A High-Quality Non-Destructive XRD Technique for Cultural Heritage

G. M. Hansford[a], S. M. R. Turner[a], P. Degryse[b] and A. J. Shortland[c]

[a]University of Leicester, Space Research Centre, Dept. of Physics and Astronomy, University Road, Leicester LE1 7RH, UK.
[b]Celestijnenlaan 200E, Division of Geology, Centre for Archaeological Science, K.U. Leuven, 3001 Heverlee, Belgium.
[c]Centre for Archaeological and Forensic Analysis, DASSR/CDS, Cranfield University, Shrivenham, Swindon SN6 8LA, UK.

Energy-dispersive X-ray diffraction (EDXRD) implemented in a back-reflection geometry is extremely insensitive to sample morphology and positioning even in a high-resolution configuration[1,2]. This technique allows high-quality XRD analysis of samples that have not been prepared in any way and is therefore completely non-destructive. The experimental technique was implemented on beamline B18 at the Diamond Light Source synchrotron in Oxfordshire, UK. The majority of the experiments in this study were performed with pre-characterised geological materials in order to elucidate the characteristics of this novel technique and to develop the analysis methods. Sample *d*-spacings were extracted from the data with a typical accuracy of 2×10^{-4} Å, enabling phase identification and the derivation of precise unit-cell parameters which yield insights into the sample material such as the position within a solid solution series. The data is of sufficient quality to allow the investigation of microstructural properties such as crystallite size and shape, and microstrain. A particular highlight was the identification of a specific polytype of a muscovite in an unprepared mica schist sample, avoiding the time-consuming and difficult preparation steps normally required to make this type of identification in a phyllosilicate-containing sample. The technique was also demonstrated in application to a small number of fossil and archaeological samples, including a Cretaceous shark tooth and a Roman glass mosaic tessera; details of these analyses will be given in the presentation.

Back-reflection EDXRD implemented in a high-resolution configuration shows great potential in the crystallographic analysis of cultural heritage artefacts and other specimens. Scientific research of archaeological objects is usually conducted either for the purposes of provenancing or as an aid to the formulation of effective conservation strategies. The avoidance of the need to extract samples from high-value and rare objects is a highly-significant advantage, applicable in other potential research areas such as palaeontology, and the study of meteorites and planetary materials brought to Earth by sample-return missions.

References

1. G. M. Hansford, "Back-Reflection Energy-Dispersive X-Ray Diffraction: A Novel Diffraction Technique with Almost Complete Insensitivity to Sample Morphology", *J. Appl. Cryst.*, **44**, 514-525 (2011).
2. G. M. Hansford, S. M. R. Turner, P. Degryse and A. J. Shortland, "High-resolution X-ray diffraction with no sample preparation", manuscript submitted to *Acta Cryst. A: Foundations and Advances* (2017).