

Transmission Kikuchi Diffraction, a powerful imaging technique for nanoscale structural characterisation of cultural heritage materials.

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TKD (Transmission Kikuchi Diffraction), also known as t-EBSD (Transmission Electron Back-Scattered Diffraction) is a structural characterisation technique that has received increasing interest in recent years [1–3]. This technique, based on the indexation of net-like patterns, first mentioned in 1928 [4], enables to identify crystalline phases and to determine their crystallographic orientation. It has since been used to analyse a variety of samples ranging from nano-crystalline materials, strained materials, corrosion of alloys to geological samples [1].

Although TKD was still never applied in the particular field of Cultural Heritage materials, the technique is completely suited to these materials, which often display complex and small crystalline structures. This study is a first demonstration of the possibilities of TKD investigation on historical materials through a study on Chinese glazed ceramics.

Iron-coloured ceramics represent a major part of Chinese ceramics, from the sky-blue or light-green celadons to silver-spotted black glazed Jian ware. This element was probably most creatively used during the Song Dynasty (960-1279), which is often seen as a high-peak in ceramic manufacturing [5]. Brown-to-black glazed ceramics from the Song Dynasty exhibit specific dendritic structures that have been identified as ϵ -Fe₂O₃ [6], a rare metastable ferric oxide first discovered in 1934 [7]. The growth of these dendrites is a key point for the understanding of the manufacturing of these ceramics. Precise micro- and nano-characterisation to identify the morphology as well as the chemical and structural nature of the crystalline phases is thus a necessity for the study of their growth.

This work aims to analyse finely dendritic structures found at the surface of traditional brown-glazed Chinese ceramics. To achieve this, preliminary experiments were implemented using a combination of Raman spectroscopy and XRD to have an overview of the structural characteristics of the sample. TKD cartography has then been implemented on Focused Ion Beam-prepared thin sections of the samples. TKD results were compared with other chemical and structural imaging techniques implemented on the same thin sections with a Scanning Transmission Electron Microscope, such as Energy Dispersive x-ray Spectroscopy (STEM-EDS) and Automated Crystal Orientation Mapping (STEM-ACOM).

The results of the preliminary analyses showed that the crystals were mainly ϵ -Fe₂O₃, with small amount of hematite crystals at the surface. However TKD and STEM-EDS analyses implemented on the thin sections at the nano-scale revealed the dendrites to be much more complex than expected. They were found to contain a mixture of ϵ -Fe₂O₃, hematite (α -Fe₂O₃) and spinel (MgAl₂O₄). Grain oriented maps were extracted from the TKD cartographies and are compared with STEM-ACOM to further understand the microstructure of the dendrites and discuss their growth.

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