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A significant reduction of ac losses in twisted Bi(2223) multifilamentary tapes with Ag sheaths has been achieved by using oxide ($BaZrO_3$ and $SrZrO_3$) barriers between filaments. These barriers have two important effects: they increase the transverse resistivity, which suppresses induced coupling currents, and they reduce filament bridging, which in pure Ag sheath tapes largely cancels the beneficial effect of filament twisting. The decoupling can be gauged by the frequency at which loss shows a maximum in a low-amplitude ac field applied perpendicular to the tape. So far, the frequency of the loss maximum, f_m, in Ag-sheathed tapes has been enhanced from 5 Hz (untwisted) to 82 Hz (11 mm in twist pitch length). Different ways to introduce oxide barriers in tapes with 19–95 filaments are presented. The critical current density in the filaments varied between 10,000 and 20,000 A/cm^2. Ac loss measurements as well as the electrical and mechanical characterization are discussed in detail. The variation of the critical current density with bending strain is shown to be similar to that of tapes without barriers.

KEY WORDS: Oxide barrier; ac losses; mechanical property; Bi(2223) tape; fabrication.

1. INTRODUCTION

Recent achievements on the fabrication of Bi(2223) multifilamentary tapes with high critical current density, both in short and long lengths [1,2], have generated a great interest for large-scale applications of high-T_c materials, such as transmission cables, transformers, motors, and generators. Since most applications imply alternating currents and fields, the reduction of the ac losses is an important task. For this reason, much research has been aimed at further development of these materials, with special attention to lowering the ac losses [3–6]. High ac losses are partially caused by the very low resistivity of the Ag sheath, resulting in important induced currents flowing through the Ag matrix. All the filaments are then electrically coupled together and the multifilamentary tape behaves as a single core tape. In addition, many 2223 grains grow out from the filaments, introducing many superconducting "bridges" between the filaments which directly connect the filaments. To date, many metals and alloys have been investigated as sheath materials in order to replace Ag. Only Mg, Ti, and Au additives to Ag have a relatively low detrimental effect on the properties of the tapes [7,8]. However, only 2–3 times higher effective resistivity can be reached with these additions, which is insufficient for a substantial lowering of the ac losses. The main reasons for the limited choice of highly resistive sheaths are twofold: (1) chemical stability of the Bi(2223) phase. Small amounts of additives to the 2223 phase precursor can significantly influence its formation and result in a contamination

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of the superconductor; (2) oxygen diffusion. Many modified sheaths slow down the 2223 phase formation and thus enhance the formation of secondary phases, probably due to the problem of oxygen diffusion through the sheaths during high-temperature treatments.

Recently, a new way to increase the resistivity of the sheath and thus to reduce ac losses has been developed by our group [9], which consists in surrounding each filament with an oxide barrier. By this method, the effective resistivity was increased by a factor of 10, and the frequency f_m at which the ac coupling losses show a maximum was shifted from 4.5 to 45 Hz. The importance of further increasing f_m is still a major task, because this value has to be shifted as far as possible above the working frequency (e.g., 50 Hz). Moreover, the introduced barrier layer electrically decouples the filaments not only by increasing the resistivity of the matrix but also by avoiding the direct contacts between filaments (bridges). $BaZrO_3$ has been found to be one of the most suitable materials for this purpose, due to its high chemical stability with high-T_c materials [9,10] and its oxygen permeability during high-temperature treatment. Further enhancement of the sheath resistivity with this material is still to be expected because it is an insulator at 77 K. In this paper, we will present different ways to introduce barrier materials and their effects on the electrical decoupling of the filaments. The optimization of the thermomechanical treatment conditions for increasing j_c will also be described. Finally, the characterization of the mechanical and electrical properties of the tapes, as well as the ac losses, will be discussed.

2. EXPERIMENTAL

Three different ways were used to introduce the oxide barrier into Ag-sheathed Bi(2223) multifilamentary tapes. The first method, as shown in Fig. 1a, consists of using billets made of two tubes: Ag as the inner one and a selected metal (e.g., Cu or Ni) as the external one. The tube is filled with pre-reacted Bi(2223) powder and deformed to a multifilamentary tape by means of the standard powder-in-tube (PIT) method [11]. The metal layers are then oxidized by a heat treatment at 500–600°C under flowing O_2. The final thickness of the oxide layer is controlled by selecting the initial size of the metal tube. The second way consists in preparing Ag-sheathed monocore wires (1 mm in diameter), painting oxide powders on the surface of the wires (see Fig. 1b), and then stacking them into another Ag tube. The last method uses two Ag or Ag alloy tubes with different diameters, the oxide powder being between the two tubes (see Fig. 1c). The oxide barrier thickness is controlled by varying the tube sizes or the oxide powder initial packing density. Some Ag alloys (e.g., Ag99Ti and Ag98Mg2) were used to replace the external tube with the purpose of obtaining a further enhancement of matrix resistivity. For improving the j_c value of the tapes, the mechanical deformation process was carefully optimized. The standard PIT method was modified by using a two-axes rolling technique on a driven turks head [12]. The improvement of the intermediate deformation condition was realized by a new technique [13], which provides a uniform reduction on the composite tape. The twisting process for a square multifilamentary wire was performed in a novel way: the "square–round–twisting–square" procedure. The details of this technique are described in Sec. 3.3.

