s5.m17.o4 Archeometric Study of Dutch Tin Spoons from Amsterdam: 1350 -1750 AD. A Neutron Scattering Study. D.Visser [a], W. Kockelmann [b,c], P. Hallebeek [d], J. Veerkamp [e], W. Krook [e], [a]*NWO-Physics, ISIS Facility, Rutherford Appleton Laboratory, Chilton Didcot OX11 0QX, UK,* [b]*Mineralogisches Institut, Universität Bonn, Poppelsdorfer Schloss, 53115 Bonn, Germany,* [c]*ISIS Facility, Ruther-ford Appleton Laboratory, Chilton Didcot OX110QX, UK,* [d]*Netherlands Institute for Cultural Heritage (ICN), Department of Conservation Science, Gabriël Metsustraa t8, 1071 EA Amsterdam, The Netherlands,* [e]*Archaeology Amsterdam, Noordermarkt 45, 1015 NA Amsterdam, The Netherlands. E-mail: d.visser@rl.ac.uk*

Keywords: Neutron Diffraction; Quantitative Phase Analysis; Texture Analysis; Archaeometry; Tomography

The Department of Archaeology from Amsterdam excavated over time more than 600 complete tin spoons and numerous incomplete parts within its town boundaries. The earliest types of these spoons date back to ca 1350 AD, while the latest are from ca 1750 AD. A large number of these spoons were investigated by X-ray fluorescence spectroscopy in order to determine their metal content. For this purpose the patina had to be removed over an area of 5 x 5 mm. The patina itself was investigated by X-ray diffraction. The objects were archaeologically dated, facilitated by hammer or rose marks on some of them. A large variation in Pb content has been found in these objects. Early spoons (1350-1450) may contain up to 40-50% Pb. Due to the health hazards of Pb, the production of tin spoons was regulated and overseen by the Guild after 1530. Only a low Pb content (~5-10 %) was allowed from then on. The X-ray data seem to suggest that this control was more or less lost after 1600.

In this paper we present first results on a time-of-flight neutron diffraction study on spoon fragments and complete tin spoons from different time periods, carried out on the ROTAX neutron diffractometer of the ISIS Facility at the Rutherford Appleton Laboratory, UK. Neutron scattering permits to perform non-destructive quantitative analyses of the metal-alloy phases present in the bulk of the spoon parts (scoop, handle), and to look at the microstructure of each of the constituent phases, that include Sn, Pb and Sn-Pb alloys as well as SnO. In addition, texture analyses on some of the objects were carried out in order to obtain information on the orientation distribution of grains and, hence, to shed light on the manufacturing methods.

The diffraction patterns of 20 dated spoons over the period 1400 - 1750 AD were obtained. Large variations in Pb content were found for different objects whilst there is little variation of the metal composition of scoop and handle of one and the same spoon. The surface patina is for most cases not visible in the neutron data.

Some spoon handles contain an iron core. Neutron-tomography has been used to visualize the distribution of iron in the spoon handle.

s5.m17.o5 Synchrotron X-ray Analysis and Diffraction Mapping in Art and Archaeology. J. L. Hodeau,[a] M. Anne,[a] I. Bardies,[c] E. Dooryhée,[a] P. Martinetto,[a] S. Rondot,[a] J. Salomon,[b] G.B.M. Vaughan,[d] P. Walter[b], [a]*Lab. de Cristallographie, CNRS-UPR 5031, 25 av. des Martyrs, BP 166, 38042 Grenoble, France,* [b]*Centre de Recherche et de Restauration des Musées de France, CNRS UMR 171, 6, rue des Pyramides, 75041 Paris, France,* [c]*Musées de la Cour d'Or, 2 rue Haut Poirier, 57000 Metz,* [d]*European Synchrotron Radiation Facility, av. J. Horowitz, BP220, 38043 Grenoble, France. E-mail: hodeau@grenoble.cnrs.fr*

Keywords: Microdiffraction; Archaeology; Powder Diffraction

Our contribution is concerned with two main issues: archaeology and art history on the one hand, and structural analysis for materials science on the other hand. We demonstrate that both the surface image of an object and its relevant structural and chemical content can be obtained by scanning a focused, intense synchrotron X-ray beam and by recording the diffraction patterns and the fluorescence emission. Synchrotron diffraction is here presented as a quantitative method and an appropriate non-invasive tool for mapping the phase and microstructure distributions on surfaces or bulk (or buried) specimens. The spatial resolution and the energy of the synchrotron X-ray probe can be adapted to the scale and heterogeneity of the material in the mm-to-micrometer range. This mapping method opens more paths for the non-destructive investigations of materials in a large variety of applications.

By exploiting the unique properties of X-rays synchrotron radiation, we can identify and map all the constituents composing the painting at the surface of an ancient object. Mapping of an archaeological image by X-ray synchrotron diffraction has not been attempted before. Thousands of Mbytes of synchrotron X-ray diffraction images has been analysed, determining the pigments composing the fresco on a fragment of a Roman wall and reconstructing non-destructively the image of a Cupid's face.

The 3600 pixel diffraction phase maps of the individual constituents allow to separate :

– underlying preparation layer of calcite → Ca;
– superficial calcite pigment → Ca;
– haematite → Fe;
– goethite → Fe;
– lead carbonate as transformed → Pb;
– Egyptian Blue → Cu;

The joined elemental and mineral maps mimic the major features of the painting. Different structural phases made of common atomic elements are differentiated. Textures and graininess are measured. This is put in relation with the provenance, the processing of the pigments and the techniques of painting in the past for a deeper interpretation of the artist's work and methods.