ILL RESEARCH PROPOSAL

Printed : 17/11/2003

Experiment Title : **Quantitative Textures of porous YBa CuO bulk prepared by infiltration a nd melt growth process**

Proposal Number

5-26-167

Proposer (to whom correspondence will be addressed)

Name and first name	Address	Phone / Fax / Email
NOUDEM JACQUES	**CRISMAT/ISMRA, CAEN** CRISMAT/ISMRA 6 BLD MARECHAL JUIN F-14050 CAEN	+33 231452915 +33 231951600 noudem@ismra.fr

New neutron user? **No**
New ILL user? **No**

Co-proposers (mark the main proposer in each laboratory with an asterisk)

Name and first name	Laboratory	Country
CHATEIGNER Daniel	**CRISMAT/ISMRA, CAEN**	**France**
MESLIN Sophie	**CRISMAT/ISMRA, CAEN**	**France**

Local contact(s) : **OULADDIAF Bachir**

Suggested keyword number **5-26**

This proposal is :

[**x**] A new proposal.
[ ] A continuation proposal.
[ ] A resubmission.

The main research area of your proposal is

[ ] Biology [ ] Chemistry [ ] Physics [**x**] Materials [ ] Methods and instrumentation
[ ] Engineering [ ] Soft condensed matter [ ] Other :

Industry : **NOT Related to industrial applications**

Instrument required	Days
D20	**2**
D1B	**7**

Requested starting time :

1. Jan/Feb **X** 2. Mar/Apr **X** 3. May/Jun
4. Jul/Aug 5. Sep/Oct 6. Nov/Dec

Unacceptable **Jul/Aug**
Dates :

Sample availability **november**

Sample description

Substance/Formula : **YBa2Cu3O7**
Y2BaCuO5

Mass (in mg) :

Size (in mm3) : **10x10x10**

State : **polycristalline**

Surface area :

Space group : **Pmmm**

Unit cell dimension : a = **3.83** b = **3.88** c = **11.68**

T (k) = **298** α = β = γ =

Sample container :

Safety aspects

No danger associated with sample.

Is there any danger associated with the proposed sample or its preparation at ILL ?

[ ] Yes [ ] Uncertain [**X**] No If Yes or Uncertain, please give details of the risks asociated :

Is the sample a transuranium sample [ ] Yes [**X**] No

Experimental details

Energy / wavelength range : **2,523**

Resolution in energy or wavelength :

Range of momentum transfer :

Resolution in momentum transfer :

Sample environment equipement (supplied by ILL)

Environment equipment : **4-circle diffractometer**

Temperature range (stability) : Pressure range :

Magnetic-field strength (stability)

Is there any danger associated with ancillary equipement ? [ ] Yes [ ] Uncertain [**X**] No
If Yes or Uncertain, please give details of the risks associated :

To be filled in by ILL

Sample environment code	Comments by Health Physics Officer and Safety Engineer
X	

Abstract

YBCO superconductors as bulk materials suffer from limitation in the heat conduction which conditions and limits transport current capabilities, as desired for practical devices like current limiters. A way of facilitating heat exchanges with the sample's exteriors is to design percolating holes in the materials in which a cryofluid could help the heat tranfers. However, designing holes may perturbate the actual structure, microstructure and texture of the material which in turns diminishes superconducting properties. Here we would like to examine to which extent holes fashioned in the initial powders perturbates the resulting growth and properties of the melt grown samples.

Quantitative Textures of porous YBaCuO bulk prepared by infiltration and melt growth process

S. Meslin, D. Chateigner and J.G. Noudem,

CRISMAT-ENSICAEN, UMR 6508 Caen

Introduction

Yttrium compounds of the so-called "Y-123" phase ($Y_1Ba_2Cu_3O_7$) are the most promising high-Tc superconductors to date, for several practical applications. Quantitative texture inputs focussed up to now on single-domain bulk and/or coated/coating conductors tapes, in view of their potential use as motors, fault current limiter (FCL), current leads [1, 2, 3] or cables [4, 5]. However, flywheels and current leads or limiters need large Y123 ceramics with a grain oriented texture. We have developed a new methodology of artificially patterned holes for bulk texturation based on an infiltration process (figure 1a). Using this technique, highly anisotropic Y123 single-domains are obtained with **c**-axes perpendicular to the sample surface, favouring the (a,b) planes current transport. Furthermore, thanks to the relative ease in the parameters control (sample composition, oxygenation, temperature, and time), the Y123 phase is amenable to a very high degree of preferred orientation, as controlled using classical X-rays, as necessary for transport applications.

