model $M_{n+1}AX_n$ *Phase* systems at a <100ms time resolution. In turn, this technique has been further refined and applied in the confirmation of a novel solid state $M_{n+1}AX_n$ *Phase* precursor design. The ability to simultaneously explore the *in situ* chemical and thermal environments of large volume samples has provided us with a means of rapidly prototyping novel synthesis techniques. More generally, time-resolved *in-situ* neutron diffraction has the potential to redefine many research techniques in both materials science and solids state physics if two experimental methodologies can be perfected; (i) concurrent experimentation and (ii) complementary analysis. More specifically, we should aim to couple *in situ* scattering with the simultaneous analysis of chemical, thermal, physical or environmental factors, while analysis using complementary techniques (e.g. neutrons & X-rays) will ideally produce higher scientific standards in characterisation. Together, these methodologies significantly reduce the development time and complexity of novel materials syntheses, while ultimately lowering associated costs. The key to achieving these goals is the design and implementation of robust *in situ* sample environments capable of exploring a wide range of simulated service environments.

Keywords: in situ, diffraction, QPA

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Nonlinearity in residual stress measurements using X-ray powder diffraction

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Residual stress has often a strong influence on material properties, such as fatigue and stress corrosion resistance. X-ray powder diffraction can be used to measure the residual stresses in polycrystalline materials. Normally, the classic d-$sin^2\psi$ method is used to derive the stress from diffraction data [1]. However, it often happens that the d-$sin^2\psi$ data shows nonlinearity, either as oscillation (Fig. 1) or curvature (Fig. 2) for industrial materials (e.g. ferritic steel). The nonlinearity has been attributed to elastic anisotropy due to strong texture (Fig. 1) and stress gradients in the sample normal direction (Fig. 2), respectively [2]. Despite texture, d *vs.* $sin^2\psi$ linear relationships are still found for biaxial stress states for cubic materials in the case where elastic models apply, when {*h*00} or {*hhh*} planes are used for the stress measurement. The d-$sin^2\psi$ function is principally linear as long as only influences of texture in connection with residual stresses are present [2]. In the case of curvature due to a strong stress gradient, grazing incidence X-ray diffraction can be applied in order to reduce the penetration depth, therefore, $\Delta z \rightarrow 0$. Thus, the stress gradient would be zero. A linear d-$sin^2\psi$ plot is shown in Fig. 3 with 10° grazing incidence angle for the ferrite (200) reflection. However, in this case the measured stress is only the very surface stress. In order to obtain the stress gradient a series of measurements with varied grazing incidence angles should be carried out and the residual stress as function of sample depth could be obtained as shown in Fig. 4. In this way the nonlinearity in residual stress measurement using X-ray powder diffraction can be avoided.

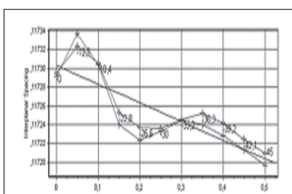

Fig. 1a. Oscillation d-$sin^2\psi$ plot using ferrite (211) reflection due to texture.

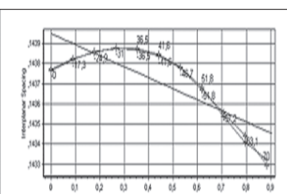

Fig. 2a. Curvature d-$sin^2\psi$ plot due to stress gradients in the sample normal direction.

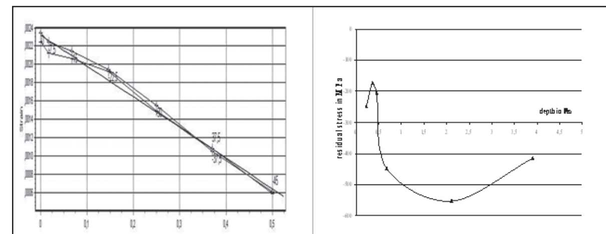

Fig. 3. d-$sin^2\psi$ plot with 10° incidence for the ferrite (200) reflection.

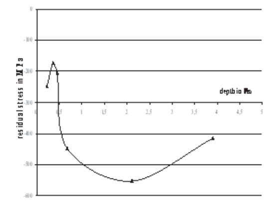

Fig. 4. Residual stress as a function of incidence angle (penetration depth) for the (200) ferritc reflection.

[1] B.D. Cullity, S.R. Stock, *Elements of X-ray Diffraction,* 3rd, edt., Prentice Hall Inc., Upper Saddle River, **2001,** 435-469. [2] H. Dölle, J. *Appl. Cryst.* **1979,** *12,* 489-501.

Keywords: residual stress, X-ray powder diffraction, material sciences

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The development of rapid tomographic energy dispersive diffraction imaging TEDDI

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Energy dispersive diffraction has been shown to be useful in producing three dimensional X-ray images of objects where each voxel within the image contains structural information [1]. This coherently scattered signal can be used to identify the material concerned or to display some feature of the sample such as a chemical change or a stress distribution. A major difficulty with attempting to produce an image with weak diffracted signals is the slowness of the method. This has been partially overcome at lower energies by producing energy sensitive pixilated detectors combined with suitable collimator arrays to eliminate unwanted scatter and to define energy resolution [2].

However the vast majority of samples we wish to image for security scanning, aerospace or chemical engineering applications require much higher energy radiation for sample penetration. This requires high energy X-ray pixilated detectors and associated high performance collimators. We describe in this paper the development of an 80 x 80 pixilated array made from CdZnTe material and its application to the non-destructive study of the internal strain distribution in Ti6246 which is a superalloy widely used in the aerospace industry. We will also present the study of the evolution of an alumina catalyst support as function of processing [3].

We conclude that TEDDI, as one of the growing family of X-ray imaging methods, can deliver signals with suitable signal-to-noise in an acceptable timescale for scale-up to industrial applications.

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