

from the X-ray powder diffraction data alone. An *ab initio* powder structure determination of the novel zeolite SSZ-82 was then performed. While it was very difficult, if not impossible, to derive the structure directly by powder charge flipping, the structure solution procedure became straightforward when the 2D-XPB approach was applied. The positions of all the framework Si atoms and most of the O atoms could be located directly from the 2D-XPB electron density maps.

This approach appears to offer a remarkably simple and powerful method for solving the structures of complex polycrystalline materials.

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Textural evolution of zinc alloys deformed by NECAE method

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One of the main methods for increasing mechanical strength is grain refinement. Ultrafine grains can be obtained by means of several methods of Severe Plastic Deformation (SPD) [1], being the most common Accumulative Roll Bonding (ARB), Torsion Straining (TS), Equal-Channel Angular Pressing (ECAP) y Milling Mechanical (MM) [2], [3]. On the current work we study another method of deformation, NECAE [4] (Non Equal-Channel Angular Pressing), by processing sheets of Zn alloys pre-rolled until 0.65 mm thickness and cut into the directions of 0°, 45° and 90° with respect to the original Rolling Direction. 21 plates of 14 mm width and 45 mm length were stacked in the channel matrix, with the plane perpendicular to the transversal direction, and deformed with a load of 5000 kg in a single pass of deformation. The matrix was configured with external and internal angles of $\Psi = 81^\circ$ and $\Phi = 150^\circ$, respectively. We selected four samples for each rolling direction corresponding to different transverse distances of the channel matrix. The samples were characterized by optical microscopy (OM), x-ray diffraction (DRX) and Electron Back Scattering Diffraction (EBSD) analysis. The purpose of the work was to study the deformation texture, microstructural parameters, such as grain size and crystal orientations and local misorientations. The main purpose of the study is a preliminary assessment of shearing deformations that will be applied to another SPD technique: asymmetric rolling of different metals and alloys.

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Structural characterisation of fluorites and pyrochlores

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Compounds with general formula $A_2B_2O_7$ may adopt the fluorite or pyrochlore structure. This requires 12.5 % anion sites to be vacant (relative to the ideal fluorite, MO_2), due to the different valency of the A and B cations. The formation of the pyrochlore structure over the fluorite structure has been widely reported to be governed by the cation radius ratio, r_A/r_B , with pyrochlore forming in the range 1.46-1.78 [1]. In the fluorite structure, cations and anions are disordered across their respective crystallographic sites, whereas in pyrochlore they are ordered.

$A_2B_2O_7$ compounds with r_A/r_B values close to the fluorite-pyrochlore boundary of 1.46 may crystallise independently in both pyrochlore and fluorite structures. The formation of pyrochlore or fluorite is influenced by the synthetic method (*i.e.* precursor or conventional solid state) and also the thermal treatment of the sample [2,3]. However, due to the closely related structure of fluorite and pyrochlore, it is often difficult to distinguish between polymorphs using average characterisation techniques, such as diffraction, alone. This is especially problematic when precursor routes have been adopted and diffraction patterns show broad peaks, indicating a small crystallite size. This may result in weak pyrochlore superstructure peaks being difficult to detect by laboratory XRD. Therefore, it is important to utilise local structure characterisation techniques for a complete knowledge of the structure adopted.

We report our recent structural characterisation of an $A_2B_2O_7$ compound, using a range of average and local probes, such as powder X-ray Diffraction and neutron diffraction, neutron total scattering, electron diffraction, Raman and NMR spectroscopy.

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Expanded austenites studied by PXRD and EXAFS

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Low temperature nitriding (< 450°C) or carburising (<520°C) of stainless steel brings about a transformation of the surface into so-called *expanded austenite* by incorporation of nitrogen and/or carbon in the FCC octahedral interstices. The result is greatly improved wear and fatigue properties without compromising the corrosion resistance. Obviously, this favourable combination of wear, fatigue and corrosion performance is technologically highly relevant as it opens up for new applications of stainless steels, but also scientifically the colossal solubility of interstitials (up to 60 % occupancy) at low temperatures is indeed intriguing. We have recently characterized a series of nitrided and carburized steels by combining powder diffraction (for phase

identification) and EXAFS (to find the local environment for Cr, Fe and Ni) [1,2,3,4]. The combination of powder diffraction and EXAFS makes it possible to find the preferences for Cr, Fe and Ni, respectively, which is crucial for the understanding of these materials.

