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Superconductive and structural properties of plasma sprayed YBaCuO and BiSrCaCuO layers

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Abstract

In this paper we discuss the influence of melt texturing on the structural and superconductive properties of plasma sprayed $Bi_2Sr_2CaCu_2O_x$ and $YBa_2Cu_3O_x$ layers on stainless steel and nickel substrates with a Yttria stabilized Zirconia (YsZ) buffer layer. The YBaCuO interacts with the YsZ during melt texturing whereas the amount of texture is relatively small. Application of the quench melt growth technique gives a better texture but there still is a strong interaction with the YsZ. For both heat treatments crack formation occurs in the YBaCuO layers, leading to relatively small critical current densities. The BiSrCaCuO layers are treated at lower temperatures and have a smaller interaction with the YsZ. Also crack formation during the heat treatment is not observed. For BiSrCaCuO layers the best superconductive properties (at 10 K) are found for texturing in a gradient of 15 K/cm with maximum temperatures ranging from 910-940 °C and velocities through the gradient ranging from 50-500 mm/h.

1. Introduction

Plasma spraying is a well known technique for the deposition of thick layers of ceramics and metals. Because of the large deposition rate large areas can be coated. Soon after the discovery of high T_c materials the plasma spray process was used for the production of thick layers of these materials [1-7].

The technical implementation of the plasma spray process and the subsequent thermal treatment for the production of high T_c layers is relatively simple. However, the deposition process, the phase relations of the superconductors and the interactions with the substrate materials make the whole process very complicated. The main effects of the deposition process are the decomposition of the superconductor into the elementary oxides, change of the overall composition of the deposit and the formation of micro cracks in the coating (as a result of the rapid cooldown) of the deposit on the substrate [8]. After deposition the coating has to undergo a heat treatment to regrow the superconducting phase. Here the relative stability of the different phases of the superconductor and the interaction of the superconductor with the substrate material is important because the heat treatment takes place at high temperatures. At the beginning of the heat treatment only the decomposed superconductor is present in the coating. Therefore the interaction between the basic oxides and the substrate also should be considered.

In previous papers we presented some studies on the interaction of plasma sprayed YBaCuO with a YsZ buffer layer [9,10] and the effect of heat treatment on the structure and superconducting properties of YBaCuO on nickel rich steels with and without YsZ buffer layers [11].

In the present paper we discuss the influence of melt texturing and quench melt growth process [12] on the structural and superconductive properties of plasma sprayed BiSrCaCuO and YBaCuO layers on YsZ buffer layers.

2. Experimental

The YBaCuO and BiSrCaCuO layers are deposited by means of vacuum plasma spraying. In this technique the deposition takes place in a vacuum chamber where a suitable background atmosphere and pressure can be chosen. For our experiments argon was used at a pressure of 200 mbar.

Powder of the coating material is fed into a plasma jet where it melts. The plasma jet transports the molten grains to the substrate where they are deposited. Both YBaCuO and BiSrCaCuO decompose in the molten state. At the high temperatures reached in the plasma jet the melt consists of the elementary oxides only. Also the high temperature causes some of the material to evaporate. For YBaCuO a deficiency of Cu and Ba can occur when the spray parameters are not suitably adjusted. For BiSrCaCuO it was not possible to prevent the loss of large amounts of Bi. Therefore we used BiSrCaCuO powder with a nominal composition of 3212. The nominal com-

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position in the deposited layer is found to be close to that of $Bi_2Sr_2CaCu_2O_x$.

As substrates we used stainless steel and nickel. For a good adherence of the coatings the substrates are first sandblasted and then coated with a bond layer of the same material as the substrate. In order to prevent an interaction between the superconductor and substrate the bond layer is covered by a YsZ buffer layer. Finally the buffer layer is coated with the superconductor material. All layers are deposited by plasma spraying.

The powders used were obtained from Hoechst AG. The YBaCuO powder had an average diameter of 30 μm. For an improvement of the flow characteristics the powder was split in two fractions: one smaller and one larger than 10 μm. The larger fraction was used for the spraying. The BiSrCaCuO powder had an average diameter of 25 μm and could be used without further processing.