The microstructure was investigated by scanning electron microscopy (SEM) and energy disperse X-ray analysis (EDX). T_c was identified by means of the ac susceptibility. j_c values at 77 K were measured by the four-probe method in applied fields up to 0.8 T using a criterion of 1 μV/cm. For the study of ac losses, two types of measurements were performed. A magnetic ac loss measurement (frequency f= 1–130 Hz, at T= 77 K) using a double hall sensor method [3] was performed on 3 cm long tapes in a sinusoidal external field ($H||c$) of varying amplitudes applied perpendicular to the tape. This experiment allows access to a wide frequency domain and is well suited for investigating the coupling loss. Another

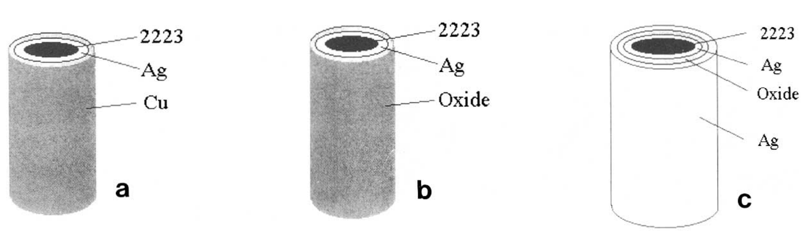

Fig. 1. Schematic drawing of three ways to introduce the oxide barrier.

type of data is obtained with standard transport ac loss measurements in the self-field, as described in, e.g., [14].

3. RESULTS AND DISCUSSION

3.1. Barrier Material Introduction

Figure 2 shows a cross-sectional view of a Cu/Ag composite sheathed tape, a typical result obtained by the first method (see Fig. 1a), with which continuous 1–5 μm thick Cu layers around every filament can be obtained easily after deformation. This method is very suitable for introducing other metals into the Ag sheath, if these metals have similar mechanical properties as Ag. Ni and Ti were also successfully combined into the matrix. Some intermediate heat treatments, 500°C × 1 h under Ar, during the deformation processes were applied to join everything together and keep the composite deforming uniformly. Cu, Ni, and Ti thin layers were completely oxidized during a treatment at 500–600°C under flowing O_2. However, although these oxides have no solubility in Ag, very strong interaction occurred destroying both the continuity of the oxide layers and superconducting filaments. The best results obtained on these tapes are an enhancement of the effective resistivity by a factor of 3 and a j_c value of 10 kA/cm^2 (77 K, 0 T) [9]. Because of the limited choice of metals with respect to their mechanical properties and chemical stability at high temperature, we started to look for stable oxides and ways to introduce them directly by other methods.

Figure 3 shows a detailed cross-sectional view of a wire made by the second method described above (see Fig. 1b). The painted oxide powder moves easily during deformation and to obtain a uniform layer between filaments is therefore quite a challenge. Moreover, as the filaments are separated by a hard oxide powder and a very small reduction per pass of drawing or rolling (<10%) is applied, it is very difficult to avoid nonhomogeneous deformation. However, since with this method it is possible to obtain a reasonable fill factor (20–25%, which is the ratio of superconductor over total tape section) and to enhance the decoupling effect (see Sec. 3.4), technical improvements of this method are underway.

Representative cross-sectional pictures of the wires made by the third method (see Fig. 1c) are shown in Fig. 4. Two uniform barrier layers between two neighboring filaments can be relatively easily obtained and the thickness of the barrier layers can be well controlled by this method. In addition, some other highly resistive metals or alloys can be used to replace the external Ag tube. Two kinds of Ag alloy tubes, Ag99Ti and Ag98Mg2, were used with $BaZrO_3$ as barrier in order to check if the barrier layers can increase the chemical stability of the alloys. These alloys were selected because of their high resistivity (3 times higher than Ag) and the fact that they show no strong reaction with Bi(2223) phase. The cross sections of the tapes after heat treatment are present in

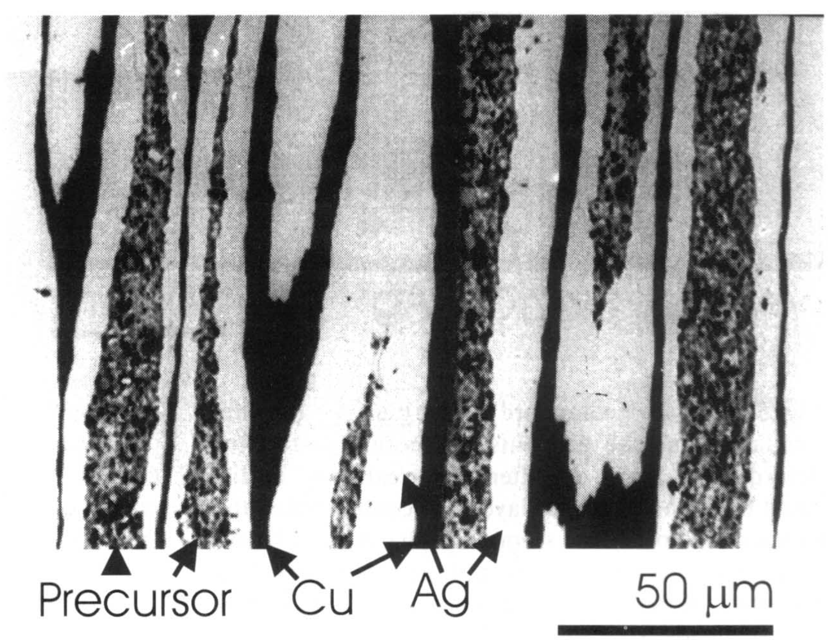

Fig. 2. A detail of the cross section of a 37-filament Bi(2223) tape with CuO barriers after deformation, but before heat treatment.

