Exploring the Heat Treatments of $Bi_2Sr_2CaCu_2O_{8+\delta}$/Ag Wires For Superconducting Properties

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Abstract

Advances in superconductivity research are impacting aspects of life today. This includes progress and innovation, for example, in medicine, transportation, communication and environmental control. In recent superconductivity research, high temperature superconductors (HTS) are being developed and explored. HTS, also known as Type II superconductors, are extremely popular because of the high critical temperature, which in most cases can be maintained using only liquid nitrogen. Now the leading HTS reach a critical temperature as high as 135K.

Within this work the HTS $Bi_2Sr_2CaCu_2O_{8+\delta}$ (BSCCO), superconducting at a critical temperature of about 85K, was used. In order to produce these materials a powder-in-tube BSCCO, a silver-encased superconductor, was drawn through a furnace as a continuous process. The process varied from 60 hours down to a 15-hour heat treatment. Two different size diameter tubes were used to determine what is efficient for use when a certain current and/or other properties are defined. The two cross-sectional areas used were 2.27 and 0.65 mm^2.

In order to examine the structural properties X-Ray Diffraction Pattern (XRD) were taken. The superconducting properties were determined by critical current (I_c), Superconducting Quantum Interference Device (SQID) measurements and magneto-optical imaging.

Introduction

Type II superconductors were discovered in 1986; HTS are the mystifying alloys that have a higher critical temperature than that of the traditional Type I superconductors. Conveniently, most of these can be maintained at a superconducting performance using only liquid nitrogen, at 77K.

The HTS, BSCCO, is a ceramic composed of bismuth, strontium, calcium, copper and oxygen. There are two types of BSCCO, 2212, 2223. The numerical identities signify the molecular make up of the elements in the compound.

Processed BSCCO has a particular molecular structure, such that the electrons positioned in the crystal create a series of insulating and superconducting layers. Hole-doping in the CuO layers contribute to the formation of Cooper pairs in the superconducting planes. The Cooper pairs make up the actual superconducting layers of the material. BSCCO is then said to perform with anisotropic superconductivity.

The samples come from a long wire of long filaments, of about 2 meters long. When a sample for processing is taken, a four-centimeter long sample is cut and is clamped at the ends in order to protect the filaments during the process. Within the BSCCO wires, the silver sheathing encases nineteen unprocessed filaments of the raw materials (for both the smaller and larger wires), such as bismuth oxide, strontium carbon oxide, etc. The filaments are created by the “powder-in-tube” method, where the filaments are deposited within the silver of the raw materials as previously mentioned. In that case, the layers need to re-solidify in the heat treatment such that the insulating and superconducting layers align. The alignment of the layers is essential for the higher critical current.

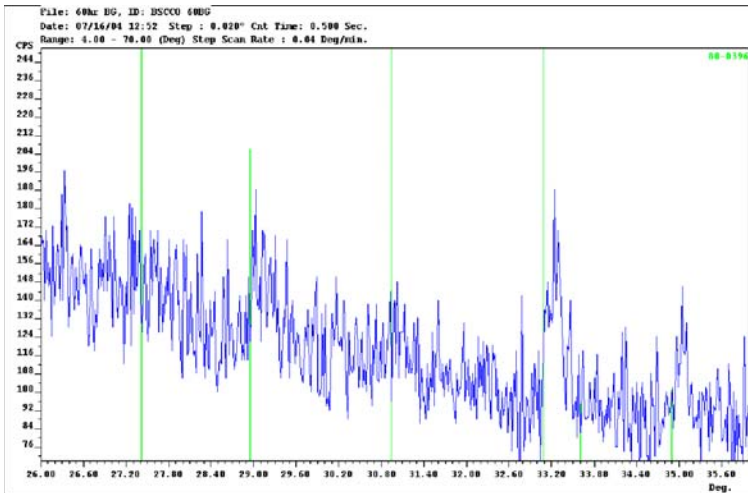
The alignment is the key reason for the heat treatment. The process melts the individual molecules down into a two-phase mixture of liquid and solid. This occurs between the temperatures of 884 and 889°C. In experimenting, my peak temperature occurred at 887°C. The temperature profile of the furnace used remained constant through experimentation, but the speed of the continuous process was varied. The oxygen supply remained constant.

The traditional heat treatment is done over a period of 60 hours. I also then ran a 30-, 20-, and 15-hour heat treatments for comparison. Some specific superconducting properties were then explored.

Experimental Methods

Evaluating the properties involved X-Ray diffraction, critical currents, magneto-optical imaging, and the critical temperature. The processed BSCCO wires were tested for the superconducting properties, which come from the aligning of the crystals, resulting from the type of heat treatment employed. (All of the tests were run on only the 20-, 30-, and 60-hour heat-treated samples, because after finding the critical currents, it was decided that the 15-hour sample was useless).

