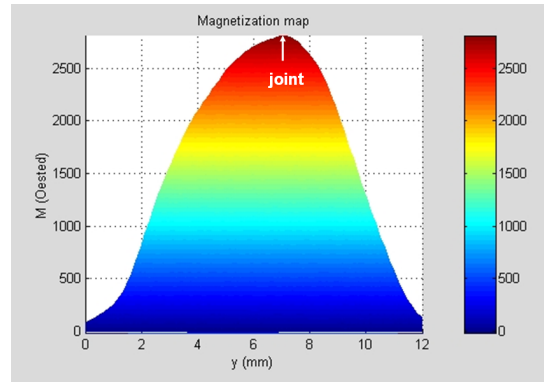


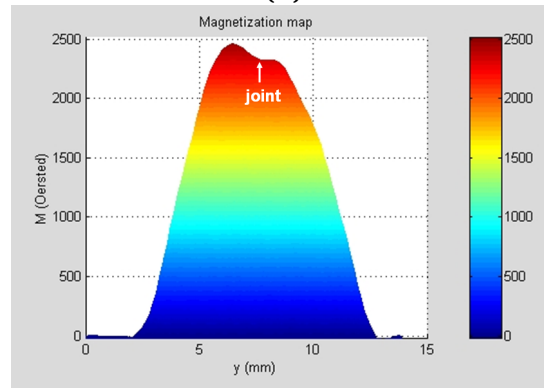
7.4.3 Homogeneity in superconducting properties of the YBCO/Ag/YBCO joints

Now, for a better understanding of Ag diffusion phenomenon into YBCO matrix, we will determine the remanent magnetization profiles and critical current densities of different sample faces which will be called hereafter: top, center and bottom. It is important to notice that, since the resolution of the measurements depends on the distance between the Hall probe and the layer in which the critical current flows, not much information about the remanent magnetization distribution at the center and bottom sides is obtained by scanning the top face of the samples. Thus, in order to do this experiment we have cut the samples in three layers along the c-axis as it was schematically shown in figure 6.43. The "top face" corresponds to the ab plane where the seed has been placed in order to obtain the YBCO monolith by TSMG process. The "center" and "bottom" faces correspond to ab planes situated at 0.3cm and 0.6cm below the "top face", respectively.

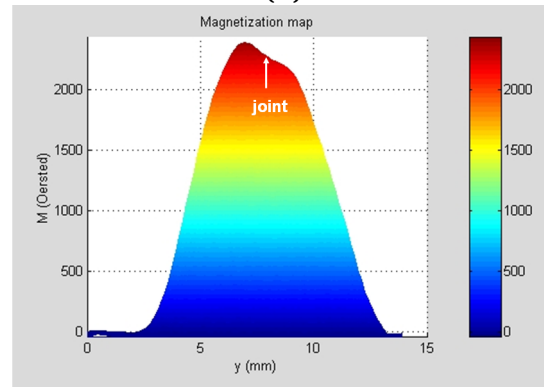
Two samples ΔT_{28} and C_3 obtained by the welding process have been chosen for the homogeneity study. The nomenclature used for the faces studied in this Section are $top_{\Delta T_{28}}$ and top_{C_3} corresponding to the top side of the samples ΔT_{28} and C_3 , respectively and $center_{\Delta T_{28}}$, $center_{C_3}$ and $bottom_{\Delta T_{28}}$, $bottom_{C_3}$ corresponding to the center and bottom sides of samples ΔT_{28} and C_3 , respectively. Sample ΔT_{28} is a joint obtained after the optimization of the welding process by employing the following parameters: $g_{Ag}=10\mu\text{m}$ thick, $T_{max}=992^\circ\text{C}$, $t_1=3\text{h}$, $T_2=973^\circ\text{C}$, $t_2=1\text{h}$, $r=0.6^\circ\text{C/h}$, $\Delta T=28^\circ\text{C}$, $T_{ox}=450^\circ\text{C}$ and $t_{ox}=168^\circ\text{C}$. On the contrary, sample C_3 is a joint obtained by using a non-optimized welding process, thus it is of a reduced critical current density. The later kind of joint is not of a technological interest but it will be interesting to determine the critical current density along its depth in order to observe the phenomenon of Ag diffusion and how does it affect the superconducting properties of the samples.



(a)



(a)



(b)

Figure 7.33: 2D remanent magnetization profiles obtained after a fc process for different faces along c-axis of the sample ΔT_{28} : a) top face, b) center face and c) bottom face. The center face is situated 0.3cm below the top face and the bottom face is situated 0.6cm below the top face. The dimension of c-axis of the top face is different from the other faces. The joint is indicated in the figure by arrows

"Top face" of the samples studied has a thickness of 0.6cm, whereas the center and bottom faces have a thickness of 0.3cm. As a consequence, the remanent magnetization at top faces cannot be compared with the remanent magnetization measured at center and bottom faces. We will use remanent magnetization measurements in order to have a general view of the quality of the final joint. For comparison purposes we will calculate the critical current density of the YBCO samples and of the final joints by using the software "Caragol".

The top face of sample ΔT_{28} was analyzed in order to optimize the parameter ΔT . It is shown that the remanent magnetization profile (figure 7.33a) exhibits only one peak, as expected for a single domain. As microstructural studies have shown (see figure 6.41), no additional phases have been formed at the interface after the welding process concluding that the profile indeed agrees with the results obtained when the microstructure was investigated. A small asymmetry is seen in this profile with respect to the joint, indicating a small difference between the critical current densities present in the sample in the left-hand side and right-hand side. Remanent magnetization profiles corresponding to center and bottom faces of sample ΔT_{28} are shown in figures 7.33(b-c), respectively, where the joint is indicated by arrows. A small reduction of remanent magnetization of the junction and of the YBCO grain from the right-side of the graph is observed in these profiles indicating that their critical current density are slightly reduced when compared with J_c of the left side YBCO grain. The inhomogeneity found in the remanent magnetization values is reflected in the critical current density values as can be seen in figure 7.34 where the blue and red symbols are associated with critical current density values corresponding to YBCO grains while the yellow symbols are associated to the critical current densities obtained for the junction.

Note that when, the top face is investigated, the critical current densities of the Grain 1 and Grain 2 are quite different. This difference make us conclude that the previous YBCO samples used for the joining process are in-

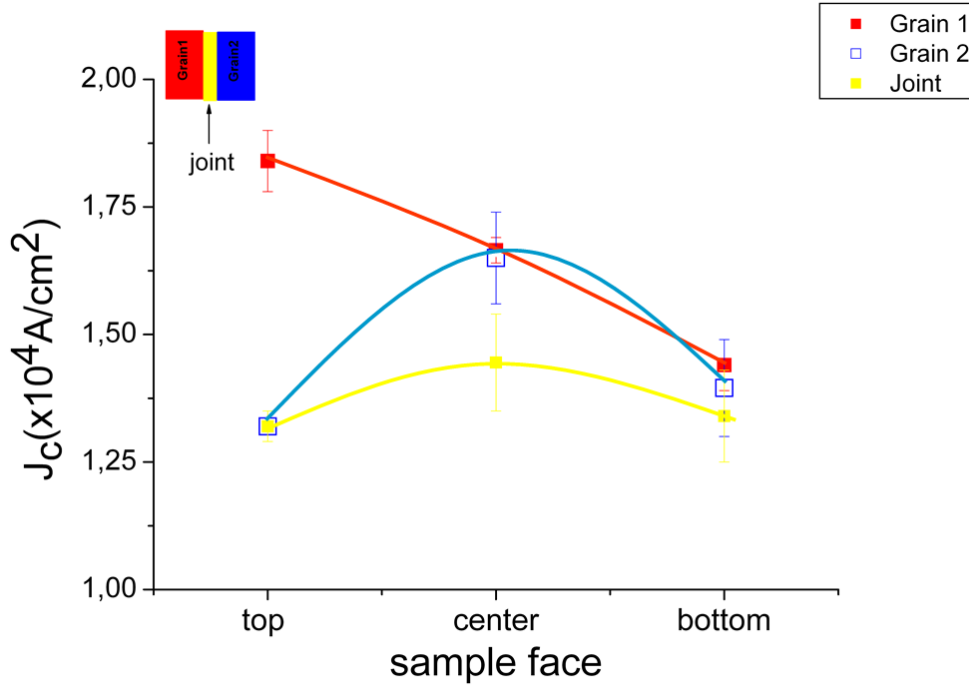


Figure 7.34: Critical current density values obtained along c-axis of the junction at different depths from the face, where the seed was located during the TS process. The sample analyzed is ΔT_{28} . Red and blue symbols indicate the critical current density dependence along the c-axis of the sample of the Grain 1 and Grain 2, respectively. Yellow symbols indicate this dependence for the joints. Error bars show the J_c values dispersion obtained by using the methodology detailed in Section 7.1.

