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BPSCCO–Ag powders by a wet method and a low-carbon content for tape preparation

G. GUIDI, M. SOLCA, P. L. VILLA*
Dipartimento di Chimica Industriale e Ingegneria Chimica, Politecnico di Milano, 20133 Milano, Italy

L. GHERARDI
Pirelli Cavi S.p.A. Divisione, Italy

The preparation of BPSCCO–Ag composites by an innovative wet method is described, which allowed admixing of silver in the form of a solution to the solution containing all other cations, and the decomposition of the organic matter in a mild and reproducible way. The citrate route was used without the use of nitrates, ethylene glycol or other reticulating or complexing agents. After drying, silver was found to be dispersed in the powder in metallic form. The powders were treated in an atmosphere containing nitrogen oxides in order to reduce the carbon content. The powders so prepared were suitable for ''powder in tube'' processing in that they were very reactive toward the 2223 formation and may contain only the desired phases: 2212, metallic silver and Ca_2PbO_4 which, when present in excess, produce a flux that helps texturing and full phase conversion. Preliminary results on the high-purity samples prepared by the powder in tube process show a steady increase of J_c, with the absence of a maximum of J_c with number of treatments, and high texturing of the powder inside the tube.

1. Introduction

The traditional techniques for BSCCO preparation involve repeated mixing, milling and sintering of the oxides and/or carbonates of bismuth, strontium, calcium and copper. With such a process, the time required to obtain acceptable homogeneity is very long and serious contamination may occur. These methods do not allow us to obtain a good interspersion of the cations and lead to high grain dimensions; resulting in low-quality and little-reproducible superconducting powders [1].

In contrast, the wet methods, which lead to highly reactive homogeneous powders, require lower-temperature processing and offer many advantages: homogeneous mixing, good stoichiometric control and the production of active submicrometre-size particles in a relatively short processing time. The lower calcination temperature with powders produced by the wet routes is even more favourable when lead is present in the powder because, in these conditions, the loss of lead by evaporation is reduced and more easily controlled.

Several techniques are reported in the literature, e.g. the coprecipitation of carbonates and oxalates and the alkoxide route. These methods, however, still hold some disadvantages. Coprecipitation suffers from incomplete precipitation, non-uniformity and material loss during precipitation and washing. The alkoxide route has many preparation steps which are all critical for the properties of the final material. Moreover, it requires expensive chemicals.

The citrate route is peculiar in that, starting from a metallic cations solution, it leads, after drying, to an amorphous powder where cations are uniformly distributed at the atomic scale and to a well-controlled stoichiometry and small grain size of the powder. This method allows for shorter reaction time without the formation of undesired phases.

Nitrate salts are often used in the traditional citrate route owing to their great solubility, but during concentration and decomposition the large concentration of nitrates in burnable organic materials can be risky, especially for industrial scale applications either for explosions or for nitrous red smoke release.

In this work an effort was made to eliminate nitrates using oxides, carbonates and acetates. Considerable difficulties were found in bismuth oxide dissolution, because bismuth does not form soluble citric acid complexes. To overcome this problem, the use of ethylene diamine tetra acetic-acid (EDTA) or ethylene glycol is proposed in the literature [2–4]. In this work, in addition to citric acid, propionic acid was also used which is a non-complexating substance readily evaporated from the mixture.

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* Author to whom all correspondence should be addressed. *Present address*: Dipartimento di Chimica, Ingegneria Chimica E Materiali, Università degli Studi di L'Aquila, 67040 Monteluco di Roio-L'Aquila, Italy.

In order to improve the characteristics of the final products, particularly where mechanical properties are concerned, and in order finally to obtain ceramic superconductors–noble metal composite powders, addition of a noble metal solution to the BiPbSrCaCu starting solution was considered.

The present research used the BSCCO–Ag system because it offers favourable mechanical properties, stability in an ambient atmosphere and is one of the few metals chemically inert with the superconductor phase. Moreover, the high oxygen diffusivity in silver permits a better oxygenation during calcination.

The technique proposed here starts from an aqueous solution of bismuth, lead, strontium, calcium and copper cations with citric acid and ammonium hydroxide. If a BPSCCO–Ag composite is required, it must be added with a solution obtained by complexing Ag_2O with citric acid and ammonium hydroxide. The resulting solution is then dried under vacuum and the solid so prepared is decomposed in a controlled atmosphere (oxygen and nitrogen) at 350 °C; the precursor thus obtained contains bismuth, calcium and copper oxides, calcium carbonate and metallic silver well interspersed.

The citrate route, as well as all the methods using organic compounds, leads to powders containing about 3 wt % residual carbon, most of which is due to strontium and calcium carbonate. Carbon is not completely eliminated during calcination and its segregation at grain boundaries seems to reduce the superconducting qualities. In order to reduce carbon content, the precursor was heated in an atmosphere containing NO_2.

2. Experimental procedure

2.1. Preparation of the precursor

The preparation method includes three steps: preparation of the solution, drying and decomposition. Because the preparation of the solution is rather a difficult task, some details about the production of 137.5 g BPSCCO with 10 wt % Ag are reported.

Some of the superconductors were prepared with high-purity chemicals in order to reduce further the impurities at grain boundaries, which is considered to be one of the principal causes of the weak link [5].

2.2. Preparation of the starting solution

2.2.1. High-purity chemicals

The chemicals used were as follows: bismuth oxide (Strem Chemicals) 99.9998%; lead acetate (Strem Chemicals) 99.999%; strontium carbonate (Strem Chemicals) 99.999%; calcium carbonate (Strem Chemicals) 99.9995%; copper acetate (Aldrich) > 99.99%; silver nitrate (Strem Chemicals) 99.9995%; citric acid (Carlo Erba) RPE; acetic acid (Aldrich) > 99.99%; Propionic acid (Carlo Erba) RPE; ammonium hydroxide 30% (Aldrich) > 99.99%; tetramethylammonium hydroxide 10% (Carlo Erba) RPE; and water (Carlo Erba) RS per HPLC.

