-O tape have been studied in the temperature (T) range 77–106 K and magnetic fields (B) up to 0.06 T. The intrinsic critical current density  $J_s$  (from ac susceptibility) is 30 000 A/cm<sup>2</sup> at 77 K whereas the transport critical current density  $J_c$  (from V-I curves) is 10 100 A/cm<sup>2</sup> at the same temperature. Both  $J_s$  and  $J_c$  showed power law variations with T and B, respectively, characteristic for intragranular flux creep. From the dissipation within the core the differential resistance  $R_f$  (associated with the flux flow) has been deduced. The magnitude and the variations of  $R_f$  with T and B are consistent with those due to flux flow in type II superconductors in low field. From  $d^2V/dI^2$  vs I curves very broad critical current distribution CCD has been deduced. The most probable  $J_c$  within the core

is  $J_{cp} \simeq 2.4 J_c$  and the maximum one  $J_{cm} \simeq 5 J_c$ . Such large  $J_{cm}$  enables further improvement of  $J_c$  in Ag-BPSCCO tapes.

# 1. Introduction

The achievement of high critical current densities  $(J_c > 10^4 \,\text{A/cm}^2)$  in magnetic fields B comparable with the upper critical field  $B_{c2}$  ( $B_{c2} > 10 \,\text{T}$ ) is necessary for the large scale applications of superconductivity. For the high temperature superconductors (HTS) these conditions are easily met in the epitaxial thin films and single crystals but not for the bulk samples [1]. In particular for ceramic HTS samples the zero field values of  $J_c$  at 77 K are up to  $10^3 \,\text{A/cm}^2$  and decrease to about tenth of this value [1] in fields as low as  $0.01 \,\text{T}$ .

A considerable improvement in  $J_c$  has been achieved for the melt-textured polycrystalline HTS samples [2]. In these samples the grain structure is more regular than in ordinary ceramics [1] and roughly resembles the brick-wall.  $J_c$  approaching  $10^4$  A/cm<sup>2</sup> in magnetic fields B < 0.1 T has been achieved but the preparation technique proved impractical for the production of large samples. The improvement in  $J_c$  has been ascribed to the strong intergrain links, associated with better grain alignment and larger and more regular grain boundaries.

In the last two years a considerable progress has been made in the production of the tapes of high temperature superconductors possesing high critical current density  $J_c$ . In particular the transport  $J_c$  up to 40 000 A/cm<sup>2</sup> at 77 K has been achieved in Ag-clad (BiPb)<sub>2</sub>Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>3</sub>O<sub>10+y</sub> (BPSCCO) tapes [3,4]. Since the magnitude of  $J_c$  and its variation with magnetic field depend sensitively on the microstructure of a given tape [4] it seems clear that the onset of dissipation is associated with depinning of vortices as in conventional type II superconductors.

However, the observed values of  $J_c$  are still below those expected to arise due to intrinsic pinning mechanism in layered HTS compounds [5]. The thermally activated flux creep and the residual weak intergranular coupling have been proposed as the  $J_c$ -limiting factors at higher [6] and lower [7] temperatures, respectively. The "brick-wall model" specific to the microstructure of HTS tapes has also been proposed [8,9]. Within this model the current passes through the bulk of the grains and large-area c-axis grain boundaries so as to bypass small a- and b-axis boundaries along the tape. Therefore  $J_c$  along the tape is determined with that along c-axis and the ratio of the length to the thickness of the grain [9].

In our opinion the available data are not sufficient in order to single out the  $J_c$ -limiting mechanism in the present-day HTS-tapes. In particular the results for intrinsic critical current density  $(J_s)$ , the differential resistance  $(R_f)$  and the critical current distribution function (CCD) which proved important for the understanding [10-14] of ceramic samples are still missing for tapes. In that what follows we present the preliminary results of the measurements of  $J_s$ , transport  $J_c$ ,  $R_f$  and CCD for a well characterised Ag-clad BPSCCO tape [4].