homogeneous. The critical current density of the Grain 1 when the "top face" was analyzed is $J_c^{grain1} = 1.84 \times 10^4 A/cm^2$ and of Grain 2 is $J_c^{grain2} = 1.35 \times 10^4 A/cm^2$. On the contrary, when the center and bottom faces are investigated, the critical current densities of Grain 1 and Grain 2 are $J_c^{grain1} = 1.67 \times 10^4 A/cm^2$, $J_c^{grain2} = 1.65 \times 10^4 A/cm^2$ and $J_c^{grain1} = 1.44 \times 10^4 A/cm^2$, $J_c^{grain2} = 1.4 \times 10^4 A/cm^2$, respectively and can be considered quite homogeneous.

Concerning the critical current density calculated at the junction we have noticed that the values determined all along its junction are similar. For the top face the critical current density is $J_c^{gb} = 1.35 \times 10^4 A/cm^2$, for the center face is $J_c^{gb} = 1.44 \times 10^4 A/cm^2$ and for the bottom face is $J_c^{gb} = 1.34 \times 10^4 A/cm^2$. Thus, we can

conclude that when the welding process is optimized, a quite homogeneous joint is obtained. For comparison purposes, we will determine the ratio J_c^{gb} / J_c^{grain} for each face studied. This ratio indicates the percentage of the reduction of the critical current density of joint (J_c^{gb}) after the welding process when compared with the lowest critical current density value obtain for the YBCO grains J_c^{grain} (see figure 7.34). The error bars in the figure quantify the dispersion on J_c^{gb} / J_c^{grain} values calculated by using the methodology described in Section 7.1 for each case and exist due mainly to the inhomogeneities found in the proper YBCO grains. For the top face note that J_c^{gb} exhibits similar value that the lowest critical current density exhibited by the YBCO grain, thus the ratio J_c^{gb} / J_c^{grain} is ~ 1 as it is indicated in the figure. On the contrary, note that for the center face, the ratio J_c^{gb} / J_c^{grain} is 0.87 which means that J_c^{gb} is reduced $\sim 12\%$ from J_c^{grain} . Even the ratio is reduced, J_c^{gb} is kept constant. This reduction is given by the J_c^{grain2} increasing and not by the J_c^{gb} reduction. When the bottom face critical current density values are investigated, the ratio J_c^{gb} / J_c^{grain} is ~ 0.96 which indicates that J_c^{gb} is reduced only 4% from J_c^{grain} values corresponding to the weakest grain. As it is shown in figure 7.34, the reduction of the J_c^{gb} / J_c^{grain} if it is compared with the ratio obtained for the top face and the enhancement of ratio value if it is compared with the ratio value obtained for the center face is mainly due the inhomogeneities in J_c values obtained for the YBCO grains.

Thus, we conclude that the J_c^{gb} is quite homogeneous along the c-axis and that the welding process used to determine this weld was the optimum one and Ag diffusion into YBCO matrix was quite homogeneous.

When the sample C_3 is investigated, differences in the remanent magnetization distribution between the top, center and bottom sides (see figures 7.36(a-c)) can be seen. All profiles exhibit two peaks indicating that the critical current which flows across the junction is disrupted by the inhomogeneities found at the interface when the microstructure has been investigated. In figure 6.47 (a-c) can be seen that some non-superconducting phases have been trapped at the

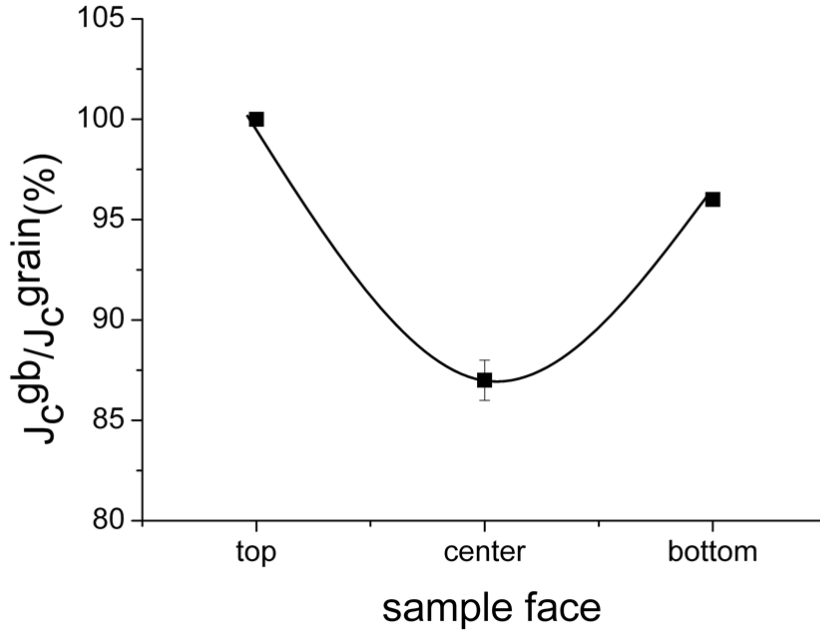


Figure 7.35: Dependence of ratio J_c^{gb}/J_c^{grain} with sample face analyzed (red and blue symbols for grains, whereas yellow symbols for the joint).

interface of the top face during the welding process. Because of the high cooling rate used to grow this sample, the Ag-rich liquid could not be completely eliminated from the interface and has solidified along with Y211 and $BaCuO_2 - CuO$ phases at the interface. On the contrary, the remanent magnetization profile corresponding to the center sample, $center_{C_3}$, shows two less differentiated peaks indicating a better connectivity between grains than the other faces (center and bottom). As in the previous case when the sample ΔT_{28} was investigated, the top face of the sample is thicker than center and bottom faces, thus we cannot use the comparison as a method to conclude which face has higher performances. Because of this, we further continue our investigation by calculating the critical current density of the YBCO sample and final joint.