2.2.2. Preparation of the solutions

Bismuth. 48.6314 g bismuth oxide was poured into 200 ml propionic acid heated to 100 °C; when the temperature reached 136 °C, a hot (90 °C) solution, prepared by dissolving 150 g citric acid in 150 ml water, was added, under stirring, and then ice-cooled. 1000 ml ammonium hydroxide was cooled in an ice-bath and the bismuth solution was then added. A transparent and colourless solution was obtained.

Lead. 25 g citric acid was poured into 75 ml water, then 17.5956 g lead acetate was added. A white suspension was obtained which was ice-cooled and added to 165 ml ammonium hydroxide in an ice-bath to obtain a transparent and colourless solution.

Strontium. 225 g citric acid was added to 10 ml acetic acid and 250 ml water. To this solution, 34.2397 g strontium carbonate was added. The whole was heated to 60 °C until the solution became clear and colourless. Addition to ammonium hydroxide, even if not detrimental, was not necessary.

Calcium. 25.5352 g calcium carbonate was added to 120.5 g citric acid previously dissolved in 375 ml water. After ice-cooling, it was poured into 200 ml cold ammonium hydroxide. The solution so obtained was limpid and colourless. This solution should be prepared last because it is the only one that presents stability problems.

Copper. 36.5 g citric acid was poured into 115 ml water and 69.4565 g copper acetate was added. The transparent solution was cooled in an ice-bath and added to 65 ml ammonium hydroxide.

Silver. 20.66 g silver nitrate was poured into 70 ml water. 121 ml tetramethylammonium hydroxide 10% was added: a brown precipitate of Ag_2O was obtained which was washed on a G4 filter until neutral pH of the filtered water was recorded. 9.5 g citric acid was dissolved in 350 ml water; Ag_2O was then added. The resulting suspension was ice-cooled and 140 ml previously cooled ammonium hydroxide was added. A clear solution containing silver was obtained.

2.3. Concentration and drying of the solution

In an ice-bath, the solutions containing calcium, lead, bismuth and strontium were added to the solution containing copper; it was important to add the strontium-containing solution carefully because this led to an increase in the temperature owing to its acidity. The final volume was about 3000 ml.

The solution was then put into a rotary evaporator at 90 °C and concentrated under vacuum until a homogeneous viscous product was obtained; this happened when the volume of the collected distillate was about 1400 ml. Then the silver-containing solution was added and the evaporation was carried out until a homogeneous viscous product was obtained and the distillate was about 1000 ml in volume. The viscous slurry so obtained was dried in a vacuum oven with nitrogen traps. After treatment at 90 °C for 15 h, the temperature was raised to 130 °C at a speed of $20\,^{\circ}C\,h^{-1}$ and left for 24 h; at the same speed the product was heated to 200 °C and the drying was completed until significant quantities of condensed matter were collected inside the traps.

2.4. Decomposition of the organic matter

The product, a black powder, was ground to a particle size < 0.297 mm and then placed in a downwards gas flow reactor. The powder was heated to 200 °C at a speed of 40 °C h^{-1} and left for 15 h in a nitrogen flow (0.5 min^{-1}). At the same speed the furnace was heated to 320 °C in nitrogen (99%) and oxygen (1%) with a total flow rate of 8 l min^{-1}. A slow oxidative decomposition was started and controlled according to two criteria: (a) the temperature difference across the reactor wall (due to reaction heat), which was kept to < 20 °C; (b) the oxygen consumption of the precursor, which was kept to $< 1\%$ of the total flow. To fulfil these criteria the oxygen content in the gas was kept at low values (1%) for most of the overall heating.

When the oxygen consumption was reduced, the external temperature was raised up to 350 °C at the rate seen before. The inlet oxygen percentage was gradually increased to 2%, 4%, 10%, 20% and finally to 100%, changing the percentage only after the oxygen consumption became less than 1%.

A typical decomposition scheme and process data are shown in Fig. 1 and Table I.

2.5. Processing of the powder in a nitrogen oxide-containing atmosphere

After the decomposition of the organic matter, a powder containing a mixture of metallic silver, calcium and copper oxides, and calcium and strontium carbonates, was obtained. The carbon associated with calcium and strontium carbonates can only be partially eliminated during calcination because carbonates decompose at equal or higher temperatures to the calcination temperature (825 °C), as shown in the thermogravimetric analysis curves (TG) in Fig. 2a and b. The residual carbon, segregated at grain boundaries of the superconducting phase, seems to be one of the most important factors limiting the critical current; therefore, it is important to reduce the carbon content before annealing.

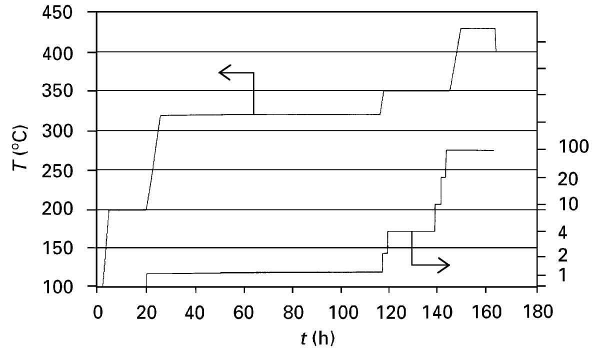

Figure 1 Typical decomposition scheme.

TABLE I Typical decomposition data

Temperature (°C)	Time (h)	Oxygen (%)	φ (cm^3 min^{-1})
200	15	0	500
320	90	1	8000
350	2	2	8000
350	19.5	4	8000
350	2	10	8000
350	1.5	20	8000
350	1	50	8000
350	1	100	8000
430	14	100	2000

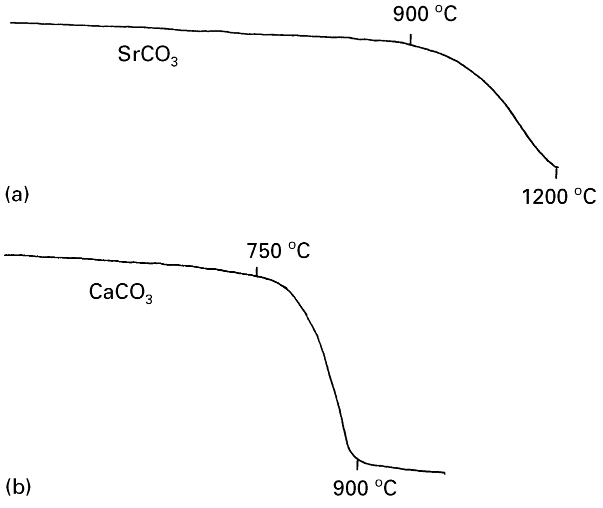

Figure 2 (a) TG curve of $SrCO_3$. (b) TG curve of $CaCO_3$.

