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Effective silver-assisted welding of YBCO blocks: mechanical versus electrical properties

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Abstract
Superconducting welding of bulk YBCO is a key technology allowing the fabrication of large, complex-shaped pieces for applications such as levitation, bearings or large magnets. Ideally, the electrical and mechanical properties of welds should be comparable to that of the joint grains. In this paper, we have investigated the correlation between the microstructural, mechanical and critical current density performances of melt-textured [001]-tilt YBCO welds fabricated by the silver welding technique. The hardness reduction across the weld, measured by nanoindentation, correlates linearly with the decrease of intergranular critical current density, measured at 77 K and self-field by magnetic Hall mapping. Remarkably, we show that high quality zero-angle welds could be fabricated with unaltered current and hardness performances across the joint, paving the way for the implementation of silver welds in large-scale systems.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

The performance of some bulk superconducting applications, such as levitation, large magnets or bearings, could be drastically enhanced by using larger YBa₂Cu₃O₇₋ₓ (YBCO) blocks. Unfortunately, YBCO pellets of only a few centimetres can be achieved by melt-texturing growth. This fact has motivated the development of alternative procedures to obtain larger tiles, like multi-seeding [1, 2] and welding [3] techniques. Silver-assisted seeding appears to be the most attractive method in order to build up or repair complex-shaped pieces by assembling primary domains previously fabricated by the standard melting technique; meanwhile, multi-seeding allows us to obtain large pieces during the same growth process. In any case, ideal efficiency is achieved only if the joint pieces are electrically and mechanically comparable to single-domain blocks.

In the scope of this research, we developed a robust superconducting welding technique based on the use of a metallic Ag foil as the welding agent [4]. The process is based on the controlled melting of the foil and diffusion of the Ag into the YBCO matrix at an annealing temperature $T_{\text{max}} = 985^\circ \text{C}$ above the peritectic temperature of the YBCO/Ag composites ($T_p \approx 980^\circ \text{C}$), but below that of YBCO ($T_p \approx 1010^\circ \text{C}$). The use of a silver foil as welding agent presents several advantages: (i) the large temperature window existing among the peritectic temperatures of pure YBCO and YBCO/Ag composite facilitates the seeding process of the molten interface from the lateral tiles to be welded; (ii) no dissimilar interfaces are created [5] and (iii) the process is easily scalable.

The silver welding technique enables us to produce both symmetric and asymmetric, [001]-tilt [6] or [110]-tilt [7] superconducting welds in a large range of misorientation angles, as required for flexible fabrication of different complex-shaped parts. Details of the microstructure were reported elsewhere [7]. The magnetic properties of melt-textured [001]-tilt YBCO welds have been thoroughly studied.
in the past, using Hall magnetic mapping [8]. The critical current density across the weld, \(J_{c}^{GB}\), and at the adjacent grains, \(J_{c}^{G}\), were simultaneously determined as a function of the applied magnetic field at 77 K. Inductive results demonstrated that high quality zero-angle welds could be fabricated, with inter-to-intragrain critical current density ratios \(J_{c}^{GB}/J_{c}^{G} \approx 1\). For misoriented welds, the dependence of the intergrain critical current density on the applied magnetic field and the angle, \(J_{c}^{GB}(\theta, H)\), was established. Guidelines for the choice of the most adequate \(\theta\) weld for each application were provided, as a function of operational field requirements.

It is worth remarking that, for superconducting welds to become functional in applications, mechanical stability in addition to high electrical performance must be guaranteed. Despite that, the mechanical properties of superconducting joints have never been studied.

In this paper, we report results on the mechanical properties of melt-textured YBCO silver welds in correlation with their microstructure and critical current density performance. We determined the nanohardness and Young’s modulus across welds of different [001]-tilt angles using a nanoindentation technique at room temperature [9]. In the last decade nanoindentation has become one of the most versatile methods for measurement of the imprint area \(A_c\). At this load, obtained values of \(H = E\) correspond to the YBCO compound and not to each of the separate Y123 and Y211 phases [13]. Table 1 summarizes other measurement parameters chosen

### Table 1. Experimental parameters for nanoindentations.

<table>
<thead>
<tr>
<th>Name</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Allowable drift rate</td>
<td>0.05 nm s(^{-1})</td>
</tr>
<tr>
<td>Maximum load</td>
<td>10 mN</td>
</tr>
<tr>
<td>Number of loads</td>
<td>5</td>
</tr>
<tr>
<td>Peak hold time</td>
<td>30 s</td>
</tr>
<tr>
<td>Unload percentage</td>
<td>90%</td>
</tr>
<tr>
<td>Time to load</td>
<td>15 s</td>
</tr>
</tbody>
</table>