Critical current density values have been determined for sample C_3 by using

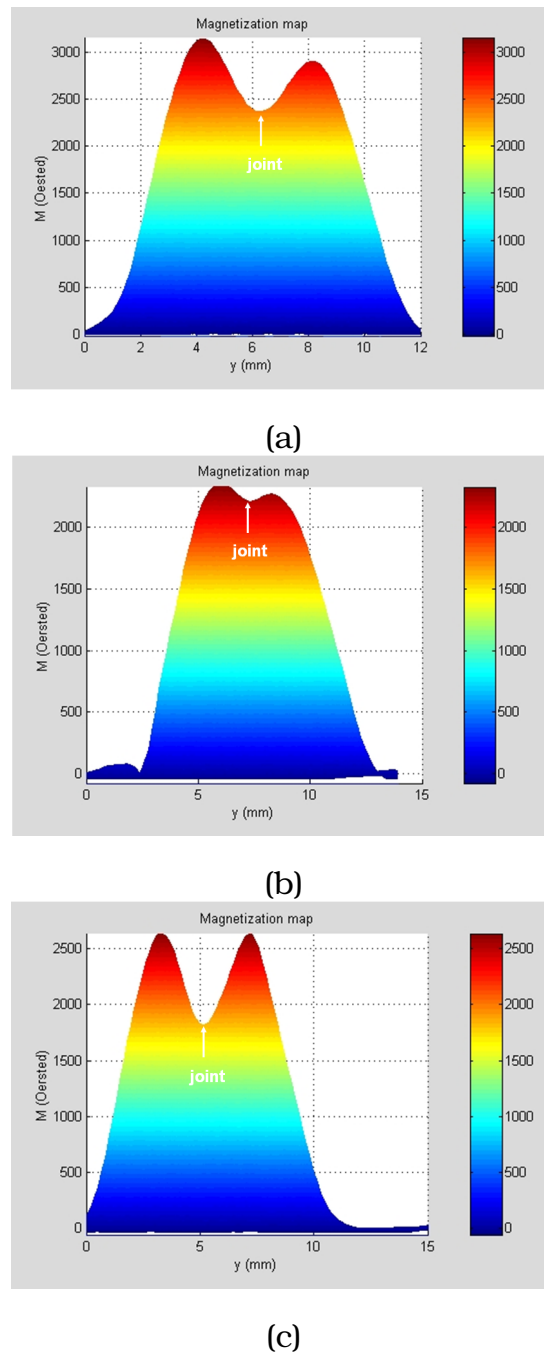


Figure 7.36: Remanent magnetization maps obtained after applying a field cooled process for different faces obtained along the c-axis of the sample C_3 . a) top face, b) center face and c) bottom face. The center face is located at 0.3cm below the top face, whereas the bottom face is located at 0.6cm below the top face. The joints are indicated by arrows in the figure.

the methodology described in Section 7.1 and by using "Caragol" software. Figure 7.37(a-c) shows the critical current density values determined for each side of sample C_3 (top, center and bottom) where red and blue symbols indicate the critical current density of the YBCO grains (Grain 1 and Grain 2) and the yellow symbols correspond to the J_c^{gb} values. Error bars in the figure quantify the inhomogeneity in J_c values found by employing this methodology. In the figure can be observed that the error bars indicating the dispersion of J_c values obtained for the YBCO grains are overlapping in all the cases. This can be interpreted as follows: this inhomogeneity in J_c values is mainly due to the inhomogeneity in the proper YBCO grains. Thus, both YBCO grains could be considered of similar superconducting properties. The critical current density of the joint J_c^{gb} is spanning between $1.01 \times 10^4 A/cm^2$ and $1.21 \times 10^4 A/cm^2$. For the "top face", the critical current density of the junction is $J_c^{gb}=1.1 \times 10^4 A/cm^2$, whereas for the "center face" is $J_c^{gb}=1.2 \times 10^4 A/cm^2$ and for the "bottom face" $J_c^{gb}=0.8 \times 10^4 A/cm^2$. Higher reduction takes place at the bottom face.

The ratio J_c^{gb}/J_c^{grain} shows the percentage of the reduction of the critical current density of joint (J_c^{gb}) after the welding process when compare with the weakest YBCO grain. The dependence of this ratio with the analyzed face is depicted in figure 7.38. It can be observed that highest reduction of the critical current density of the joint took place for the bottom face of the J_c^{gb}/J_c^{grain} is ~ 0.53 which means that J_c^{gb} is reduced $\sim 47\%$ from the J_c^{grain} value corresponding to the lowest value of critical current density corresponding to YBCO grains. For the "top face", this ratio is ~ 0.69 , which means that J_c^{gb} value is reduced $\sim 31\%$ from J_c^{grain2} value of sample C_3 where the ratio, whereas for the "center face", the figure shows that the J_c^{gb} was reduced $\sim 23\%$ from the J_c^{grain2} value. As it was observed in figure 7.37, the critical current density values corresponding to the top and center faces of the junction are quite similar. On the contrary, the critical current density values corresponding to YBCO grain to the same faces showed a small inhomogeneity. Thus, we conclude that the difference in ratio

values determined for the top and center faces of this sample is governed by the inhomogeneity in J_c values corresponding to proper YBCO grain and not to the differences in superconducting properties of both YBCO grains used for joining.

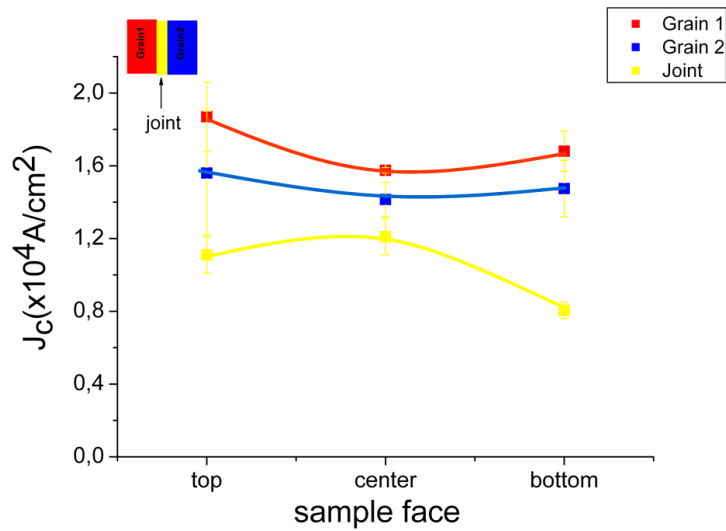


Figure 7.37: Critical current density values obtained for each YBCO grain (Grain 1 indicated by red symbols and Grain 2 indicated by blue symbols) and for the final joint (indicated by yellow symbols) along the c-axis of the joint. Top, center and bottom faces corresponding to the sample C_3 are investigated.

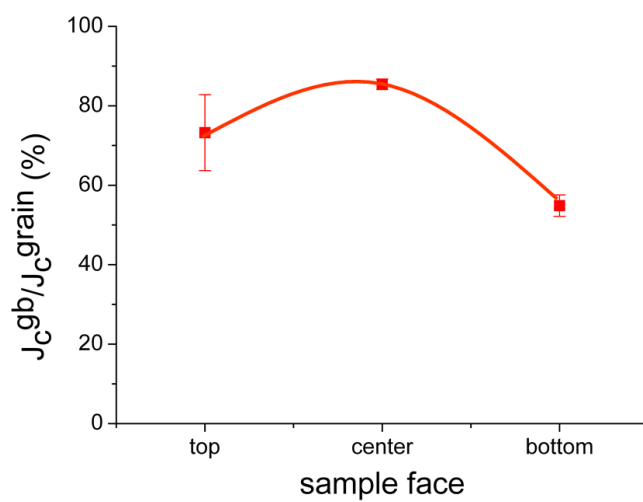


Figure 7.38: Ratio J_c^{gb}/J_c^{grain} along the c-axis of the sample analyzed. Top, center and bottom faces are investigated.

7.4.4 Conclusion of Chapter 7

The remanent magnetization distribution was investigated on samples joined by the welding process described in Chapter 5 by using a Hall probe imaging system. These measurements allow us to deduce the magnitude of the critical current densities by solving the inverse problem, i.e Biot-Savart law, as well as the homogeneity and spatial scale on which they flow.