# 2. Experimental procedure

Powders were prepared by code composition of metal nitrate solutions having the cation ratio Bi : Pb : Sr : Ca : Cu = 1.85 : 0.35 : 1.90 : 2.03 : 3.05. The powders were calcined at 820°C for the 10 h, uniaxially pressed into pellets and sintered at 840°C for 20 h. X-ray diffraction patterns obtained using a Siemens D5000 diffractometer showed that the major phase was (BiPb)  $_2$ Sr $_2$ Ca $_1$ Cu $_2$ Os $_{+z}$ (2212), the high  $_2$ Ca phase (2223) has not been formed at this stage.

A powder-in-tube method [3,4] was used to prepare Ag-sheated Bi-2223 wires. The wires were rolled into tapes of overall thickness  $\simeq 0.1$  mm and width  $\simeq 4$  mm. The resultant tapes were heat treated at 830°C–838°C for varying times up to 60 h in air. The tapes were then uniaxially pressed at 1 GPa and heat treated under the same conditions for 120 h. This treatment was sufficient to form nearly single-phase 2223. The x-ray diffraction pattern revealed about two percent of Bi-2212 phase in the investigated tape.

The experimental methods for the measurements of the resistance and V-I curves have been given previously [11-14]. Since our pulse-current setup is limited to  $I \leq 6$  A a 1.8 mm wide strip was cut from the edge of original (4.5 mm wide) tape. The V-I curves have been measured over the temperature range 80–106 K in magnetic fields up to 60 mT.

The high-resolution ac susceptibility measurements were performed with the setup described in some detail elsewhere [10]. The rest of the tape (left after cutting the resistance sample) has been used in these measurements. The measurements were performed in the temperature range 77–110 K with ac magnetic field amplitudes  $10^{-6}$ – $10^{-2}$  T at the frequency 28.4 Hz and in dc fields up to 0.015 T.

## 3. Results and discussion

The resistive transition of the tape centered around 106.8 K is very narrow ( $\Delta T \leq 1\,\mathrm{K}$ ) and rather symmetrical (Fig. 1). In particular the characteristic "foot" [15] for ceramic BPSCCO samples (associated with the percolative transition in an array of weak intergranular links [1]) is absent in a tape. The other features indicating that the approach to R=0 in tape is not dominated with weak links are rather homogeneous broadening (as in single crystals) of the transition in magnetic field and the insensitivity of the transition on the measuring current ( $I \leq 20~\mathrm{mA}$ ).

As shown in the inset in Fig. 1 the resistance (R) of our sample decreases linearly with temperature between 300 K and 110 K and extrapolates to zero at 28 K (as that of reasonably pure Ag). Indeed because of the unfavourable ratios of resistivity and cross-sections of Ag and BPSCCO core the resistance of our sample between 110 K and 300 K is practically that of Ag-sheating. From the resistance measurements on the intact strip and that with badly damaged core (showing no  $I_c$ ) we estimated that the resistance of the core at 300 K is about 400 times larger than that of the strip.

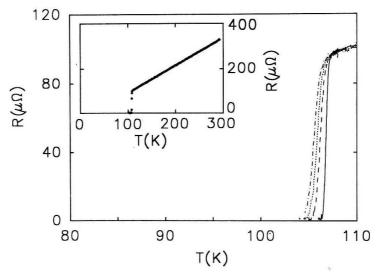


Fig. 1. Resistive transition of BPSCCO/Ag tape at applied fields B=0 (-), 0.02 (--), 0.04 (··) and 0.06 T (-·-), respectively. The inset: resistance vs. temperature for the same tape.

