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High-pressure phase transition of Fe₂O₃.

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Iron oxide and iron-bearing compounds exist in significant abundance in the Earth's mantle and play an important role in determining the physical properties of the Earth's interior. The investigation of hematite, Fe₂O₃, is useful in providing information that can lead to a better understanding of the group of compounds that crystallize in similar structures. It is generally known that MgSiO₃ perovskite, which is a dominant phase in the lower mantle, can contain a significant Fe₂O₃ component. In addition, Fe₂O₃ hematite (*R-3c*) transforms to a perovskite-type structure (*Pbnm*) at high pressures [1,2]. This high-pressure phase of Fe₂O₃ is an isostructure of MgSiO₃ perovskite. The similarity of the structure is likely to affect the solubility of the Fe₂O₃ component and the dissolved Fe₂O₃ must change the stability of MgSiO₃ perovskite. In this study, structural phase transitions of iron oxide (Fe₂O₃) have been investigated at pressures up to 100 GPa and temperatures above 2500 K. High-pressure X-ray diffraction experiments were performed using a laser-heated diamond anvil cell (LHDAC) high-pressure apparatus. The samples were heated with a TEM01-mode YLF laser or a multi-mode YAG laser to overcome potential kinetic effects on possible phase transitions. The sample temperature was measured using the spectroradiometric method. The samples were probed using an angle-dispersive X-ray diffraction technique at the synchrotron beam lines BL10XU, SPring-8 and BL13A, PF in Japan [3,4]. A phase transition between alpha-Fe₂O₃ (hematite) and perovskite-type Fe₂O₃ was observed at about 30 GPa. At pressures higher than 60 GPa, we also observed the occurrence of a new high-pressure phase of Fe₂O₃[5]. The new phase showed an orthorhombic symmetry (space group: *Cmcm*) that are denser than other known Fe₂O₃ phases and was confirmed to remain stable to 100 GPa. The structure of the new phase was same as that of CaIrO₃. The volume change from perovskite-type to the CaIrO₃-type phase is about 7 %. Because Fe₂O₃ is an analog material of MgSiO₃, it is possible that this new CaIrO₃-type structure, *Cmcm*, is also the post-perovskite structure in MgSiO₃. Therefore, there is a possibility that the seismic anomaly at the base of lower mantle, D'' layer, is attributable to the transformation from the perovskite to the CaIrO₃-type structure in MgSiO₃.

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Neutron Diffraction Analyses of Intact

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Neutron diffraction as an archaeometric technique has a particular strong case if any material sampling such as cutting or coring is out of question for intact and unique archaeological artifacts and museum objects. Being both a phase and structure sensitive technique, information about the phase content, the microstructure, the bulk texture, and internal stresses can be non-destructively obtained. The knowledge of the variation of these material properties over different parts of big objects is important for the understanding of ancient production methods. The determination of the mineral phase abundance in ceramics, for example, may provide information on firing conditions such as firing temperatures. Microstructural analyses of metal objects, such as ancient tools, weapons or coins, may hint to specific manufacturing steps like plastic deformation processes or thermal treatments. A texture analysis provides pole figures that may be regarded as fingerprints of the making history. In case the historic production methods are known, the texture information may help to distinguish genuine from fake objects.

Here we report on two recent archaeometric neutron diffraction studies on the time-of-flight diffractometer ROTAX at ISIS at the Rutherford Appleton Laboratory, UK: quantitative phase analysis of 18th century Böttger stoneware from the State Art Collections Dresden and texture analysis of 16th century silver coins from the Kunsthistorisches Museum Wien. Böttger stoneware represents a unique technological development and is characterised by a particular hardness which makes it suitable for surface polishing. The diffraction analyses aimed at distinguishing Böttger objects from other types of red stoneware from China (Yixing), Holland (Ary de Milde) and Plau in Germany on the basis of the mineral phase content. Another neutron application concerns the texture analysis of silver/copper Taler coins aiming at identifying different 16th century minting techniques in order to distinguish between genuine and fake coins.