We have proposed a methodology allowing the determination of the critical current density of the final joints from remanent magnetization profiles obtained by employing a Hall probe imaging system after a FC process. Following the Bean model which predicts that J_c is constant all over the sample, we have used only the y component of J_c (J_{cy}) to further determine the critical current density of the YBCO grains and of the junction at the same time. We have observed that the current distributions patterns obtained by using "Caragol" software [30, 96] agree well with the current distribution profile predicted by the Bean model.

In this Chapter we have evaluated the limitations of the Hall probe imaging system, employed to determine the remanent magnetization profiles, and of the calculation of critical current densities by using the software "Caragol". In this way we have used a totally non-superconducting joint, obtained by simply gluing two YBCO parts. We have demonstrated that when the reduction in remanent magnetization of the joint with respect with YBCO grains, is higher than 60%-70%, the junction will be considered of a very low quality. Moreover, any sample exhibiting J_c values across the joint that have been reduced more than 60% after the welding process and an angle α higher than 33° , will be considered to have a low critical current density and we will not be able to quantify more precisely the J_c across the joint.

After the analysis performed on studied samples, we have observed that parameters such as: oxygenation time, cooling rate and window temperature are very important to optimize the quality of the superconducting joint. After the systematic analysis of the welding process, the samples obtained using the opti-

mized parameters had pyramidal shaped remanent magnetization profiles, i.e they exhibit only one peak, indicating that the connectivity between the domains was good (example: sample ΔT_{28} and $C_{0.6}$). These samples exhibit only one current loop. When the welding process was not optimized some segregation of non-superconducting phases at the interface was observed with microstructural analysis. This segregation consists of a mixture of Ag-rich liquid which was not pushed completely from the interface, Y211 and $BaCuO_2 - CuO$ phases which were trapped at the interface for different reasons which were explained in Chapter 6. Therefore, the critical current density flow was strongly limited at the junction and the remanent magnetization locally decreased (example: sample C_6). Consequently, some peaks and valleys appeared in the remanent magnetization profiles, which correspond to regions where the critical current flow was or was not limited, respectively. We have observed too that the difference in superconducting volume exhibited by the both YBCO grains used for joining is reflected in the remanent magnetization profiles obtained by using the Hall probe imaging system after a field-cooled process.

We have determined that: $t_{ox}=168-240h$, $r \leq 1.8^\circ C/h$ and $\Delta T=28^\circ C$ are the optimum parameters needed for the obtention of a high quality superconducting joint and the critical current density of the junction determined by using the methodology described in Section 7.1 was $J_c^{gb}=1.35 \times 10^4 A/cm^2$, the same as the mother blocks. Moreover, it has been shown that the critical current density of the junction along the c-axis of the sample is quite homogeneous. On the contrary, J_c^{grain} of the YBCO material is quite inhomogeneous along c-axis. This inhomogeneity in J_c values is provided from the inhomogeneity in the microstructure of the starting samples. It has been shown that the inhomogeneity in the YBCO material is increasing while moving away from the top face of the sample, being the face where the seed has been placed during the TS method.

Chapter 8

Conclusions

The main goal of this PhD thesis has been to develop a technology which would be able to achieve superconducting pieces of large dimensions and complex geometries, with the purpose of integrating them into different superconducting devices by fabricating artificial superconducting joints.

The first step in achieving high temperature superconducting YBCO joints was to find a suitable welding material. We have investigated two Ag based materials as welding agents: Ag_2O powder and Ag thin foil. Microstructural analysis along with transport and in-field Hall mapping measurements have been performed to find the conditions to reach a high quality superconducting joints. Considering the quality limitations found with the use of Ag_2O powder as a welding agent, giving rise to the formation of a large amount of porosity at the interface, and the promising results achieved for Ag thin foils, we scheduled a systematic study of the YBCO joints parameters by using Ag thin foils as welding elements obtained by a cold rolling process.

A deep study of the influence of different parameters on the microstructure and on the superconducting properties, i.e remanent magnetization and critical current density of the final joints, generated using a YBCO/Ag/YBCO architecture has been performed in order to understand the role of the Ag diffusion and to optimize the welding process. It has been shown that these parameters influence in one way or another the microstructure and superconducting properties

of the final joints. In order to optimize these parameters, we have performed two kinds of experiments: quench experiments and slow cooling experiments. By quench experiments we have succeeded in controlling the Ag diffusion process into the YBCO matrix. Parameters such as: melting time (t_{melt}), Ag foil thickness (g_{Ag}) and weld configuration have been investigated and optimized. On the other hand, the influence of parameters such as: cooling rate (r), processing temperature (T_{max}) and window temperature (ΔT), on the microstructure of the final joints has been analyzed by means of slow cooling experiments.

It has been shown that the microstructure of the starting YBCO monoliths is very important since a strong Ag liquid migration from the interface occurs through the pre-existing macrocracks perpendicular to the interface. Hence, special care was taken to avoid the existence of such defects in the microstructure of the starting YBCO monoliths. We have used previously non-oxygenated samples which are mainly free, or have a reduced concentration of micro and macrocracks.

We have succeeded in controlling the Ag diffusion process into the YBCO matrix by employing a melting time of 3 hours, a Ag foil thickness of $10\mu\text{m}$ and a welding configuration (100)/(100). By using these conditions, the Ag diffusion is kept homogeneous and reduced in dimensions into the YBCO matrix.

By slow cooling experiments we have found that a $T_{max}=992^{\circ}\text{C}$, a cooling rate of $r=0.6^{\circ}\text{C/h}$ and a window temperature of $\Delta T=28^{\circ}$ are the optimum parameters for our samples, and thus they, should be used in order to obtain a clean welded interface free of impurities and pores and high critical current density joints. WDS analysis at the interface and far away from it have been performed. When the joint is obtained after applying an optimized welding process, no Ag precipitates have been detected neither at the interface nor into YBCO matrix. On the contrary, when the joint is obtained by using a non-optimized welding process, Ag-rich and Cu-rich precipitates have been detected at the interface.

Therefore, a 2D Ag diffusion model has been proposed after these analysis

which establishes that Ag rich liquid is pushed away from the inner part of the sample towards its edges along the YBCO growth interface. The crystallization is first completed in the inner part where the Ag-rich liquid was completely expelled. It turns out, then, that we should use a cooling rate slow enough to expel all the Ag from the interface. Accumulation of non-superconducting phases, such as: Ag precipitates, Y211 and $BaCuO_2$ phases, at the edge of the junction occurs when the cooling rate (r) is too high and the time for recrystallization is too low.

The homogeneity in the microstructure of the final joints along the c-axis of the sample was analyzed. It was demonstrated that the microstructure at the interface was quite homogeneous along its depth.

The influence of welding process parameters on the superconducting properties of the final joints has been studied by determining the remanent magnetization profiles of each sample and calculating from them the critical current density of the joint and YBCO grains for comparison. Moreover, the ratio J_c^{gb}/J_c^{grain} , indicating the reduction of critical current density of the joint with respect to the critical current density of the YBCO grains, has been calculated.

The remanent magnetization distribution was investigated on samples joined by the welding process by using a Hall probe imaging system. These measurements allowed us to deduce the magnitude of the critical current densities by solving the inverse problem by using the "Caragol" software, as well as the homogeneity and spatial scale on which they flow.

We have proposed a methodology in order to determine the critical current density of the final joints from the results of "Caragol". We have observed that the current distributions patterns obtained by using "Caragol" software agree well with current distribution profile predicted by Bean model.

We have determined the limitation of the Hall probe imaging system allowing the determination of the remanent magnetization profiles and of the calculation of the critical current densities by using the software "Caragol" by employing a

glued sample. We have demonstrated that when the reduction in remanent magnetization of the joint with respect to the YBCO grains is higher than 60%-70%, the junction will be considered to be of a very low quality. Moreover, any sample exhibiting J_c values across the joint that have been reduced more than 60% after the welding process, will be considered to have a too low critical current density and we will not be able to quantify more precisely the J_c across the joint.