The diamagnetic transition of BPSCCO-Ag tape is shown in Fig. 2. The transition in low ac field  $(B_0=5\cdot 10^{-5}\ T)$  is very narrow with the midpoint at 106.5 K. In higher fields the transition was broadened but two steps characteristic for ceramic samples [1] did not appear up to the highest available field  $(B_0\simeq 10^{-2}\ T)$ . Accordingly the imaginary part  $(\chi'')$  of the ac susceptibility showed a single maximum which was shifted to the lower temperature on increasing  $B_0$  (Fig. 2). These results do not support the interpretation of a tape in terms of two phases (grains and intergranular links) possesing different superconducting properties. For a superconductor in a form of a long thin plate the critical current density  $J_c''(H_{\rm max},T)=\sqrt{2}H_{\rm max}/d$  where  $H_{\rm max}$  is the ac field amplitude for which  $\chi''$  has the maximum at the temperature T and d is the thickness of the plate [10] (BPSCCO core). When the plate is very thin  $(d=0.026\ {\rm mm}$  for our tape)  $H_{\rm max}$  is low hence  $J_c''(H_{\rm max})$  is practically the zero field (intrinsic) critical current density [10]  $J_s$ . Indeed for our tape  $J_c''(H_{\rm max},T)=J_s(T)$  has been observed for  $T\geq 80\ {\rm K}$ . In particular at  $T=80.5\ {\rm K}$  both measurements yielded  $\simeq 28\,000\ {\rm A/cm}^2$ .

Typical V-I curves measured at 80.5 K are shown in Fig. 3.  $J_c$ , estimated from the width and average thickness of the core (determined from the magnetic susceptibility measurements) was 6000 A/cm<sup>2</sup> at 80.5 K. Since the rectangular core-section was assumed in this estimation wheras the actual cross-section near the edge of the tape is more triangular-like [7] the actual  $J_c$  is probably in good agreement with  $J_c = 10\,100$  A/cm<sup>2</sup> obtained on the intact tape at 77 K. Even so the transport  $J_c$  is almost three times lower than  $J_s$  or  $J_s''$  (determined from the maxima in  $\chi''$ ). A similar difference between  $J_c$  determined from transport

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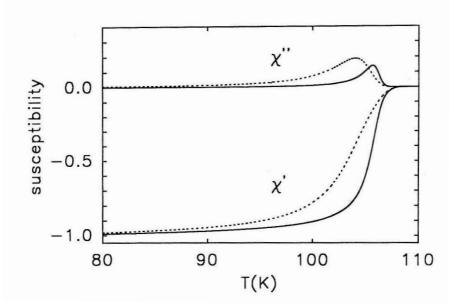


Fig. 2. Variations of real  $(\chi')$  and imaginary  $(\chi'')$  part of ac susceptibility of BPSCCO/Ag tape with temperature for ac field amplitudes  $B_0 = 0.05$  (-) and 0.23 mT (--), respectively.

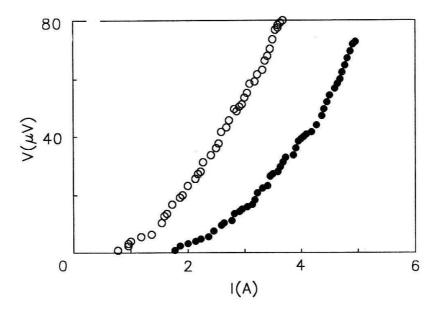


Fig. 3. V-I characteristic of BPSCCO/Ag tape at 80.5 K in magnetic fields B=0 ( $\bullet$ ) and 0.06 T ( $\circ$ ), respectively.

and magnetisation measurements has also been observed for other Ag-BPSCCO tapes as well as in conventional superconductors with reduced cross-section [7]. In our opinion this arises mainly because magnetization and transport take different averages over the current path in material which results in different sensitivity to inhomogeneities, inherent to present-day tapes. In particular, if some grains have c-axis along the length of the tape this would sizably reduce the transport  $J_c$ , but would hardly affect  $J_s$ .

The variations of the  $J_c$ ,  $J_c''$  and  $J_s$  (normalized to their respective T=80.5 K values) with temperature are shown in Fig. 4. Apparently all these critical currents show qualitatively the same variation with temperature. The data for  $J_c$  and  $J_c''$  (which are more detailed) are replotted in the inset in Fig. 4 in a form more suitable to deduce the exponent n of a power law variation  $J_c \sim (1 - T/T_c)^n$  expected to arise due to flux creep in type II superconductor.