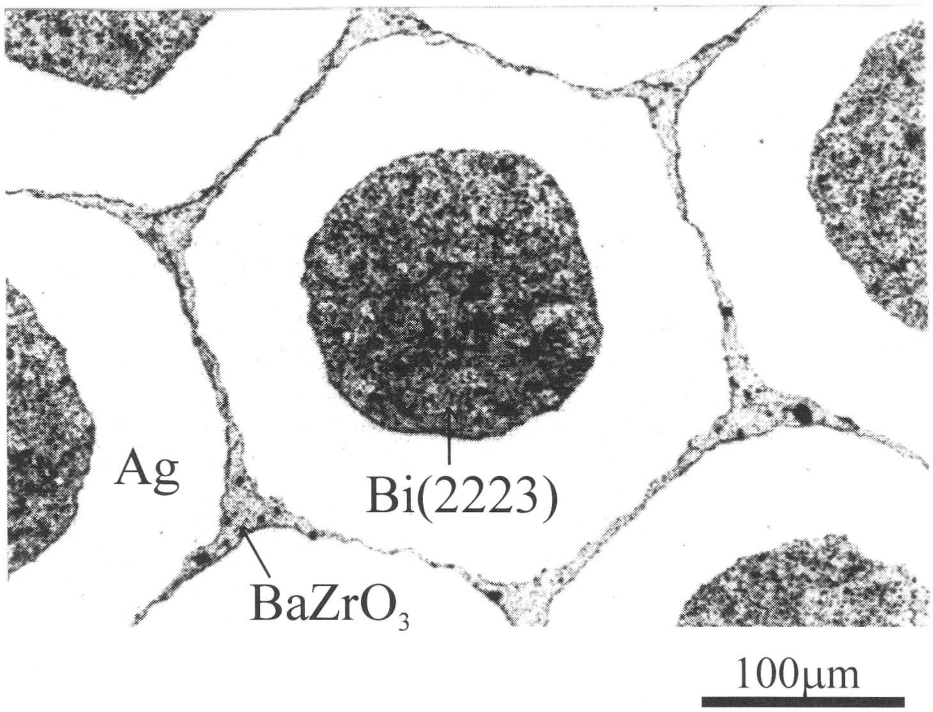

Fig. 3. A detail of cross section of a 37-filament Bi(2223) wire with $SrZrO_3$ barriers at an intermediate stage of deformation.

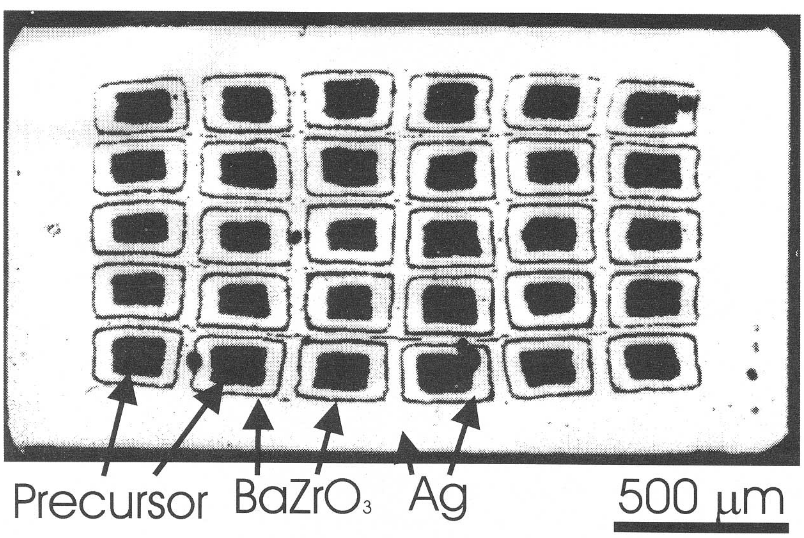

Fig. 4. SEM picture of the cross section of a 30-filament Ag/$BaZrO_3$-sheathed Bi(2223) made by a modified PIT method, barriers at an intermediate stage of deformation.

Fig. 5. Since these alloys are harder than Ag, the barrier layers and the filaments are more regular after deformation. However, after heat treatment, Ti diffused through the barrier layer and contaminated the superconductor. More secondary phases were found in the filaments (see Fig. 5a), and the superconducting transition of the tape was very broad, $\Delta T_c \approx 30$ K. For the tape made from Ag98Mg2 alloy, an even stronger diffusion of Mg appeared, which caused neighboring layers of $BaZrO_3$ to join together in most parts and induced many secondary phases (see Fig. 5b). The j_c values of these tapes are much lower than the normal tapes, only 3–6 kA/cm^2 (77 K, 0 T). Furthermore, no remarkable enhancement of the effective resistivity was found on this type of tapes.

Another important benefit of oxide barriers is that they can stop the outgrowth of 2223 grains, as shown in Fig. 6. Such outgrowths can form bridges

