glycol (PEG) concentration on the structural characteristics of Uox uncomplexed and complexed with 8-azaxanthine (AZA) using powder diffraction data collected on ID31 [2] at the ESRF.
Previously unknown phases of Uox were observed depending on the presence and type of salt whereas PEG and pH variation had a minor affect on the cell dimensions. All phases have been successfully indexed, and the known I222 orthorhombic phase of Uox complexed with AZA was solved by molecular replacement using software designed for single crystal diffraction data [3]. The phase diagram of Uox and its relevant crystallizing conditions will be presented.

[1] Margiolaki, I., Wright, J. P. *Acta Cryst.* **2008** A64, 169-180. [2] Fitch, A. N. **2004**. J. Res. Natl Inst. Stand. Technol. 109, 133-142. [3] Collaborative Computational Project, Number 4 **1994**. Acta Cryst. D50, 760-763.

Keywords: protein crystallography; powder diffraction analysis; phase diagrams

FA5-MS01-P14

Use of Rietveld Method in Quantitative Analysis of Weldments in Duplex Stainless Steels. Jorge L. Garin[a], Rodolfo L. Mannheim[a], Manuel A. Camus[a]. [a]*Department of Metallurgical Engineering, Universidad de Santiago de Chile, Santiago, Chile.*
E-mail: jorge.garin@usach.cl

Duplex stainless steels (DSS) are an important class of engineering materials, currently been considered for welding applications among many other industrial requirements. They have approximately equal proportions of the body-centered cubic ferrite and face-centered cubic austenite phases in their microstructure. The main advantage of DSS over conventional stainless steels are strength, chloride stress-corrosion cracking resistance and pitting corrosion resistance. Although the weldability of DSS is generally good, the high alloy content and the existence of a ferritic matrix render SDD susceptible to embritlement and loss of mechanical properties due to precipitation of sigma-phase in the microstructure. This phase is a complex intermetallic compound of Fe and Cr, based upon an ideal stoichiometric composition AX_2, Pearson's code tP30 and space group P_2/mnm. Owing to the usually complex diffraction pattern of these alloys, which disclose many overlapping reflections and strong preferred orientations caused by the welding process, the Rietveld method was used to resolve those difficulties in welded joints of commercial duplex stainless steels (21-23 Cr - 4.5-6.5 Ni). The Rietveld refinements were performed based upon typical measurement and global parameters. The powder diffraction patterns of the weldments resulted in strong preferred orientation effects due to the uniaxial solidification of the weld metal-pool, which was corrected in the Rietveld refinement by using the March-Dollase function. The pseudo-Voigt function was used for the simulation of the peak shapes, while the background was modeled by a 3rd order polynomial in 2θ with refinable coefficients. A total of three phases, namely ferrite (Cr,Ni), austenite (Ni,Cr) and sigma phase (Fe_7Cr_6) were identified and considered in the quantitative analysis. The results obtained have assessed the application of the Rietveld method to quantify the microstructural components of weldments in duplex stainless steels. The main advantage of this methodology was the use of the March-Dollase model for correcting the strong texture effects on the diffraction pattern, which yielded the lower R-values and much better represented the relative amount of phases in the samples.

Keywords: duplex stainless steel; rietveld; sigma phase

FA5-MS01-P15

Laboratory X-ray Microdiffraction - Limits and Applications in Forensic Science. Ivana Jebavá[a], Viktor Goliáš[a], Marek Kotrlý[b]. [a]*Institute of Geochemistry, Mineralogy and Mineral Resources, Charles University in Prague.* [b]*Institute of Criminalistics Prague.*
E-mail: Iva.Jebava@seznam.cz

The aim of this work was to evaluate and prove abilities and limits of laboratory x-ray powder microdiffraction and to create the methodical procedure for applying of this technique in forensic science.
X-ray powder microdiffraction is a progressive non-destructive analytical method that allows analysing very small area on a sample. X-ray beam was in our case focused by monocapillary with an exit diameter 100 or 800 μm. Monocapillary is a hollow glass tube in which there is a total reflection of X-ray beam.
Several problems were investigated. Microdiffraction technique was compared with standard powder diffraction method in Bragg-Brentano instrumentation on identical small-volume samples and capabilities of identification of more phases in mixture were verified. Results showed that microdiffraction reflections are 2-3 times broader than the Bragg-Brentano ones (FWHM 0.13 vs. 0.27° 2Θ for (012) line of corrundum). Peak shape can be fitted by conventional profile functions approach only with difficulties. However, in case of analysing of small-volume samples, microdiffraction technique usually identify more phases.
The next task was to determine limit of microdiffraction with respect to grain size in a sample. This was carried out on commercially produced alumina of several different granularities and results were compared with scans from imaging plate. Studied material contains pure alumina (α-Al_2O_3) and minor concentration of β-Al_2O_3 that was used for evaluation of quality of quantitative analyse from microdiffraction patterns. Upper grain size limit for 100 μm capillary was determined 10-15 μm (static sample) and 25 μm (rotated sample). For 800 μm capillary the upper granularity limit is 50 μm (static sample) and 100 μm (rotated sample).
The determination of detection limit of minority phase in mixture was our next step. We studied the mixture of quartz and fluorite in several concentrations. The detection limit was defined as 0,5 wt. % of fluorite.
The optimized step was defined 0,05º 2Θ for both 100