FA5-MS40-T01

Hard X-ray diffraction scanning tomography with sub-micrometer spatial resolution Hervé Palancher^a, Rémi Tucoulou^b, Pierre Bleuet^c, Eleonore Welcomme^a, Anne Bonnin^{a,b}, Peter Cloetens^b, ^aCEA, DEN, DEC, Cadarache, F13108 Saint Paul lez Durance, France. ^bESRF, ID22/ID22Ni, BP220, F38043 Grenoble Cedex,France.^cCEA, LETI, MINATEC, F38054 Grenoble, France. E-mail: herve.palancher@cea.fr

To perform 3D crystallographic phases imaging inside polycrystalline single phase but also multi phases samples, a new method combining X-ray powder diffraction with scanning tomography has recently been proposed [1-3]. One major advantage of this technique (written here Xray Diffraction-Computed Tomography (XRD-CT)) is that contrary to others methods no a priori knowledge on the phases present in the sample (crystallographic structure ...) is required. The spatial resolution of this technique is directly linked to the beam size incoming on the sample and micronscale resolution has already been demonstrated [1]. The capability of the method to reach higher spatial resolution and therefore to access nanomaterials characterization is linked to the focusing capabilities of the beamline. Since the XRD-CT technique is based on powder diffraction methods, Rietveld refinement can be partly included in the data processing just like for 2D-XRD. A previous study based on a quite similar approach for XRD-CT data treatment has been recently proposed [2].

We report in this paper the first attempt to perform XRD-CT measurements on the ID22NI hard X-ray nanoprobe of the European Synchrotron Radiation Facility (ESRF) with a beam size of 150×220 nm [4]. The studied sample is a small spherical (about 50 µm in diameter) annealed UMo particle and a multiphases interface buried about 5 µm under the surface has been especially characterized. Moreover the interest of Rietveld method for analyzing the XRD-CT data will be evaluated and the possibility to derive from such an approach the weight fraction of the various phases inside each voxel will be discussed. Finally, the advances provided by this experiment on our understanding of the UMo/Al metallurgy will be presented.

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Keywords: diffraction tomography, 3D-characterization, sub-micrometer resolution

FA5-MS40-T02

3D grain structures from X-ray diffraction contrast tomography. <u>Wolfgang Ludwig</u>^{a,b}, Andrew King^c, Peter Reischig^d, Michael Herbig^a, E.M. Lauridsen^e. ^aUniversité de Lyon, MATEIS, CNRS, France. ^bESRF, France. ^cGKSS, Germany, ^dKIT, Germany. ^eDTU, Risoe, Denmark. E-mail: ludwig@esrf.fr Combining the principles of X-ray diffraction imaging (topography) and image reconstruction from projections (tomography), it has recently become possible to map the 3D grain microstructure in a range of polycrystalline materials [1,2]. Associating this 3D orientation mapping with conventional attenuation and/or phase contrast tomography yields a non-destructive characterization technique, enabling time-lapse observation of crystal growth, deformation and damage mechanisms in the bulk of structural materials. The capabilities and limitations., as well as future perspectives of this new characterization approach will be discussed and illustrated on selected application examples.

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Keywords: 3D grain structure, X-ray tomography, X-ray diffraction

FA5-MS40-T03

Recent developments in white and monochromatic xray microdiffraction. <u>P. Gergaud</u>^a, P. Bleuet^a, J. Villanova^b, O. Sicardy^b, P Lamontagne^c, L Arnaud^a, O. Robach^d, J.S. Micha^d, O. Ulrich^d, X. Biquard^d, F. Rieutord^d. ^aCEA, LETI, MINATEC, F38054 Grenoble, France. ^bCEA, LITEN, F-38054 Grenoble, France. ^cST Microelectronics, Grenoble, France. ^dCEA, INAC, SP2M/NRS, F38054 Grenoble, France. E-mail: <u>patrice.gergaud@cea.fr</u>

Polychromatic microdiffraction has been developed for more than ten years. It is a powerful local probe of crystal structure made possible by the progress in ultra-brilliant X-ray synchrotron sources, in optics and in detectors. The technique is now available in many synchrotrons all over the world (ALS, APS, ESRF, SLS, ...).

The technique consists in scanning in x and y a polycrystalline sample in front of a broadband polychromatic x-ray beam (i.e. white) of submicrometer section, while recording, on a 2D detector placed around 2theta = 90° , the Laue patterns produced by the illuminated grains. This allows to map in 2D, with a spatial resolution around one micron laterally, the crystalline orientation near the surface of a polycrystalline material. The probing depth varies from a few microns to a few 100 microns depending on the sample's transparency.

The white beam mode allows to measure the deviatoric part of the elastic strain tensor (i.e. the local lattice parameters b/a, c/a, alpha, beta, gamma), with an accuracy in the 10^{-4} range (at least for "well-crystallized" grains), by analyzing precisely the relative positions of the center of masses of the diffraction spots. The simultaneous recording of a large number of spots also allows analyzing the distribution of orientation and/or strain inside plastified grains, from the broadening / splitting of the spots (microstrain and / or microrotations). The unit cell's lattice expansion is also accessible through an additional measurement of the energy of one or several Laue spots. For this, the incident beam is monochromatized, and the incident energy giving the maximum intensity in the selected spot is measured.

A large amount of work was dedicated to improving the instrument and the experimental methods. It concerns: