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Development of YBCO coatings by atmospheric plasma spraying

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Abstract

Superconducting Y–Ba–Cu–O thick films were produced by the atmospheric plasma spraying method. The effect of processing parameters (powder characteristics, spraying parameters) on the coatings properties was studied. X-ray diffraction analysis, SEM studies combined with EDS microanalysis and scratch test experiments were carried out in order to characterize the adhesion of the coatings to the substrate, the coatings morphology the thickness and crystalline structure as well as the powder phase transformations during spraying. For restoring the superconducting phase after deposition, the coatings were heated in oxygen in the temperature range 750–930°C. It was shown that the quality of the coatings and the adhesion to the substrate are greatly dependent on the deposition conditions. By calcining in oxygen under the appropriate conditions coatings consisting of the pure superconducting phase can be obtained. © 2000 Published by Elsevier Science Ltd. All rights reserved.

Keywords: Calcination; Microstructure; Oxide superconductors; Plasma spraying; Superconductivity

1. Introduction

After the discovery of superconductivity numerous efforts have been performed not only to expand the possible application fields of superconductors but also to come to commercially available forms of these materials. One of the most promising materials in the field of superconductivity is the $YBa_2Cu_3O_7$ (<123>) oxide. Especially the development of thin and thick superconducting <123> films can be employed in high-quality factor filters, delay lines, Josephson junctions for high speed, low power switching, bolometers, motor applications, magnetic field sensors, magnetic shielding, microwave cavities, etc.[1–4] Recently, research efforts are focussed on the development of thin films on flexible substrates and foils with the aim of producing coils of high critical current density.[5] The techniques for the development of thin films such as magnetron sputtering or pulsed laser ablation deposition[6,7] require the development of high quality YBCO targets.

Like most ceramic materials, YBCO is brittle and thus difficult to shape into intricate objects. Thermal spraying for the development of thick YBCO coatings could be a viable solution to this problem. This technique is very promising for YBCO processing due to the fact that it offers high deposition rate, non-vacuum operation, use of a wide variety of materials as substrates and the ability to coat large areas as well as substrates with complicated shapes.[8–10] Thus, the atmospheric plasma spraying technique has been investigated[11–14] for processing superconducting materials for industrial applications (magnetic shielding, targets for thin film deposition techniques, etc.). It can produce coatings that are well adhered to the substrate offering a solution to the inherent problem of brittleness of these materials in the bulk form.[15]

However, although the plasma spraying technique offers many advantages, the development of high quality YBCO coatings requires a very careful selection of processing parameters. The feedstock powder characteristics (morphology, particle size distribution) are important factors. Powder selection has to take into account the rheology problems that might occur from pneumatic powder transportation to the plasma spraying gun as well as the plasma-particulate interaction during spraying in terms of thermal exchange for the melting of the particles.[16,17] The influence of other parameters that have to do with the spraying itself such as internal feeding of the powder into the plasma or powder feeding from outside the plasma gun (external feeding) and the spraying angle, on coating quality is reported in the literature.[18,19] The selection of the right

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substrate has to consider the thermal expansion coefficients of both coating and substrate materials since thermal expansion mismatch leads to internal stresses, crack formation or even detachment of the coating during deposition.

Concerning plasma spraying of YBCO coatings, research efforts have focussed mainly on the development of coatings of the pure superconducting phase with good superconducting properties after a post thermal annealing process, since it has been shown that $YBa_2Cu_3O_{7-x}$ phase decomposes during spraying.[20–23] Thus, very few works refer to a parametric study in order to obtain good quality coatings in terms of microstructure and adhesion to the substrate, factors, which are very important especially when high power targets for the thin film deposition techniques (magnetron sputtering) are to be developed.[24,25]

In this paper, the development of thick YBCO coatings on Cu and stainless steel substrates with the atmospheric plasma spraying technique for the manufacturing of high power magnetron industrial YBCO targets is studied. Coating quality is correlated with both starting powder characteristics and plasma processing parameters. This study forms part of the Brite Euram industrial project "MUST" dealing with the development of a cost effective multi-functional flexible high-temperature superconducting tape using sputter deposition of YBCO films on metallic substrates.

2. Experimental

For the production of thick Y–Ba–Cu–O coatings a Miller-Thermal 80 kW equipment was employed with Ar as the plasma and carrier gas. Flat rectangular Cu and Stainless Steel 304 specimens (30×30×2 mm, 30×15×2 mm, 15×15×2 mm) were used as substrates. Prior to spraying the substrates were grit blasted with Al_2O_3 grit (1000+500 μm) and ultrasonically cleaned.

A powder consisting of pure <123> YBCO phase composition was used as a feed powder for the coatings production. It was produced by spray drying (in a laboratory ICF spray dryer with a capacity of 6 l/h) of aqueous solutions of nitrate salts of Y, Ba and Cu (10% w/w). The spray dried powder obtained was subsequently calcined at 870°C for 40 h for the formation of the $YBa_2Cu_3O_{7-x}$ phase. After calcination, it was sieved in four different fractions (−100, −100 +63, −63 +40 and −40 μm) and the particle size distribution was measured by a laser particle size analyzer (Malvern 3600 E type). The fractions obtained were also characterized by X-ray diffraction (XRD) analysis (Siemens D-500, Cu K_α radiation) and scanning electron microscopy (SEM) studies (Jeol JSM 6300). These fractions were used as feeding powders in order to investigate the effect of the particle size distribution on the produced coatings quality. The powder was fed into the plasma through an external nozzle of 3 mm in diameter with a feeding rate adjusted at 8 g/min. The plasma power employed was 26 kW.

In addition, the effect of both the carrier gas pressure (30–85 psi) and the spraying distance (6–11 cm), on the coatings quality was studied. Plasma spraying gun was moved in two axes (a horizontal and a vertical one) with a velocity of 1.65 m/min in both of them.

During spraying, the temperature of the substrate was kept below 60°C by air-cooling. For this purpose, compressed air was blown through external fittings attached to the gun 3–4 cm below the exit of the powder.

After deposition, the microstructure of the coatings was investigated under scanning electron microscopy (SEM) (Jeol JSM 6300) combined with energy dispersive X-ray microanalysis (EDS) (LINK ISIS ultra thin window PENTAFET detector). Both unpolished top surfaces of the as sprayed coatings and polished cross sections of them were examined. The SEM studies on polished cross sections permitted also to characterize the homogeneity and thickness of the deposited layers. The deposition rate of the different applied conditions was measured by dividing the total thickness of the coating measured under SEM by the number of passes of the spraying gun over the substrate surface. Coating phase composition was studied with X-ray diffractometry (Siemens D-500 Cu K_α radiation). Adhesion of the coatings to the substrate was investigated carrying out scratching test measurements (CSEM REVETEST Scratch tester) with progressively increasing loads in the range 0–200 N. The load was applied by a diamond stylus vertical to the specimen which was moving with a speed of 9.79 mm/min and had a loading rate of 106.55 N/min. The point and consequently the load at which total loss of adhesion occurred was determined using scanning electron microscopy. The depth of the scratching test track before total detachment from the substrate was measured using a roughness profilometer (HOMELLTESTER T1000).

The sprayed coatings were subsequently heat treated in the temperature range of 750–930°C for 0 up to 20 h under flowing oxygen atmosphere and microstructural as well as phase composition characterizations were also carried out. In addition adhesion of the thermally treated coatings was investigated by scratching tests and track depth measurements as described above.

3. Results and discussion

3.1. Feedstock powder characterization

The production of coatings by atmospheric plasma spraying requires the use of appropriate powders in terms of morphology and size. Since pneumatic transportation is used for the powder to enter the plasma, powders with angular shapes and very small particle size, which is the situation for most commercially available YBCO powders, are unacceptable. That is why powders produced by spray drying were used in this study. Their morphology is shown in Fig. 1. They present a spherical homogeneous shape, each granule consisting of nanoparticles. In addition, their particle size distribution is totally controlled by the spray drying conditions, which makes them potentially superior in terms of rheological properties compared to powders prepared by the conventional solid state reaction route. The four different powder fractions used in this investigation for the coatings production had different particle size distributions and mean particle sizes (Fig. 2). The −100 μm powder fraction exhibits a broad particle size distribution with particle sizes ranging between 5 and 100 μm and a rather low mean size of 40 μm. The other three fractions are of very narrow particle size distributions. These fractions have been selected with the aim of investigating the effect of both the particle size distribution and of the mean particle size on coating properties. All four powders, after their calcination, consisted of a pure superconducting < 123 > phase along with some traces of $BaCuO_2$ and Y_2BaCuO_5 phases (Fig. 3a). The calcination procedure is an one step procedure at relatively low temperatures (870°C) and this is attributed to the fact that the nanoparticles within each spray dried granule react rapidly to form the < 123 > phase.

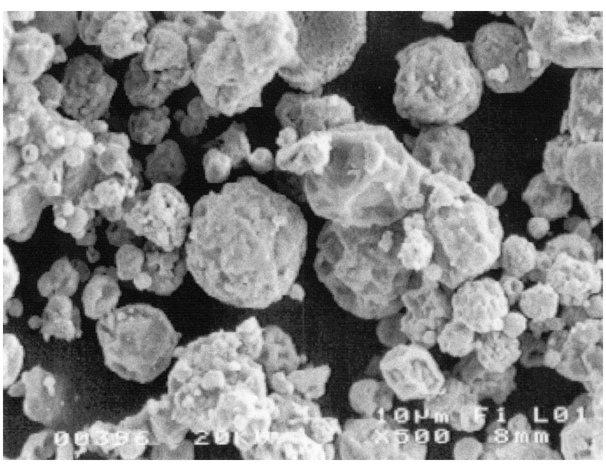


Fig. 1. SEM study of the powder produced by spray drying of nitrate salts of Y, Ba and Cu before calcination.

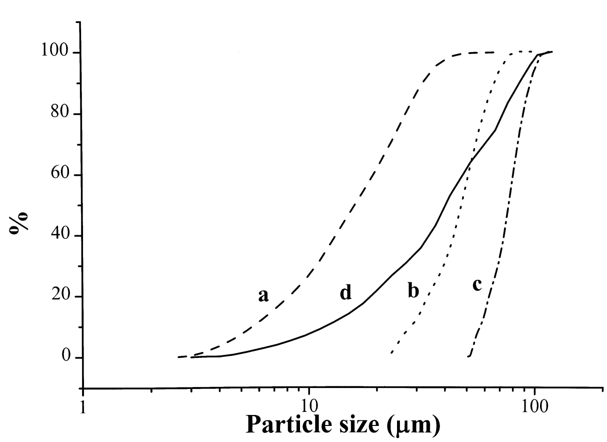


Fig. 2. Particle size distribution of the four different fractions (a) −40 μm, (b) −63 + 40 μm, (c) −100 + 63 μm and (d) −100 μm used as feedstock powders.