An effort was made to transform carbonates into nitrates using appropriate thermal treatment in an NO_2 containing gas flow, here termed nitrification. In this case, the reaction atmosphere was obtained by mixing two gases; the first was oxygen enriched with ozone by allowing it to flow through an ozonizer consisting of four Berthelot tubes whose electrodes were charged at 15 kV; the second one was a mixture of nitrogen and nitrogen oxide.

In order to eliminate the nitrates from the precursor, the nitrified samples were subjected to a thermal treatment called denitrification in fluent oxygen. Unlike carbon, nitrogen is easily eliminated at temperatures lower than 825 °C, as shown in the TG curves in Fig. 3a and b.

The two treatments were held at the same T_{max} (760 °C) and for 24 and 12 h, respectively.

2.6. Calcination – sintering

During this phase the oxide mixture was transformed into the superconducting oxide $Bi_2Sr_2Ca_2Cu_3O_x$. The annealing was performed inside a quartz reactor in a nitrogen and 7% oxygen atmosphere. The precursor was calcined in powder or pellet form; the powder was cold pressed into 13 mm diameter pellets by applying a uniaxial pressure of 100 MPa.

The sintering temperature was 825 °C; this parameter is critical because the 2223 phase formation occurs in a narrow interval.

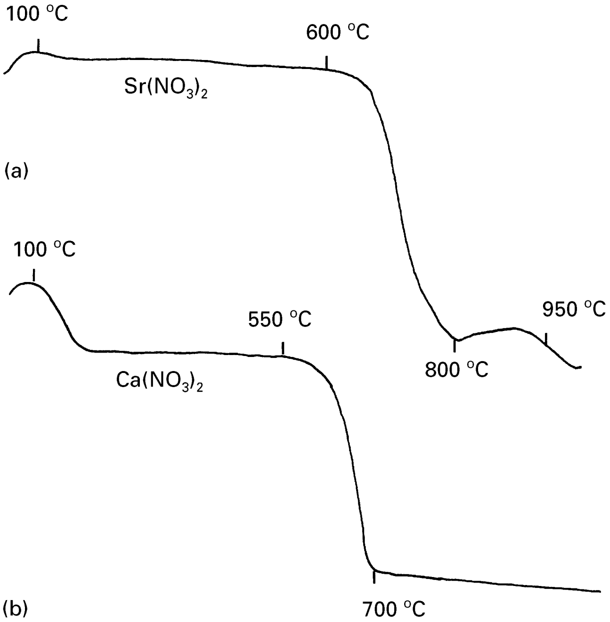

Figure 3 (a) TG curve of $Sr(NO_3)_2$; (b) TG curve of $Ca(NO_3)_2$.

2.7. Preparation of tapes

Critical current measurements were made on tapes prepared with two precursors, one with 10% Ag and one without; high-purity chemicals were used in order to reduce the impurities at grain boundaries. Tapes of different thicknesses were prepared and subjected to several thermal treatments, each one followed by a mechanical compression; all of them showed a remarkable improvement of the J_c value after each annealing.

The thin tape was drawn to 100 μm thickness and subsequently subjected to 6000 MPa with a further thinning to 95 μm. For the thick ones, the parameters used were 280 μm, 2500 MPa and 250 μm.

The first thermal treatment consisted in an annealing for 24 h at 825 °C plus 50 h at 810 °C in a nitrogen and 8% oxygen atmosphere.

After a compression equal to the first one, a second thermal treatment was performed similar to the first at 815 °C for 100 h.

The third and the fourth anneals consisted in 24 h at 825 °C, plus 100 h at 815 °C.

3. Results and discussion

3.1. Preparation of the solution

Some of the details of the dissolution of the various elements may seem trivial, but they are essential for a successful operation.

In some cases (bismuth, lead, calcium, copper, silver) the solutions are ice-cooled ($T \approx 10\,^{\circ}\mathrm{C}$); this is necessary to prevent the formation of a precipitate before the correct complex with citric acid is formed.

Pouring the main solution into cold ammonia and not vice versa (bismuth, lead, calcium, copper, silver) allows for a very sudden and relevant increase of pH which also helps the dissolution.

The dissolution of bismuth can be obtained in different ways, as found in the literature [2–4], using ethylene glycol, which tends to form reticulate complexes with citric acid and thus might present some problems in decomposition, or EDTA which, besides being an expensive reagent, is hard to eliminate. In this work, bismuth oxide was dissolved in propionic acid which is a cheap chemical and easily distilled off under vacuum.

We suppose that silver is forming, at least initially, a true solution with the other citrate solution, because of its limpid and transparent appearance. The segregation of metallic silver seems to occur at least partially before the decomposition of the organic matter, e.g. during drying.

3.2. Drying of the solution

After the drying step, an amorphous powder was obtained as shown by the X-ray diffraction (XRD) pattern of the powder dried at 200 °C (Fig. 4). Only the metallic silver peaks may be observed, with an estimated crystal size of about 30 nm (Scherrer method). The structure of the other elements is clearly in the amorphous state which is an indication of a good interspersion of the metallic cations.

