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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], [a]*CEA, DEN, DEC, Cadarache, F13108 Saint Paul lez Durance, France.* [b]*ESRF, ID22/ID22Ni, BP220, F38043 Grenoble Cedex,France.*[c]*CEA, LETI, MINATEC, F38054 Grenoble, France.*
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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 micron-scale 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

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3D grain structures from X-ray diffraction contrast tomography. Wolfgang Ludwig[a,b], Andrew King[c], Peter Reischig[d], Michael Herbig[a], E.M. Lauridsen[e].
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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

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Recent developments in white and monochromatic x-ray microdiffraction. P. Gergaud[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]. [a]*CEA, LETI, MINATEC, F38054 Grenoble, France.* [b]*CEA, LITEN, F-38054 Grenoble, France.* [c]*ST Microelectronics, Grenoble, France.* [d]*CEA, INAC, SP2M/NRS, F38054 Grenoble, France.*
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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:

1) the reduction of the beam size (a few 0.1 micron) with a new set of focusing mirrors; data collection speed thanks to new 2D detectors and increase of the incident flux, …
2) the setting up of the monochromatic mode, to obtain a good beam stability over an extended energy range, and a reproducibility of the beam size and position when shifting between white and monochromatic beam,
3) the setting up of an alternative method, and the associated analysis tools, for measuring the lattice expansion, via a direct measurement of the spot energy in the white beam mode, using an energy-resolved detector,
4) the development of in situ measurements with various sample environments (oven, stress rake, electrical test bench).
5) the development of a new technique for 3D strain imaging
Each of the above point will be detailed and examples of results will be shown.

Keywords: Microbeam, Laue diffraction

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Recent Developments in Scanning Micro and Nanobeam Diffraction Techniques. Manfred Burghammer[a], Sebastian Schöder[a], Silvia Santucci[b], Emanuela Di Cola[a], Richard Davies[a], Aurelien Gourrier[a,c], Christian Riekel[a]. [a]*European Synchrotron Radiation Facility, Grenoble, France.* [b]*CNR-IOM/ TASC Basovizza, Trieste, Italy.* [c]*Laboratoire de Physique des Solides, UMR8502 CNRS Université Paris Sud, Orsay, France.*

X-ray microbeam diffraction experiments have evolved into a well established technique since third generation synchrotron radiation sources became available [1][2]. The method has been applied to a considerable variety of scientific problems in material science, soft matter research, surface science, biology, and others. Recent developments are trying to explore scanning diffraction combined with complex sample environments e.g. allowing for optical tweezers based particle manipulation [3] or stroboscopic generation of ballistic microdrops [4]. The application of X-ray nanobeams, typically in the 100 nm range, has become a very important topic during the past few years opening new possibilities in the field of high resolution scanning diffraction and scattering [5][6]. Instrumental developments at the Microfocus Beamline (ID13, ESRF) aim at pushing the limits in terms of spatial resolution while keeping enough modularity to establish routine operation for a broad user community. Related problems and concepts will be discussed.

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Structure analysis of polyphasic nano-mixtures by automated electron diffraction. Enrico Mugnaioli[a], Tatiana Gorelik[a], Ute Kolb[a], Christina S. Birkel[b], Martin Panthöfer[b], Wolfgang Tremel[b], Mauro Gemmi[c], Johannes Fischer[c]. [a]*Physical Chemistry, Johannes Gutenberg University Mainz, Germany.* [b]*Inorganic and Analytical Chemistry, Johannes Gutenberg University Mainz, Germany.* [c]*Earth Science, University of Milano, Italy.*
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Many natural and synthetic materials appear as nano-crystalline polyphasic mixtures. Their structure, necessary for understanding their genesis and properties, is often problematic to access through X-ray powder diffraction. Apart from eventual preferred orientation, peak overlap is the biggest problem for detection and structural investigation of unknown phases, especially for low-symmetry structures.
Electron diffraction provides high resolution structural data from single nano-crystals, but is often compromised by strong dynamic effects and a limited number of sampled reflections. Automated diffraction tomography (ADT) [1, 2] is a new method delivering almost complete intensity data sets from a single nano-crystal with size down to 20 nm. Moreover, ADT data are less dynamical than conventional electron diffraction, as single diffraction patterns are collected *off zone*. Usually ADT intensity data sets allow *ab initio* structure solution by common X-ray routines in a fully kinematical approach [3, 4].
Structure analysis of two polyphasic mixtures is presented. The first is a high pressure synthetic rock in MgO-Al_2O_3-SiO_2-H_2O system having composition close to a clinochlore. The sample mostly consisted of piropo and olivine, plus a third minor monoclinic phase. This unknown phase could not be detected neither by microprobe micronalysis since the grains were too small (<1 μm), nor by X-ray powder diffraction. Yet, ADT delivered immediately unambiguous cell parameters and structure solution of the new phase from a single nano-crystal. The knowledge of the crystal structure was used to tune the chemical composition of the starting mixture in order to increase the yield of the new phase and facilitate subsequent structure refinement.
The second example is a mixture of synthetic Zn_xSb nano-particles. Conventional TEM analysis revealed the existence of two phases. Beside the known Zn_1Sb_1, a new compound was identified. Its cell parameters and symmetry could not be attributed to any known phase in the Zn_xSb system [5]. ADT allowed cell parameter determination and *ab initio* structure solution in space group P-1.

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