3.2. Coatings characterization

The aim of this study is the production of high quality thick YBCO coatings. This means that the coatings must be dense, without any microcracks and with the appropriate phase composition. For this purpose a parametric study of the plasma spraying process was undertaken.

In Fig. 4 SEM studies of the top surfaces of coatings produced using feedstock powders with different particle size distributions are shown. These coatings were produced keeping all the other plasma spraying parameters constant (26 kW plasma power, 7 cm spraying distance and 80 psi carrier gas). It is worth mentioning that on the top surfaces no microcracks are visible irrespective of the particle size of the powder used. In all four cases, large melted areas are observed around which spherical droplets appear. It is clear, that as the mean particle size of the starting powder decreases, the melted areas become larger and wider, while the number of spherical droplets reduces. In this way the number and the size of pores within the coating is reduced. This indicates that on hitting the substrate the smaller particles create better quality lamellae and therefore the use of powders of smaller mean particle size leads to the development of denser coatings. On the other hand, it is observed that the use of the feedstock powder with the

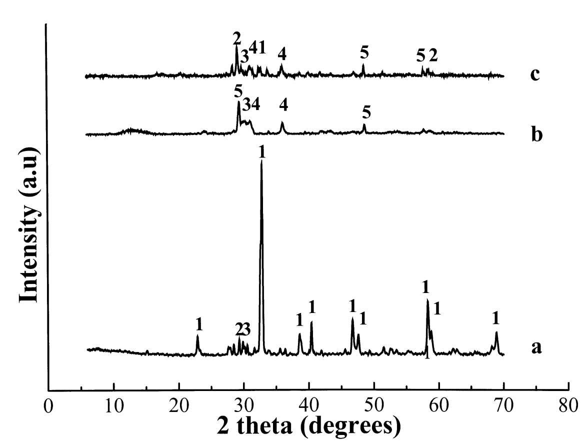


Fig. 3. X-ray diffraction pattern (a) of the powder used as feedstock material and (b), (c) of coatings developed using plasma power 26 kW, spraying distance 7 cm and carrier gas pressure 85 and 30 psi respectively. (1: < 123 >, 2: $BaCuO_2$, 3: < 211 >, 4: $CuYO_2$, 5: Y_2O_3).

broad particle size distribution (−100 μm) leads to coatings with better top surface morphology compared to the other powder fractions, where a significant reduction in the number of pores and also in the number and size of the small round particles that exist around the melted areas is observed. It seems that the large number of small particles existing in this powder as well as the broad range of particle sizes contribute to this positive result since both these factors permit a more compact packing of the melted droplets after hitting the substrate.

These observations are confirmed by studying the coating cross sections using the SEM. In Fig. 5 the polished cross sections of the developed coatings are shown. Indeed, the use of powders with smaller particle size leads to denser coatings. In the case of the fraction of −100+63 μm the developed coating has a low quality as no lamellae are formed and it has a significant amount of porosity. In the case of the −63+40 μm fraction, formation of lamellae is observed and decrease in the number of pores but at the same time a lot of microcracks appear within the coating. The use of the −40 μm fraction led to coatings of better quality, since the number of microcracks and pores are reduced but yet the phases appearing within the coating are less homogeneously dispersed. The best results concerning the coatings microstructure were obtained by using the fraction −100 μm. In this case the coatings become homogeneous with very thin lamellae, while elimination of microcracks and very few pores both in number and size are observed. The coating thickness in all cases ranged between 100 and 200 μm.

Since the −100 μm powder fraction, for the production of which there is no need of an extensive sieving procedure, leads to almost equivalent or better results than the fine powder fraction (of −40 μm), it was decided to continue the study employing this powder. The plasma spraying parameters investigated were the carrier gas pressure and the spraying distance. There was a very strong influence of the processing parameters on the deposition rate as well as on the microstructure and morphology of the coatings. Studying the cross-sections