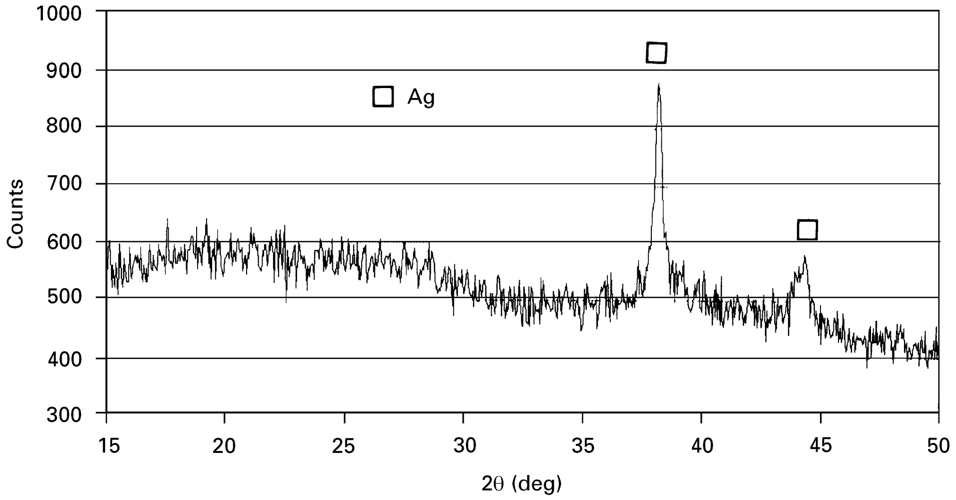

Figure 4 XRD pattern of a dried sample.

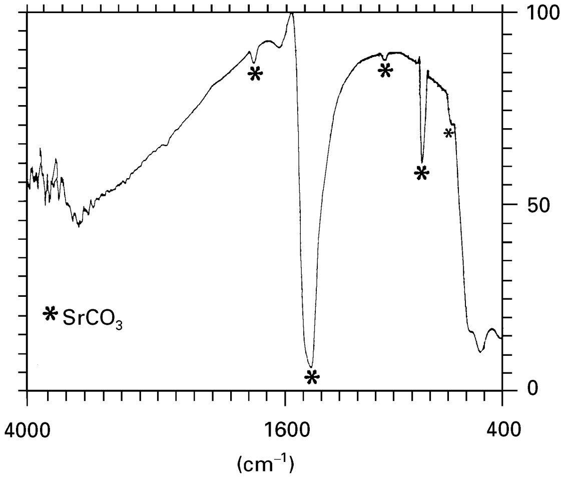

Figure 5 Infrared pattern of a decomposed sample.

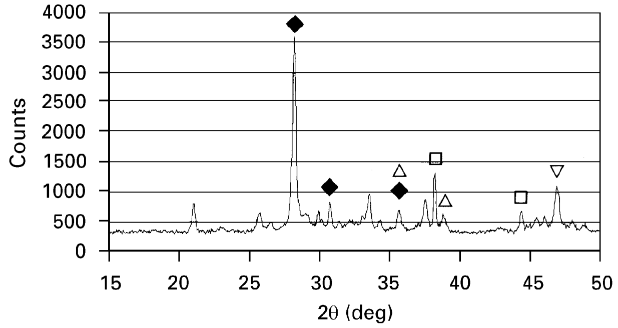

Figure 6 XRD pattern of a decomposed sample. (◆) Cu_4O_3, (△) CuO, (□) Ag, (▽) Bi_2O_3.

3.3. Decomposition of the organic matter

This is one of the critical unit operations of the preparation: in order to carry it out in a smooth and reproducible way, the oxygen content in the NO_2 flow is initially only 1% in order to control the heat evolution due to the oxidation. At the end of this step, the powder contains bismuth, calcium and copper oxides, calcium and strontium carbonates and eventually metallic silver (Figs 5 and 6).

3.4. Reduction of the carbon content

The decomposed powders were divided into several portions, each of which was submitted to different thermal treatments in order to optimize the conditions for a minimum residual carbon. The nitrification tests were carried out in a wide temperature range (460–810 °C), because the reactions involved give rise to some uncertainty about the selection of the temperature.

Indeed, while the oxidation of NO to NO_2 increases both for thermodynamic and kinetic reasons as the temperature decreases, the gas–solid reactions and the phase transformation of the powder should speed up with temperature.

Fig. 7 shows the variation of the carbon content with the nitrification temperature. Two different batches of powder were used, prepared with a slightly different procedure. In fact, while the preparation used for sample M5 made some use of nitrates [6], the second powder (GM2) was prepared according to the recipe reported here. In both cases, the best results were obtained in the 710–810 °C range.

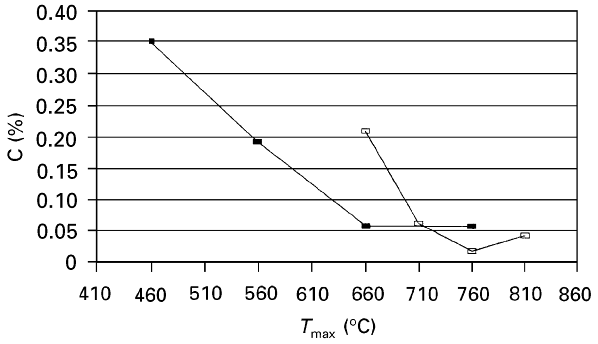

Figure 7 Carbon content versus T_{max} of nitrification.

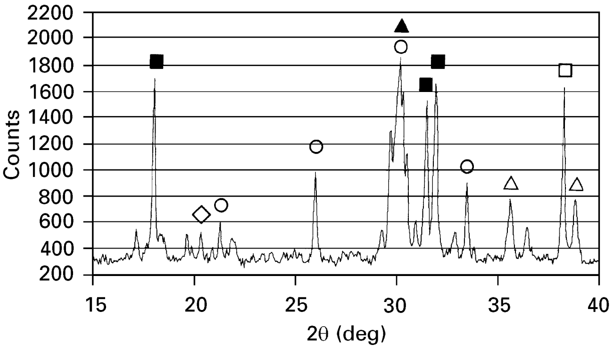

Figure 8 XRD pattern of a sample nitrified at 710 °C. (■) Ca_2PbO_4, (◇) $SrPbO_3$, (○) 2201, (△) CuO, (□) Ag.

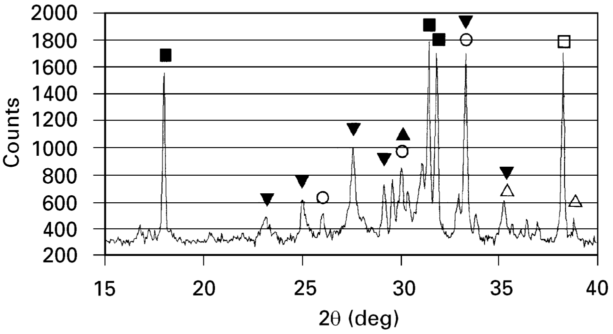

Figure 9 XRD pattern of a sample nitrified at 760 °C. (■) Ca_2PbO_4, (▲) Sr_2PbO_4, (▼) 2212, (△) CuO, (○) 2201, (□) Ag.