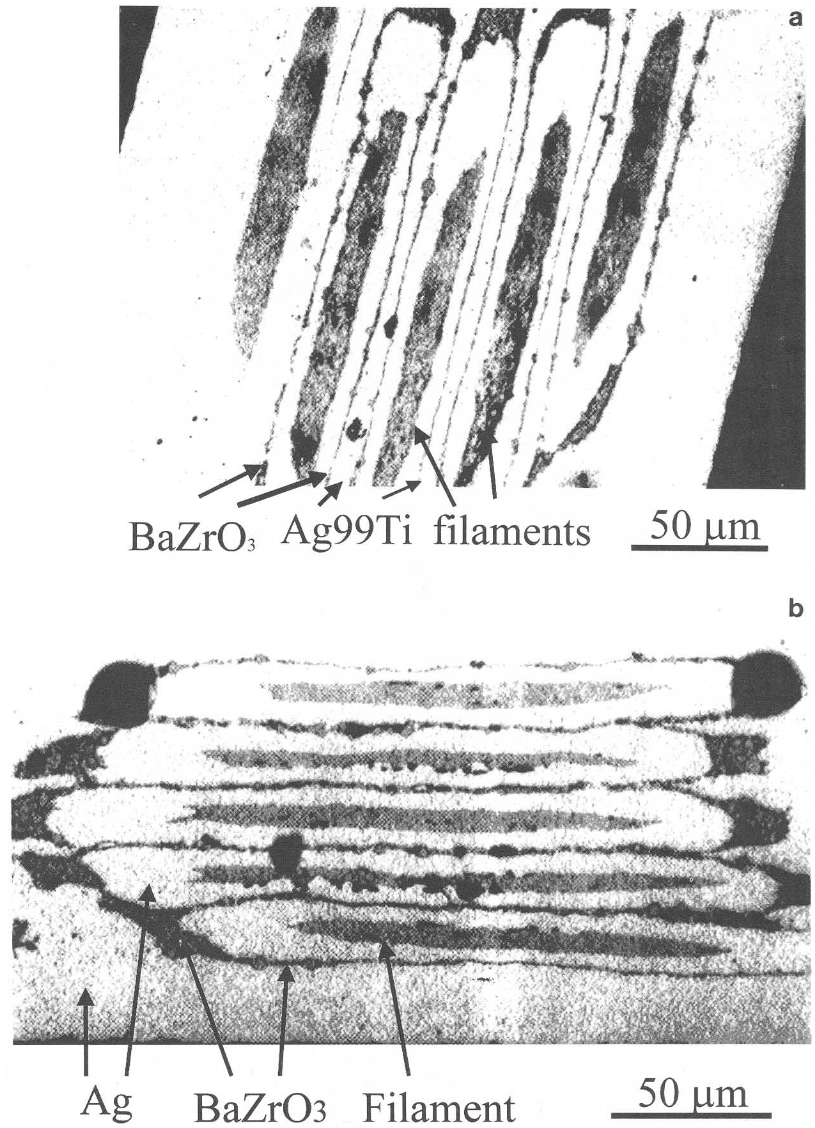

Fig. 5. Cross section of a 30-filament Bi(2223) tape sheath after heat treatments (a) with Ag99Ti/$BaZrO_3$/Ag sheath and (b) with Ag98Mg2/$BaZrO_3$/Ag sheath.

between filaments and yield a direct superconducting coupling [15].

Several different types of metal–ZrO_2 have been studied for use as barrier materials. It has been found that $SrZrO_3$ has similar chemical stability as $BaZrO_3$ and thus produces high-quality, low-ac-loss tapes. However, $CaZrO_3$ and $PbZrO_3$ react strongly with the 2223 precursor, since Ca and Pb are substituted by Sr in the metal–ZrO_2 [16].

3.2. Improvement of Deformation Condition

We have succeeded in producing Bi(2223) tapes with different numbers of filaments well encapsulated by $BaZrO_3$ barrier layers by the standard PIT method [9]. The value of T_c for these tapes is always close to 110 K, regardless of the thickness of the barrier layers. However, if the barrier thickness was over 1 μm, j_c values are lowered from 13 to 5 kA/cm^2 (77 K, 0 T) because the hard barrier oxide powder influences the workability of the tape and yields strong sausaging of the filaments.

For this reason, we have modified the PIT method using a two-axes rolling technique, where no tensile force is applied during the wire forming and every filament is deformed equally during rolling. Consequently, the sausaging problems are diminished and j_c values are increased, up to 15 kA/cm^2 (77 K, 0 T), compared to those of tapes made by the normal PIT method with barrier thickness exceeding 1 μm. In order to further enhance the j_c value, we recently paid more attention to the intermediate deformation process, because after heat treatment at 820–840°C, the Ag sheath is softened whereas the barrier layer

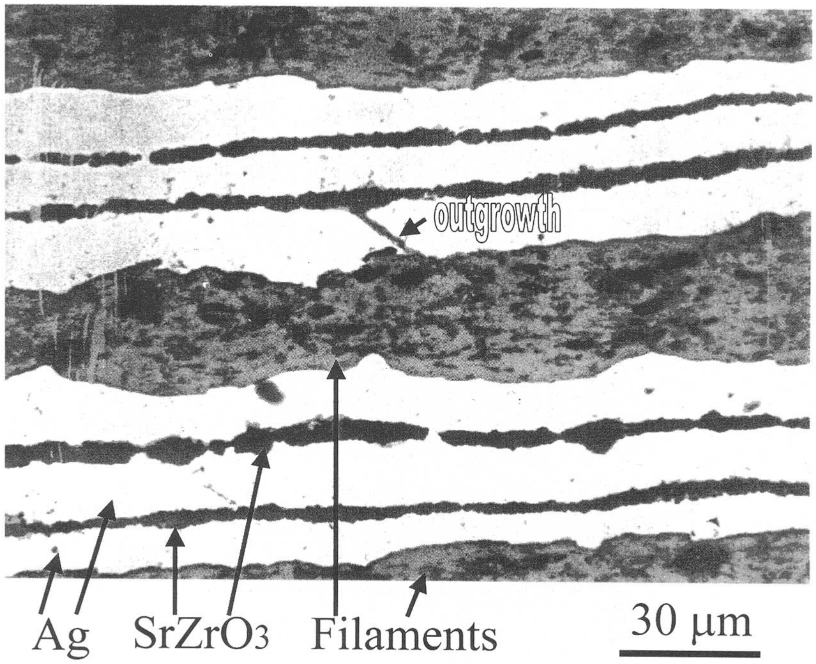

Fig. 6. A detail cross section of a 30-filament Bi(2223) tape with $SrZrO_3$ barrier, showing the outgrowth stopped by the barrier layer.

and superconducting core are sintered, which means that the difference in the mechanical properties of the three materials is getting bigger and cracks will appear more easily. Using a new prototype set-up [13], we could apply a more uniform deformation and improve the homogeneity both in the filaments and the barrier layers (see Fig. 7a). The j_c value was increased from 15 to 20 kA/cm^2 and the field dependence of j_c was also improved, as shown in Fig. 7b.

