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# SCANNING ELECTRON ACOUSTIC MICROSCOPY OF $Bi_2Sr_2CaCu_2O_x$

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The temperature dependence of scanning electron acoustic microscopy (SEAM) and cathodoluminescence signals of $Bi_2Sr_2CaCu_2O_x$ samples have been measured. The evolution of SEAM signal with temperature between 90 and 300 K indicates the capability of SEAM to detect structural changes.

ACOUSTIC methods have been recently used to detect structural phase transitions at low temperatures in several high-$T_c$ superconductors (for instance [1–3]). In particular in [1] the photoacoustic technique is used for detection of the superconducting transition. These results suggest that scanning electron acoustic microscopy (SEAM), based on the generation of acoustic waves in the sample by an incident electron beam, could also be used to characterize superconductors and to study their structural transitions. Since the intensity of the SEAM signal depends on thermal and elastic properties of the sample, its evolution with temperature can provide information about structural changes involving changes of the mentioned properties. A major property of SEAM as compared with macroscopic acoustic technique is that the information is obtained with a space resolution of about one micron. SEAM has been used in the recent years to characterize different materials [4–6] but has not been to our knowledge applied to investigate high-$T_c$ superconductors. In the present work the temperature dependence of SEAM signal has been measured in $Bi_2Sr_2CaCu_2O_x$ samples in order to investigate the capability of SEAM technique to detect structural changes in this material. Since SEAM can detect different phases or inhomogeneities in material properties, SEAM images of the samples have been also obtained. Cathodoluminescence (CL) measurements on the same samples have been performed in order to compare both, CL and SEAM, scanning electron microscopy techniques.

Sintered $Bi_2Sr_2CaCu_2O_x$ samples from Colorado Superconductors with nominal $T_c$ of 110 K were used. The samples were observed with SEAM and CL techniques in a Hitachi S-2500 or in a Cambridge S4-10 scanning electron microscopes at accelerating voltages between 20 and 30 keV and at temperatures between 300 and 90 K. SEAM technique is based on the production of acoustic waves in the specimen due to the interaction between a solid and the electron beam. The acoustic waves are detected by a transducer and used to form a scanned image or to provide the total output signal. In the experimental SEAM arrangement of this work the microscope electron beam was pulsed at frequencies from 40 kHz to 240 kHz and the electron acoustic signal was detected by a piezoelectric transducer. For CL measurements a lens was used to focus the luminescence emission onto a photomultiplier attached to a window of the microscope. A data acquisition system was used to record the CL or SEAM intensity as a function of the temperature.

Figure 1 shows the temperature dependence of SEAM signal intensity. In general, the dependence of SEAM intensity with temperature in other materials reveals a complex evolution [7] as a consequence of the temperature dependence of several material parameters such as elastic constants, coefficient of

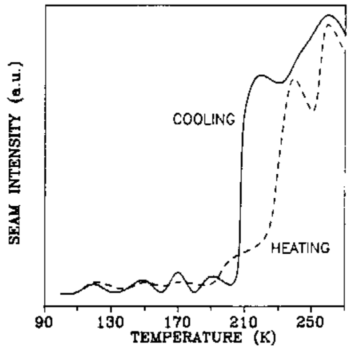

Fig. 1. Temperature dependence of SEAM intensity.

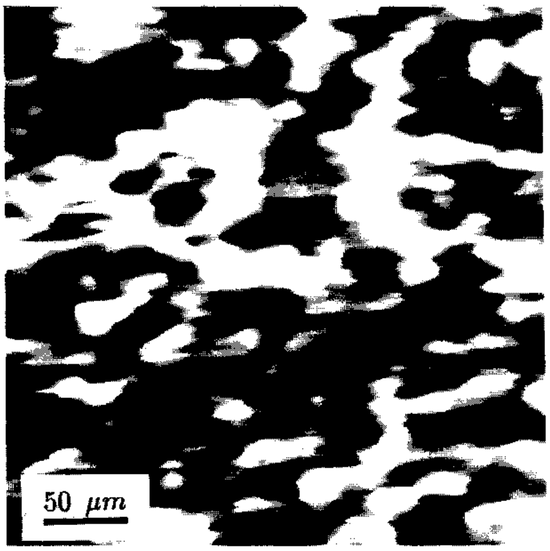

Fig. 2. SEAM image of a BSCCO sample at room temperature.

thermal expansion, heat capacity and thermal conductivity. The changes of SEAM signal are due to changes in some of these parameters. However, since many physical parameters are involved, there is not a complete quantitative theory of the SEAM signal and contrast even for less complicated materials. For this reason we cannot quantitatively relate the observed SEAM signal change at a given temperature to changes of specific physical parameters. In spite of this shortcoming our results show the great applicability of SEAM to detect structural changes in high-$T_c$ superconductors. In particular, the rapid change of the SEAM signal between 200 and 230 K is consistent with the observation of phase changes at 210 K [1] and with the heat capacity changes appeared between 200 and 250 K [8]. It is interesting to point out that in [8] the heat capacity presents a hysteretic behaviour between heating and cooling runs markedly similar to the hysteresis of the SEAM intensity represented in Fig. 1. Hysteretic behaviour in the same temperature range is also described in [1]. In addition, Fanggao *et* al. [9] found anomalous elastic effects and hysteresis in the range about 190–240 K in BSCCO by means of velocity and attenuation of longitudinal and shear ultrasonic waves. It is most remarkable the similarity between sound attenuation as a function of temperature reported in [9] and the SEAM signal evolution shown in Fig. 1. This supports our claim that the generation of SEAM signal is due to thermal and elastic properties of the BSCCO. Figure 2 shows a typical SEAM image of the sample. The general appearance of dark and bright regions is similar to that observed by CL in $Bi_2Sr_2CaCu_2O_x$ films [10] as well as in this work and is related to different phases and inhomogeneities in the properties of the sample.

Figure 3 shows the temperature dependence of CL intensity. Besides the general trend of CL increase by decreasing temperature a peak at about 120 K is observable. Although the origin of the 120 K peak is unknown the possibility that is related to some kind of transition can be considered. Some authors [11, 12] have suggested that CL is sensitive to structural transitions in superconductors. Some of the transitions reported in the literature could be magneting ordering phenomena as described theoretically in [13]. Such possibility has been studied by Litvinchuk *et al.* [14] who found a correlation in the temperature evolution of the phonon self-energies and magnetic order. For this reason one can speculate that transitions could involve luminescence changes as in some simple antiferromagnetic compounds [12]. A study of this possibility in YBCO is under way. In the case of BSCCO we can only point out with the available data that CL peak appears close to the temperature (110 K) at which Qinium Xu *et al.* [15] detected a structural change by internal friction and specific heat measurements.

In conclusion the present results show the presence of SEAM signal intensity changes at temperatures at which other authors have detected phase transitions using different techniques. This can be explained by the fact that SEAM signal depends on elastic and thermal properties whose value change in the transition.The evolution of the SEAM signal in BSCCO represents an example of a property (specific heat) that influences markedly the value of SEAM signal above 200 K. CL signal presents a different temperature dependence with significative changes in the low temperature (110–140 K) range.

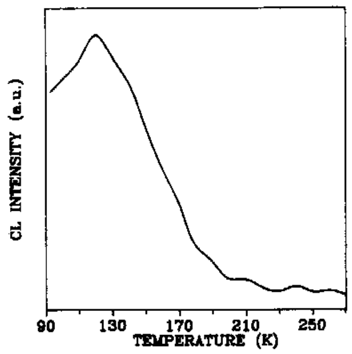

Fig. 3. Temperature dependence of CL intensity.

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