To determine the best temperature, we followed a criterion based on phases present, phase ratios and magnetic susceptibility of the sintered pellets obtained at 825 °C for various treatment times.

The patterns reported in Figs 8–10 show that the only differences are between the superconducting phases: phases 2201 and 2212 appear at 710 and 760 °C. Phase 2223 appears at 810 °C, which is undesirable before tubing.

With the precursor nitrified at the three different temperatures, pellets were made and calcined for increasing times (25, 50, 100, and 150 h) and the susceptibility was measured at different external magnetic

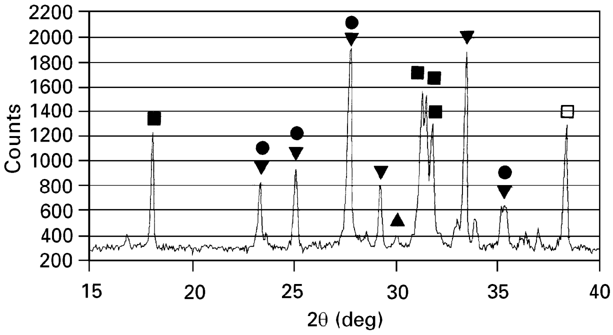

Figure 10 XRD pattern of a sample nitrified at 810 °C. (■) Ca$_2$PbO$_4$, (▲) Sr$_2$PbO$_4$, (●) 2223, (□) Ag, (▼) 2212.

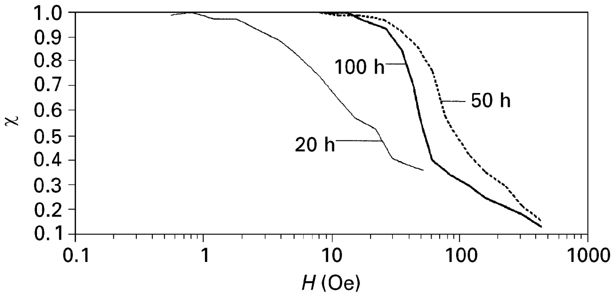

Figure 11 Characteristic χ versus *H* of pellets calcined for increasing times prepared with powder nitrified at 710 °C. (—) GM2P71A, (– – –) GM2P71B, (—) GM2P71C.

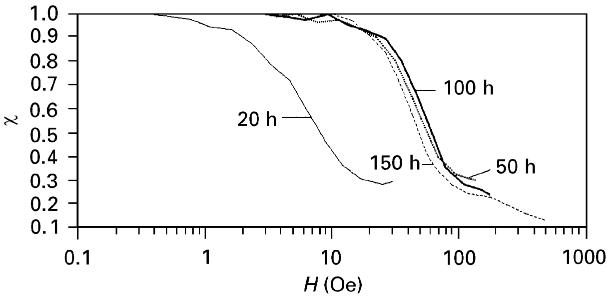

Figure 12 Characteristic χ versus *H* of pellets calcined for increasing times prepared with powder nitrified at 760 °C. (—) GM2P76A, (···) GM2P76B, (—) GM2P76C, (– – –) GM2P76D.

fields. In Fig. 11 it appears that, for the pellets made with powder nitrified at 710 °C, χ regresses after the third annealing (100 h); for the pellets prepared with powder nitrified at 760 and 810 °C (Figs 12 and 13) the regression appears only after the fourth annealing (150 h) and is less relevant for the pellet calcined at 710 °C, which is more evidently shown in Fig. 14.

In order to evaluate the growth rate of the 2223 phase, the XRD patterns made on the ground pellets were used; the 2223 versus (2212 + 2223) phase intensities ratio is representative (Fig. 15): this evaluation was made using the 0 0 1 0 peak of the 2223 phase and the 0 0 8 peak of the 2212 phase.

In conclusion, in low residual carbon phases present in the nitrified powder, their stability after prolonged annealing and critical magnetic field indicate 760 °C as the nitrification temperature.

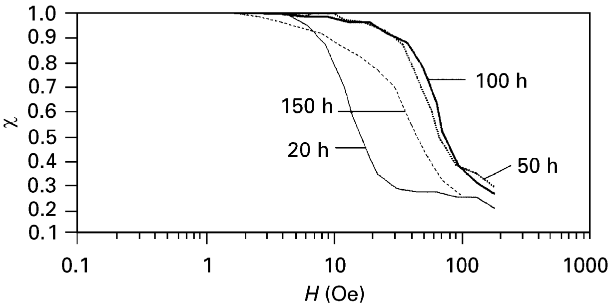

Figure 13 Characteristic χ versus *H* of pellets calcined for increasing times prepared with powder nitrified at 810 °C. (—) GM2P81A, (···) GM2P81B, (—) GM2P81C, (– – –) GM2P81D.

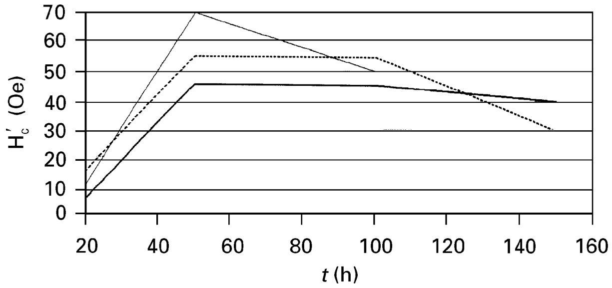

Figure 14 Characteristic H'_{co} versus t_{att} at (—) 710 °C, (—) 760 °C, and (– – –) 810 °C.

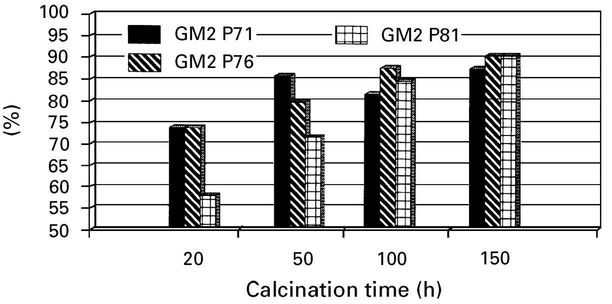

Figure 15 2223/(2223 + 2212) phase intensities ratio at various t_{att}: this evaluation was made using the 0 0 1 0 peak of the 2223 phase and the 0 0 8 peak of the 2212 phase.