3.3. Twisting Process

It is well known that twisting the multifilamentary tape with a very small twist pitch length is even more efficient to reduce ac coupling loss [3]. Recently, we developed a convenient way to twist our tapes which, due to the two-axes rolling process, have a rectangular shape. As shown in Fig. 8, a square wire ($1.6 \times 1.6\,mm^2$ in cross section) is first drawn into a round wire (1.6 mm in diameter). This round wire is annealed at 500°C for 1 h and then twisted down to the required length. The minimum pitch length which could be achieved without breaking the wire was 3 mm. After the process, the wire is rolled down again to a rectangular tape of $0.2 \times 2.0\,mm^2$ by the two-axes rolling process. The effect of twisting on the j_c value is present in Fig. 9. For a tape twisted in 11 mm pitch length, the j_c value was lowered only by 18%, which shows that the tape with oxide barrier can still be twisted with a very small twist pitch length without lowering the j_c value to an unacceptable level. Similar results were also found by other groups twisting pure Ag-sheathed Bi(2223) tapes [17]. The j_c values of this type of tapes are still relatively low, 4–6 kA/cm^2, because the intermediate deformation was performed by rolling. A further improvement is expected by using our new uniform deformation technique [13].

3.4. Characterization

3.4.1. Mechanical Properties

The mechanical properties were checked by bending tapes on cylinders with different diameters, from 20 to 60 mm, and measuring j_c after straightening them out again. Figure 10 shows the results obtained on two tapes, one with a $BaZrO_3$ barrier of 1 μm thickness and one without barrier. The tapes are $0.2 \times 2.0 \times 20\,mm^3$ in size, 20 mm in twist pitch length, and 5 kA/cm^2 in initial j_c value. It can be seen in this figure that the critical bending strain, below which the j_c value is still reversible after bending, is $> 0.3\%$ for tapes both with and without oxide barrier. After a bending strain of 0.6%, j_c retains 75% and 85% of its initial value for the tapes with and without oxide barrier, respectively. This bending behavior is comparable to the values reported in the literature [18].