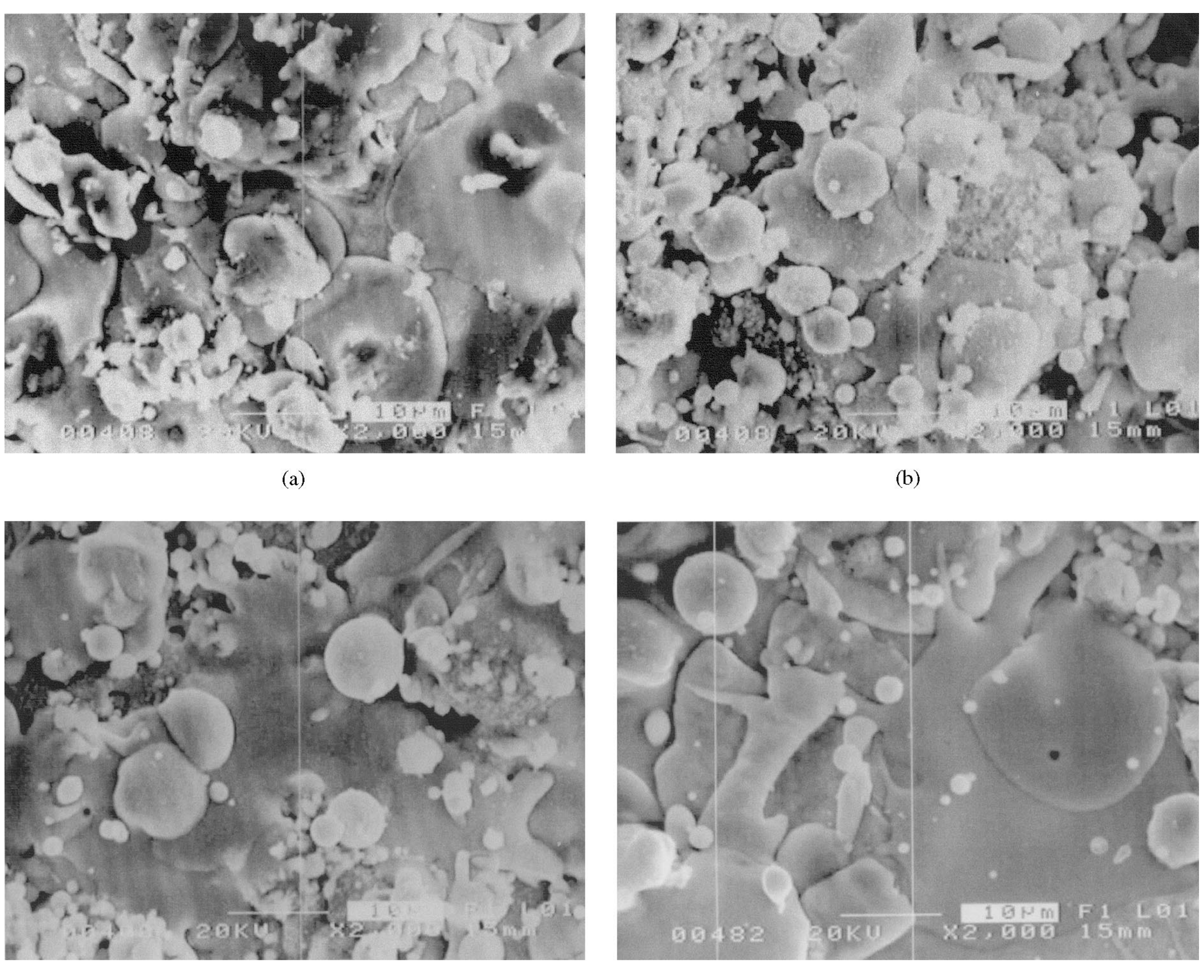


Fig. 4. SEM studies of top surfaces of coatings prepared with different feedstock powders (a) −100+63 μm, (b) −63+40 μm, (c) −40 μm and (d) −100 μm keeping all the other plasma parameters constant (26 kW plasma power, 7 cm spraying distance and 30 psi carrier gas pressure).

of the fractured surfaces of the coatings using SEM, it was observed that as the carrier gas pressure decreases (85–20 psi), the number of unmelted particles is reduced, leading to the formation of denser coatings with less defects and improved adhesion (Fig. 6). The spraying distance has a similar effect and for each powder fraction there is an optimum distance leading to best results in terms of morphology and adhesion. As the spraying distance approaches the optimum, the unmelted particles reduce in number and the size of microcracks is reduced. Fig. 7 shows the polished cross sections of coatings developed under three different spraying distances where it can be very clearly observed that both the number and the size of microcracks depend to a great degree on the spraying distance. At these optimized conditions concerning both the carrier gas pressure and the spraying distance, a maximum deposition rate is also obtained (Fig. 8). These results were achieved using both Cu and Stainless Steel 304 substrates.

Scratch test measurements showed quite good adhesion for coatings deposited on Cu and stainless steel substrates. The critical load (load at which total loss of adhesion to the substrate occurs) in the as sprayed state was found to be 163 N for Cu substrate and 185 N for stainless steel substrate. As it can be seen in Fig. 9a, the depth of the diamond stylus within the coating was very low at an applied load of 87 N (less than 20 μm) compared to the total thickness of about 150 μm.

Investigation of the YBCO coatings by X-ray diffraction analysis showed decomposition of the <123> phase during spraying (Fig. 3b and c). The as sprayed coatings contain a considerable amount of amorphous phase, $BaCuO_2$, Y_2O_3, <202>, <211> (green phase) and in a few cases small amounts of undecomposed <123> phase. Small differences concerning phase composition within the coating have been observed depending on the initial plasma spraying parameters employed. For the restoration of the crystallinity and the <123> phase, the samples were calcined in air