The XRD (X-Ray diffraction) was used in comparing the phase pattern of each of the heat-treated samples. The phase pattern is the way a certain material diffracts light through a series of angles. This pattern is unique to the material. The diffraction pattern used was from an angle of 4 to 70°. The "noise" in the background refers to the vibrations seen. This is due to the silver sheath surrounding the actual superconducting material, impurities in the BSCCO, and the poor crystallization. However, the samples can be compared when a small portion of the graph was enlarged around the theoretical peaks of the BSCCO (light green lines shown). The 60-hour sample had the best alignment with the theoretical peaks. (Shown here, from 26 to 36°) The light green lines are the theoretical peaks; the blue lines are the experimental. There are five points here to compare, the 30- and 20-hour samples were not as consistent as the graph shown, but much more noise was displayed, especially with the 20-hour. This is due to the lack of time in the heat

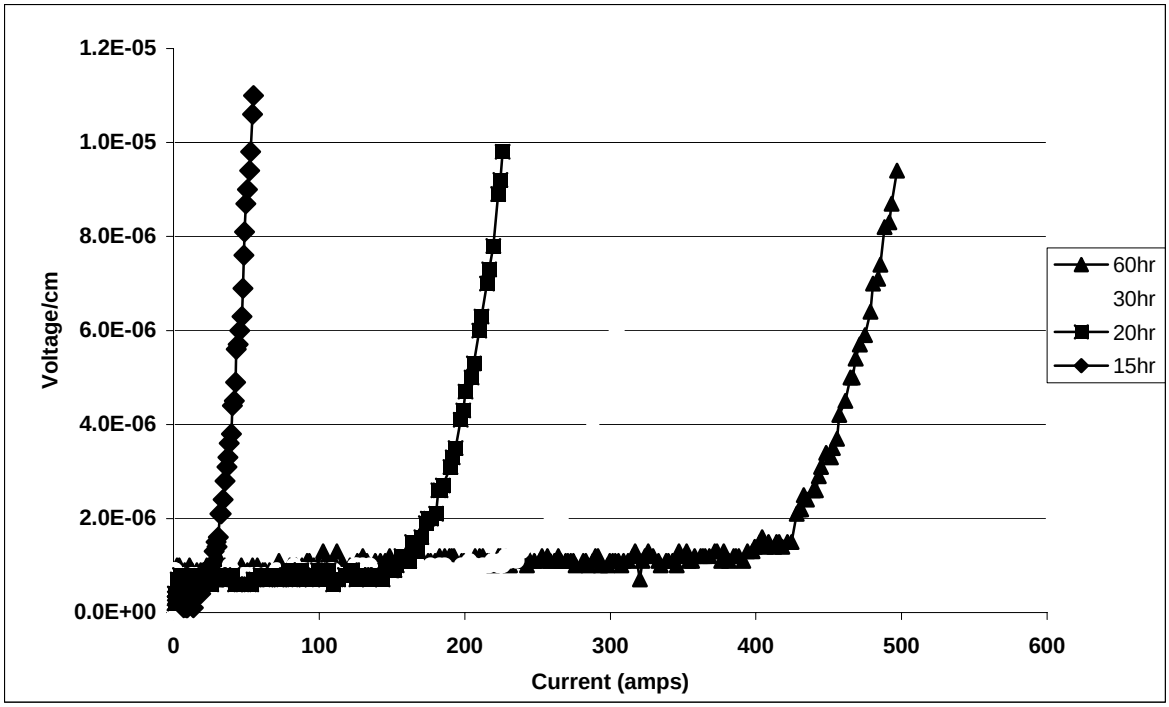


treatment to homogenize the mixture of the raw materials and the poor crystallization during the cooling down/re-solidification of the BSCCO.

The critical current of a superconductor is the maximum current that can be sent through a superconductor when it is cooled down to below its critical temperature. These data are the essential values for comparing the superconducting properties. The samples were soldered onto a probe with current leads, a voltmeter, and a temperature sensor. The probe was then lowered into liquid helium, cooled below the material's critical temperature, here using liquid helium at the NHMFL. Current was run through the samples. Using Ohm's Law, the voltage difference should theoretically stay at zero when superconducting, because of the negligible resistance of a superconductor. Ohm's Law, stated here:

$$V=IR$$

tells us that the voltage is equal to the current multiplied by the resistance. Any increase in voltage is detected due to an increase in resistance, and that is where the current has peaked for that superconductor, known as the "critical current". Shown below is a comparison of critical currents of the 2.27mm^2 cross sectional area samples.



