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The anisotropic expansion and contraction of $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ (YBCO) grains during fabrication could lead to internal stresses that may be relieved by microcracking resulting in low critical currents. Neutron diffraction techniques have been used to examine the internal strain as a function of grain size in samples of YBCO. The full width at half maximum (FWHM) of the Gaussian shaped diffraction peaks are used for the strain analysis. The FWHM in the strain distribution for the coarse grain material is found to be about 0.05%. The strain variation for the fine grained material on the other hand is significantly larger and is about 0.3%. The results of this study suggests the strains are relieved by microcracking in coarse grained YBCO.

The discovery of superconducting materials with high transition temperatures such as $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ (YBCO) has led to a considerable effort to understand and improve the mechanical properties and current-carrying capacity which has been a limiting factor for practical applications. The anisotropic expansion and contraction of the YBCO grains during fabrication result in internal stresses. These stresses are expected to increase with increasing grain size and result in microcracking at a critical grain size. This microcracking results in

degraded mechanical and superconducting properties [1]. Therefore, an understanding of the effects of grain size on the magnitude of internal stresses and extent of microcracking will be very critical for developing microstructures with improved properties. In this paper we report the results of a study to apply neutron diffraction techniques to examine the effect of grain size on strain distributions in samples of YBCO.

Neutron diffraction techniques have been applied to the study of microstrains using the Intense Pulsed Neutron Source (IPNS) and General Purpose Powder Diffractometer (GPPD) at Argonne National Laboratory. Fast neutrons are moderated and produce beams at various instruments such as the GPPD, which is used for powder diffraction experiments. The GPPD is about 20 m from the target. Neutrons are detected with banks of ^3He proportional counters (140 total) encircling the sample chamber on a 1.5-m scattered flight path, at 20° , 30° , $\pm 60^\circ$, $\pm 90^\circ$ and $\pm 148^\circ$ relative to the neutron forward direction. Thermal neutrons with velocities of up to 1000 m/s are of interest for neutron diffraction. At these energies, the wavelengths are on the order of the lattice spacing and the Bragg Law of diffraction can then be applied to neutrons. In the experiments described here, Bragg's Law is used first to determine the lattice spacing d for a particular hkl reflection averaged over the sample. The variation in strain is then determined from the full width at half maximum (FWHM) of the various peaks.

The full width at half maximum (FWHM) of the Gaussian shaped diffraction peaks are used for the strain analysis as described in the following section. In this paper the diffraction pattern is presented in time-of-flight units. Time-of-flight is related to neutron wavelength by $\lambda = ht/mL$ where λ is the neutron wavelength, h is Planck's constant, t is the time-of-flight for a neutron to reach a detector after leaving the neutron source, L is the flight path for the neutron from the source to the detector and m is the neutron mass. Bragg's law, $2d\sin\Theta=\lambda$, can then be written as follows:

$$t = (2dmL/h)\sin\Theta \quad (1)$$

where d is the lattice spacing and Θ is the diffraction angle.

The widths of the Bragg peaks in the neutron diffraction spectrum have been analyzed by the integral breadth method [2,3] which assumes the peak shapes and strain contributions are Gaussian. The contributions to the width of the Bragg peak are from instrumentation, variation in lattice spacing (strain) and/or particle size distribution. The FWHM is corrected for instrument broadening using

$$F^2_{YBCO} = F^2_{exp} - F^2_{standard} \quad (2)$$

where F_{YBCO} , F_{exp} and $F_{standard}$ are the adjusted, experimentally measured and standard values of the FWHM acquired using a silicon

standard. Particle size and strain both can contribute to the broadening in the following way [3]:

$$F^2_{YBCO} = (\lambda/2D \sin\theta)^2 + (4\langle\epsilon\rangle \tan\theta)^2 \quad (3)$$

where D is the particle size and $\langle\epsilon\rangle$ is the FWHM of the strain distribution in the material.

