Journal of Alloys and Compounds, 211/212 (1994) 276–278
JALCOM 018

Low temperature torsion pendulum measurements on YBCO superconductors

P. Devos
Institute of Materials Science (IMS), University of Antwerp, RUCA, Middelheimlaan 1, B-2020 Antwerp (Belgium)
Materials Division, VITO, Boeretang 200, B-2400 Mol (Belgium)

R. De Batist
Institute of Materials Science (IMS), University of Antwerp, RUCA, Middelheimlaan 1, B-2020 Antwerp (Belgium)

J. Cornelis and F. Servaes
Materials Division, VITO, Boeretang 200, B-2400 Mol (Belgium)

Abstract

We report low temperature torsion pendulum measurements on two types of $YBa_2Cu_3O_{7-\delta}$ superconducting samples made from different precursors. The internal friction and modulus of the samples show in some aspects different behaviour than reported in the literature so far: a broad maximum of the internal friction around room temperature and a strong reduction of the anomalies near T_c. In addition, in both samples a peak becomes apparent when the samples are cooled below a temperature around T_c. The maximum for this peak is at 145 and 200 K for the two samples (for a frequency of about 10 Hz). Possible mechanisms for the peaks are discussed; however, details of the exact mechanism for these anomalies remain rather unclear.

1. Introduction

The low temperature internal friction spectrum of $YBa_2Cu_3O_{7-\delta}$ has been studied in various frequency ranges by several groups [1–7] and presents several damping maxima. The most notable of these for a 1 Hz vibration are a double peak around 50–100 K (with activation energies of 0.12 and 0.18 eV) and a double broad peak between 200 and 300 K in which the lower temperature (200–220 K) part appears to be rather frequency independent. In addition, further damping effects are observed between 120 and 170 K. Most of these phenomena are interpreted in terms of oxygen (in)stability in this thermodynamically unstable material; however, the detailed physical mechanisms remain as yet rather unclear. The low activation energies of the 50–100 K processes can be attributed to thermally activated jumps between the off-centre positions of the O(1) atom or to thermal hopping of carriers in the CuO_2 plane. The low temperature part of the 200–300 K process has been associated with a phase transition, although details and confirmation from direct structure measurement techniques are still missing. If these materials have (anti)ferroelectric behaviour, the destruction of (anti)ferroelectric ordering of the oxygen atoms in the off-centre positions in the CuO zigzag chains has been suggested to be responsible for the 120–170 K anomaly [5].

2. Experimental details

In this paper, additional measurements on $YBa_2Cu_3O_{7-\delta}$ material are presented. Two types of sample were prepared from different commercial precursor materials (sample 1 from Hoechst powder and sample 2 from Rhône-Poulenc powder). The samples were treated in slightly different ways. The main steps of the thermal treatment for both samples are a peritectic melting step, a brief temperature rise in oxygen as a sintering step and a final oxygenation step, similar to a thermal treatment reported elsewhere [8]. The peritectic melting process has been shown to improve the quality of the grain boundaries, which results in higher critical currents [8]. The critical temperature of both materials was 92 K and the critical current (direct transport current) of the specimens was about 370 A cm^{-2} at 77 K for sample 1 and about 420 A cm^{-2} at 77 K for sample 2, indicating good coherent grain boundaries. A.c. susceptibility measurements of parts of the pellets for both samples are shown in Fig. 1, from which one can infer the good quality of the grain boundaries.

0925-8388/94/$07.00
SSDI 0925-8388(93)00018-T

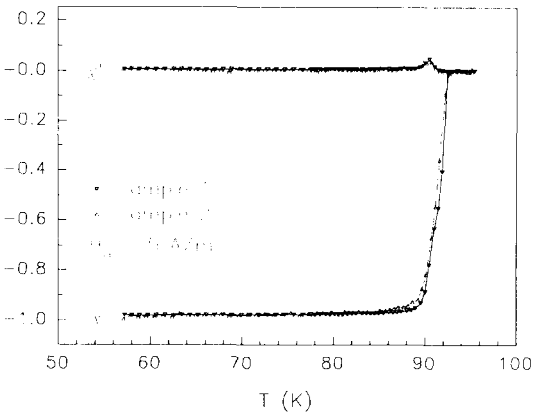

Fig. 1. A.c. susceptibility for both samples.

The structure of the material was checked by X-ray diffraction (XRD). Only for sample 2 were traces of additional phases (CuO and Y_2BaCuO_5) detected. The microstructure of sample 1, from optical microscopy, reveals heavily twinned large grains (50–100 μm). The grains of sample 2 are also heavily twinned but are smaller and more plate-like (10–50 μm). The oxygen concentration of the material was measured by thermogravimetric or iodometric measurements [9]: $\delta = 0.21$ for sample 1 and $\delta < 0.25$ for sample 2. From the pellets, bars with typical dimensions of $35 \times 1 \times 1$ mm^3 were prepared. These were measured in an automated torsion pendulum working in resonance (frequency around 10 Hz) and at constant strain amplitude. The applied strain was about 10^{-5}. A very small amount of epoxy steel paste was used to improve the clamping and facilitate mounting of the specimens. The specimens were cooled fairly rapidly and during heating of the specimens the internal friction and resonance frequency were measured from freely decaying vibrations of the specimens.