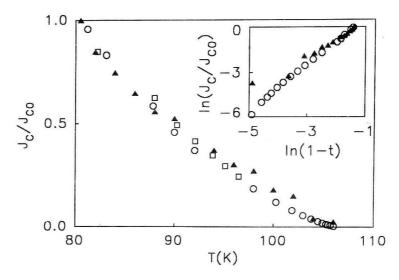


Fig. 4. Variations of critical current densities ( $\square$  intrinsic,  $\triangle$  transport and  $\circ$  determined from  $\chi''$ ) normalized to their respective T+80.5 K values with temperature for BPSCCO/Ag tape. The inset: plot showing  $J_c \sim (1-t)^n$  variation with  $t=T/T_c$  and n=1.4.

The observed n=1.4 is somewhat lower than the Ginzburg-Landau value n=1.5 for  $T\to T_c$  (usually observed in thin films and single crystals of HTS compounds) but is still quite different from n=1 or n=2 expected to arise due to different types of weak links [16]. We note however that the transport measurements cannot give intrinsic  $J_c(T)$  dependence for  $T\to T_c$  since the sample is not fully superconducting at these temperatures (Fig. 2) which may result in an enhanced increase of  $J_c$  on lowering T (hence lower n). Similarly  $J''_c(T)$  is affected with increasing  $H_{\rm max}$  on lowering T which also results in somewhat lower n. Because of

this a thorough discussion whether the Ginzburg-Landau theory is applicable to  $I_c$  of HTS compounds (for  $T \to T_c$ ) or not will be possible when the variation of the intrinsic critical current density becomes available.

The magnetic field (B) dependence of  $I_c$  and  $I_s$  at 80.5 K are shown in Fig. 5. The observed variations are qualitatively the same as the observed for other BPSCCO tapes [3,4,6,7] and can be fitted rather well with  $I_c \sim B^{-0.5}$  within the explored field range. Although much weaker than in ceramic HTS sample the observed B-dependence is quite strong because  $t = T/T_c$  is quite high and the flux pinning is not so strong in our sample. (Indeed considerably weaker  $I_c(B)$  dependence has been observed in BPSCCO tapes exhibiting enhanced flux pinning [3,4]).

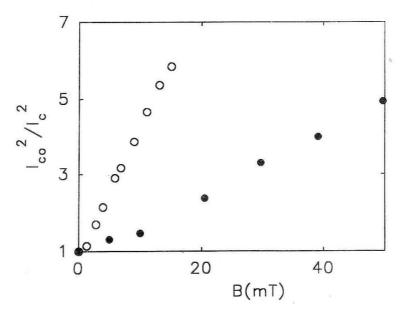


Fig. 5. Plots of intrinsic (o) and transport ( $\bullet$ ) critical currents of BPSCCO/Ag tape showing  $I_c \sim B^{-0.5}$  variations with magnetic field.

However  $I_c$  and  $I_s$  are reduced to about half of their respective zero field values at very different fields (0.05 and 0.012 T, respectively, Fig. 5). At present we have no proper explanation for such a large difference between the rates of decrease of  $I_c$  and  $I_s$  with B. This difference could arise from the anisotropic flux pinning [5] within the oxide core (note that  $I_s$  flows both parallel and perpendicular to the face of core). Alternatively as regards  $I_c$ , the possible residual weak links [7] may become resistive on the onset of dissipation at  $I_c$  (hence  $I_c < I_s$ ) and therefore contribute a little to the variation of  $I_c$  with B. The measurements of the angular field dependence of  $I_c$  and  $I_s$  as well as the extension of the present measurements to higher fields and lower temperatures are required in order to elucidate these findings. However, even without such a detailed analysis, the observed variations

of  $J_c$ ,  $J_c''$  and  $J_s$  with T and B are more consistent with the intragranular flux creep than with the intergranular weak links [1]. The magnitude of critical current  $I_c$  is however associated with the worst properties of a given sample and therefore the residual weak links or more generally the inhomogeneities may be the factor limiting transport  $I_c$  of the present-day tapes.

In order to assess the average and the best properties of the particular sample a detailed analysis of its V-I curves (extending to  $I\gg I_c$ ) is required [13]. Such analysis is for composite superconductor more involved than that for a pure one because in the dissipative region  $(V\neq 0)$  the current is carried both through the core and the Ag-sheating. Therefore one has to single out the dissipation due to current I actually flowing through the core from the observed one which is caused by the total current  $I_T$  along the tape.