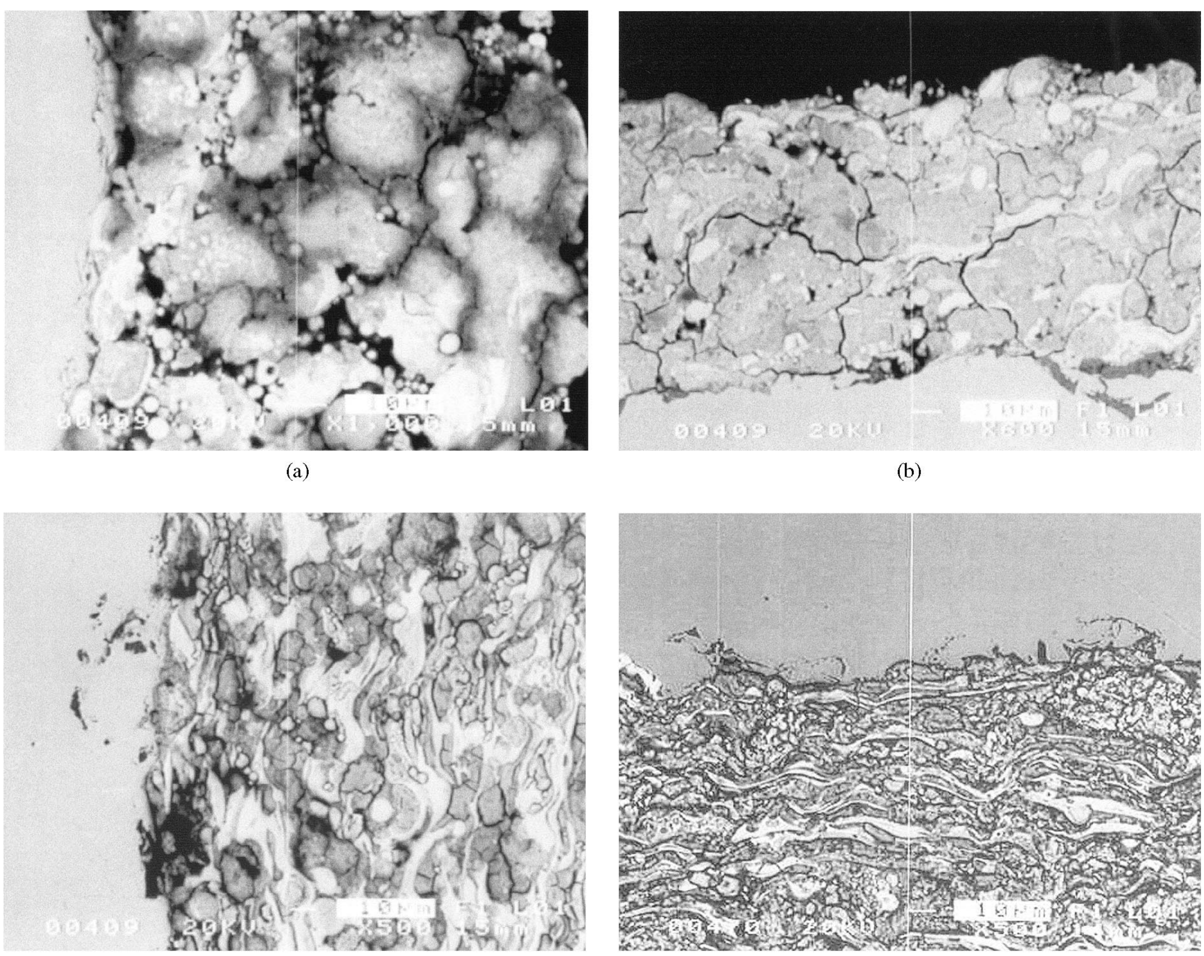
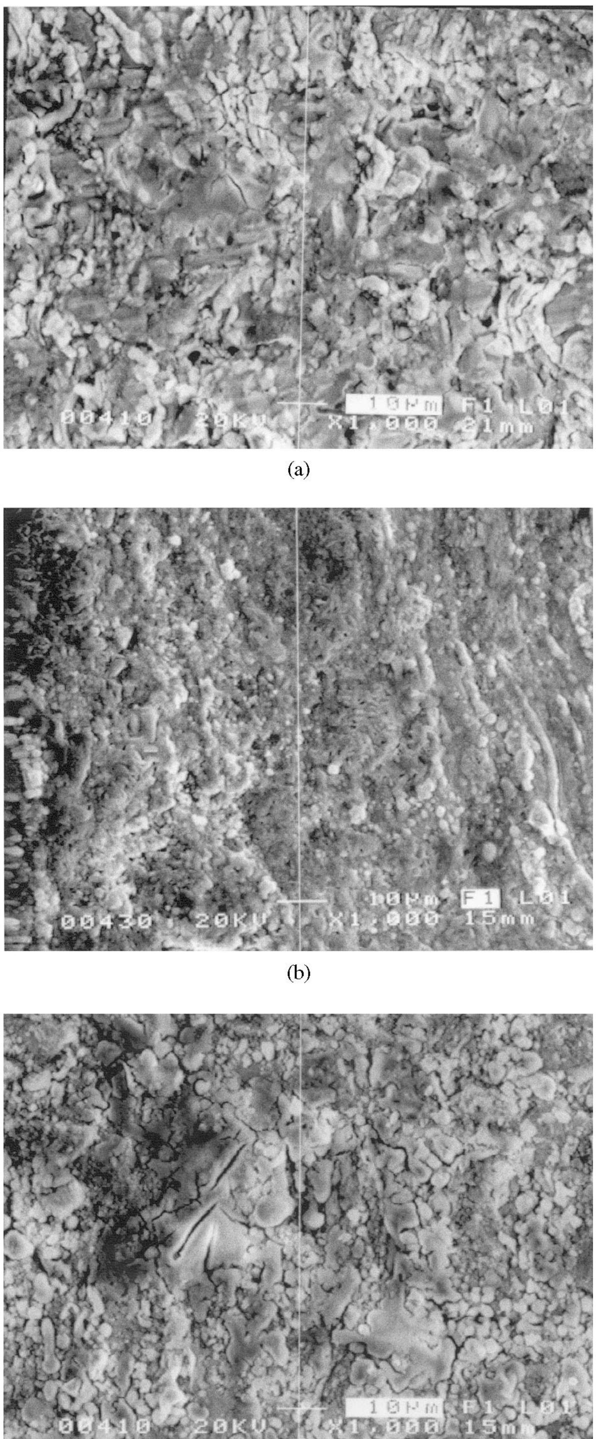


Fig. 5. SEM studies of the coatings polished cross sections developed using different feedstock powders (a) −100+63 μm, (b) −63+40 μm, (c) −40 μm and (d) −100 μm and keeping all the other plasma parameters constant (26 kW plasma power, 7 cm spraying distance and 30 psi carrier gas pressure).