Nanoindentation measurements at room temperature were performed with a Nanoindenter XP\(^{®}\) system (Agilent Technologies) equipped with TestWorks 4 Professional level software and a Berkovich tip. In a typical indentation process, the diamond indenter was repeatedly pushed and withdrawn from the material at a given position, and the load \((P)\) versus tip penetration \((h)\) was recorded (figure 1). The maximum applied load was chosen as 10 mN, small enough to produce net imprints which allowed the accurate measurement of the imprint area \(A_c\). At this load, obtained values of \(H\) and \(E\) correspond to the YBCO compound and not to each of the separate Y123 and Y211 phases [13]. Figure 1. (a) Load versus indenter displacement curves after five loading/unloading processes with a maximum applied force of \(P_{\text{max}} = 10\) mN for reference bulk sample in the tetragonal and orthorhombic phases. On top: the projected area \(A_c\) is obtained from an FE-SEM image of the nanoindentation imprint: (b) tetragonal phase and (c) orthorhombic phase.
for nanoindentations. The loading/unloading $P$–$h$ curve characterizes the elastic/plastic response of a sample. The elastic recovery is calculated from the difference between the total indentation depth ($h_t$) at maximum indented load ($P_{\text{max}}$) and depth of residual impression upon unloading ($h_r$).

From the analysis of the $P$–$h$ curves, the nanohardness ($H$) and Young’s modulus ($E$) at each point was obtained using the Oliver and Pharr [17] relations:

$$ h_c = h_t - \epsilon \frac{P_{\text{max}}}{S}, $$

$$ A_c = A(h_c) = 24.56 \cdot h_c^2, $$

$$ H = \frac{P}{A_c}, $$

$$ E_{\text{eff}} = \frac{\sqrt{\pi} S}{2 \sqrt{A_c}}, $$

$$ \frac{1}{E_{\text{eff}}} = \frac{1 - \nu^2}{E_1} + \frac{1 - \nu^2}{E_2}, $$

where $\epsilon$ is the strain (0.75 for the Berkovich indenter), $A_c$ is the projected area under the load, $E_{\text{eff}}$ is the effective Young’s modulus, $S$ is the elastic constant stiffness (obtained from the load/unload curve $dP/dh$), $E$ is the Young’s elastic modulus, $\nu$ is Poisson’s ratio of the tested YBCO material ($\nu = 0.3$ [18]), $E_1 = 1141$ GPa and $\nu_1 = 0.07$ are the elastic modulus and Poisson’s ratio of this nanoindenter [9]. The tip was calibrated against a fused silica standard before measurements.

In order to investigate the variation of mechanical properties across the welds, we performed nanoindentation scans on the $ab$ plane in a range of 2 cm across each boundary. The separation between imprints was 50 $\mu$m in order to isolate the plastic behaviour generated under the residual imprint (as a general rule, the imprint separation should be 20–30 times the maximum penetration depth). Six hundred indentations were performed for each studied sample in order to achieve statistical significance. Each of the $E$ and $H$ values plotted in subsequent figures is the average of 15 scans performed across the boundary, separated 50 $\mu$m from each other (figure 1(a)).

Nanoindentation imprints were observed with a field emission Hitachi H-4100 scanning electron microscope (FE-SEM). These images served a threefold purpose: (i) to discard nanoindentation measurements performed on sample pores; (ii) to measure the contact area $A_c$ required for the $E$, $H$ determination, thus avoiding over- or underestimations due to sink-in or pile-up events [19] and (iii) to investigate the different fracture mechanisms activated during the indentation process.

A magnetic Hall probe scanning system with 160 $\mu$m spatial resolution was used to determine $B_z(x, y)$ maps of the $ab$ plane of welds in the remanence at 77 K. Details of the system can be found elsewhere [20–22]. The grain and intergrain critical current densities, $J_c^G$ and $J_c^{GB}$, were simultaneously determined from inversion of the measured $B_z$ map, following the methodology described in [23, 24].

Figure 2 depicts as well the $H$ measured across a $\theta = 0^\circ$ weld (b), and the low quality $0^\circ$ weld (c).

3. Results and discussion

First, the mechanical properties of a melt-textured sample were measured to have a reference of the material characteristics prior to the welding procedure. Figure 1 displays typical loading/unloading $P$–$h$ curves measured for the reference sample in the tetragonal and orthorhombic phases. As a result of the oxygenation process, the penetration depth in the orthorhombic phase is larger than in the tetragonal phase, and thus, according to equation (3), the hardness is smaller in the former phase. The FE-SEM image of the nanoimprint in the orthorhombic phase (figure 1, inset) shows superficial defects such as cracks at the corners of the imprint and a chipping effect, which were activated during the indentation process.

The nanohardness values for the bulk reference sample in the tetragonal phase, $H = 10.6 \pm 0.4$ GPa, and orthorhombic phase, $H = 7.6 \pm 0.4$ GPa are shown in figure 2. As expected, the hardness is smaller in the less compact crystalline phase. However, the Young’s modulus remains constant in both phases, within the error ($E = 120 \pm 5$ GPa). This value is in good agreement with different values reported in the literature for melt-textured YBCO [25, 26] and smaller than those reported for Bridgman-grown YBCO at similar applied loads [13]. The Young’s modulus depends basically on the composition of the material, thus $E$ does not change significantly with the crystalline phase, but it is influenced by the texturing process affecting the microstructure.