The quantities derived from such analysis of V-I curves are shown in Figs. 6 and 7. The dynamic resistance of the core  $\mathrm{d}V/\mathrm{d}I = (\mathrm{d}V/\mathrm{d}I_T) \cdot (R_{\mathrm{Ag}}/(R_{\mathrm{Ag}}-R))$  where  $R_{\mathrm{Ag}}$  and R are the resistance of Ag-sheatings and whole tape, respectively, is shown in Fig. 6. We note that  $\mathrm{d}V/\mathrm{d}I$  increases rather slowly with I and eventually enters

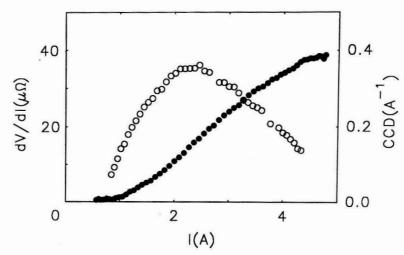


Fig. 6. The dynamic resistance  $\mathrm{d}V/\mathrm{d}I$  ( $\bullet$ ) of the oxide core of BPSCCO/Ag tape and the corresponding critical current distribution function CCD ( $\circ$ ) vs current through the core at 85 K.

the linear V-I regime (characteristic for the viscous flux flow in the conventional type II superconductors [17]) at  $\geq 4I_c$ . The variation of the differential resistance  $R_f$  ( $R_f \equiv \mathrm{d}V/\mathrm{d}I$  in the linear V-I regime) with temperature is shown in Fig. 7. For the sake of comparison the resistance of Ag-sheating ( $R_{\mathrm{Ag}}$ ) is shown with dashed line. Up to 96 K  $R_{\mathrm{Ag}}$  is about two times larger than  $R_f$ . Since the cross-section area of the core is over five times smaller than that of the Ag-sheating, the corresponding flux flow resistivity ( $\rho_f$ ) is at least ten times smaller than that of silver at the same

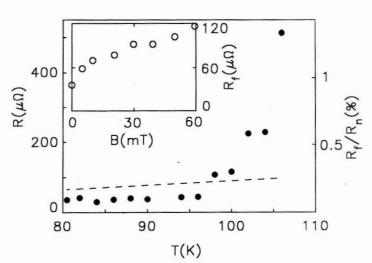


Fig. 7. Variations of the differential resistance  $R_F$  (•) of BPSCCO core and the resistance of Ag-sheating (--) with temperature. Right scale gives  $R_f/R_n$  with  $R_n$  the resistance of the core at 110 K. The inset: variation of  $R_f$  with magnetic field at 85 K.

temperature. More interesting is the ratio between  $R_f$  and the resistance  $R_n$  of the core in the normal state (T = 110 K). This ratio as seen from Fig. 7 (right scale) increases from about  $8 \cdot 10^{-4}$  (80 K) to about  $1.3 \cdot 10^{-2}$  (106 K). These values of  $R_f/R_n$  are much smaller than those of ceramic HTS samples (typically [11-14] 0.1) and seem consistent with those arising from the flux flow in the conventional type II superconductors in very low field [17]. The strong increase of  $R_f/R_n$  above 96 K is also not consistent with the temperature independent  $R_f/R_n$  observed in ceramic HTS samples [11].

The variation of  $R_f$  with magnetic field (B) at 85 K is shown in the inset in Fig. 7. Whereas in ceramic high  $T_c$  samples  $R_f$  practically does not depend on B (or decreases somewhat due to redistribution of current within the sample [11-14]) here  $R_f$  increases monotonically with the magnetic field. From the average slope of  $R_f/R_n$  vs B we estimated [17] the upper critical field  $B_{C2}$  of about 60 T. This value is sizably lower than that attributed to ab-plane of Bi-2223 single crystals ( $\simeq 250$  T). Since our estimate of  $R_n$  (based on resistance measurements on the intact tape and that with badly damaged core) is not very accurate (probably low), a more detailed discussion of our result for  $B_{C2}$  would be premature. We note however [12] that  $B_{C2} \simeq 50$  T estimated from  $R_f$  vs B variation for an YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7- $\delta$ </sub> sample at 77 K was also sizably lower than that for the ab-plane of this compound ( $\simeq 150$  T).