Since the diffraction angle is fixed and the wavelength varies, the equation above can be shown to reduce to the following form

$$(\Delta t/t)^2 = (\lambda/2D \sin\theta)^2 + 4\langle\epsilon\rangle^2 \quad (4)$$

where it can be shown that $(\Delta t/t)^2 = (\Delta\lambda/\lambda)^2$ and a plot of $(\Delta t/t)^2$ vs. $(t)^2$ results in a line with intercept $4\langle\epsilon\rangle^2$ and a slope related to D, the particle size.

YBCO samples were fabricated with different grain sizes. YBCO powder (3-5 μm) was produced by vacuum calcination then pressed at 140 MPa into bars 3x6x10 mm [4]. The bars were then sintered in flowing oxygen. The small grained material was fabricated by sintering at 910°C for 10 hours reaching densities of 80-85%. The large grain samples were made by sintering at 950°C for 20 hrs with resulting densities of 85-90%. The dominant grain size was about 100 μm for the coarse grain material while the fine grain samples had grains on the order of 5 μm . Twinning was visible in the coarse grain material.

The small grain size samples should have more twinning present than the large grain samples and this could lead to broader lines in the small grain YBCO spectra [5]. Therefore, the line broadening could be the result of twinning and/or strain. The evaluation of coarse grain material indicates that in spite of the presence of twinning there was minimal line broadening. Thus, it is likely that the contribution to the line broadening due to twinning is small and that the line broadening in the small grain YBCO is an indication of the strain present. Some shifts in the position of the diffraction peaks were observed. These shifts are attributed to variation in stoichiometry between the small and large grain material.

Figure 1 shows the plot of corrected $(\Delta t/t)^2$ vs. $(t)^2$ for the coarse grain YBCO material. A linear fit to the data is shown. Figure 2 shows the results for the fine grained YBCO sample. The scatter in the data for the small grained samples is much larger and as a result the intercept is estimated by eye in this case.

The strain ($\langle e \rangle$) in the coarse grain material is very small as can be seen in Fig.1. The strain ($\langle e \rangle$) calculated from Eq.1 using a linear fit to the data to obtain the intercept is about 0.05% (the essentially zero slope implies particle size effects are not significant). The strain variation for the fine grained material on the other hand is significantly larger as can be seen from Fig.2. Here an approximate value of 10^{-5} is used for the intercept. The resulting strain ($\langle e \rangle$) is about 0.3%. The anisotropic thermal expansion coefficients could lead to significant residual microstresses in the large grain YBCO yet the

strains are less than in the small grain material. It is likely that the strains are relieved by microcracking much more in the coarse grained YBCO though another possibility is that the residual stresses cause the generation of dislocation arrays to relieve the strain. The microcracking scenario is consistent with the discussion of Richardson and De Jonghe {1} who studied acoustic emissions from YBCO during processing. They present evidence indicating smaller grain sized YBCO would have less microcracking (they also point out that aligning grains may be the most effective way to reduce microcracking). The strains in the smaller grained samples and resultant stresses do not exceed the fracture strength of the material and thus are not relieved by cracking.

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Figure 1. A representation of the FWHM vs. lattice spacing for the coarse grain YBCO material for a number of diffraction peaks. The data is presented as a plot of $(\Delta t/t)^2$ vs. $(t)^2$. A linear fit to the data is shown. The intercept is $4\langle\epsilon\rangle^2$ where $\langle\epsilon\rangle$ is the FWHM of the strain distribution in the material.

Figure 2. A representation of the FWHM vs. lattice spacing for the fine grain YBCO material for a number of diffraction peaks. The data is presented as a plot of $(\Delta t/t)^2$ vs. $(t)^2$. The intercept is $4\langle\epsilon\rangle^2$ where $\langle\epsilon\rangle$ is the FWHM of the strain distribution in the material. The intercept is estimated in this case because of the scatter in the data.

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