We have observed the influence of the following parameters: oxygenation time, cooling rate and window temperature on the remanent magnetization and critical current density of the final joints. We have determined that: $t_{ox}=168-240\text{h}$, $r \leq 1.8^\circ\text{C/h}$ and $\Delta T=28^\circ\text{C}$ are the optimum parameters needed for the obtention of high quality superconducting joint and the critical current density of the junction determined by using the methodology here proposed was $J_c^{gb}=1.35 \times 10^4 \text{A/cm}^2$, same as the mother blocks. These results agree with the results obtained when the microstructure of the final joints was investigated. Moreover, it has been shown that the critical current density of the junction along the c-axis of the sample is quite homogeneous. On the contrary, J_c^{grain} of the YBCO material is quite inhomogeneous along the c-axis. This inhomogeneity in J_c values is provided from the inhomogeneity in the microstructure of the starting material. It has been shown that the inhomogeneity in the YBCO starting material is increasing while moving down from the top face of the sample, being the top face where the seed was placed during the top seeding process.

In summary, by employing the new welding methodology developed in the present work, we have been able to obtain YBCO superconducting joints having a clean and crystallographic coincident microstructure and with critical current densities through the joint similar to those of the YBCO monoliths.

List of Figures

1.1	Schematic drawing of the localization of the Ag based welding material and orientation of the YBCO single crystalline ceramic tiles. (a) Two pieces of YBCO having the same crystallographic orientation, 1 and 2, are joined by the Ag based welding materials, 3. The joined interface is parallel to the c-axis. (b) Three pieces of YBCO having the same crystallographic orientation, 1, 2 and 3 are joined by Ag based welding material, indicated by 4, in such a way that the joined interface is perpendicular to the c-axis. (c) Ring-shaped configuration where the individual YBCO elements (1) can have different crystallographic orientations. In one case the c-axis points towards the central part of the ring and in the second configuration all the c-axis of the elements are parallel and directed perpendicular to the ring. The silver based materials are also indicated (2).	4
1.2	Working plan followed in this PhD thesis.	11
2.1	a) Temperature dependence of electrical resistivity of the oxide superconductor $YBa_2Cu_3O_{7-\delta}$; b) Expulsion of a weak, external magnetic field from the interior of the superconducting material.	14
2.2	Magnetic phase diagram (H(T)) for: a) Type I superconductors: one critical field H_c exists; b) Type II superconductors: where two critical fields exist (lower critical field (H_{c1}) and upper critical field (H_{c2})).	15
2.3	Representation of the mixed state, where each vortex is surrounded by shielding currents.	16
2.4	The Lorentz force F_L on flux lines in the presence of the current.	17

2.5	Magnetization process for an infinite cylinder based on the Bean critical state model. Schematic representation of magnetic field and current distribution inside the sample. a)-c) increasing the magnetic field; d)-f) decreasing the magnetic field.	21
2.6	Magnetization process for a slab after a fc process. a) initial stage when an magnetic field of $H > H^*$ is applied before the cooling of the sample below its T_c ; b) decreasing the applied magnetic field; c) reducing the applied field down to $H=0$.	22
2.7	Schematic representation of the remanent magnetization profile after a fc process when the applied field is lower than H^* , the full penetration field. In this case the remanent magnetization is lower than the saturated magnetization.	22
2.8	Position of irreversibility line $H_{irr}(T)$ in the H-T phase diagram. $H_{irr}(T)$ is below the $H_{c2}(T)$ and divides the states where the critical current density J_c of the superconductor is characterized by $J_c = 0$ and $J_c > 0$.	23
2.9	The irreversibility line in various cuprates. The dashed line represents the respective H_{c2} .	24
2.10	Sketches of the unit cell of the a) tetragonal and b) orthorhombic phases of YBCO [23]. Representative lattice parameters for the orthorhombic phase are $a=3.827\text{\AA}$, $b=3.882\text{\AA}$ and $c=11.682\text{\AA}$.	26
2.11	a) Phase diagram of $YBa_2Cu_3O_{7-\delta}$ system as a function of oxygen content. T_N represents the Niel temperature of the antiferromagnetic phase and T_c the critical temperature of the superconducting phase. b) Variation of T_c with oxygen content.	27
3.1	Scheme of Scanning Electronic Microscope	31
3.2	Scanning Hall Probe imaging set-up	37
3.3	Comparison between the $B_{measured}$ (■)- red line and $B_{calculated}$ (●)-black line for a joint obtained by the welding process. The junction is indicated in the figure by an arrow.	40
3.4	In-field Hall microprobe remanent magnetization profile measurements of an YBCO sample with the applied magnetic field parallel to the [001] axis: Remanent magnetization profile after a field cooling process: a) original YBCO sample; b) glued sample.	41

4.1	Pseudo-binary phase diagram for YBCO material	45
4.2	Change in the free energy ΔG and its components during the formation of a nucleus [42]	48
4.3	The configuration used to obtain YBCO monoliths: a Nd123 seed is situated on the top of the YBCO pellet. The compacted powder is located on a support consisting of Yb_2O_3 and CeO_2	55
4.4	Temperature profile to which the YBCO pellets have been subjected: the texturation has been done in air and the superconducting behavior of the sample has been reestablished by oxygenation.	56
5.1	a) Peritectic temperature of the melt-textured YBCO-Ag composite as a function of Ag_2O addition; b) Microcrack spacing (■) and microcrack density (□) as a function of A_2O content in the starting mixture.	60
5.2	Steps to follow for the preparation of YBCO/Ag/YBCO joints: the single domain to be joint is extracted from an YBCO pellet obtained by top seeding melt textured growth method. Then, is cut perpendicular to the ab plane so that the Ag based welding agent is inserted between two ac planes. The Ag based welding agent is obtained by a standard rolling process if a Ag foil is used or by suspension in acetone if a Ag_2O powder is used and the thickness used in this study is spanning between 10 and 100 μm . Finally the assembly is submitted to thermal process and a clear and incident superconducting joint is obtained.	63
5.3	Schematic illustration of the experimental setup. The temperature of the furnace was monitored by two termocouples situated close to the sample. For the high temperature process, the assembly was surrounded by a silicon carbide tube in order to achieve an isothermal region around the sample. The junction is situated in the horizontal direction.	64

- 5.4 General schema of the thermal cycle followed to fabricate a superconducting joint. The temperatures T_{max} , T_2 and T_3 can be varied within a certain range depending on the microstructure of the initial YBCO ceramics. T_{ox} and t_{ox} , where T_{ox} is the maximum temperature of the oxygenation process and t_{ox} is the time spent by the sample at T_{ox} , must be optimize in order to reestablish the superconducting behavior of the final joints. 64
- 5.5 SEM micrograph corresponding to the ac plane of a joint obtained by using a $50\mu\text{m}$ thick Ag_2O suspension. Arrows indicate where the joint is. 67
- 5.6 SEM micrograph of an ac plane of a joint when the welding agent is a $\approx 50\ \mu\text{m}$ thick pressed Ag_2O powder: a) an abundant porosity is observed; b) a better connectivity between grains is achieved but some porosity still can be seen. The interface is indicated by arrows. 68
- 5.7 SEM micrograph showing a general view of the microstructure of the ac plane of the joint obtained by using a $50\mu\text{m}$ Ag foil. The joint is indicated by the arrows. . . 70
- 5.8 Observation at higher magnification of the ab plane of the joint area by using BSE. A perfect continuity is reached without porosity and non-superconducting phases. The joint is indicated by arrows. 71
- 5.9 SEM image of a $\{100\}$ plane of an YBCO/Ag/YBCO joint where a perfect matching of the two YBCO monoliths is observed without any sign of segregated metallic Ag at the interface. The arrows in the figure indicate the welding interface. 72
- 5.10 In-field Hall microprobe remanent magnetization profile after a field-cooled process of a joint YBCO sample with the interface and applied magnetic field parallel to the $[001]$ axis. The remanent magnetization at the center of the joint is of $M_{rem} \simeq 800$ Gauss. The joint is indicated by arrow. 73
- 5.11 Geometry arrangement for the magnetoresistance measurements. Dark grey areas represent the contacts. The junction is indicated in the figure by dash line. The magnetic field is applied parallel to the c-axis and to the joint. The current is injected parallel to the ab plane and perpendicular to the joint. 74

5.12 a) Temperature dependence of the magnetoresistance measured in fields of 0, 0.1, 1.3, 6 and 9T, applied parallel to the c-axis, of a single domain and to the joint. b) Irreversibility lines measured for the grain and for the final joint at the same time: black line for the bulk and magenta line for the weld.	76
6.1 Quench experiment: T_{max} is a temperature between the peritectic temperature of YBCO and the melting point of YBCO/Ag composite: t_{melt} is the time spent at T_{melt} by the YBCO/Ag interface.	79
6.2 a) Schematic illustration of the configurations investigated in this study. a) (100)/(100) weld; b) (001)/(001) weld. The YBCO tiles are represented by dark grey color and the Ag foils by light grey.	81
6.3 SEM detail of the melted zone in a quenched sample where the Y211 and Ag particles can be identified together with the quenched liquid BaCuO ₂ -CuO.	82
6.4 EDX analysis at the YBCO/Ag/YBCO interface in the molten area. a)analysis corresponding to the amorphous regions (dark grey) in figure 6.3 showing the presence of liquid phase at the interface; b)analysis corresponding to light grey particles formed at the interface showing the presence of Ag at the molten region.	82
6.5 Silver diffusion length dependence with the melting time. Red symbols: (001)/(001) joint; blue symbols: (100)/(100) joint. Error bars in figure quantifies the inhomogeneity found in the Ag diffusion length.	83
6.6 Optical micrograph of an ac plane corresponding to a sample quenched for two hours at $T_{max}=1000^{\circ}\text{C}$: a)(100)/(100) weld; b)(001)/(001) weld at the maximum temperature. The junction is indicated by arrows and the molten region is delimited by white lines.	84
6.7 Polarized optical micrograph of a YBCO/Ag/YBCO sample after quenching it from 1000°C and held for 3 hours. The welded plane was (100)/(100) and the observation plane is parallel to the c-axis. The molten region is delimited by white lines.	86

- 6.8 Microstructure of a (100)/(100) YBCO/Ag/YBCO interface of a sample quenched after an annealing at 1000°C during 3h. The initial thickness of the Ag foil was 50 μ m: SEM micrograph of the plane a) parallel to the c-axis; b) perpendicular to the c-axis. The molten region is limited by the white lines. 88
- 6.9 SEM micrograph of a YBCO/Ag/YBCO interface of a quenched sample, where a detail of the Ag diffusion along the macrocracks existing in the TSMG YBCO samples can be observed. 90
- 6.10 SEM micrograph of an ac plane of a (100)/(100) YBCO/Ag/YBCO interface of the sample Ag_{25} quenched after an annealing at 1000°C during 3h. The initial thickness of the Ag foil was 25 μ m, the welding plane was {100} and the observed plane is: a) parallel to the c-axis; b) perpendicular to the c-axis. The molten region is limited by the white lines. 92
- 6.11 SEM micrograph showing a higher magnification at the edge of the ac plane corresponding to the sample Ag_{25} 93
- 6.12 BSE micrograph of a (100)/(100) YBCO/Ag/YBCO interface of a sample quenched after an annealing at 1000°C during 3h. The initial thickness of the Ag foil was 10 μ m, the welding plane was {100} and the observed plane is parallel to the ab plane. 94
- 6.13 Electronic microprobe analysis of a YBCO/Ag/YBCO interface of a quenched sample: a) map of Ag-rich region; b) map of Cu-rich region. The initial thickness of the Ag foil was 10 μ m, the welding plane was {100} and the observed plane is perpendicular to the c-axis. The junction is indicated by arrows. 95
- 6.14 Elemental concentration found in the sample Ag_{10} , inside and outside the molten area: red line Cu; yellow line Ag; blue line Y; magenta line Ba. 96
- 6.15 Schematic temperature profile used to optimize the cooling rate. The sample is heated up to 1000°C and held at this temperature during 3 hours. This temperature lies between T_p (YBCO) and T_p (YBCO/Ag). The arrangement is cooled down with different cooling rates of 6, 3, 1.8 and 0.6°C/h in a temperature range of $\Delta T=33^\circ\text{C}$ 98

6.16 General view of the microstructure of the YBCO/Ag/YBCO interface obtained by cooling it down at 6°C/h and obtained by BSE. The junction is indicated by arrows. 99

6.17 BSE micrographs corresponding to the ab plane of the sample C_6 grown by employing a cooling rate of $r=6^\circ\text{C}/\text{h}$. The junction is indicated by arrows. a) BSE micrograph corresponding to the central part of the junction. Some Y211 particles can be observed at the interface; b) BSE micrograph corresponding to the lateral part of the junction where some agglomerations can be observed. 100

6.18 Electron microprobe analysis obtained by scanning a $512\mu\text{m}\times 488\mu\text{m}$ region corresponding to the edge of the sample C_6 grown by using a cooling rate of $r=6^\circ\text{C}/\text{h}$: a) map of Ag-rich region; b) map of Cu-rich region. The junction is indicated by arrows. 101

6.19 General view of the microstructure of the YBCO/Ag/YBCO interface obtained by cooling it down at 3°C/h and obtained by optical microscopy. The junction is indicated by arrows. 102

6.20 BSE micrographs corresponding to the ab plane of the sample C_3 grown by employing a cooling rate of $r=3^\circ\text{C}/\text{h}$. The junction is indicated by arrows. a) BSE micrograph corresponding to the central part of the junction. Some Y211 particles can be observed at the interface; b) BSE micrograph corresponding to the lateral part of the junction where some agglomerations can be observed. 103

6.21 Electron microprobe analysis obtained by scanning a $608\mu\text{m}\times 580\mu\text{m}$ region corresponding to the edge of the sample C_3 grown by using a cooling rate of $r=3^\circ\text{C}/\text{h}$: a) map of Ag-rich region; b) map of Cu-rich region. The junction is indicated by arrows. 103

6.22 General view of the microstructure of the YBCO/Ag/YBCO interface obtained by cooling it down at 1.8°C/h and obtained by SEM. The junction is indicated by arrows. 105

