microbatch-style crystallization experiment with a different chemical composition. The MPCS allows the researcher to use the entire protein sample in crystallization experiments for efficient exploration of crystallization phase space by combining sparse matrix with gradient screening in one comprehensive hybrid crystallization trial. Furthermore, individual crystallization optimization trials can be prepared using highly granular gradients of protein and optimization reagents such as precipitation agents, ligands, or cryo-protectants. The MPCS produces Diffraction-Ready crystals that can be removed from the Peel-Apart CrystalCard for traditional cryocooling and diffraction.

Keywords: microfluidic crystallization, nanovolume, *in-situ* X-ray diffraction"

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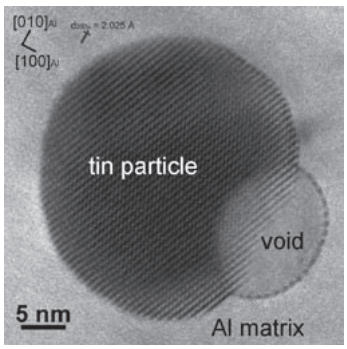
Three-dimensional void-like defects associated with tin nano-particles in aluminium

Laure Bourgeois[1,2,3], Matthew Weyland[1,2], Barry Muddle[2,3]
[1]Monash University, Monash Centre for Electron Microscopy, Building 81, Clayton, Victoria, 3800, Australia, [2]Monash University, Department of Materials Engineering, Victoria, 3800, Australia, [3]Monash University, Centre of Excellence for Design in Light Metals, Victoria, 3800, Australia, E-mail：laure.bourgeois@mcem.monash.edu.au

Defects, such as lattice defects (vacancies, dislocations, stacking faults) or extrinsic defects (solute atoms clusters) play a critical role in the nucleation and growth of precipitate phases in precipitation-strengthened aluminium alloys. Defects often act as heterogeneous nucleation sites for phases that nucleate with difficulty. Defects, vacancies in particular, may also influence or even control the kinetics of nucleation and growth. This work reports the finding and characterisation of three-dimensional defects commonly associated with tin nano-particles in aluminium. The shape, structure and composition of the defects and their surroundings were investigated using a variety of transmission electron microscopy imaging, diffraction and analytical techniques. The three-dimensional defects were deduced to contain a significant number of vacancies, hence their description as void-like. These void-like defects were found to occur in isolation at the interface between the tin precipitate and the aluminium matrix. An example of one such void observed at high magnification is shown; in this case tin can be seen to decorate the void-matrix interface.



Keywords: defects, transmission electron microscopy, aluminium alloys

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Microstructure of surface-tailored platinum nanocrystals

Emmanuel Garnier[1], Matteo Leoni[2], Paolo Scardi[2], Ken Beyerlein[3], Robert L Snyder[3]
[1]University of Poitiers UMR6503, Equipe d'Electrocatalyse, 40 Avenue du Recteur Pineau, POITIERS CEDEX, Vienne, 86000, France, [2]University of Trento, Department of Materials Engineering and Industrial Technologies, via Mesiano 77, 38100 Trento, Italy, [3]Georgia Institute of Technology, Materials Science and Engineering, 771 Ferst Drive, N.W. Atlanta, GA 30332-0245 U.S.A., E-mail：Emmanuel.Garnier@univ-poitiers.fr

Nanoscaled Pt-based materials have potential applications as electrocatalysts in fuel cells. Fundamental interest in the mechanisms of model reactions has led to the development of synthesis routes allowing a fine tuning of particle shape, mean size and size distribution. However, an effective control of the surface structure seems more important, as the majority of the reactions taking place in fuel cells are structure sensitive or site dependent (1). This fine control over surfaces is allowed by the water-in-oil or by the colloidal routes: single clean-surface nanocrystals with defined shapes and tailored percentages of {100} and {111} surface domains can be produced (2). In particular, the colloidal method allows cubes, octahedra, tetrahedra and cuboctahedra to be preferentially obtained (depending on the Pt precursors and the hydrogen bubbling time), whereas more rounded crystals are formed via water-in-oil. All nanocrystals are almost defect-free and nearly monodisperse, as confirmed by TEM and by X-ray diffraction Whole Powder Pattern Modelling (WPPM)(3). An analysis of the microstructure is proposed, based on the modelling of nanocrystal shape, size distribution, defects and surface effects, following the WPPM and the Debye equation approaches.