3. Results

The mechanical spectra of both samples are shown in Fig. 2. The figure shows the final spectra of a series of measurements with various start temperatures. For both specimens the following observations can be made.

In the 50–100 K temperature range the double peak is hardly observable.

In the 145–200 K range mechanical anomalies in damping and modulus are observed which are governed by the thermal history of the specimen. In Fig. 3 this anomaly is shown in detail for sample 2. The behaviour of sample 1 was analogous, except that the temperature of the anomaly was somewhat different.

In the 200–220 K temperature range a maximum in the damping is observed, in agreement with other measurements [e.g. 1, 2, 4], where the peak temperature

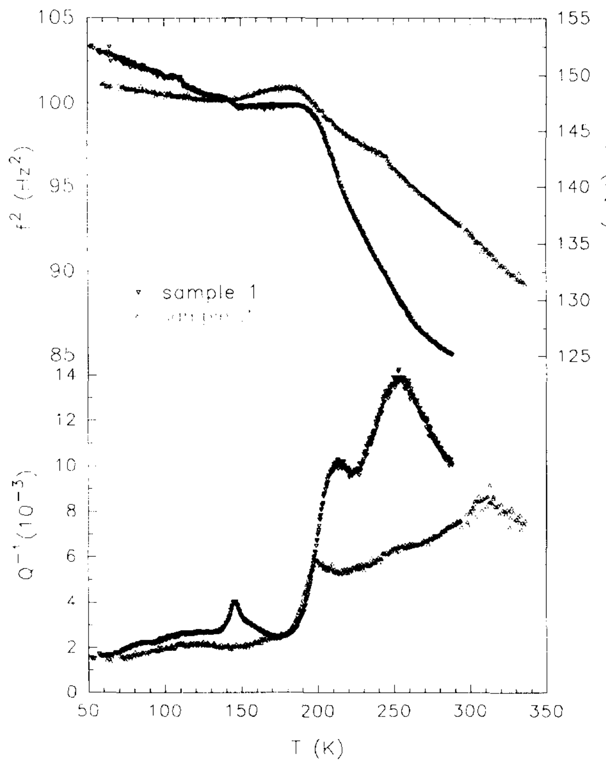

Fig. 2. Q^{-1} and f^2 (proportional to the modulus) *vs.* temperature for both samples.

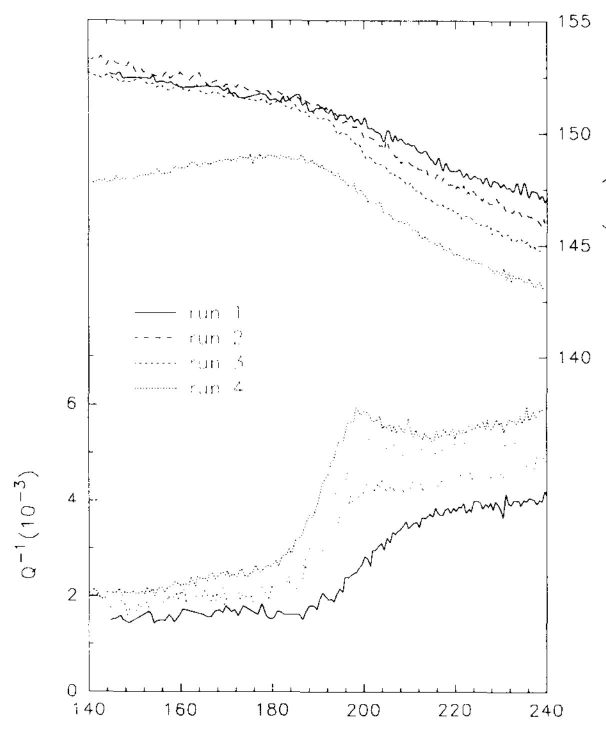

Fig. 3. Growth of the Q^{-1} peak and decrease in f^2 near 200 K for sample 2 for various runs starting from different temperatures: run 1, 120 K; run 2, 110 K; run 3, 80 K; run 4, 20 K. The effect is somewhat masked by the other anomalies.

has been found to be independent of frequency, indicating the peak to be of a phase transition nature.

Around room temperature for both samples a pronounced peak is observed, with the maxima at fairly distinct temperatures.

