s5.m1.p1 Orientation relationships of hematite layers on ceramic substrates investigated by TEM.

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Thin polycrystalline films (thickness ~ 40 – 70 nm) of Fe_2O_3 on single crystalline alumina platelets and on amorphous silica platelets (diameter ~ 20 – 50 µm, thickness ~ 0,2 – 0,5 µm) are investigated by transmission electron microscopy (TEM) and X-ray diffraction (XRD). The surface of the alumina substrate is perpendicular to the c-axis. The influence of the substrate structure on the ongrowing film structure and microstructure is studied.

The films are prepared wet-chemically by precipitation of Fe_2O_3 from aqueous solutions of $FeCl_3$ on the substrates. Preparation parameters such as temperature, pH and concentration of the iron chloride need to be thoroughly adjusted in order to allow for only heterogeneous nucleation on the platelets. After precipitation, samples are dried in a desiccator and calcined at several temperatures.

A suitable preparation technique for TEM cross-sections of the platelets is developed. It consists of sandwiching the platelets between two glass slides in order to get an in-plane-orientation. Further preparation involves sectioning, grinding, polishing and finally ion thinning down to electron transparency.

TEM investigations and XRD measurements show a strong preferred orientation of the hematite grains on the alumina substrates even in the dried state. Many grains grow epitaxially with (001) $Fe_2O_3 \parallel$ (001) Al_2O_3 and <100> $Fe_2O_3 \parallel$ <100> Al_2O_3.

The microstructure of the films on alumina substrate changes from grains with sharp tips (pointing away from the substrate) for the dried state to a smooth film with hardly viewable grains in calcined samples.

Preferred orientation is less pronounced or even completely absent on the SiO_2 substrates. Also the change in microstructure is not as distinct as in the case of the alumina substrate.

Preferred orientation and epitaxy are already known to occur in polycrystalline Fe_2O_3 films precipitated from solution on single crystalline muscovite platelets as a substrate[1,2].

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[1] Hildenbrand, V.D.; Doyle, S.; Fuess, H.; Pfaff, G.; Reynders, G. "Crystallisation of thin anatase coatings on muscovite", Thin Solid Films, (1997), 304: 204 - 211.
[2] Hildenbrand, V.D. "Untersuchungen an dünnen Titandioxid- und Eisenoxidschichten auf verschiedenen Substraten", Doctoral Thesis, TU Darmstadt, 1995.

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s5.m1.p2 Identification of forbidden reflections from microdiffraction, CBED and LACBED patterns.

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Identification of the kinematical forbidden reflections is very important to characterize the space group of a crystal.

This identification is very difficult from conventional Selected Area Electron Diffraction (SAED) due to the dynamical behavior of electron diffraction. As a result, the forbidden reflections nearly always appear on the diffraction patterns (except for in very thin specimens) from multiple diffraction and therefore they cannot be distinguished from the allowed reflections.

Despite this difficulty, this identification can be performed, at a nanoscopic scale, from electron microdiffraction patterns, Convergent Beam Electron Diffraction (CBED) patterns and Large Angle Convergent Beam Electron Diffraction (LACBED) patterns.

On microdiffraction patterns, the forbidden reflections due to glide planes can be identified by observing, on Zone Axis Patterns (ZAP) perpendicular to the glide planes, the shifts between the reflections situated in the Zero Order Laue Zone (ZOLZ) and in the First Order Laue zone (FOLZ). The forbidden reflections due to screw axes can also be identified by tilting the specimen in order to suppress the multiple diffraction paths. A systematic method based on these observations has been proposed to identify, through the Buerger extinction symbol, the possible space groups[1].

On CBED patterns, the forbidden reflections can be identified from observations of the Gjonnes and Moodie lines of dynamic absences observed in the forbidden reflections when a glide plane or a screw axis is respectively parallel or perpendicular to the incident beam. Nevertheless, this method requires a very accurate specimen orientation.

On Dark Field (DF) LACBED patterns, the Gjonnes and Moodie lines are also observed in a much simpler way. They can be separated from the forbidden reflections due to special Wyckoff positions or to special values of the atomic positions in the unit cell. The forbidden reflections can also be identified from Bright Field (BF) LACBED patterns where they are only visible at the intersections with other allowed lines for which multiple diffraction paths to the forbidden reflections are possible.

Examples of these various methods used to identified the forbidden reflections will be given.

[1] J.P. Morniroli and J. Steeds, "Microdiffraction as a tool for crystal structure identification and determination", Ultramicroscopy, (1992), 45, 219-239