All melt texturing experiments followed the same procedure. First the sample was heated in a gradient furnace to a maximum temperature. Then it was moved through a gradient to a temperature well below the peritectic melting temperature of the material. Finally the sample was slowly cooled to room temperature with a stop of a few hours at ~500 °C for reoxygenation of YBaCuO. Some YBaCuO samples received a Quench Melt Growth treatment [12]. In this process the sample is first heated to ~1100 °C, kept there for 15 min. and then cooled at a rate of 600 K/h to ~1020 °C. From this point on the previously described melt texturing procedure was followed.

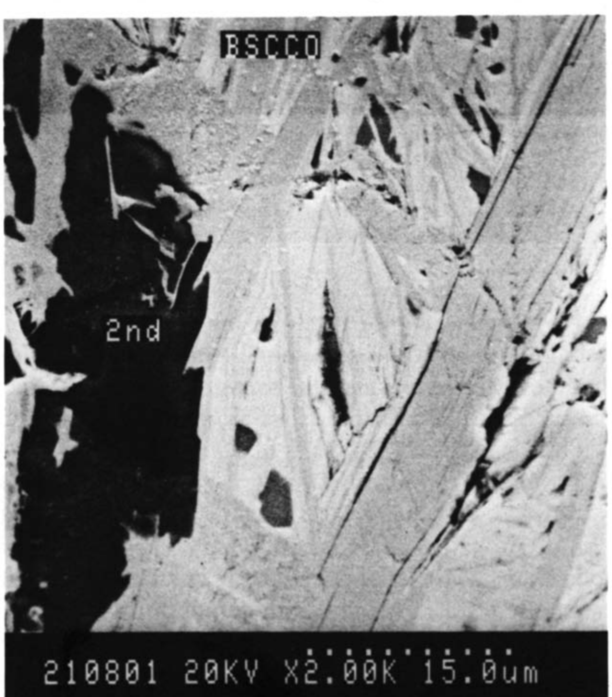

Figure 1 SEM cross section of a BiSrCaCuO layer on a YsZ diffusion barrier textured in a gradient of 15 K/cm with a maximum temperature of 950 °C and a pulling speed of 90 mm/h.

The micro structure was studied using a Hitachi 800-S scanning electron microscope with EDX facilities. Resistance and critical current measurements were performed using the standard four point technique. A 1 μV criterion was used for determination of the critical current. With a typical distance of 0.8 cm between the voltage contacts this gives an electric field criterion of 1.25 μV/cm. The electrical contacts for both YBaCuO and BiSrCaCuO were prepared by polishing the surface and then depositing 1 μm thick silver pads by thermal evaporation. Wires were connected to the silver pads using indium solder.

The contact resistances varied from ~0.01 mΩ to ~100 mΩ. Since the largest contact resistance occurred in the samples with the lowest critical currents we had no problems with heating of the samples due to dissipation in the contacts.

3. Results and discussion

3.1 BiSrCaCuO

BiSrCaCuO layers were melt textured by pulling them at a velocity ranging from 10-3000 mm/h through a temperature gradient of 15 K/cm with maximum temperatures ranging from 890 to 950 °C. In figure 1 the microstructure of a layer textured with a maximum temperature of 950 °C and a pulling speed of 90 mm/h is shown. The bright phase has the 2212 composition. Large (grey) grains of a secondary phase with reduced Bi content are present and a large (black) CuO grain is visible. For lower temperatures the secondary phase grains are smaller and less numerous. In figure 2 a pseudo phase diagram is shown for the occurrence of superconductivity in melt textured BiSrCaCuO layers as a function of the maximum temperature and pulling speed. As a measure for the quality of the superconducting layers the critical current density, J_c, at 10 K is used. J_c values have been divided in three ranges, each represented by its own symbol: (+) $J_c = 0$ A/mm², (x) $J_c < 0.5$ A/mm² and (□)$J_c > 0.5$ A/mm². The largest J_c's are found for temperatures ranging from 910-940 °C and pulling speeds ranging from 50-500 mm/h. This region is indicated in figure 2 by the rectangular box.