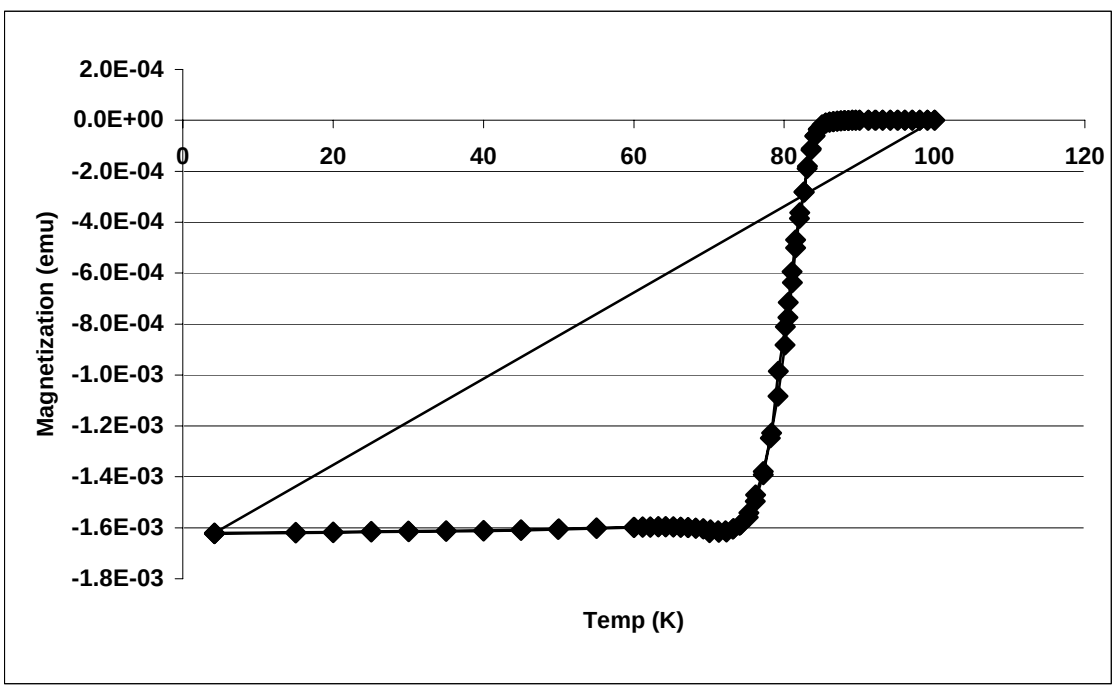
This is a very convenient comparison showing that the longer the heat treatment, the greater the critical current, and consistently decreasing from there.

Magneto-Optical Imaging (MOI) is a cryogenic microscope to convert magnetic information into optical images. When using the MOI, the sample is mounted onto to cryostat, and cooled down, with liquid helium and a series of thermal couples, to bring it to its superconducting state, below the critical temperature. A small magnetic field is then induced, surrounding the superconductor. Ideally, the superconductor will repel the field. Where the magnetic field penetrates into the material is the sites in which superconducting properties are weak, where crack propagation is found, or impurities in the material exist. On top of the superconductor is a garnet, like a small gold mirror. The procedure starts with a light source that is directed to the polarizer, where only the light of a certain polarized vertical direction may pass through. That light then hits a semi permeable mirror that will direct the light at a 90° angle, sending it downwards to the sample. Just before the light hits the sample, it passes through a garnet that appears to the garnet. The light then reacts with the garnet. The “reaction” is the rotation of the light depending on the magnetic field at each point. Where there is no field (i.e. pure superconductor where no field will penetrate) the light will not rotate at all, however,

where there is a field the light will be rotated. The light then is sent upwards, though the semi permeable mirror, but this time just passing straight through at 180° (straight up towards the CCD camera). The analyzer just before the camera is the separation of the vertical and horizontal components of how much the light has been bent by the rotated light. The vertical component is washed out (the original position of the polarized light). The horizontal component of the rotated light is directly related to the intensity (i.e. the greater the horizontal component, the more intense the field at that point). The intensity is the information that the camera captures. The light green areas are the locations of the strongest magnetic field, and the dark area is that of the superconducting area.

The MOI was an excellent procedure; the comparisons were obvious. The image of the 60-hour sample had a dark rectangular figure where no field could penetrate in. Within the image of the 30-hour sample, found were filaments running through that were dark areas. However there was no "block" of material that repelled the field, but weak filaments. The 20-hour sample showed almost no area that could repel the field. The images were an excellent comparison of the superconducting areas that formed in each one.

The last superconducting property tested was the critical temperature. Like the critical current, this is a criterion for the material to perform at a superconducting state.



The theoretical critical temperature for BSCCO (2212) is 85K. The machine used for this property was the SQUID, or the Superconducting Quantum Interference Device. This is an extremely sensitive device to detect the magnetization in a material, while adjusting other variables. After inducing a field, the superconductor's response in magnetization is recorded and plotted versus the immediate temperature. Shown below is the SQUID's plot of the magnetization of the BSCCO in temperatures from 4.2K (liquid helium) to 100K. The sudden decrease in magnetization creates a steep slope with an X-intercept of 86K. (Negative values due to the diamagnetism of the material.) This is the temperature at which the material stops responding to the field, (i.e. no magnetization in the superconductor), where the field has penetrated into the material completely, and hence the material is not superconducting any longer. This is known as the critical temperature of the superconductor. The sample used for this test was the 60-hour sample. No more data was needed, because this value will be the same for the other samples. No matter how much BSCCO crystals were formed, it should keep the same critical temperature, of either the 20-, 30-, or 60-hour sample.

Conclusion

Through little observation, it can be shown that the process duration is essential for the superconducting phase. By increasing the process time, the result is a higher critical current. The 60-hour process had ample time for the raw BSCCO compounds to uniformly intersperse and create homogeneous crystalline layers. The 30- and 20-hour processes are not as efficient. A new challenge is the manufacturing process given the brittleness of the BSCCO; silver sheathing is an expensive route.