Fast characterisation method for strongly textured domains

Authors:

D. Grossin and D. Chateigner (CRISMAT-ENSICAEN, Caen)
B. Ouladdiaf (ILL)
J.-G. Noudem and C. Henrist (IUT Caen)

Texture analysis is being increasingly recognised as an important tool in the characterisation of many polycrystalline materials to understand their anisotropic properties. The development of textured ceramics is required to improve physical macroscopic properties. Texture is usually characterised by means of powder x-ray or neutron diffraction, or electron backscattering diffraction. We illustrate here that, for strong multicomponent textures stabilised in large domains, classical characterisations requiring prohibitive counting time, can be avoided using neutron Laue diffraction.

Transport and levitation applications demand large superconducting, strongly three-dimensionally textured, bulk samples, and among high-Tc superconductors, $YBa_2Cu_3O_{7-\delta}$ (YBCO) appears to be the most suitable [1]. Large domains of TSMTG (Top-Seeded Melt Textured Growth) YBCO [2] were chosen because classical neutron, x-ray and electron quantitative texture analyses were already available, allowing comparison with those using the backscattering-Laue diffractometer OrientExpress.

Using x-ray or neutron diffraction, a medium resolution (using typically 5° x 5° scanning grids on a 4-circle diffractometer) classical texture experiment can be carried out in one day using large spanning 1D-detectors. However, for large samples, the low penetration of x-rays prevents bulk analysis except in cases where many sample sections can be cut and then carefully positioned relative to each other. Even here, x-rays may not provide enough statistical information [3]. For strongly textured materials, both x-ray and neutron techniques need larger scan resolutions requiring unacceptably long counting times. Electron Back-Scattering Diffraction (EBSD), although offering the great advantage of looking at the material at a local scale, suffers even more in bulk analyses due to low x-ray penetration. Moreover, back-scattered electrons are strongly sensitive to the surface topology.

For samples containing few large single crystals, using the usual statistical approach of the Orientation Distribution Function (ODF), we find that refinement vanishes, although a few misorientations can be detrimental for the resulting properties. Neutron Laue characterisation is a good alternative to show the individual orientations, taking typically less than a minute, with analysed volumes changed by collimation and micrometric XY-stage translations. EBSD and classical x-ray analyses ***(figure 1)***, using {001} pole figures, are compared with neutron Laue analysis of YBCO bulk textured samples ***(figure 2)***.

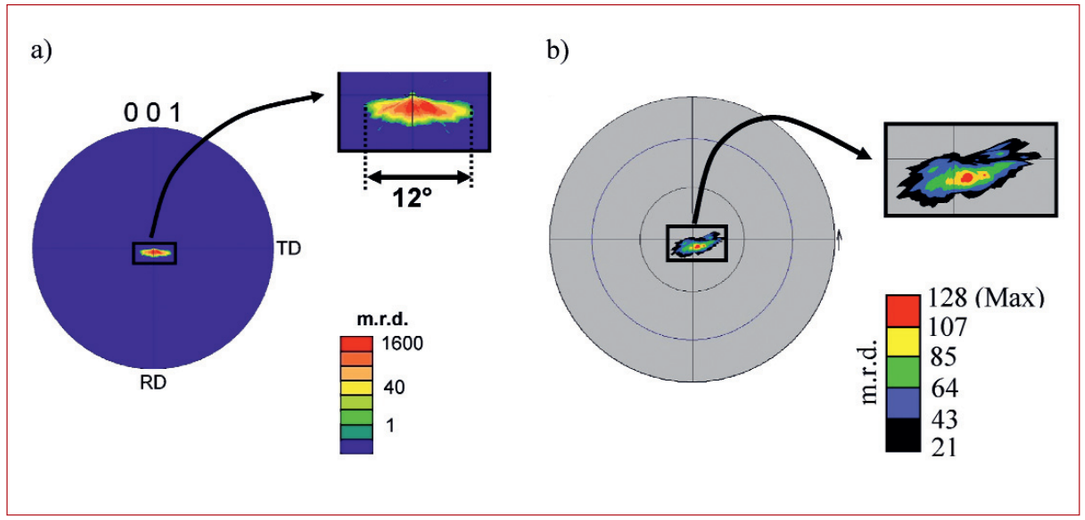

Figure 1: *{001} pole figures reconstructed from ODF refinement based on a) EBSD and b) x-ray data.*

EBSD and x-ray pole figures (figure 1a and 1b respectively) reveal the same very strong orientation, with c-axes perpendicular to the analysed sample plane and spread along the X and Y directions in a dispersion of approximately 12° and 5° at the 1 m.r.d. (multiple of a random distribution) level respectively. However, the maximum densities refined are about one order of magnitude different, which may denote large grains with insufficient statistics. These analyses come from ODF smoothing in order to retain acceptable counting times. Smoothing may mask single domain orientations present in the material.