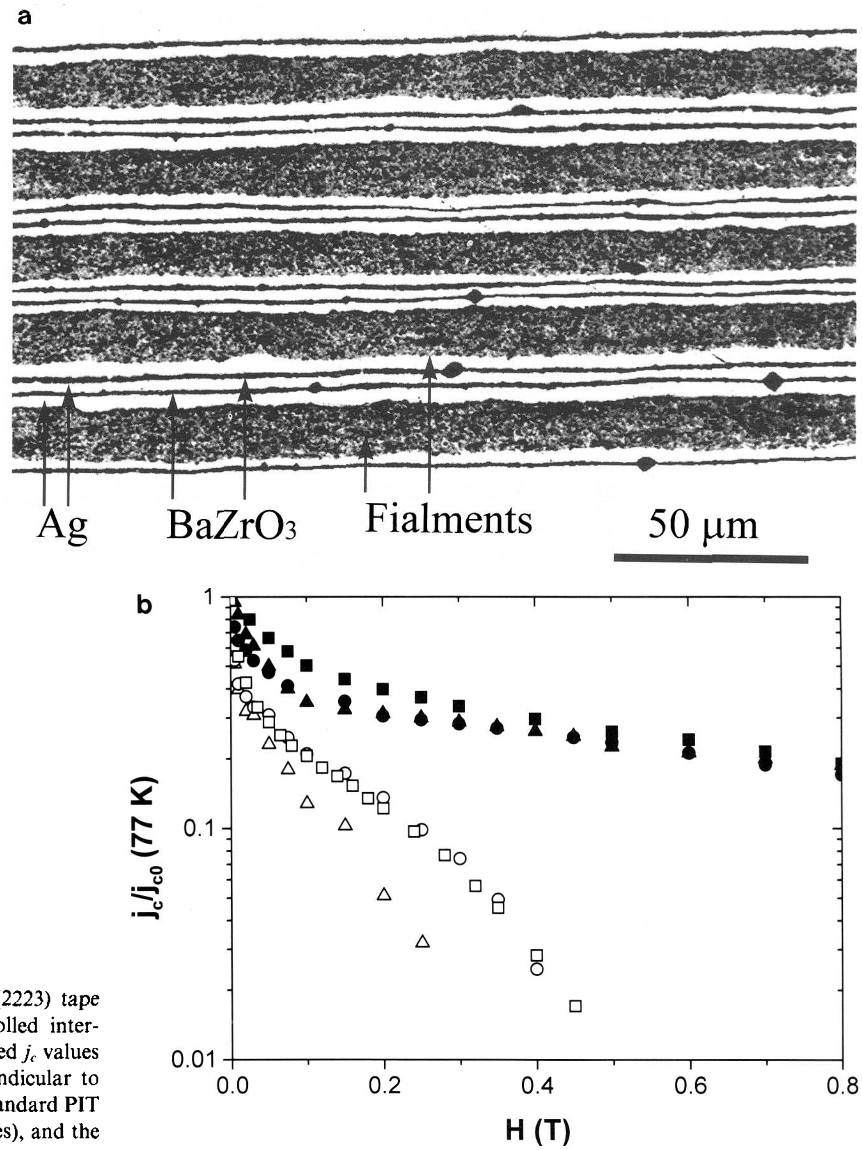

Fig. 7. (a) Cross section of a 30-filament Bi(2223) tape with $BaZrO_3$ barriers made by a well-controlled intermediate deformation process and (b) normalized j_c values vs. applied field parallel to (solid) and perpendicular to (open) tape plane, for the tapes made by the standard PIT method (triangles), the modified method (circles), and the new method (squares).

3.4.2. *ac Losses*

Figure 11 makes a comparison at $T = 77$ K and $f = 50$ Hz of the amplitude dependence of the total loss per cycle Q between two 19-filament tapes with and without barriers. Figure 11a shows magnetization data while Fig. 11b presents transport self-field losses. At high amplitudes the magnetic data shows how saturation losses are significantly reduced by the barriers. In the incomplete penetration regime the losses are hysteretic in the pure Ag-sheathed tape, $Q \propto (\text{amplitude})^n$, with $n = 3$ (magnetic) or $n = 4$ (transport), but this dependence changes to the square of the amplitude after insertion of barrier layers. The latter behavior is characteristic of an ohmic dissipation mechanism, the coupling loss. In order to understand why the coupling loss dominates the hysteretic loss-component at lower amplitudes in the barrier tape, we need to consider the frequency dependence of Q in the incomplete penetration regime [19].

To get low coupling losses, we need conductors with a small coupling current decay time constant τ, with $\tau \propto l_t^2/\rho_e$ for twisted tapes and $\tau \propto l^2/\rho_e$ for untwisted tapes. l_t is the twist pitch length, l is the sample length, and ρ_e is the effective interfilament transverse resistivity. The proportionality constant is

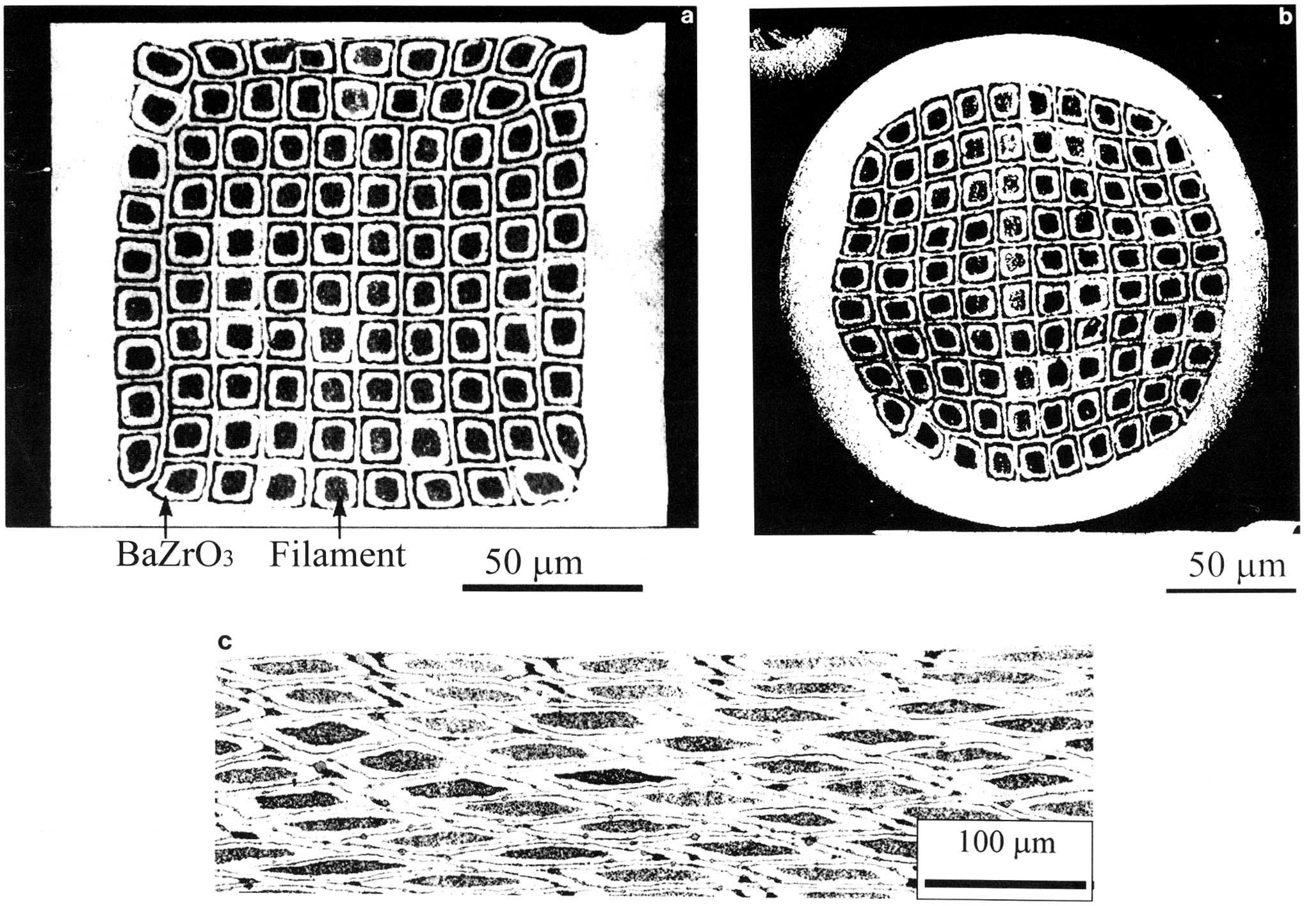

Fig. 8. Cross section of a 94-filament Bi(2223) wire with $BaZrO_3$ barriers, showing different steps of the twisting process.