Expanded austenite does not represent a thermodynamically stable state and will decompose into nitrides or carbides provided that time and/or temperature allow diffusion of the substitutional elements, viz. Cr. This meta-stable state is established due to a strong affinity between nitrogen – and to a lesser extent carbon atoms – and chromium, which explains that large quantities of interstitials (N and C) can be dissolved. We will present the results from the as prepared nitrogen and carbon expanded austenites as well as from nitrides and carbides formed during controlled decomposition.

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Crystal structure and non-linear properties of LaEr(MoO₄)₃

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Sm-Ho molybdates show the usual β'-Gd₂(MoO₄)₃ ferroelectric-ferroelastic structure [1]. In particular Gd₂(MoO₄)₃ has attracted attention since it has shown to be an efficient frequency doubling medium for laser diode pumping [2]. Also, it is of great interest for the synthesis of crystallized glasses consisting of this type of ferroelectric materials [3]. Recently a new interesting compound of this family: LaEr(MoO₄)₃ has been investigated by the processes of energy transfer NIR to visible (upconversion) [4]. LaEr(MoO₄)₃ has been prepared by the conventional solid-state synthesis, and X-ray powder diffraction has been collected using a PANalytical X'Pert Pro diffractometer at room temperature. LaEr(MoO₄)₃ features a distorted β'-Gd₂(MoO₄)₃ structure under normal conditions. In this work we have applied a new alternative way of treating distorted structures using a symmetry modes analysis [5] which gives us an input file to perform with FullProf [6] the Rietveld refinement of the amplitudes of such modes, instead of the atomic positions. This new procedure will let us discuss the distorted ferroelectric-ferroelastic structure in comparison with other member of this family of materials [7]. The distorted structure has thus been decomposed in three adapted symmetry-adapted modes from the paraelectric phase. Furthermore we have performed a theoretical analysis of these modes. Also of interest is the high luminescent efficiency under the IR excitation that La₂(MoO₄)₃ phosphors co-doped with Er³⁺-Yb³⁺ feature [8]. We have thus analyzed the factors affecting the up-conversion fluorescent intensity by comparing different concentrations of Er³⁺ in matrices of La_xEr_{2-x}(MoO₄)₃ with different crystal structures: 1) β'-Gd₂(MoO₄)₃ structure-

type and 2) modulate scheelites. A two-photon excitation mechanism was proposed to explain the observed experimental results. Change of Er³⁺ symmetry, phase structure and/or concentration enhanced the upconversion emissions.

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Characterization of MOFs by single crystal and powder X-Ray diffraction

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Metal Organic Framework (MOF) structures are well known for their many interesting applications, such as gas storage and separation, chemical sensing and catalysis [1]. The same structural properties that allow for such excellent chemical properties [2] cause difficulty in characterization by crystallographic methods. Owing to the highly porous nature of MOFs, crystalline samples tend to lose solvent rapidly, degrading the quality of diffraction. Additionally, a high degree of disorder over long ranges frustrates single crystal structure determination efforts. In order to characterize MOF samples, our lab has investigated the complementary techniques of single crystal and powder diffraction. Series of MOF complexes with similar framework structures but subtle chemical substitutions have been studied by single crystal X-ray diffraction and compared to powder X-ray diffraction patterns obtained using a single-crystal X-ray diffractometer equipped with a CuKα microsource. Powder samples have been run in capillaries with and without solution, and in nylon cryoloops in order to determine the most advantageous scenarios for data collection. Powder XRD patterns are then compared to single-crystal datasets (both observed and calculated intensities) to judge not only the composition of the bulk, but the diffraction produced (and often subsequently removed) by disordered solvents.

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