As mentioned above, the flux flow is established at currents several times larger then  $I_c$ . This results in very broad distribution of the critical currents within the core CCD  $\equiv R_f^{-1}(\mathrm{d}^2V/dI^2)$  shown in Fig. 6. The most probable critical current (corresponding to the maximum of CCD curve [13]) is  $I_{cp} \simeq 2.4I_c$  and the maximum one  $(I_{cm})$  around  $5I_c$ . (This estimate of  $I_{cp}$  is consistent with  $I_s \simeq 2.8J_c$ 

obtained at 80.5 K.) We note here that the inhomogeneity of the core (such as the variation in the cross-section area and/or quality of the core) could limit  $I_c$  but cannot produce such a broad CCD. Indeed much narrower CCD's have been observed in inhomogeneous fillamentary superconductors [18]. The observed variation in  $I_c$  within the core could however arise from the wide distribution of the flux-pinning energies within the material as deduced from the flux noise measurements on BSCCO single crystal [19]. This possibility can be verified by measuring CCD on tapes exhibiting enhanced flux-pinning [4].

### 4. Conclusions

The results presented above prove that the behaviour of critical current and differential resistance in Ag-clad BPSCCO tapes is very different from that observed for ceramic samples, including those of the same compound [20]. The variations of  $J_c(B,T)$  and  $R_f(B,T)$  are however consistent with those for conventional type II superconductors and hence are probably determined by the intragranular flux-pinning. The observed critical current distribution is however much broader than those observed in conventional type II superconductors. This has tentatively been ascribed to a broad distribution of the flux-pinning energies within the material. The measurements on tapes exhibiting an enhanced flux-pinning [4] may elucidate this question.

The results obtained for a single tape apparently cannot single out the mechanism limiting the magnitude of critical current in present-day Ag-clad BPSCCO tapes. However, systematic studies on carefully prepared and well characterised tapes [3,4] are likely to achieve this goal. Rather large values of  $J_{cp}$  and  $J_{cm}$  are promising for the applications of such tapes.

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#### SUPRAVODLJIVA SVOJSTVA TRAKE (BiPb)<sub>2</sub>Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>3</sub>O<sub>10+y</sub> U SREBRU

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Proučavana su magnetska susceptibilnost u izmjeničnom polju i V-I krivulje dobro karakterizirane trake Bi-Pb-Sr-Ca-Cu-O prekrivene srebrom u području temperatura 77–106 K i magnetskom polju do 0.06 T. Intrinsična gustoća kritične struje  $J_s$  (iz magnetske susceptibilnosti) je 30 000 A/cm² na 77 K dok je gustoća transportne kritične struje  $J_c$  (iz V-I krivulja) 10 100 A/cm² na istoj temperaturi.  $J_s$  i  $J_c$  ovise po zakonima potencija svojstvenim puzanju magnetskih vrtloga u supravodičima II vrste, o temperaturi i magnetskom polju. Analizom dinamičkog električnog otpora oksidne jezgre određen je njezin diferencijalni otpor  $R_f$  (povezan sa viskoznim tečenjem magnetskih vrtloga). Veličina i ponašanje  $R_f$  u skladu su s onim za klasične supravodiče II vrste u malom magnetskom polju. Iz ovisnosti d²V/dI² o I uočena je vrlo široka raspodjela veličina kritičnih struja unutar trake. Najvjerojatnija kritična struja  $I_{cp}$  je 2,4 $I_c$  a najviša uočena  $I_{cm}$  oko 5 $I_c$ . Tako visoka vrijednost  $I_{cm}$  ukazuje na mogućnost daljnjeg povišenja kritičnih struja u trakama sa Bi-Pb-Sr-Ca-Cu-O jezgrom.