Further attempts were made to improve the nitrification process by (a) evaluation of the best oxygen percentage in the nitrifying mixture, (b) double nitrification, (c) humidification and further nitrification, (d) using a nitrifying atmosphere containing only N$_2$ and O$_2$.

(a) The NO$_2$ amount used for nitrification depends on the equilibrium constant of the reaction NO + 1/2 O$_2$ = NO$_2$; at constant temperature, this constant depends on the partial pressure of the gases and presents a maximum when oxygen is around 33% of the total mixture.

On reducing the oxygen to 33% and keeping 760 °C as the nitrification temperature, we did not observe a reduction of residual carbon but a considerable simplification of the phase composition (Figs 16 and 17), indeed only the desired phases appear, i.e. 2212, Ca$_2$PbO$_4$ and metallic silver.

(b) A second nitrification was performed either on a calcined or simply on a once-nitrified powder.

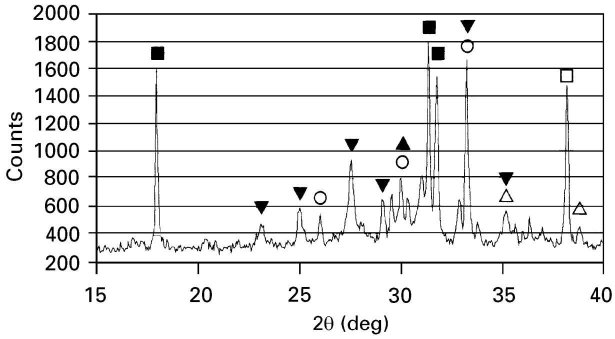

Figure 16 XRD pattern of a sample nitrified with 50% oxygen. (■) Ca$_2$PbO$_4$, (▲) Sr$_2$PbO$_4$, (▼) 2212, (△) CuO, (○) 2201, (□) Ag.

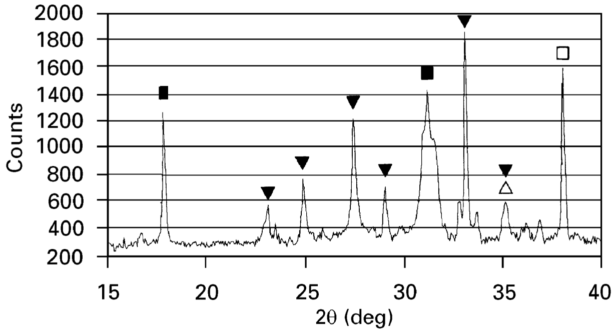

Figure 17 XRD pattern of a sample nitrified with 33% oxygen. (■) Ca$_2$PbO$_4$, (△) CuO, (▼) 2212, (□) Ag.

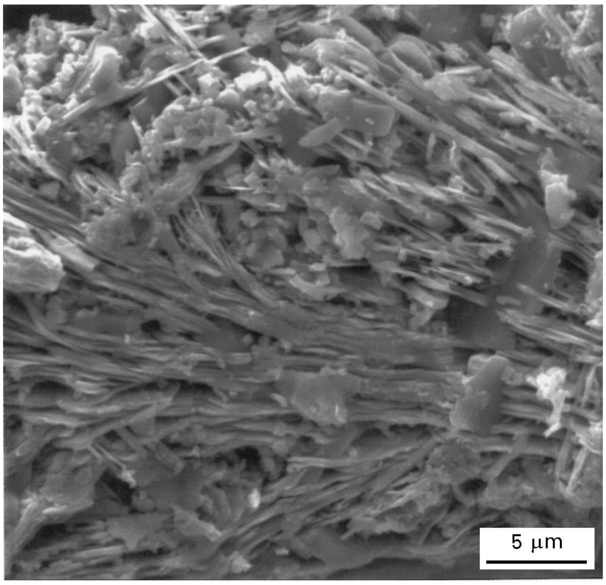

Figure 18 Scanning electron micrograph of a nitrified sample.

The results did not justify such a complication. Indeed, in the former sample, no change was recorded, while in the latter powder, a small carbon reduction was noted together with a deterioration of the morphology, as the flakes reduction shows (Figs 18 and 19).

(c) Considering that residual carbon in the precursor powder is located at the grain boundaries as superficial carbonates, a method to reduce the carbon content could be to humidify the samples. It is commonly known that water vapour and CO$_2$ often compete for the same surface sites. Therefore, a nitrified sample was put in a water vapour-saturated atmosphere at 40 °C for 24 h.

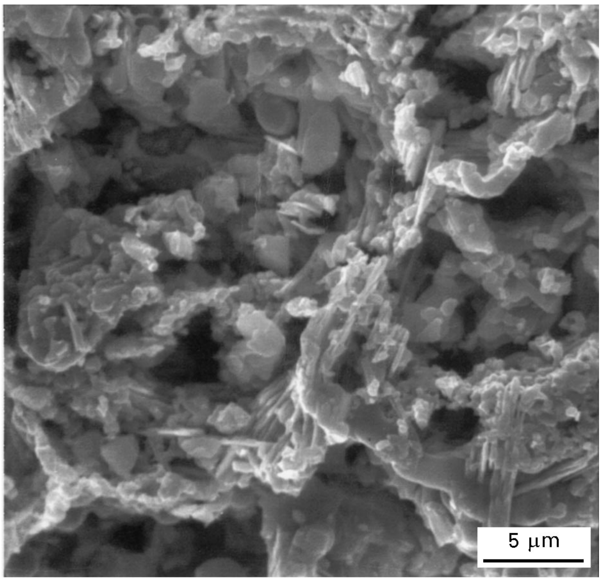

Figure 19 Scanning electron micrograph of a sample after a second nitrification.