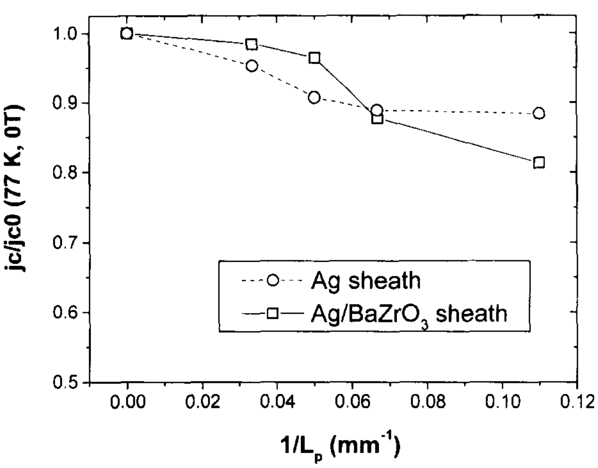

Fig. 9. Normalized j_c values vs. twisting pitch length.

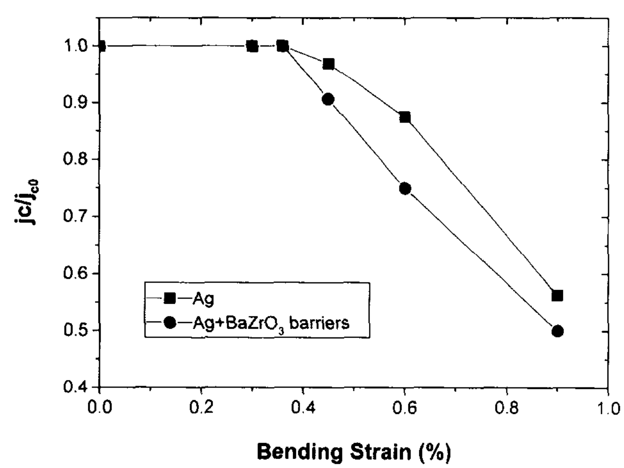

Fig. 10. Normalized j_c values vs. bending strain.

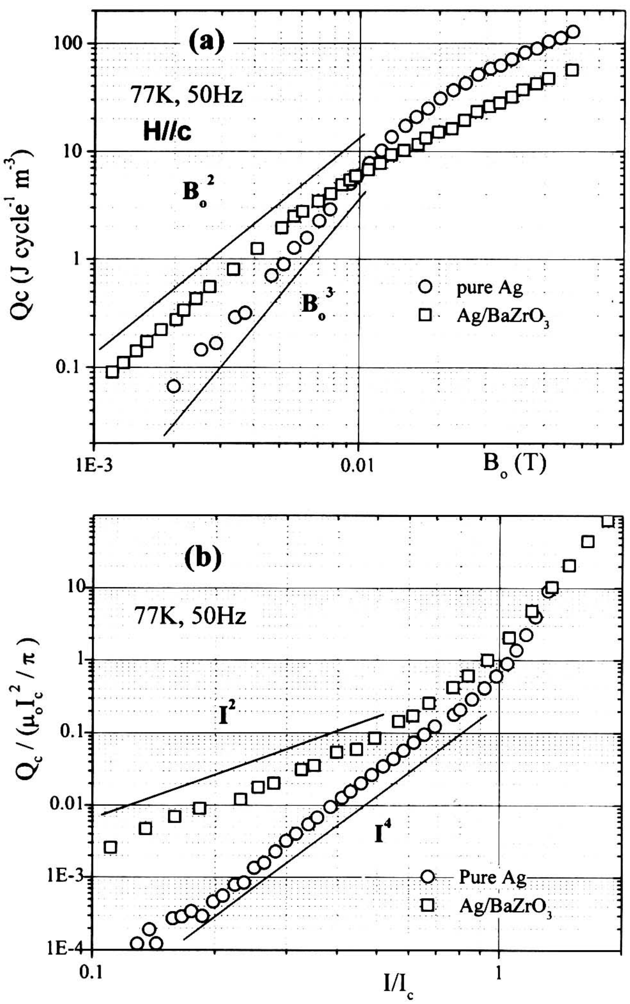

Fig. 11. Comparison of the amplitude dependence of the ac losses between 19-filament Ag- and Ag/$BaZrO_3$-sheathed tapes. (a) Magnetically determined loss-data, in an external ac field; (b) the transport self-field loss.