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(2) J. Solla-Gullon, F.J. Vidal-Iglesias, E. Herrero, J.M. Feliu, A. Aldaz, Electrochem. Comm. 8 (2006) 189.
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Keywords: nanocrystals, platinum, microstructure

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Crystallite dimensions obtained with Rietveld refinement and Delaunay triangulation

Xim Bokhimi
Universidad Nacional Autonoma de Mexico, Instituto de Fisica, A. P. 20-364, Mexico D. F., Distrito Federal, 01000, Mexico, E-mail：bokhimi@fisica.unam.mx

Crystallite dimensions and morphology of phases were obtained by refining crystalline structures with the Rietveld method. Of special interest was the case where average crystallites were modeled in reciprocal space with a linear combination of normalized spherical harmonics; the coefficients that weighted the harmonics' contribution were refined to fit the breadth of the diffraction peaks. The crystallite dimensions obtained in reciprocal space were used to calculate the corresponding ones in real space, generating a set of vertices that described crystallite surface. These vertices were used to generate a mesh of the surface using the Delaunay triangulation, which made possible to get crystallite surface area, and to generate a Delaunay tetrahedralization that was used to calculate crystallite volume. The density of each phase, determined from the Rietveld refinement, together with the determined volume were used to get crystallite mass and its specific surface area, which, for comparison, can be determined with other experimental techniques. Since in nanocrystalline materials peak breadth is mainly determined by crystallite size and microstrain, the aberrations of the diffractometer can be neglected, which is not the fall for microcrystalline materials.

Although the obtained crystallite dimensions and morphology are affected by the approximations of the diffraction pattern model used in the refinement, the technique described in the present work is always valid. Crystallite dimensions, however, approximate more to their real values, as the model used for the Rietveld refinement is improved.

Keywords: Rietveld refinement, crystallite dimensions, Delaunay triangulation

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X-ray powder microdiffraction and its limits in forensic practise

Marek Kotrly[1], Veronika Grunwaldova[2], Ivana Turkova[1]
[1]Institute of Criminalistics Prague, PO box 62/KUP, Strojnicka 27, Praha 7, 170 89, Czech Republic, [2]Zentiva, a.s. Praha, U Kabelovny 130, 102 37 Praha 10, Czech Republic, E-mail：kup321@mvcr.cz

X-ray diffraction is an important technique of forensic practise for accurate phase analysis of soil, pigments, explosives, drugs, etc. Recently, X-ray Powder Microdiffraction (micro-XRPD) has been applying predominantly, allowing analyses of very small samples. The analysed area is approaching to a size of surfaces examined by microscopic techniques, light microscopy, SEM/EDS, etc., routinely used in forensic practice. Combination of these techniques provides complementary information. Micro-XRPD allows in the forensic field a direct phase analysis recovered from traces, for which XRPD was not feasible. To employ micro-XRPD for microscopic fragments and abrasions it was necessary to carry out tests of different ways of sample fixation allowing the performance of other analyses by different methods (SEM/EDS, optical microscopy, FTIR, etc.) on the same carrier. These sample treatment eliminate either the possibility of loss or trace contamination. Classical aluminum specimen mounts for SEM with carbon tape, zero-background silicon sample holders, foils designed for XRF sampling, etc., were analysed. Each of different fixation methods brings some advantages and some drawbacks for each kind of analysed traces. Another limiting factor tested for micro-XRPD is the size of monocrystalic areas in a sample. In the case of samples containing micro/nano particles and nanocomposites plays a pivotal role the issue of detection limits for different types of these materials and their comparisons with different configuration limits of classical XRPD. Both mixture models and real materials from different areas were tested.
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Keywords: forensic microanalysis, microdiffraction, nanophases