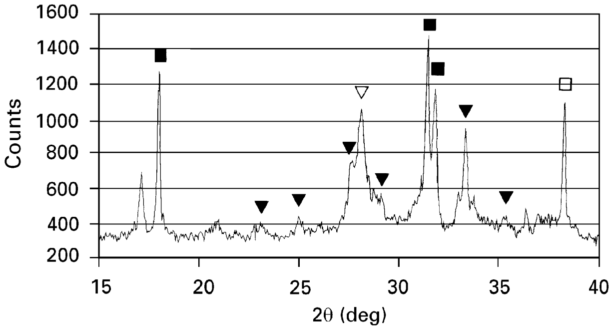

Figure 20 XRD pattern of a sample after nitrification and humidification. (■) Ca$_2$PbO$_4$, (□) Ag, (▼) 2212, (▽) Bi$_2$O$_3$.

XRD and microscope analysis show that humidification itself at 40 °C causes deep modification in the phase composition (Fig. 20) and morphological variations (Fig. 21). After a further nitrification, carbon was reduced from 0.05% to 0.03%, but the initial phases did not reform (Fig. 22), and morphological deterioration, due to the second nitrification, appeared (Fig. 23).

(d) To verify the efficacy of the NO$_2$ gas, a blank thermal treatment was performed in an atmosphere composed of nitrogen and 33% oxygen, with no NO$_x$.

The XRD pattern (Fig. 24) and scanning electron micrograph (Fig. 25) show that phase composition and morphology differ from the nitrified sample; the residual carbon is 1.5 times greater.

3.5. Critical current measurements

J_c measurements on tapes are reported in Fig. 26. After each thermal treatment, a considerable J_c improvement was recorded and a further increase could be presumed with another annealing. Tapes prepared

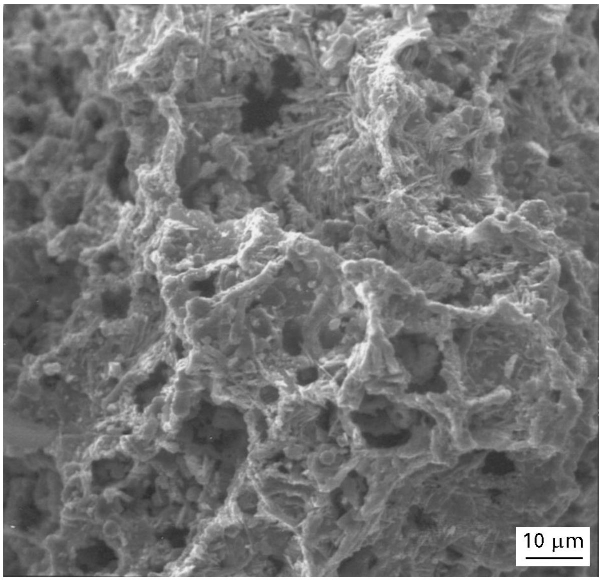

Figure 21 Scanning electron micrograph of a sample after nitrification and humidification.

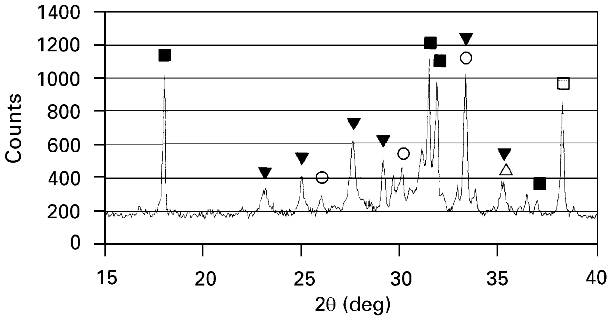

Figure 22 XRD pattern of a sample after nitrification, humidification and a second nitrification. (■) Ca_2PbO_4, (△) CuO, (▼) 2212, (□) Ag, (○) 2201.

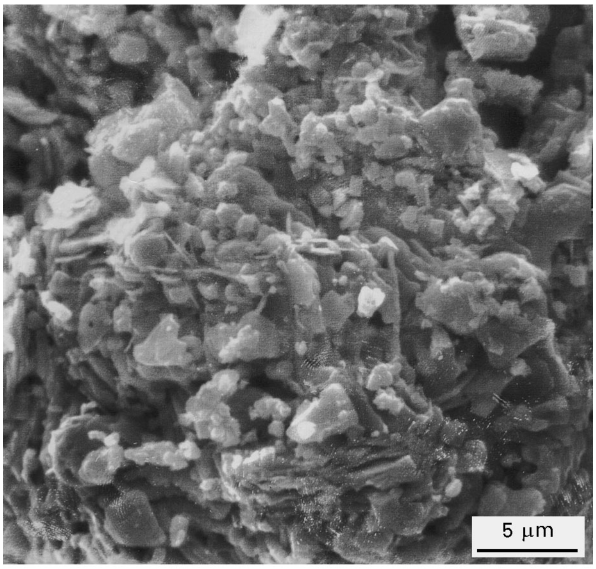

Figure 23 Scanning electron micrograph of a sample after nitrification, humidification and a second nitrification.

with silver-containing powder show higher critical current than tapes containing powder without silver; in thin tapes, higher J_c than in thick tapes were detected, most likely because of the higher texturing achievable on the former, which has a more favourable geometry and comparatively larger silver–superconductor interface area.

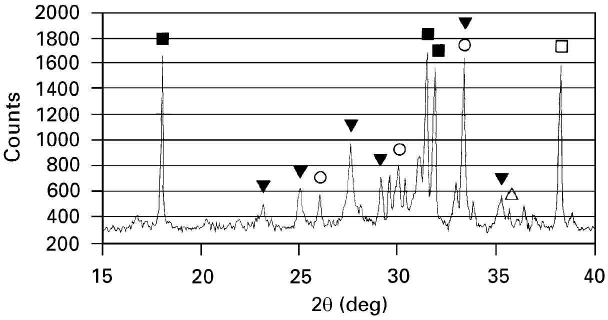

Figure 24 XRD pattern of a sample after a thermal treatment performed with no NO_x. (■) Ca_2PbO_4, (△) CuO, (▼) 2212, (□) Ag, (○) 2201. GM5 = 0% Ag; GM6 = 10% Ag; A, B, thin; C, thick.