under different conditions (750–930°C, 0–20 h). Total restoration of the <123> YBCO phase is possible under the appropriate conditions. For less severe conditions (lower temperature or less dwell time at the final temperature) than the optimum ones, $BaCO_3$ as well as some small quantities of $BaCuO_2$ and in some cases CuO are observed indicating incomplete restoration. For temperatures or calcination times higher than the

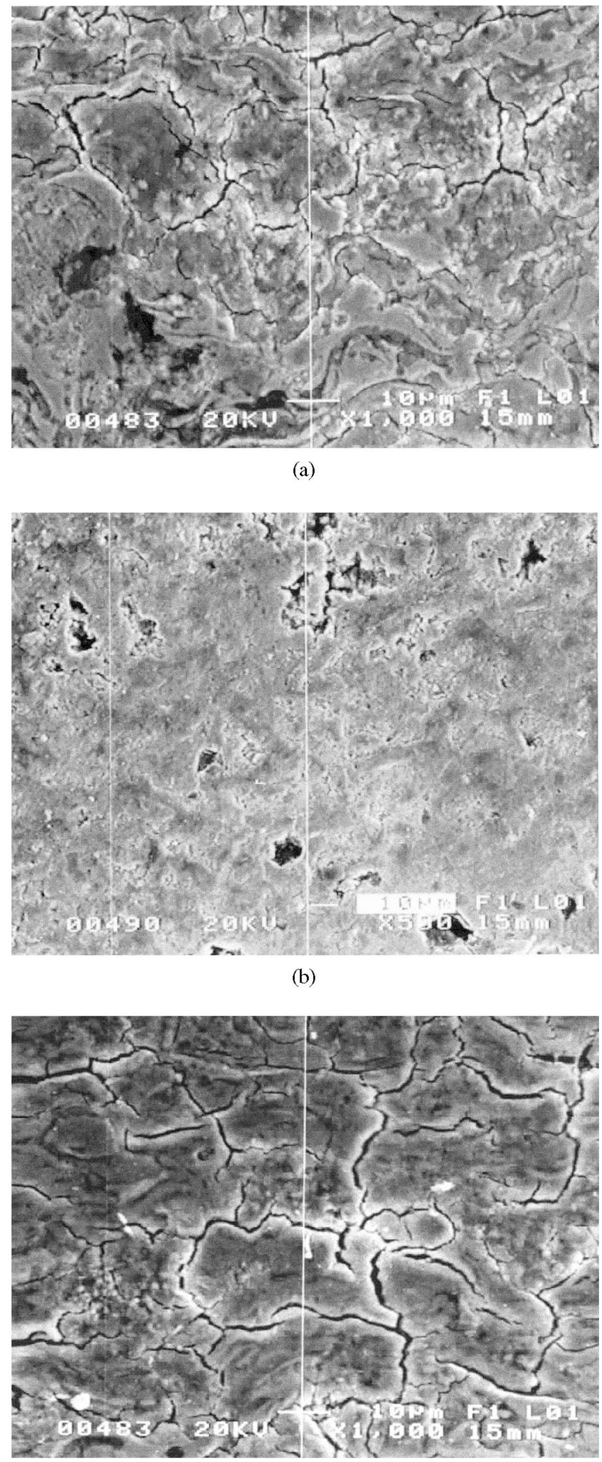


(a)

(b)

(c)

Fig. 6. SEM studies of the fractured surfaces cross sections of the coatings developed from the −100 μm powder fraction using 26 kW plasma power, 7 cm spraying distance and (a) 60 psi, (b) 30 psi, (c) 20 psi carrier gas pressure.



(a)

(b)

(c)

Fig. 7. SEM studies of the coatings polished cross sections developed from the −100 μm powder fraction using 26 kW plasma power, 30 psi carrier gas pressure and (a) 6 cm, (b) 7 cm, (c) 10 cm spraying distance.

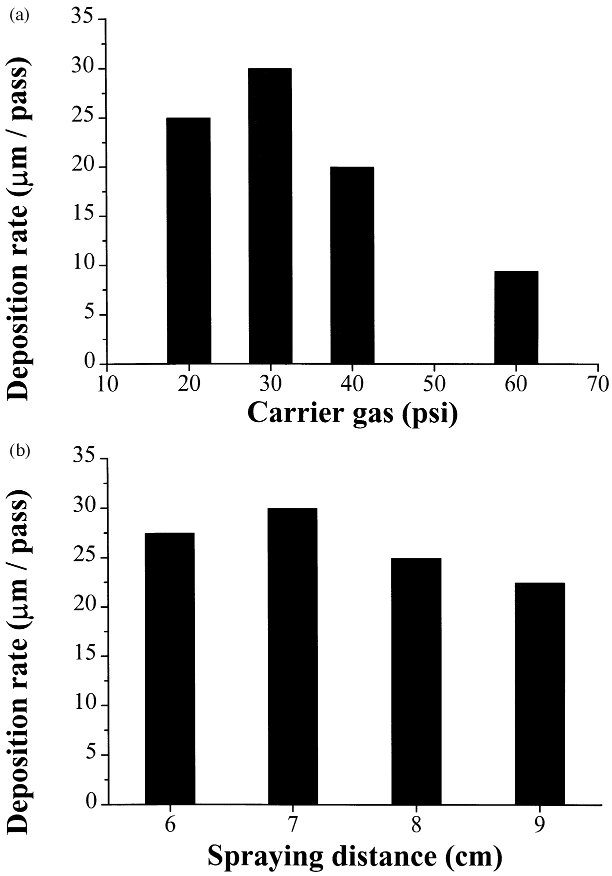


Fig. 8. Deposition rate of coatings developed from the −100 μm powder fraction using 26 kW plasma power versus (a) the carrier gas pressure and (b) the spraying distance.

