Experimental Report

09/09/2004

Proposal: **5-26-157** **Council:** 4/2002

Title: Quantitative Textures of Bi-2223 bulk, high-Jc, sinter-forged superconductors

This proposal is a new proposal

Researh Area: Materials

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Samples: Bi,Pb)2Sr2Ca2Cu3O10+delta
Bi2Sr2CaCu2O8

Instrument	Req. Days	All. Days	From	To
D1B	3	3	22/11/2002	25/11/2002

Abstract:

Combined texture-structure-phase-microstructure as a way to extract diffraction-accessible parameters of real samples in a stable way: examples of superconducting multiphase ceramics

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Texture, microstructure, structure and phase ratio analysis of sinter-forging multiphase bulk superconductors of the (Bi,Pb)-Sr-Ca-Cu-O family are investigated. In such materials the presence of several low crystal-symmetry textured phases makes the complete analysis a complex problem, unsolvable when working with a point detector, and the necessary bulk characterisation implies the use of neutrons. Thanks to the full characterisation of the real materials using a combined Rietveld-WIMV algorithm, the macroscopic critical current densities are correlated to the samples anisotropy.

The development of texture in polycrystalline materials remains a handicap in the characterisation of materials using diffraction techniques, as soon as non-destructibility is involved (as for expensive or rare samples). However, even if the sample can be powderised, its destruction implies the elimination of any possible correlation between measured parameters (e.g. texture and strains) that may be of interest [1]. Furthermore, it removes any possibility to make use of sample's anisotropy: since diffraction patterns of textured samples exhibit simpler signal than powder patterns, they give access to better reliabilities of the refined parameters. This is particularly true for low symmetry phases and multiphase samples. Textured samples range between powders, in which angular relationships between atomic bonds cannot be probed directly using diffraction, and ideal single crystals for which these angular values are directly measured using four-circle diffractometry. As such, texture analysis probes for angular relationships via the calculation of the Orientation Distribution (OD) of crystallites. The OD is a 3-dimensional object that self-consistently depends on the crystal structure, which provides a large number of constraints in the refinement, hereby removing parts of the correlation existing between refined parameters and ensuring, when enough independent measurements are available, a better convergence of refinement procedures.

We would like to illustrate this on 4 complex multiphase samples as encountered in high-Tc superconductivity. Samples are ceramics of the (Bi,Pb)-Sr-Ca-Cu-O system, prepared by the newly developed sinter-forging method [2] using 20, 50, 100 and 150 h of forging dwell times respectively. This method provides bulk samples exhibiting among the best superconducting properties [2]. The samples are mixtures of 2 superconducting phases, $(Bi,Pb)_2Sr_2Ca_2Cu_3O_{10}$ (Bi2223) and $(Bi,Pb)_2Sr_2CaCu_2O_8$ (Bi2212), and one alkaline earth cuprate phase $Ca_{8.2}Sr_{5.8}Cu_{23.62}Bi_{0.38}O_{41}$ (14-24). The two former are strongly textured which ensures optimum properties while the latter did not show any orientation, which renders the analysis even more complex. The phase ratio is of interest in order to optimise the elaboration process and the properties [2]. The 2θ diagrams need to be measured in a large number of sample orientations (we measured 1368 patterns for as many orientations) in order to provide as much constrains as possible. This kind of acquisition is only reasonable in time using a position sensitive detector as the one used here at the D1B line.

Figure 1 shows a selection of diagrams (χ-scans) which illustrates the refinement reliability. In this case we refined the cell parameters, mean crystallite sizes, OD and phase ratio using the combined Rietveld and WIMV [3] formalisms implemented in the MAUD program [4]. Table 1 summarises all the refined parameters. Releasing microstrains and anisotropic crystallite sizes did not provide neither a significantly better refinement nor divergence of the program. The OD allowed to recalculate the (0010) and (020) pole figures of both Bi2212 and Bi2223 (figure 2) that best refined the diagrams. These latters show the characteristic [00l] fiber texture of our samples with an orientation level as high as around 27.2 times the random powder for the maximum of the (00l) pole figure of Bi2223, which facilitates current carrying in the (a,b) planes of the superconducting phases. The textures of Bi2212 and Bi2223 are very closely linked indicating the strong growing interaction between these phases.