Figure 2 depicts as well the $H$ measured across a $\theta = 0^\circ$ weld. We observe a decrease of around 10% in the grain.
Figure 3. YBCO silver weld with a \( \theta = 14^\circ \) angle. (a) Optical micrograph showing the decrease in the density of Y211 particles in a ‘welding path’ of around 50 \( \mu \)m, after oxygenation process; (b) nanohardness scan \( H(x) \) across the weld in the tetragonal (♦) and orthorhombic phase (♣), and FE-SEM images far from the weld and at the weld region.

Figure 3 shows the nanohardness measured across a \( \theta = 14^\circ \) weld in the orthorhombic phase. The \( H \) values at the grains were similar for all measured welds, in the order of \( H = 9.42 \pm 0.39 \) GPa (in tetragonal phase), with a decrease of 25–30% in the orthorhombic phase. A decrease of 40% in the hardness in a 50–100 \( \mu \)m region around the weld is observed. A microstructure analysis (figure 3(a)) reveals that the density of Y211 particles vanishes in a welding path of approximately the same width. The existence of this welding path in silver welds has been explained by the higher Y content in the melt produced by diffusion and dissolving of solid bulk phases in melted Ag during heating, and by leaking of this melt out of the weld [7]. The nanohardness of the Y211 phase is almost a factor of two larger than that of the Y123 phase (\( H_{Y211} \approx 2H_{Y123} \)), as concluded from nanoindentation measurements at very low loads [13]. Thus, the absence of Y211 particles in the welding path produces a reduction of the YBCO compound hardness measured at the weld region.

Occasionally, the oxygenation process induced microcracks in certain weld areas arising from the boundary and extending over widths of the order of 200–1000 \( \mu \)m (figure 4(b)). In that case, the nanohardness was reduced in a region of that size, broader than the welding path (figure 4(a)). Moreover, in some cases large macrocracks developed at both sides of the weld as a result of the oxygenation, as observed in figure 5(a). The nanohardness across the weld decreased then in a broad area of approx. 3 mm (figure 5(b)). Notice that the \( H(x) \) reduction is asymmetric, the steeper reduction corresponding to the grain affected by the major crack. The FE-SEM imprint image in a region affected by macrocracking presents unusual fracture effects (figure 5): there seems to be a stress field dragging material inside the imprint region, but due to the small force applied, there is not enough energy...
to produce chipping. This effect is only observed in welds affected by macrocracks and not in neat regions (figure 3).

Concerning the Young modulus, values of \( E = 120 \pm 5 \) GPa (in tetragonal and orthorhombic phases) were measured for all samples, without noticeable variation across the weld. It should be noted that \( E \) is basically determined by the interatomic bonds, i.e. the chemical composition of the material, and secondarily, on its inhomogeneous microstructure. The gradient of Y211 particles or presence of micro/macroracks at the weld region did not significantly modify \( E \), within our determination error.

Finally, we studied the relation between the electrical and mechanical performance of YBCO silver welds, the later described in terms of the ratio \( H_{GB}^{YBCO} / H_{G}^{YBCO} \) between the minimum nanohardness value at the weld region and that of the grains. The intergrain-to-intragranular critical current density of each weld, \( J_{c}^{GB} / J_{c}^{G} \), was determined from the magnetic field distribution \( B_z(x, y) \) at 77 K, in the remanence (see, e.g., figure 6), following the procedure already described in [23].

We have found that the nanohardness ratio at the weld \( H_{GB}^{YBCO} / H_{G}^{YBCO} \) linearly correlates with the inter-to-intragranular critical current density ratio \( J_{c}^{GB} / J_{c}^{G} \) at 77 K, in self-field (figure 7). The extrapolation of the linear fit tends to the expected nanohardness ratio for a YBCO/Ag/YBCO sandwich sample, \( H_{GB}^{Ag} / H_{G}^{YBCO} \approx 0.2 \). This result has a potential practical implication: by measuring a mechanical property at room temperature, one can obtain the current performance in the superconducting state, thus avoiding the use of cryogenics tests which are expensive and cumbersome to perform in industry.

For the non-disoriented weld, the critical current density and the mechanical properties across the weld remain unchanged (\( H_{GB}^{YBCO} / H_{G}^{YBCO} = 1, J_{c}^{GB} / J_{c}^{G} = 1 \)). This result shows that mechanically robust, high quality \( 0^\circ \) welds for system applications can be readily achieved by the silver welding methodology.

In this work we presented nanoindentations at room temperature. This represents an initial, non-destructive way of evaluating the mechanical properties of welds, which already provides useful information about the superconducting current performance across the weld through the correlation...
function shown in figure 7. In the future, more work should be done to gain knowledge of the mechanical properties of welds at superconducting temperatures. Nanoindentation at cryogenic temperatures still presents many technological problems. However, tensile [28] and three-point bending [29] tests could be used to deduce the Young’s modulus, fracture strength and toughness at superconducting temperatures.

4. Conclusions

Superconducting joining represents a key enabling technology for the fabrication of large YBCO blocks, allowing an enhancement in the performance of several large-scale applications. Efficient joints must prove not only high electrical connectivity but also mechanical stability. We have evaluated the mechanical and critical current density properties across the joint. This represents an important step forward towards the implementation of welds in bulk, large-scale systems.

Acknowledgments

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