Problematic

The critical current of such compounds remains closely sensitive to the quality of the grain boundaries, somehow linked to such factors like growth rate, textural and microstructural relationships between phases, composition variations ..... However, if diffraction gives access to the structure, texture, particle sizes, microstrains, phase ratio, residual stresses ... all these influencing parameters have to be treated together in a non destructive way in order to understand the behaviour of real, sophisticated samples such as ours. The use of the so-called "combined" approach, which we developed for some years now [6], is then essential in order to take account of all the above-mentioned contributions. In order to prepare this work aiming to relate texture-microstructure-structure and physical properties, we operated preliminary 4-circle XRD measurements using a curved position sensitive detector at our laboratory. The {005/104/014} multipole figure measured on the surface of disc samples revealed a strong single-domain like texture with **c**-axes parallel to the axial pressure (figure 1b). However, only poor grain and phase representativity (statistics) could be obtained using X-rays, mainly because of a too much low number of grains in the probed volume, strongly highlighting the necessity of neutron investigations. Also, if the surface characterisation will still be probed with x-rays, samples cores are very fastidious to reliably characterise this and the additional information of both probes will be a plus to detect depth-related variations. We then want to determine on our samples, the textures, microstructures, structures, phase ratios, of all the phases in presence, in order to correlate them to the resulting macroscopic properties (transport critical current density, resistivity, magnetization and trapped field measurements).

Samples and required beam time allocation

Approximately 40 samples prepared in various temperature, p_{O2} and annealing conditions have been obtained, and their transport critical current densities and magnetic properties measured. From the the x-ray estimated texture strength and thanks to our experience of such systems at ILL (D1B), each samples will represent approximately 4 hours of acquisition time, mounting and motor position dead-time included. These scanning estimates allow the 40 samples to be measured in approximately 7 days. If the motor dead-time could be reduced,

each won second (actually 7 seconds of positionning are needed) would be 0.4h of gain per sample, i.e. some 2 days for 3 sec/sample for the whole series. We then ask for 7 days of beam time allocation for, aiming in finding by then a solution to this difficult dead time problem (which represents nearly 80% of the allocated time for such samples). Three experimentalists will relay on the experiment. The sample batch is composed of three series. The first one represents samples obtained by conventionnal Melt textured growth. The second series consists of samples infiltrated at various temperatures and source composition. Finally, a series of various porous Y123 textured bulk have been prepared.

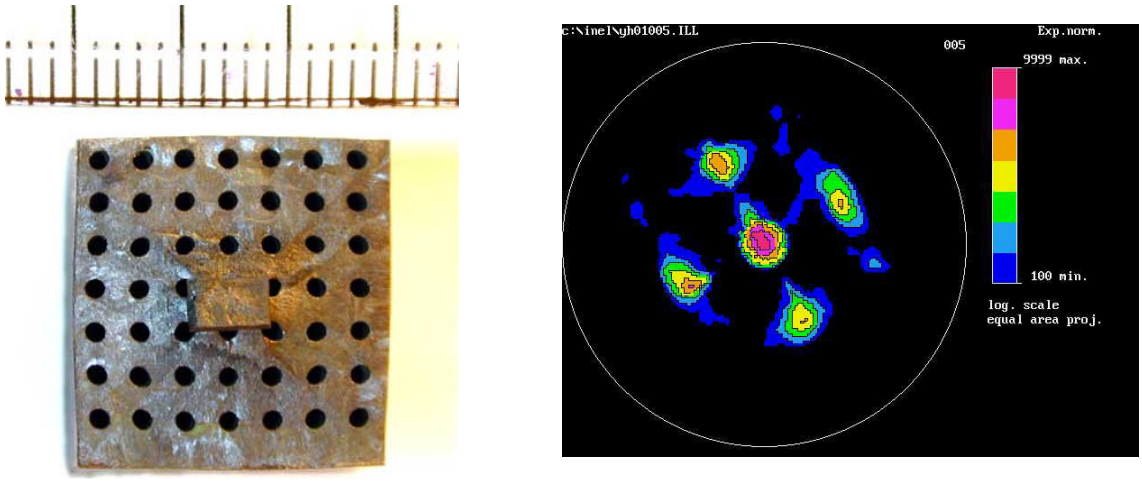

Figure 1 : a) Melt textured porous Y123 ceramic material and b) X-ray diffraction {005/104/014}multipole figure, with **c**-axes perpendicular to the sample surface (projection plane).

References

[1] J.G. Noudem, D. Bourgault, J.M. Barbut, P. Tixador and R. Tournier : *"Possible application of bulk textured Bi:2223 for current limitation"* Physica C 349 (2001) 47.