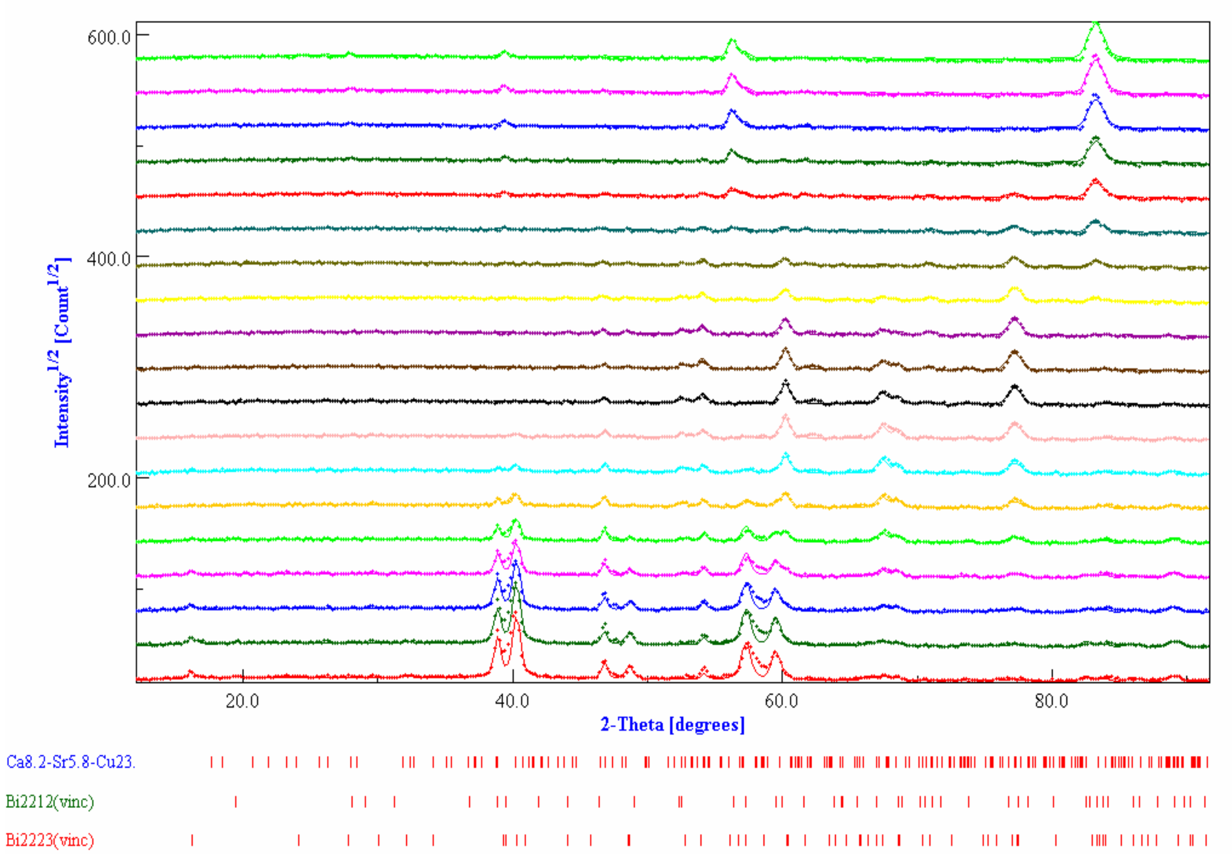

Figure 1: 2θ scans and Rietveld fits of a sample for selected diagrams measured at χ positions from 0° to 90°.

Sinter-forging dwell time (h)	Distribution density (m.r.d.)	% Bi2223	Cell parameters (Å)		Bi2223 crystallite size (nm)	J_c (A/cm^2)	Reliability factors	
			Bi2223	Bi2212			Rp	Rw
20	20.7	60(2)	a=5.419(3) b=5.391(3) c=37.168(3)	a=5.414(3) b=5.392(3) c=30.803(3)	205(7)	12500(500)	15.38	10.86
50	24.4	73(3)	a=5.419(3) b=5.408(3) c=37.192(3)	a=5.415(3) b=5.3959(3) c=30.806(3)	273(10)	15000(500)	16.08	10.89
100	25.2	84(5)	a=5.410(3) b=5.405(3) c=37.144(3)	a=5.411(3) b=5.403(3) c=30.752(3)	303(10)	19000(500)	12.93	7.77
150	27.2	87(4)	a=5.417(3) b=5.403(3) c=37.199(3)	a=5.413(3) b=5.407(3) c=30.792(3)	383(13)	20000(500)	15.39	9.16

Table 1: Summery of parameters refined in MAUD, correlated to measured J_c values.

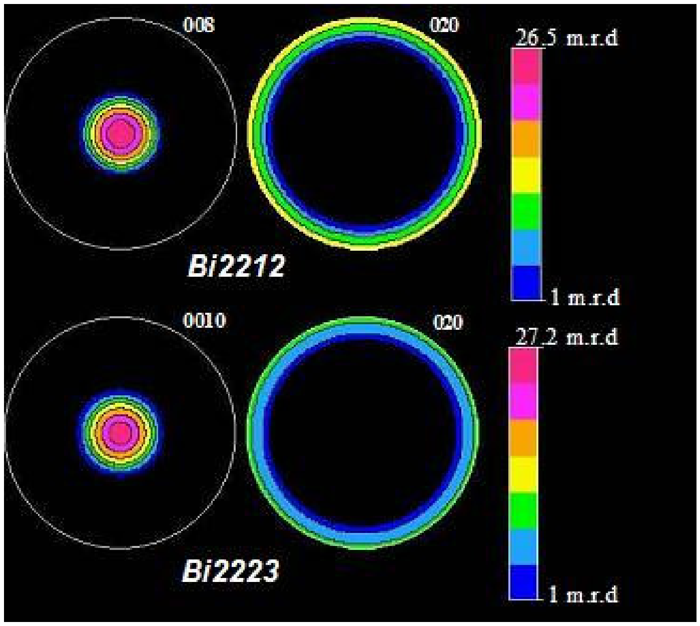

Figure 2: {0010} and {020} recalculated pole figures of Bi2212 (top) and Bi2223 (bottom) phases.

The remarkable correlation existing between the evolution of each refined parameter and the sinter-forging dwell time (Table 1) indicates that, while no significant variation of the cell parameters is observed (the phases are formed in their usual state), the improvement of the critical current densities is closely related to an increase of distribution density, Bi2223 phase content and crystallite size. The circulation of the current is not only facilitated by a better alignment of grains and a larger percentage of Bi2223 but also by a larger crystallite size which limits consequently the number of grain boundaries in the material.

References :

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[4] L. Lutterotti et al., "Textures of Materials, vol. 2" (Ed J.A. Szpunar), NRC Research Press, Ottawa 1999, 1599