Texture is only observed in the top part of the BiSrCaCuO layer [13]. In the bulk some secondary

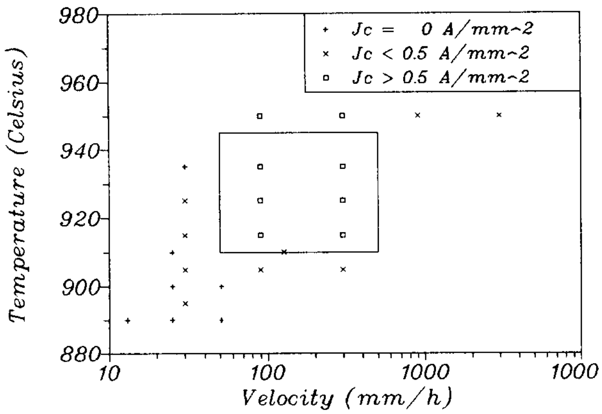

Figure 2 Pseudo phase diagram for the occurrence of superconductivity in plasma sprayed melt textured BiSrCaCuO as a function of maximum temperature and pulling speed. For details see text.

phase is always present and no evidence of texture is found. In order to make the bulk of the layer single phase and increase the texture in the bulk the heat treatment has to be improved.

3.1 YBaCuO

The influence of different melt texturing treatents was studied. Samples were heated up to a maximum temperature of 1030 or 1070 °C and then cooled down to ~980 °C by moving them through a gradient of 12 K/cm at a velocity of 30 mm/h. Some samples were just cooled down from 890 °C at a rate of 60 K/h without a temperature gradient. Samples treated with either of the above parameters showed some grain alignment and a typical grain size of 20 μm. All samples showed a strong interaction of the superconductor with the YsZ buffer layer and a considerable crack formation.

Some samples were subjected to a QMG treatment [12]. In all treatments the sample was first heated to 1100 °C, kept there for 15 min. and then cooled to 1020 °C. After this the melt texturing procedure was followed in a temperature gradient of 12 K/cm and varying velocities: 30 mm/h (QMG-2), 10 mm/h (QMG-3), 3 mm/h (QMG-4) and 1 mm/h (QMG-5). At the lowest velocity the superconductor reacted completely with the YsZ. For the two highest velocities the microstructures were very similar. A typical example is shown in figure 3. The grain alignment in these samples is much better than in the melt textured samples. As can be seen in figure 3 the cracks form under an angle of ~50° with the substrate. Figure 4 shows the structure of a sample after treatment QMG-4. The cracks are now parallel to

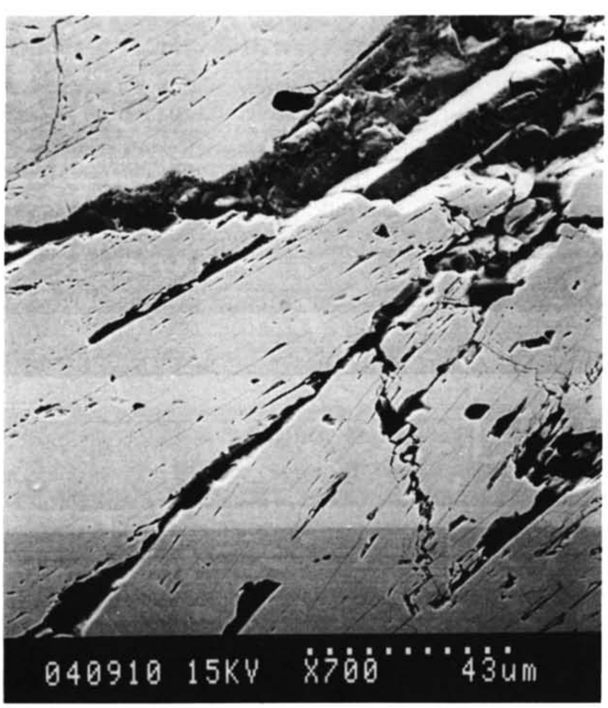

Figure 3 SEM cross section of YBaCuO layer after quench melt growth treatment QMG-3. The substrate runs parallel to the right side of the photo.

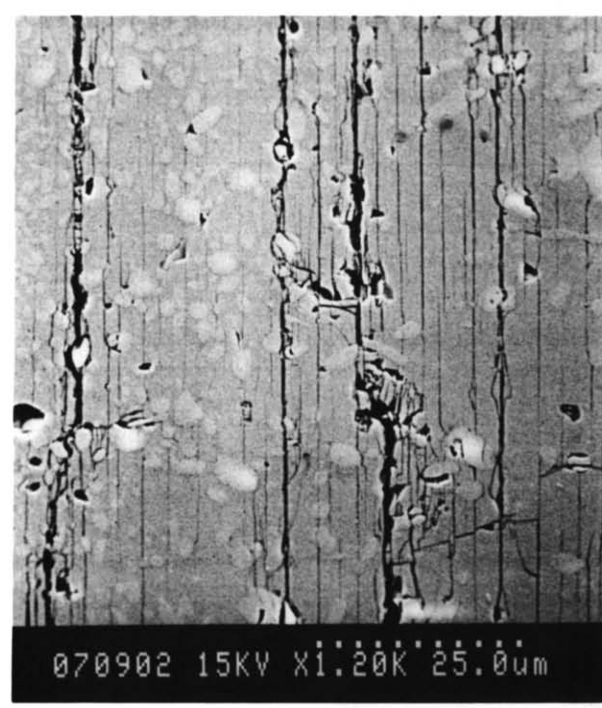

Figure 4 SEM cross section of YbaCuO layer after quench melt growth treatment QMG-4. The substrate runs parallel to the right side of the photo.

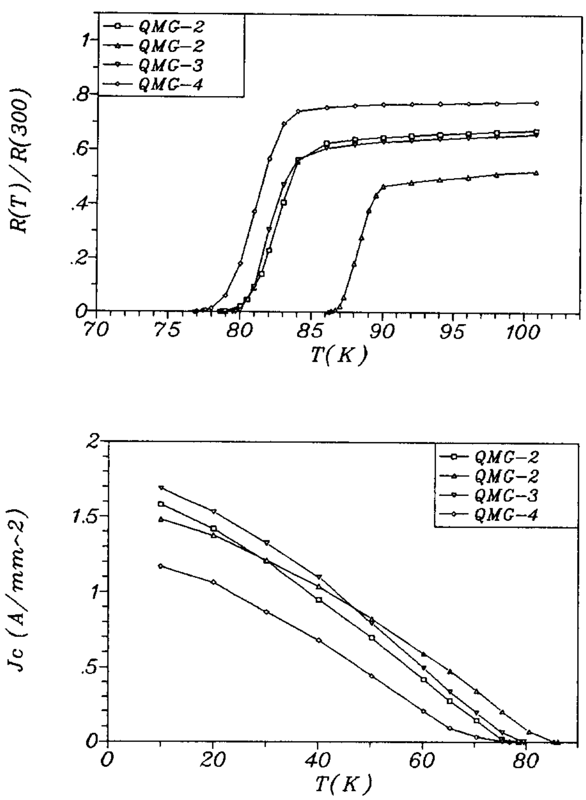

Figure 5 (a) Scaled resistance and (b) critical current density of YbaCuO layers after quench melt growth treatment.

the substrate. The superconductive transitions and temperature dependence of J_c for different QMG treatments are shown in figure 5.

Despite the considerable difference in microstructure and grain alignment the observed superconductive transitions and critical current densities of the melt textured and QMG samples are very similar. It seems that the extensive crack formation present in all the YBaCuO samples determines the superconductive properties. This is probably due to the large thermal mismatch between YBaCuO and YsZ combined with the presence of microcracks in the sprayed layers.

4. Conclusions

Crack formation due to thermal mismatch between YBaCuO and YsZ seems to determine the superconductive properties. Together with the strong interaction during melt texturing this makes YsZ unsuitable as diffusion barrier for plasma sprayed YBaCuO layers.

The heat treatment of plasma sprayed BiSrCaCuO layers has to be improved in order to make the layer single phase and increase the texture of the bulk. The interaction of BiSrCaCuO with YsZ during melt texturing is very small and no serious crack formation occurs during the heat treatment.

Acknowledgements

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