- 6.23 BSE micrographs corresponding to the ab plane of the sample $C_{1.8}$ grown by employing a cooling rate of $r=1.8^{\circ}\text{C}/\text{h}$. The junction is indicated by arrows. a) BSE micrograph corresponding to the central part of the junction. Some Y211 particles can be observed at the interface; b) BSE micrograph corresponding to the lateral part of the junction where some agglomerations can be observed. 105
- 6.24 BSE micrograph corresponding to a section of $89\mu\text{m}\times 71\mu\text{m}$ of the sample $C_{1.8}$ located outside the interface showing the microstructure of the YBCO matrix. In the left part of the figure it is shown by the white circle the region who was analyzed. 106
- 6.25 BSE micrograph of the joint obtained when the cooling rate is $0.6^{\circ}\text{C}/\text{h}$. The junction is indicated by arrows. 106
- 6.26 BSE micrograph showing a higher magnification of the joint obtained when the cooling rate is $0.6^{\circ}\text{C}/\text{h}$. The junction is indicated by arrows. No sign of the junction is seen. 107
- 6.27 The influence of the cooling rate on the high quality joint dimensions. 108
- 6.28 Liquid losses dependence with the cooling rate. If the sample losses more than 2% from its weight it will correspond to the lost of all the Ag used for joining. 111
- 6.29 Electron microprobe analysis obtained for a joint cooled down at $0.6^{\circ}\text{C}/\text{h}$ 111
- 6.30 Optical micrographs corresponding to the (a,c) planes of joints obtained at different cooling rates: a) $6^{\circ}\text{C}/\text{h}$; b) $3^{\circ}\text{C}/\text{h}$; c) $0.6^{\circ}\text{C}/\text{h}$ 112
- 6.31 Optical micrograph of the ac plane of the sample C_6 showing the microstructure of the joining zone with more detail. The joint and the oxygenation bands are indicated in the figure. 114
- 6.32 a) Optical micrograph of the ac plane of the sample C_3 showing the microstructure of the joining zone with more detail. The joint is indicated by arrows and the oxygenation bands are indicated also in the figure; b) BSE micrograph at the edge of the sample C_3 showing the presence of Ag and $\text{BaCuO}_2\text{-CuO}$ phase which was pushed away by the growth interface. The joint and the molten and resolidified region are indicated in the figure. 115

- 6.33 The map of elements of a YBCO/Ag/YBCO interface of the sample C_3 : a) Ag precipitates; b) Cu precipitates. 116
- 6.34 Optical micrograph showing the microstructure of the ac plane of the joint $C_{0.6}$ cooled down at $0.6^\circ\text{C}/\text{h}$. The joint is indicated by arrows and the dashed line indicates the oxygenation bands which are perpendicular to the junction. 116
- 6.35 Schematic view of the pushing effect observed during the welding process. The dark grey areas represent the YBCO matrix whereas the light grey areas represent the $\text{Ag} + \text{BaCuO}_2 - \text{CuO}$ precipitates which were pushed by the growth front towards the edges of the sample. 118
- 6.36 Optical micrograph of the ab plane of a joint obtained by heating it to $T_{max} = 985^\circ\text{C}$ and held for. The initial thickness of the Ag foil was of $g_{Ag} = 10\mu\text{m}$. The white area represents metallic Ag remained at the interface. 119
- 6.37 Optical micrographs corresponding the sample T_{992} : a) ab plane; b) ac plane. The joint is indicated in the figure by white arrows. 121
- 6.38 Optical micrographs showing the microstructure of the ab planes corresponding to the sample T_{1005} . The joint is indicated by arrows. The central part presents some phases which have been trapped by the interface during the welding process. 121
- 6.39 Optical micrographs corresponding the sample ΔT_{18} : a) ab plane; b) ac plane. The joints are indicated in figure by arrows. 123
- 6.40 Optical micrographs showing the microstructure of sample ΔT_{23} : a) ab plane; b) ac plane. The interfaces are indicated in figure by arrows. 124
- 6.41 Optical micrographs showing the microstructure of the sample ΔT_{28} for: a) ab plane; b) ac plane. The interfaces are indicated in figure by arrows. 125
- 6.42 Optical micrographs showing the microstructure of the sample ΔT_{33} for: a) ab plane; b) ac plane. The interfaces are indicated in figure by arrows. 126
- 6.43 Schematic drawing of YBCO/Ag/YBCO joints cut into two slices parallel to the ab planes. 128
- 6.44 Microstructure of an ab plane of a YBCO/Ag/YBCO joint obtained by using a cooling rate of $r = 6^\circ\text{C}/\text{h}$. a) top face; b) center face; c) bottom face 129

6.45	Detail of the YBCO/Ag/YBCO interface when the joint is grown by using a cooling rate of $r=6^{\circ}\text{C/h}$; the interface is indicated by white arrows.	130
6.46	EDX analysis inside an YBCO/Ag/YBCO interface corresponding to the sample A obtained by using a cooling rate of $r=6^{\circ}\text{C/h}$. a) Ag precipitates and b) BaCuO_2 phase.	130
6.47	Microstructure of an ab plane of a YBCO/Ag/YBCO joint obtained by using a cooling rate of $r=3^{\circ}\text{C/h}$. a) top face (optical micrograph corresponding to a section of $0.24 \times 0.1 \text{ cm}^2$); b) center face (SEM micrograph corresponding to all the length of the joint); c) bottom face (SEM micrograph corresponding to all the length of the joint). The joint is indicated by arrows.	133
7.1	Critical current density distribution for a fully-penetrated magnetic flux single domain in the Bean model for a: a) square shaped superconductor; b) rectangle geometry. The d lines are indicated in the figure.	139
7.2	Sketch of the critical current density distribution at a grain boundary with a ratio $J_c^{gb} / J_c^{grain} = 0.5$ assuming Bean's model. α is the angle formed by the d^+ lines with the grain boundary and is determined by equation 7.1.	140
7.3	Dependence of the angle α with the ration J_c^{gb} / J_c^{grain}	141
7.4	a) Current distribution calculated for two rectangular bicrystals with: a) $J_c^{gb} / J_c^{grain} = 0.5$; b) $J_c^{gb} / J_c^{grain} = 0.25$. The angle α is indicated in the figures. The blue lines indicate intergrain critical current and the red lines the intragranular critical current loops.	141
7.5	Representation of x and y component of critical current density in a bicrystal. Granatum color indicates the y component, J_{cy} , whereas blue color indicates the x component J_{cx} . The both components contribution is indicated by pink color in the figure. Dark color reflects the negative component of J_c and light color the positive component.	143

- 7.6 Determination of critical current density of YBCO grains and joint. a) J_{cy} of YBCO grains: section the profile parallel to the weld (outside of the rhombus area) where the y component corresponds to the full intragrain current. b) Localization of the joint: J_{cy} components profile is sectioned perpendicular to the joint along which the grain and the weld critical current contributions exist; c) J_{cy} of joint: cut the profile parallel to the weld along which J_{cy} component is associated of the current crossing the weld. 144
- 7.7 a) 2D remanent magnetization profile corresponding to a low quality superconducting "real joint". At the left side of the profile it is shown a section of this map perpendicular to the junction. The presence of two peaks can be observed. b) Critical current density pattern for the y component of the J_c obtained for a low quality "real" joint by using the Caragol software. The superposed figure corresponds with the theoretical critical current density pattern determined for the y component of J_c . The areas indicated in the figure with (a), (b), (c) and (d) correspond with the zones where the J_c^{grain} can be determined, whereas the areas (e) and (f) correspond with the zones where the J_c^{gb} is determined. c)-d) section of the critical current density pattern parallel to the joint, outside the rhombus area where only the grain contribution exists (J_c^{grain}) ((a), (b), (c) and (d)); e) section of the critical current density pattern along the joint where only the weld contribution exists (J_c^{gb}) ((e) and (f)). 149