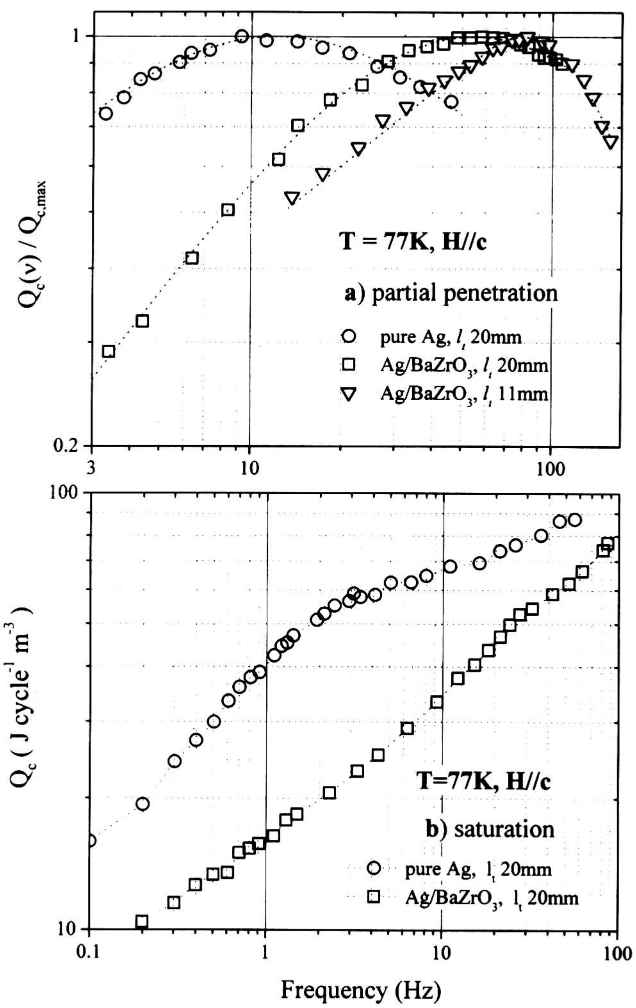

Fig. 12. The frequency dependence of the losses in external field: (a) the low-amplitude regime, where coupling losses dominate, and (b) the high-amplitude saturation regime.

different in both cases. To get the individual τ values of the samples, we measured the frequency dependence of the loss/cycle Q at amplitudes $\Delta B < \Delta B_{ps}$. ΔB_{ps} is the composite saturation field. The main contribution to Q are the coupling losses Q_c. Q_c, Q, and also the volume-normalized loss q have a maximum at frequency f_m for which

$$2 \cdot \pi \cdot f_m \cdot \tau = 1 \qquad (1)$$

By measuring f_m, using Eq. (1) and the inverse proportionality of ρ_e to τ, we get information on how the barrier thickness changes ρ_e [3, 20].

Figure 12a clearly shows such maximum in Q in the case of three twisted Bi(2223) tapes. In the first sample (Ag sheath, 30 filaments, $l_t = 20$ mm) $f_m \approx$ 10 Hz. The second tape (Ag/$BaZrO_3$, 30 filaments, $l_t = 20$ mm) has $f_m \approx 60$ Hz. As the geometry of both tapes is identical, this increase in f_m must be related to a corresponding increase in ρ_e. By shifting f_m from 10 to 60 Hz we reduced the coupling loss below ~20 Hz but actually increased its contribution to the overall losses at 50 Hz, which explains the change in amplitude dependence shown in Fig. 11. When f_m is pushed up to even higher frequencies, the loss at 50 Hz will start to decrease again. At high amplitudes Fig. 12b demonstrates how the suppression of the coupling currents already leads to a significant reduction of the saturation losses regardless of the frequency [19].

The third tape in Fig. 12a (Ag/$BaZrO_3$, 94 filaments, $l_t = 11$ mm) has a Q maximum at $f_m = 82$ Hz.

Although this is to our knowledge the first time f_m has been raised markedly above 50 Hz in Bi(2223) tapes, the increase with respect to the $l_t = 20$ mm sample is significantly less than expected. The reason for this only modest increase is still under investigation, but we suspect it might be due to some residual filament bridging [21] which we hope to reduce by further optimization of the tape fabrication procedure.

The effective resistivity ρ_e increases linearly with barrier thickness [19], but too thick barriers eventually hamper "current healing" around local weak spots in the filaments and thus decrease the overall tape j_c. Measurements of the current transfer length in these tapes, as yet unpublished but similar to those in [22], show the resistivity of the barrier layer to be $\sim 10^5$ times higher than the silver resistivity at $T = 77$ K. The fact that in comparison the increase in *effective* matrix resistivity ρ_e is modest is explained by the unhampered Ag path at the disposal of coupling currents once they crossed a single barrier. We are therefore focusing our efforts on the second preparation method described in Sec. 2 (Fig. 1b).

4. CONCLUSIONS

1. The new concept of an oxide barrier has been successfully realized, producing a strong effect on electrically decoupling filaments and reducing ac losses of Bi(2223) tapes. This new type of tapes also has relatively good transport and mechanical properties.

2. The j_c values of the tapes have been improved by a modified powder-in-tube method using a two-axes rolling process and by a new intermediate deformation technique, which lead to a very uniform deformation.

3. The square-shape wires and tapes with the oxide barrier made by the two-axes rolling method can be twisted down to 3 mm and 11 mm in twist pitch length, respectively, by a novel and scalable way. The twisted tapes still have a relatively high j_c value.

4. The mechanical properties were checked by bending the tapes to different radii and measuring their j_c values. For the tapes with and without oxide barrier, the critical bending strain is 0.32% and the j_c values at 0.6% bending strain are 75 and 85% of the initial value, respectively.

5. Several experiments were used to demonstrate that electrically insulating $BaZrO_3$ barrier layers around the filaments of Bi(2223) tapes are effective in reducing the coupling currents between them. At present a corresponding ac loss reduction is still limited either to low frequencies or to the saturation loss at high amplitude and at all frequencies, but the realistic prospect of further suppression of coupling currents leads us to believe that the loss reduction at 50 Hz will be achieved for all current or field amplitudes in the foreseeable future.

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