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. <u>Manfred</u>

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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.

 Riekel C., *Rep. Prog. Phys.*, 63(3) 2000, p. 233-262. [2] Paris O., *Biointerphases*, 3(2) 2008, p. FB16-FB26. [3] Cojoc D., Ferrari E., Garbin V., et al., *Appl. Phys. Lett.*, 91(23) 2007, 234107. [4] Graceffa R., Burghammer M., Davies R.J., Riekel C., *Rev. of Sci. Instrum.*, 79(8) 2008, 086106. [5] Hanke M., Dubslaff M., et al., *App. Phys. Lett.*, 92(19) 2008, 193109. [6] Schropp A., Boye P., Feldkamp J.M., et al., *Appl. Phys. Lett.*, 96(9) 2010, 091102

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Structure analysis of polyphasic nano-mixtures by automated electron diffraction. <u>Enrico Mugnaioli</u>^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. ^bInorganic and Analytical Chemistry, Johannes Gutenberg University Mainz, Germany. ^cEarth Science, University of Milano, Italy.

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Many natural and synthetic materials appear as nanocrystalline 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-Al₂O₃-SiO₂-H₂O 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 nanoparticles. 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.

Kolb U., Gorelik T., Kübel C., Otten, M.T., Hubert D., Ultramicroscopy 2007, 107, 507. [2] Kolb U., Gorelik T., Otten, M.T., Ultramicroscopy 2008, 108, 763. [3] Mugnaioli E., Gorelik T., Kolb U., Ultramicroscopy, 2009, 109, 758. [4] Kolb, U., Gorelik, T., Mugnaioli, E., Mater. Res. Soc. Symp. Proc. 2009, 1184-GG01-05. [5] Mozharivskyj Y., Pecharsky A.O., Bud'ko S., Miller G.J. Chem. Mater. 2004, 16, 1580.

Keywords: electron diffraction techniques, mixture, crystal structure solution