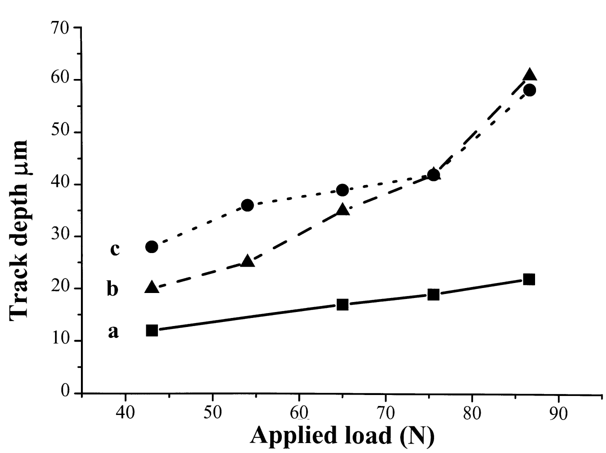


Fig. 9. Depth of the scratching test track versus increasing applied load for coatings (a) in the as-sprayed state, (b) thermally treated at 920°C for zero firing time and (c) thermally treated at 920°C for 10 h. Processing conditions: powder fraction −100 μm, 26 kW plasma power, 7 cm spraying distance and 30 psi carrier gas pressure.

optimum ones, phases indicating decomposition of the <123> YBCO phase such as $BaCuO_2$, <211> and CuO are detected. This phenomenon is confirmed in Fig. 10, where the XRD patterns of a coating developed from the −100 μm powder fraction and annealed under different conditions are presented. The <123> YBCO phase in the as sprayed coating has been totally decomposed (Fig. 3c). After annealing at 750°C for 2 h the <123> phase has already started to reform, while after annealing at 900°C for 2 h the <123> restoration is almost complete. Total restoration is obtained at 920°C for 10 h. Calcination at a higher temperature than the optimum one (930°C for 10 h) led to a clear decrease of the main peak of the <123> phase at 33 degrees and to the <123> decomposition, as peaks of <211> phase and a glassy phase can be detected. The optimum heat treatment conditions differ slightly among the coatings and depend in some way on the coating thickness and initial processing conditions. Investigations of the coating microstructures by SEM proved that during calci-

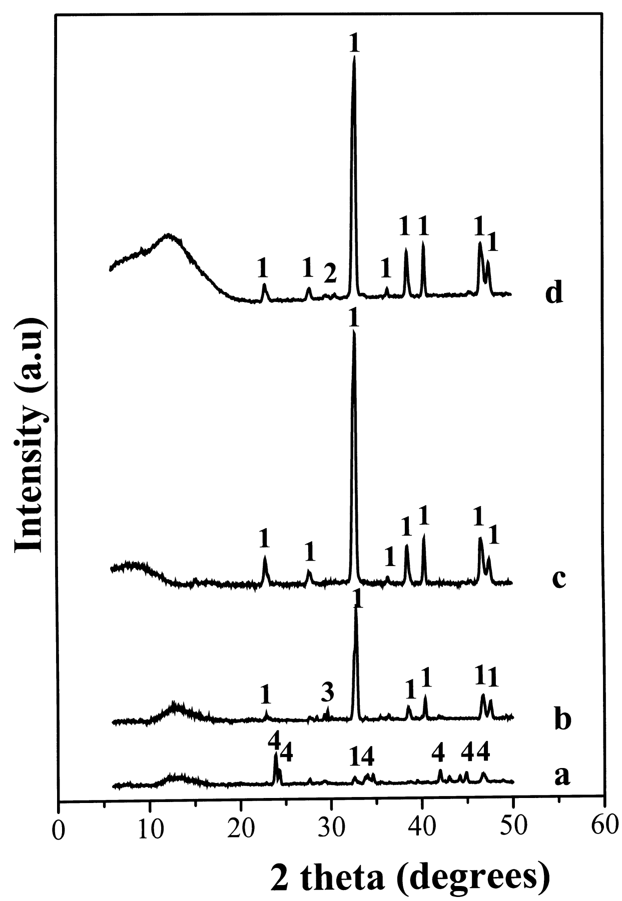
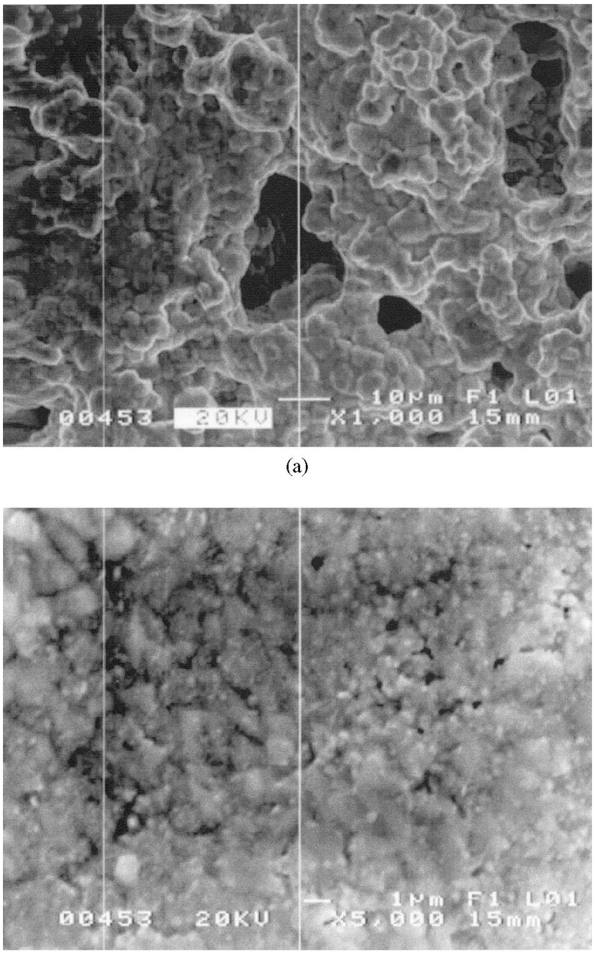


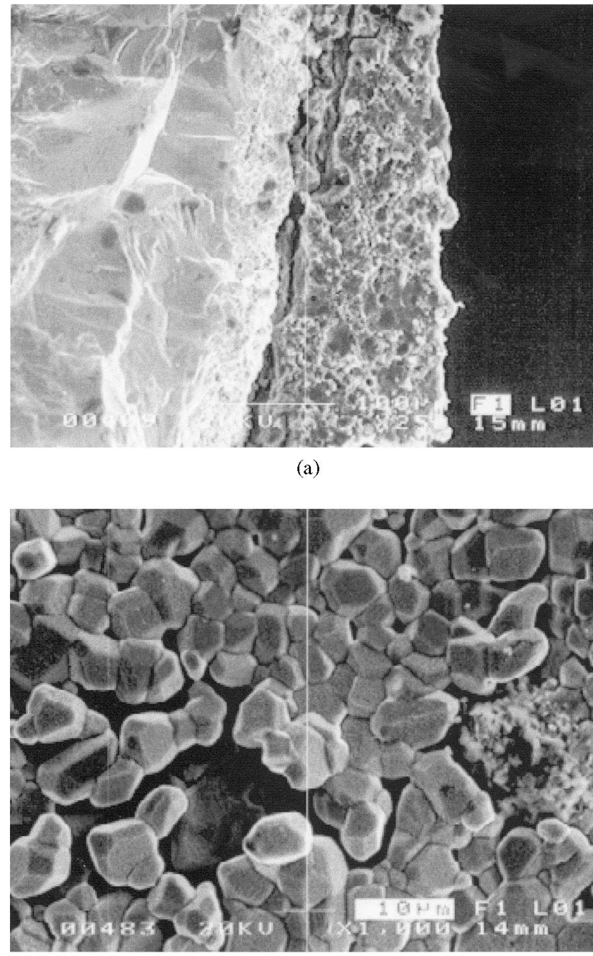
Fig. 10. X-ray diffraction patterns of a coating developed from the −100 μm powder fraction with 26 kW plasma power and 7 cm spraying distance heat treated at (a) 750°C for 2 h, (b) 900°C for 2 h, (c) 920°C for 10 h and (d) 930°C for 10 h. (1: <123>, 2: <211>, 3: $BaCuO_2$, 4: $BaCO_3$).



(a)

(b)

Fig. 11. SEM studies of the coatings top surfaces after heat treatment at 920°C for 10 h. (a) Magnfication ×1000; (b) magnification ×5000.



(a)

(b)

Fig. 12. SEM studies of the CuO formation during post-thermal treatment. (a) Magnfication ×250; (b) magnification ×1000.

nation for the formation of the < 123 > phase, sintering occurs altering the coating microstructures. In Fig. 11 the microstructure of a coating annealed at 920°C for 20 h and consisting of pure < 123 > YBCO phase is shown. It is observed that sintering has occurred and the pores have been enlarged.

However, copper was found to be unsuitable as a substrate since it oxidizes during annealing. Due to CuO formation in the Cu-coating interface, the adhesion of the coatings to the substrate is reduced (in certain cases total loss of adherence is observed) (Fig. 12). On the other hand, coatings deposited on stainless steel substrates proved quite good. Their properties in terms of adhesion are inferior compared to the coatings adhesion in the as sprayed state but they remain in acceptable levels. The critical load after the heat treatment procedure was measured at 111 N instead of 185 N in the as sprayed state (stainless steel substrate). In addition, a slightly higher depth in scratching track was measured (Fig. 9b and c). However, the total restoration of the superconducting phase in it compromises this effect.

4. Conclusions

This investigation has shown that the atmospheric plasma spraying technique is an effective technique for the production of high quality thick YBCO coatings. The particle size distribution of the feedstock powder significantly affects the quality of the deposited coatings. The plasma spraying parameters (such as carrier gas pressure, spraying distance) exhibit an optimum value within a narrow range where both coating quality and deposition rate reach a maximum. At these optimized conditions good adhesion of the coating to the substrate (both Cu and stainless steel) was achieved.

Although the < 123 > YBCO powder decomposes during deposition to $BaCuO_2$, < 211 > and Y_2O_3, total restoration of the $YBa_2Cu_3O_{7-x}$ is possible under the appropriate post thermal treatment conditions. Copper substrates proved unsuitable for the production of pure $YBa_2Cu_3O_{7-x}$ superconducting coatings due to the formation of CuO in the copper-coating interface during annealing and the consequent loss of adhesion.

However, post thermal treatment under the appropriate conditions was successful for coatings on stainless steel substrates as both the <123> phase was restored and the adhesion of the coating to the substrate was kept to satisfactory levels.

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