[2] W. Paul, M. Lakner, J. Rhyner, P. Unternahner, Th. Baumann, M. Chen, L. Widenhorn and A. Guérig : "Test of 1.2 MVA high- superconducting fault current limite" Supercond. Sci. Technol. 10 (1997) 914.

[3] J.G. Tuttle, T.P. Hait, R.F. Boyle, H.J. Muller, J.D. Hodge, and S.R. Breon : *"A High Tc Superconducting Current Lead Assembly for the the XDS Helium Cryostat".* Advances in Cryogenic Engineering 43, ed. P. Kittel, p. 965-972 (1998).

[4] Jin S, Mavoori H, Bower C, van Dover RB : *"High critical currents in iron-clad superconducting MgB2 wires"*. Nature. 2001 May 31;411(6837):563-5.

[5] X.D. Su, G. Witz, K. Kwasnitza and R. Flükiger : *"Fabrication of square and round Ag/Bi(2223) wires and their ac loss behaviour"*. Supercond. Sci. Technol. 15 No 8 (August 2002) 1184-1189

[6] D. Chateigner, L. Lutterotti & T. Hansen: *"Quantitative phase and texture analysis on ceramics-matrix composites using Rietveld texture analysis"*. *ILL Highlights **1997**,* 1998, 28-29.

Papers linked to texture at ILL:

- D. Chateigner, H.-R. Wenk & M. Pernet: Orientation analysis of bulk YBCO from incomplete neutron diffraction data. *J. Applied Crystallography*, **30**, 1997, 43-48.

- D. Chateigner, J. Ricote, X. Chaud, P. Gautier-Picard, E. Beaugnon, J.-L. Soubeyroux, C. Leblond & I. Monot: Levitation force to texture correlation in bulk Y-Ba-Cu-O. In "Textures of Materials, vol. 1" (Ed J.A. Szpunar), NRC Research Press, Ottawa 1999, p457-462.

- D. Chateigner, H.-R. Wenk & M. Pernet: Orientation Distributions of low symmetry polyphase materials using neutron diffraction data: application to a rock composed of quartz, biotite and felspar. *Textures & Microstructures* **33**, 1999, 35-43.

- Rivoirard S., Chateigner D., de Rango P., Fruchart D., Perrier de la Bathie R., Soubeyroux J.-L.: Texture investigation of hot-forged Nd-Fe-B magnets: *Philosophical Magazine*. **A80**, 2000, 1955-1966.

- J.G. Noudem, E. Guilmeau, D. Chateigner, S. Lambert, E. S. Reddy, B. Ouladdiaf, and G.J. Schmitz: Properties of $YBa_2Cu_3O_y$-textured superconductor foam. Accepted *Physica C 2003*

- J.G. Noudem, E. Guilmeau, D. Chateigner, B. Ouladdiaf and D. Bourgault : Performance of hot stacked-sinter forged Bi2223 ceramics : M2S-HTSC (2003) Rio de Janeiro-Bresil, accepted to Physica C (2003)

- E. Guilmeau, D. Bourgault, D. Chateigner, J.G. Noudem, B. Ouladdiaf: Performance of hot stacked-sinter forged Bi2223 ceramics. Accepted *Physica C 2003*

- E. Guilmeau, S. Lambert, D. Chateigner, J. Noudem, B. Ouladdiaf: Combined quantitative texture-phase-structure analysis of polyphased oxydes by diffraction: example of Bi-2223 sinter-forged ceramic and Y123 foam superconductors. Accepted *Materials Science and Engineering B 2003.*

Publication

17/11/2003

Proposal: **5-26-167** **Council:** 10/2003

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This proposal is a new proposal

Researh Area: Materials

Industry: **Not related to industrial application**

Main proposer: **NOUDEM JACQUES**

CRISMAT/ISMRA
6 BLD MARECHAL JUIN
F-14050 CAEN

Phone: +33 231452915
Fax: +33 231951600
E-mail: noudem@ismra.fr

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- E. Guilmeau, D. Bourgault, D. Chateigner, J.G. Noudem, B. Ouladdiaf: Performance of hot stacked-sinter forged Bi2223 ceramics. Accepted Physica C 2003
- E. Guilmeau, S. Lambert, D. Chateigner, J. Noudem, B. Ouladdiaf: Combined quantitative texture-phase-structure analysis of polyphased oxydes by diffraction: example of Bi-2223 sinter-forged ceramic and Y123 foam superconductors. Accepted Materials Science and Engineering B 2003.