4. Discussion

A remarkable result of our measurements is the pronounced maximum near room temperature. As already suggested by Weller [1], a relaxation mechanism at the twin boundaries can be responsible for this peak. Transmission electron-microscopy (TEM) studies of the precise atomic arrangement at these twin boundaries (10, 11] indicate that the twin interface can consist of ledges (twinning steps) where twinning dislocations are present. Such dislocations at the twin boundary have been observed [11]. A synchroshear motion of these ledges along the twin boundary has been proposed by Van Tendeloo *et al.* [10] and consists of a small displacement of the twinning dislocation along the twin boundary and a resulting oxygen rearrangement ("reshuffling"). This results in a limited displacement of the twin boundary. In addition to the dislocations at the twin interface, other dislocations at the tips of the twin lamellae are also expected to be present.

At present we believe that the room temperature peak is the result of a relaxation mechanism based on the synchroshear motion of twinning dislocations at the twin boundaries. Under a cyclic mechanical shear stress ledges will move back and forth around their equilibrium position, giving rise to a dissipation of mechanical energy. The reason for the strong enhancement of this peak in our materials could be the high degree of twinning and the existence of internal stresses leading to a high concentration of ledges, both the result of the quite large grains and the coherent grain boundaries. The variation in the peak temperature between the two samples could be due to a difference in activation enthalpy or pre-exponential factor, strongly dependent on the precise oxygen arrangement near the twinning dislocations. The motion of the twinning steps (and especially the oxygen reshuffling) is presumably facilitated by the presence of oxygen vacancies along the twin boundaries. The dimensions of the defects involved (*e.g.* the length of the twinning dislocations) could also play a role.

Bonetti *et al.* [12] have already proposed a mechanism related to twin boundary movements for a peak they observed at much higher temperatures (560 K for 10 Hz). This is not necessarily in contradiction with our measurements, in the sense that twin boundaries can move in ways other than by synchroshear motion, especially at higher temperatures. For example, the twin boundary could move by means of the dynamical nucleation of kinks in the twinning dislocations, resulting in a more extended global movement of the twin boundary. Such a mechanism could be expected at the tips of the twin lamellae, for example.

The mechanical anomalies in the 145–200 K range, governed by the thermal history of the specimens, are of the same nature as reported by Cannelli *et al.* [5], though in a somewhat different temperature range. The anomalies appear only following cooling below a temperature in the vicinity of T_c. The peaks are fairly narrow and of the order of 1×10^{-3}. These peaks are assumed to arise from the ferroelastic (and possibly (anti)ferroelectric) ordering of the O(1) atoms in their double-potential-well positions.

It is important to note that in both samples no thermally activated peaks in the 50–100 K range are observed, or at least they are strongly reduced. The reason for this reduction remains unclear.

Acknowledgments

The authors want to thank S. Hoste (RUG, Gent) for the thermogravimetric and iodometric measurements, A. Buekenhoudt (VITO) for the a.c. susceptibility measurements and P. Diels (VITO) for the XRD analysis and optical microscopy.

This work is performed in the framework of the "Incentive Programme on High-Temperature Superconductors" supported by the Belgian State, Prime Minister's Service, Science Policy Office.

References

1 M. Weller, *Mater. Sci. Forum, 119–121* (1993) 667.
2 G. Cannelli, R. Cantelli, F. Cordero and F. Trequattrini, *Supercond. Sci. Technol., 5* (1992) 247.
3 Y. Mi, R. Schaller, H. Berger, W. Benoit and S. Sathish, *Physica C, 172* (1991) 407.
4 L. Sun, Y. Wang, H. Shen and X. Cheng, *Phys. Rev. B, 38* (1988) 5514.
5 G. Cannelli, M. Canali, R. Cantelli, F. Cordero, S. Ferraro, M. Ferretti and F. Trequattrini, *Phys. Rev. B, 45* (1992) 931.
6 B. Kusz, L. Murawski, R. Barczynski, I. Davoli, O. Gzowski and S. Stizza, *J. Mater. Sci., 25* (1990) 2125.
7 J. Jiang, H. Yin, X. Wang, Y. Sun, F. Zeng and J. Du, *Mater. Sci. Eng. B, 7* (1990) 227.
8 H. Weyten, W. Adriansens, A. Buekenhoudt, R. Leysen and J. Cornelis, *J. Alloys Comp., 195* (1993) 31.
9 H. Vlaeminck, H.H. Goossens, R. Mouton, S. Hoste and G.P. Van der Kelen, *J. Mater. Chem., 15* (1991) 863.
10 G. Van Tendeloo, D. Broddin, H.W. Zandbergen and S. Amelinckx, *Physica C, 167* (1990) 627.
11 Y. Zhu and M. Suenaga, *Philos. Mag. A, 66* (1992) 457.
12 E. Bonetti, E.G. Campari, V. Luprano, S. Mantovani, S. Casagrande and P. Cammarota, *Mater. Sci. Forum, 119–121* (1993) 689.