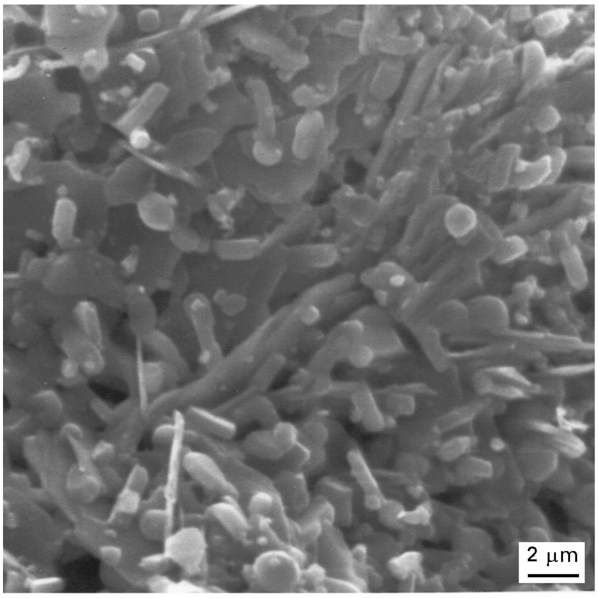

Figure 25 Scanning electron micrograph of a sample after a thermal treatment performed with no NO_x.

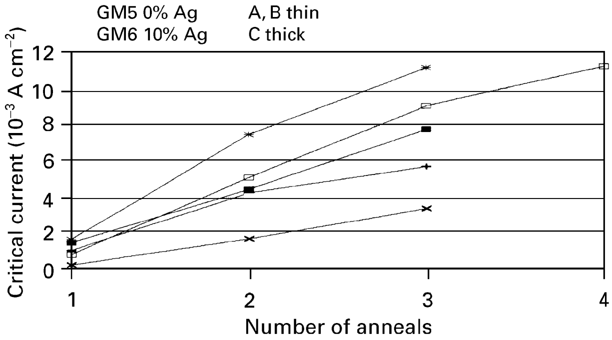

Figure 26 J_c measurements. (■) GM5A, (+) GM5C, (∗) GM6A, (□) GM6B, (×) GM6C.

High density and orientation were, in fact, shown (see Figs 27 and 28) to be a typical feature of the superconductor obtained following the procedure reported in this work.

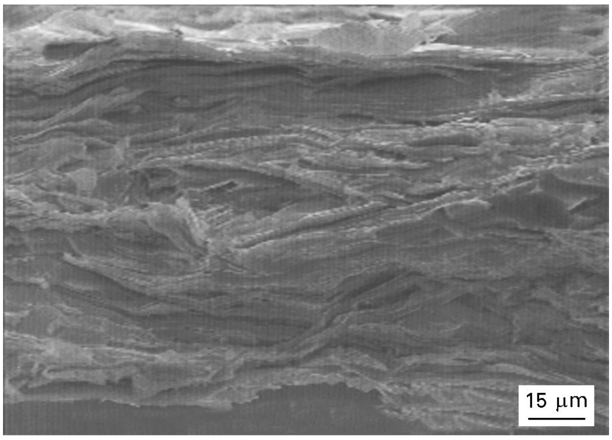

Figure 27 Scanning electron micrograph of the powder inside a tape.

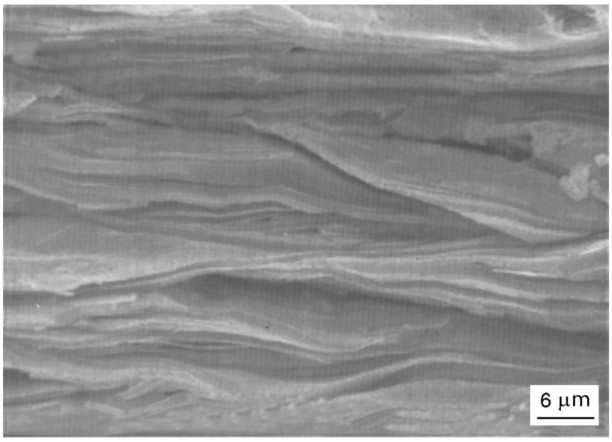

Figure 28 Scanning electron micrograph of the powder inside a tape.

4. Conclusion

One of the remarkable aspects of this work is the realization of a wet method following the citrate route for BPSCCO superconductors preparation, with eventual silver addition, but without making use of nitrates. This procedure, which has been patented [7], leads to excellent interspersion of the cations, and of silver, which has been added as a solution.

In order to improve the critical current, considerable efforts were made to obtain clean grain boundaries, by using high-purity chemicals and aiming at the reduction of residual carbon, which seems to be one of the most important factors that reduce the critical current.

A nitrifying treatment was developed to reduce carbon content and to simplify phase composition (Figs 16 and 17) obtaining only the desired phases: 2212, Ca_2PbO_4 and metallic silver. The results of the sample characterization showed that increasing the nitrification temperature leads to a residual carbon reduction until a minimum value of 0.019% at 760 °C.

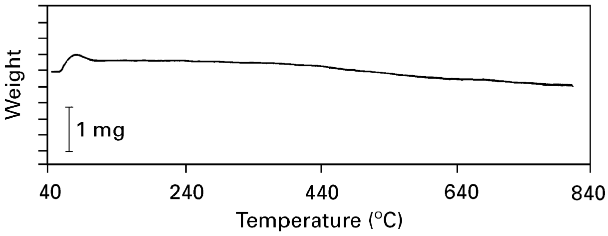

Figure 29 TG curve of a nitrified sample (260 mg).

A further positive result is the high reactivity of the powders in the superconducting phase formation; the 2223 phase is 90% of the total after 100 h, as shown in Fig. 15; usually, much more time is required to obtain the same results with other methods.

The TG curve of a nitrified sample brought to the annealing temperature (Fig. 29) shows very little weight loss during calcination. This is an important characteristic in tapes production because it reduces porosity during calcination. In fact, morphological analysis by SEM on the sintered material showed a very dense and well-aligned microstructure. J_c values above 10^4 A cm^{-2} were repeatedly measured on tape specimens fabricated with the silver-containing powder, and confirmed the homogeneity and good connectivity of the resulting superconducting material.

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