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Pyrite (FeS<sub>2</sub>) is one of the most abundant sulphur minerals on earth and plays an important role in many natural processes, e.g. acid mine drainage and heterogeneous catalysis. Its surface adsorption properties to organic molecules in hydrothermal setting were shown by Lindgren et al. [1] and are important in the iron-sulphur world scenario published by Wächtershäuser et al. [2].

Non-specular crystal truncation rods (CTR) of the pyrite surface under ambient conditions, that provide information about the periodic order of the vertical and lateral surface and the water-pyrite interface structure, were collected with grazing incidence X-ray diffraction (GIXRD) at the ESRF, Grenoble, France. The surface/interface structures of the (100) face in dry and aqueous ambient conditions were investigated using integrated and corrected intensities in a least square fit.

Different models for the surface cell dimensions and the site occupation factors (sofs) were tested. The (100) face in dry conditions shows a defect structure resulting from S and Fe vacancies with sofs of 0.1-0.3 for the topmost atom layer, decreasing with increasing depth. The surface cell is bulk structure terminated with only small relaxations in the topmost surface layers.

For the (100) face covered with a water film a pyrite/water interface model was ascertained by refining models with different start parameters: site occupation factors, positions and occupation factors of O-atoms mimicking water molecules. The pyrite surface is also bulk terminated with a defect structure resulting from S- and Fe-vacancies. It shows only small relaxations of <0.1 Å for the bottom and 0.5 Å for topmost surface layers without reconstruction.

In good agreement with results from molecular dynamic [3] and ab initio [4] simulations three layers of H<sub>2</sub>O molecules are adsorbed to the surface. Additionally, water molecules occupy the Fe-vacancies of the topmost Fe-layer and a transitional zone up to 8 Å, above the topmost Fe-layer, between adsorbed and bulk water can be found. The first H<sub>2</sub>O-layer is located 1.9(1) Å above the topmost Fe-layer and is bound to the iron atoms. The second layer interacts with the topmost sulphur layer and is located 3.0(3) Å above the topmost Fe-layer. The third layer interacts with the second H<sub>2</sub>O-layer and the transitional zone and can be found at a distance of 5.4(4) Å to the topmost Fe-layer. There is an electrostatic interaction in x-direction, whereas the interaction in y-direction is more dynamic, because of the surface anisotropy of the (100)-face of pyrite.

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**Keywords:** surface\_X-ray\_diffraction, pyrite\_surface, crystal\_truncation\_rod

## MS21.P12

*Acta Cryst.* (2011) A67, C339

### X-ray characterization of sol-gel TiO<sub>2</sub> thin films

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TiO<sub>2</sub> thin films deposited on a Ti-6Al-4V alloy by means of the sol-

gel dip-coating technique were characterized using X-ray synchrotron radiation. The crystalline structure and thickness of the films were obtained and related to the parameters of the sol-gel process.

The process involves immersing the substrate (Ti-6Al-4V) in a colloidal dispersion (sol), withdrawing the substrate at constant low velocity, drying of the coating and heat treatment. For the dispersion preparations, titanium butoxide, isopropanol, ethyl acetoacetate, HCl and distilled H<sub>2</sub>O were used. Several withdrawing velocities and number of layer were used, these ranged from 1 to 3 cm/min and from 1 to 3, respectively. Two values of titanium butoxide/isopropanol molar ratio were used in the dispersions: 1/20 and 1/10. Dispersions with aging times ranged from 1 to 10 days were used. The samples received a heat treatment for 1 h at a fixed temperature of 500 C to each layer of coating and heated to 10 C/min and cooling inside the furnace. The morphology of the coatings was observed by optical microscopy and SEM. The crystalline structure was determined by X-ray diffraction with a glancing incidence angle of 1° and the thickness was determined by the X-ray reflectometry technique. These experiments were carried out at the D12A-XRD1 beamline of the LNLS (Campinas, Brazil).

The coatings were homogeneous, compact and smooth to the optical microscope, and of different colors according to their thickness. With increase of the withdrawing velocity, the number of layers, the titanium butoxide/isopropanol ratio or the aging time of the dispersions, the thicknesses of the coatings increased, all in a range from 25 nm to 142 nm. Cracks were observed on films with thicknesses thicker than 100 nm. By X-ray diffraction of the monolayer and multilayer coatings was determined the presence of anatase and rutile.

The results show a relation between thickness and structure. In the thinnest films no crystal structure was detected, while for films with intermediate thickness rutile phase was detected, and for the thickest films both rutile and anatase phases were detected. The brookite structure was not detected in any film.

**Keywords:** sol-gel, film, synchrotron

## MS21.P13

*Acta Cryst.* (2011) A67, C339-C340

### Characterization of TiO<sub>2</sub> nano-films obtained by anodic oxidation of Ti-6Al-4V

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Anodic oxidation of Titanium and Titanium alloys produce a TiO<sub>2</sub> coating with better mechanical properties and corrosion resistance than the substrate. In addition, this TiO<sub>2</sub> coating is a film with biocompatible properties [1], [2].

In the present report, we determined and analyzed the effects of different acid electrolytes on the color, thickness, morphology and crystalline structure of the coating. The electrolytes used were sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) 0.1M to 4M and phosphoric acid (H<sub>3</sub>PO<sub>4</sub>) 1M, and the substrates were plates of Ti-6Al-4V alloy; the oxidation voltages used ranged from 10V to 100V.

The thicknesses of the films were determined by X-ray reflectometry with synchrotron radiation (D12A-XRD1 beamline of the Brazilian Synchrotron Light Laboratory, LNLS). The morphology of the films was observed using optical and scanning electron microscopy and the crystalline phases of the oxides formed were determined by the glancing incidence angle X-ray diffraction technique with a glancing angle of 1°.

The oxide films produced different interference colors depending