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Analysis of atomic structure and structural imperfections of ZnTe and (Zn,Mn)Te nanowires

Wolfgang Neumann[1], Holm Kirmse[1], Slawomir Kret[2], Piotr Dluzewski[2], Elzbieta Janik[2], Adam Presz[2], Grzegorz Karczewski[2], Tomasz Wojtowicz[2], Wojciech Zaleszczyk[2]
[1]Humboldt University of Berlin, Institute of Physics, Newton Street 15, Berlin, Berlin, 12489, Germany, [2]Institute of Physics, PAS, Al. Lotnikow 32/46, Warsaw, 02-668, Poland, E-mail：wolfgang.neumann@physik.hu-berlin.de

ZnTe and (Zn,Mn)Te NWs were grown on GaAs substrates by molecular beam epitaxy via a vapour/liquid/solid mechanism (VLS) where nano-sized droplets of a gold-based eutectic act as catalysts [1]. The morphology, atomic structure and chemical composition of the NWs were investigated by transmission electron microscopy (TEM) [2]. The wires grew along the <111> directions pointing out of the (001) GaAs. The length of the wires amounts to some microns depending on the growth time. The mean diameter ranges between 30 and 60 nm depending on the size of the gold droplet located at the tip of the NWs. The NWs are single crystals. The majority of the NWs exhibit numerous two-dimensional crystal defects (stacking faults, microtwins) with only a few {111} monolayers sequence perpendicular to the NW axis as revealed by high-resolution TEM. The different types of defects were analysed in detail. The NWs consist of a core-shell structure as detected by electron energy loss spectroscopy (EELS). The shell of the NWs is formed by ZnO. The gold spheres at the tip of NWs additionally contain gallium, zinc, and tellurium. The gallium is incorporated during the initial formation of the eutectic droplets at the GaAs substrate. The distribution of the Mn along and across the NW in (Zn,Mn)Te NWs is homogeneous as detected by EELS measurements. The formation process of the NWs can be understood as a two-step process. The first step is the one-dimensional growth along the wire axis by consuming all the material deposited near the droplet. In a second step, facets are formed due to lateral growth of the NW.
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Keywords: crystal defects, nanowires, TEM characterization

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Edgeworth-series description of anisotropic microstrain broadening in powder-diffraction patterns

Andreas Leineweber
Max Planck Institute for Metals Research, Heisenbergstrasse 3, Stuttgart, Baden-Wuerttemberg, 70569, Germany, E-mail：a.leineweber@mf.mpg.de

The diffraction effects of non-Gaussian microstrain distributions within the Stokes-Wilson approximation are described by anisotropic Edgeworth series, which can - for each reflection hkl - be quantified by the 3rd, 4th ... Nth cumulants of the microstrain distribution projected on the diffraction vector of each reflection hkl. The diffraction-vector (hkl) dependence of these Nth cumulants can be described by 2Nth-rank tensors. The situation considerably simplifies in case of univariateness of the microstrain distribution, leading to equal (or inverted) shapes of the line-broadening contribution for all different hkl reflections. The model was applied to re-evaluate previously presented X-ray powder diffraction patterns of two somewhat inhomogeneous epsilon-iron-nitride powder batches, epsilon-FeN0.433 [1] and epsilon-FeN0.407 [2]. The data had revealed [1, 2] anisotropic and partly asymmetric microstrain(-like) broadening due to the N-content dependence of the lattice parameters of these hexagonal iron nitrides [3]. The specific origin of the microstrain leads to univariateness of the microstrain distribution [1, 4]. As a result of the data re-evaluation using the model of anisotropic Edgeworth series for description of the observed microstrain broadening, Edgeworth series-type probability-density functions for composition of these two powders have been determined, quantifying the inhomogeneity of the two powder batches, respectively.
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