- 7.8 a) 2D remanent magnetization profile corresponding to a high quality superconducting "real joint". In the right side of the profile is shown a section perpendicular to the junction. The existence of only one peak corresponding to the center of the sample is shown. b) Critical current density pattern for the y component of the J_c obtained for a high quality "real" joint by using the "Caragol" software [96]. The superposed figure corresponds with the theoretical critical current density pattern determined for the y component of J_c of a single domain. The areas indicated in the figure with (a) and (b) correspond with the zones where the J_c^{grain} and J_c^{gb} can be determined as in the previous case; c) section perpendicular to the junction in region (b); d)-e) section parallel to the junction where the J_c^{grain} from the upper and bottom parts, respectively, are determined; f) section along the junction where only J_c^{gb} can be determined. 150
- 7.9 3D remanent magnetization map for a non-superconducting joint obtained by gluing two YBCO single domains after a fc process. The joint and inhomogeneities associated with macrocracks are indicated by arrows. 152
- 7.10 Calculated current distribution superposed with the remanent magnetization after a fc process of a sample prepared artificially by gluing two YBCO single domains. The junction is indicated in the figure and the limit of the sample are indicated for clarity. 153
- 7.11 Calculated critical current density for YBCO grains and junction for a glued-sample. The red and blue columns indicate the J_c^{grain} values and the yellow column indicates the J_c^{gb} values obtained by employing the methodology described in Section 7.1. Error bars quantify the dispersion of J_c values. 154
- 7.12 Schematic drawing illustrating the annealing cycle performed under 1 bar of oxygen flow. 156
- 7.13 Remanent magnetization maps corresponding to different oxygenation times: a)120 hours; b)168 hours; c)240 hours and d)310 hours. In all maps we can observe two differentiated peaks showing the weak link behavior of the joint. Sample dimensions are of $\simeq 0.96 \times 1 \times 0.6 \text{ cm}^3$ 157

- 7.14 Red line: Section of the remanent magnetization profile obtained for the sample. Blue lines: Remanent magnetization profile obtained for each YBCO grain after the fitting of experimental data. Yellow line: Gaussian fit of the experimental remanent magnetization profile. The sample studied is oxygenated for 168h at 450°C. . . . 160
- 7.15 Remanent magnetization of the YBCO/Ag/YBCO joints versus the annealing time under 1 bar oxygen at 450°C. The Grain 1 and Grain 2 are represented by the red and blue symbols respectively. The joint is represented by the yellow symbols., A schematic representation of the configuration of the grains and joint is presented in the inset of the figure. 161
- 7.16 Critical current density of the YBCO/Ag/YBCO joints as a function of the annealing time in oxygen atmosphere. The red and blue lines correspond to the YBCO grains and the yellow line to the joint. The error bars quantify the dispersion on J_c values for both YBCO grain and joint. 162
- 7.17 Dependence of the ratio J_c^{gb} / J_c^{grain} with the oxygenation time. The J_c^{gb} represents the critical current density of the joint and J_c^{grain} represents the lowest critical current density value exhibited by the YBCO grains. This ratio gives us the percentage of the reduction of the J_c^{gb} after the welding process. The error bars in the figure quantify the dispersion of J_c^{gb} / J_c^{grain} values. 163
- 7.18 Configuration of the samples studied in this work before the welding process, were both YBCO monoliths and joint are well defined. 166
- 7.19 Section perpendicular to the weld of the fc remanent magnetization for:(a) C_6 ; b) C_3 ; c) $C_{1.8}$ and d) $C_{0.6}$ samples. Note that the absolute remanent magnetization of the samples is not comparable since the sample dimensions were no exactly the same. 167
- 7.20 Red line: Section of the remanent magnetization profile obtained for the sample C_6 . Blue lines: Remanent magnetization profile obtained for each YBCO grain after the fitting of experimental data. Yellow line: Sum of remanent magnetization profiles corresponding to YBCO grains. 168

- 7.21 Red line: Section of the remanent magnetization profile obtained for the sample C_3 . Blue lines: Remanent magnetization profile obtained for each YBCO grain after the fitting of experimental data. Yellow line: Sum of remanent magnetization profiles corresponding to YBCO grains. 169
- 7.22 Dependence of critical current density on the cooling rate for: Grain 1 (red symbols), Grain 2 (blue symbols) and for the joint (yellow symbols). The error bars quantify the dispersion of J_c values. 172
- 7.23 Current distribution calculated from Hall magnetization map in the remanence after a fc process of the samples: a) C_6 , b) C_3 , c) C_{18} , d) C_{06} . The position of the weld and the sample limits are indicated in the figure for clarity. 173
- 7.24 Dependence of ratio J_c^{gb} / J_c^{grain} with the cooling rate. 174
- 7.25 Dependence with the cooling rate of angle α which describes the angle between d^+ lines and the junction line. The limit detection of the Hall probe imaging system used is indicated in the figure. 176
- 7.26 2D remanent magnetization map after a fc process for:(a) ΔT_{28} ; b) ΔT_{23} ; c) ΔT_{18} . Note that the absolute remanent magnetization of the samples is not comparable since the sample dimensions were no exactly the same. The joint is indicated by arrow. 179
- 7.27 Red line: Section of remanent magnetization profile corresponding to the sample Δ_{18} ; Blue lines: Remanent magnetization profile corresponding to each YBCO grain if it is analyzed separately after a gaussian fit of the experimental profile. Yellow line: Sum of remanent magnetization profiles obtained after the gaussian fit. 180
- 7.28 Red line: Section of remanent magnetization profile corresponding to the sample ΔT_{23} ; Blue lines: Remanent magnetization profile corresponding to each YBCO grain if it is analyzed separately after a gaussian fit of the experimental profile. Yellow line: Sum of remanent magnetization profiles obtained after the gaussian fit. 181

- 7.29 Circulating current vector calculated from flux mapping shown in figure 7.26(a-c) corresponding to the samples: a) ΔT_{28} , b) ΔT_{23} and c) ΔT_{18} respectively. The sample limits and the junction are shown in figure for clarity. 183
- 7.30 Critical current density values corresponding to the Grain 1 (red symbols), Grain 2 (blue symbols) and joint (yellow symbols) for each temperature window investigated. 184
- 7.31 Dependence of ratio J_c^{gb} / J_c^{grain} with the temperature window. 185
- 7.32 Dependence of the angle α with the temperature window. 186
- 7.33 2D remanent magnetization profiles obtained after a fc process for different faces along c-axis of the sample ΔT_{28} : a) top face, b) center face and c) bottom face. The center face is situated 0.3cm below the top face and the bottom face is situated 0.6cm below the top face. The dimension of c-axis of the top face is different from the other faces. The joint is indicated in the figure by arrows 188
- 7.34 Critical current density values obtained along c-axis of the junction at different depths from the face, where the seed was located during the TS process. The sample analyzed is ΔT_{28} . Red and blue symbols indicate the critical current density dependence along the c-axis of the sample of the Grain 1 and Grain 2, respectively. Yellow symbols indicate this dependence for the joints. Error bars show the J_c values dispersion obtained by using the methodology detailed in Section 7.1. 190
- 7.35 Dependence of ratio J_c^{gb} / J_c^{grain} with sample face analyzed (red and blue symbols for grains, whereas yellow symbols for the joint). 192
- 7.36 Remanent magnetization maps obtained after applying a field cooled process for different faces obtained along the c-axis of the sample C_3 . a) top face, b) center face and c) bottom face. The center face is located at 0.3cm below the top face, whereas the bottom face is located at 0.6cm below the top face. The joints are indicated by arrows in the figure. 193
- 7.37 Critical current density values obtained for each YBCO grain (Grain 1 indicated by red symbols and Grain 2 indicated by blue symbols) and for the final joint (indicated by yellow symbols) along the c-axis of the joint. Top, center and bottom faces corresponding to the sample C_3 are investigated. 195

- 7.38 Ratio J_c^{gb} / J_c^{grain} along the c-axis of the sample analyzed. Top, center and bottom faces are investigated. 196

List of Tables

6.1	An overview of the samples studied in this work along with the parameters investigated and the value of respective parameters.	78
7.1	Dimensions of the surfaces of the YBCO monoliths submitted to the welding process and scanned by using a Hall probe. The YBCO monoliths welded of each sample studied are denoted as Grain 1 and Grain 2.	165
7.2	Dimensions of the surfaces of the YBCO monoliths submitted to the welding process and scanned by using a Hall probe. The YBCO monoliths welded of each sample studied are denoted as Grain 1 and Grain 2.	178

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