ESRF	**Experiment title:** ASAXS study of noble metal clusters in silcate glasses and sulphides	**Experiment number**: HS2638
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Report:

Speciation of noble metals (Platinum group elements, Au, Re) in mantle phases is of considerable fundamental and applied interest. It was shown that in some experiments on noble metals solubility in silicates and sulphides submicroscopic clusters are formed and the PGE are mostly confined to these clusters, termed nuggets. The distribution of the PGE in natural rocks and glasses - as true solid solution or as nuggets - is still matter of debates [1]. In theis work we attempted to clarify the issue using Anomalous Small-Angle X-ray Scattering technique at the ID01 beamline. Large number of mineral species, known to contain PGE at variable concentrations were examined. The samples originate mostly from South African ore deposits and thin sections, comprising large mineral grains of olivine, chromite etc. were studied in transmission geometry. Several experimental charges were also investigated.

Small-Angle Scattering patterns are obvioulsy sample-dependent, but qualitatively many of them are dominated by strong halo around the primary beam and several strongs spikes. Due to low PGE content the ASAXS measurements alone do not permit to reliably establish whether the PGE are concentrated in the planar defects (precipitates), giving rise to the spikes or in other crystalline sites. Moreover the composition of the observed precipitates still have to be determined in details and this work is now underway. We have recently acquired high quality EXAFS data on the same samples, are at present the ASAXS and the EXAFS data are used to obtain the information about the mode of the PGE distribution.

Besides investigation of the PGE speciation, we have performed measurements of defects in natural and synthetic diamonds of different origin. First results of an extensive

Small-Angle X-ray Scattering study of diamonds with different types and concentration of nitrogen defects were reported in [2]. It was shown that during annealing of diamonds a new type of extended defects, giving rise to SAXS signal of variable intensity, is produced. These defects span broad size range from 8 to 60 nm and it was speculated that they are due to clusterisation of impurities with vacancies and interstitials. However, details of the formation process and structure of the defects were unclear.

During our beamtime at ID01 we have obtained new important results. For this study not only natural crystals with different defects were investigated, but also well-characterised diamonds were subjected to annealing at widely different pressure-temperature conditions and in variable deformation fields. Use of 2D detectors revealed anisotropic distribution of scattered intensity in some of the annealed crystals. Presumably, this indicates impurity segregation along different crystallographic directions or formation of precipitates with crystallographically well-defined faces. The defects are 60-80 angstroms in size. The defects observed in deformed synthetic diamonds might be related to metal-rich inclusions, presence of which is suggested by infra-red absorption spectra. On-going investigation of X-ray diffuse scattering around different nodes of the reciprocal lattice will shed more light onto nature of the precipitates.

The current work confirms earlier suggestion about importance of annealing conditions in production of the SAS-active defects. For the first time the SAS investigation is performed on heavily deformed and impact diamonds. Detailed consideration of the observed features will be given in the presentation. Existence of planar segregations of nitrogen atoms and carbon interstitials in cube planes in annealed diamonds - so-called platelets - is well-known. Current SAS work clearly shows that the platelets are not the only important extended defect in diamonds. Rich and complex systematic of extended defects deserves extensive investigation.

References

[1] Tredoux, M., et al., (1995). The fractionation of platinum-group elements in magmatic systems, with the suggestion of a novel causal mechanism. *South African Journal of Geology* **98**, 157–167
[2] Shiryaev A.A. et al., Small-angle X-ray scattering in diamonds. *J.Appl.Cryst.* **36** (2003), 420

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