Neutron Laue measurements performed on the same sample ***(figure 2)*** show a three-domain orientation volume. For the first measurement ***(figure 2a)***, the neutron beam is oriented perpendicular to the longest direction, with the second measurement at 90° to this orientation ***(figure 2b)*** using a 10 second exposure time and 50 mm of sample to detector. Both measurements highlight one major domain, but two strongly textured subdomains are also observed, at 1 - 2° from the major component (arrows in ***figure 2a***). The three domains are coherent with the elongation of the {001} pole observed in classical texture analysis, but cannot represent the elongated dispersion by themselves. On the Laue patterns, small crystals are present between the main Laue spots, which could account for the pole dispersion, together with the effect from ODF smoothing. In order to fully interpret the Laue diagrams in terms of orientation distributions in the future, image analysis has to be done to separate the different diffracting crystals, similar to the analysis performed on monochromatic Debye-Scherrer patterns or individual crystal Laue diagrams using synchrotron x-rays [4].

This new methodology opens the way for Laue quantitative texture characterisations using neutron diffraction for fast measurements of large-volume and strongly textured domains, avoiding scans with large numbers of measured points, and also for large-grains samples using local diffraction and texture mapping.

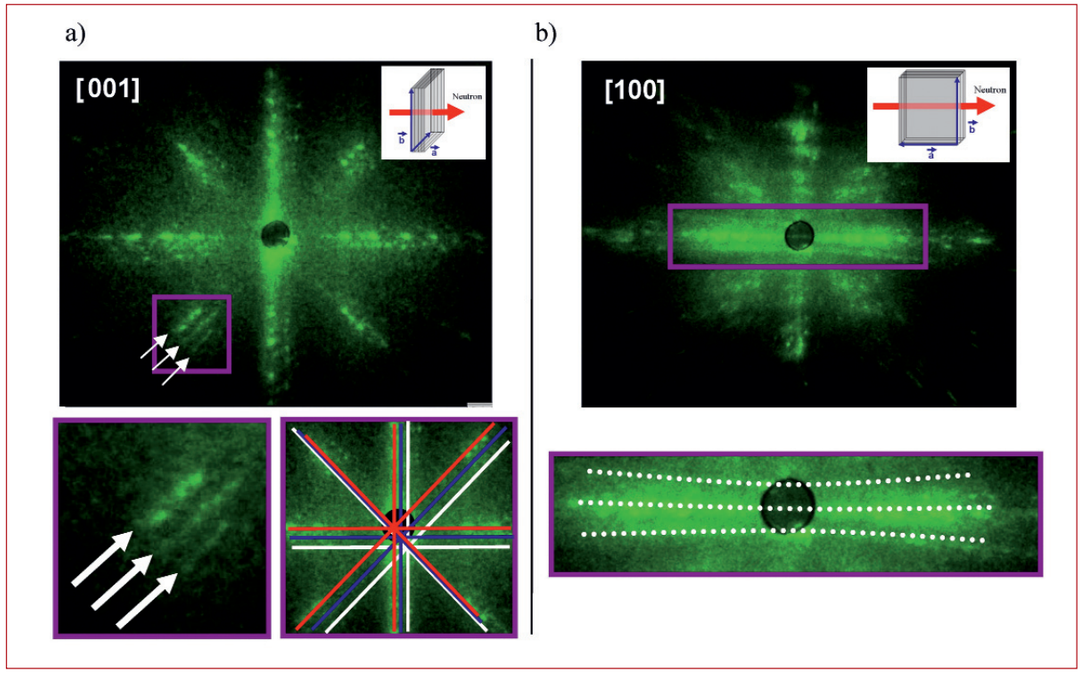

Figure 3: *Laue diagrams (top images) measured a) along the c axis and b) along the a/b axes of YBCO. Insets show the three main orientation components. Red, blue and white crosses in the bottom centre image show the c-axis orientation of all the domains and their misorientation along an elongated-like dispersion, to be compared to the {001} pole.*

References:

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