

X-RAY DIFFRACTION STUDY OF Pt AND Pd ELECTROLYTIC DEPOSITS ON POLYCRYSTALLINE SUPPORTS

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Electrolytic deposits are widely used in electrochemistry and electrocatalysis. Investigation of the real structure of electrolytic deposits is of great importance as they usually possess high defectiveness that could be the reason of unusual electrocatalytic properties. The example of the exhibition of structural effects in electrocatalysis could be the dependence of the activity of Pt and Pd electrolytic deposits on the deposition potential.

High-precision X-ray diffraction studies of Pt and Pd electrolytic deposits on various supports (Pt/Pt, Pd/Pt, Pt/Au) obtained at different deposition potentials were performed. The effect of the deposition potential on the structure of the deposit has been evaluated. It was shown that electrodeposited Pt and Pd have the particle size in the range from eight to twenty five nm. For the Pt electrolytic deposits systematic decrease of the Pt particle size and an increase of the concentration of the grain boundaries with the deposition potential have been found. For Pt/Pt and Pd/Pt the deposition potential influences the texture of the deposit: an increase of the deposition potential leads to the formation of more textured deposit. A decrease of the lattice parameter of nanocrystalline Pt and Pd in comparison with the value for bulk metals has been found to be the common characteristic of the electrolytic deposits. For Pt/Au systematic decrease of the Pt lattice parameter with deposition potential was observed. Thus the deposition potential is really an important variable, which could determine the structure of electrolytic deposits.

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Keywords: NANOPARTICLES, ELECTROLYTIC DEPOSITS, X-RAY POWDER DIFFRACTION

STRUCTURES OF FLUCONAZOLE AND ITS MONOHYDRATE FROM LABORATORY POWDER DIFFRACTION DATA

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Fluconazole [2-(2,4-difluorophenyl)-1,3-bis(1H-1,2,4-triazol-1-yl)-propan-2-ol] is a widely used antifungal drug. Two polymorphs (A and B) were found by Gu and Jiang (1995) (J. Pharm. Sci., 84, 1438-1441) using Raman spectroscopy and X-ray powder diffraction. In addition to these polymorphs we also obtained a monohydrate (Hy) and were able to determine the crystal structures of B and Hy. The 5-50° 2 θ portions (0.02° steps) of the powder patterns were used. Unit-cell dimensions were determined with TREOR90. Both compounds are triclinic (*P*-1, *Z* = 2), with *V* = 688.6(4)/761.7(5) Å³ (for B/Hy). The structures were solved in direct space using Monte Carlo simulated annealing techniques, with 11/14 (for B/Hy) degrees of freedom (5 torsional plus 6/9 translational). The Rietveld refinements, using pseudo Voigt profile functions and March Dollase corrections for preferred orientation, so far gave *R*_p = 0.148/0.137, *R*_{wp} = 0.195/0.107 for 31/33 parameters. Calculations were carried out using the Compass and Reflex Plus modules of the Materials Studio programs package. In B and Hy the fluconazole molecules display very similar conformations, in spite of the different hydrogen bond patterns (in B: O-H...N₄; in Hy: O-H...Ow, and two Ow-H...N₄).

Keywords: ANTIFUNGAL DRUG FLUCONAZOLE POWDER DIFFRACTION

GRAZING INCIDENCE X-RAY STUDY OF ION IMPLANTED Sb NANOCRYSTALS FORMATION

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Metallic and semiconducting nanocrystals embedded in dielectrics materials are attracting considerable interest due to their novel properties such as single electron effects or luminescence. In order to understand such properties, structural characterization of the formation of nanocrystals is required. Such study is generally performed using transmission electron microscopy (TEM) and Rutherford backscattering (RBS). These techniques are complementary and destructive. It would then be interesting to develop the use of a single non-destructive approach that would allow the determination of the presence and dimension of nanocrystals. In order to form nanocrystals, Sb⁺ ions were implanted into the SiO₂ matrix at a fluence of 5*10¹⁵ cm⁻², with the energy of 10 keV. The sample was then annealed at 1000°C for 30 s in dry N₂ ambient. We have used grazing incidence x-ray diffraction and reflectivity to monitor the formation of antimony nanocrystals. The measurements have been performed on the same instrument equipped with a single point detector and a position sensitive detector. The shape of antimony ion distribution is evaluated from the x-ray reflectivity analysis. It is found to be Gaussian with the maximum at the center of the layer, in agreement with Monte-Carlo simulation of implantation. The complementary of the two techniques reveals the formation of Sb nanocrystals after the thermal treatment. The nanocrystals are found to be metallic and their diameters are obtained. These results are also in good agreement with TEM and RBS analysis.

Keywords: NANO CRYSTALS GRAZING INCIDENCE REFLECTIVITY

PHARMACEUTICAL INDUSTRY BEAMLINE AT SPring-8

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The Pharmaceutical Consortium for Protein Structure Analysis (PCProt) was established in April 2001. This consortium is composed of 22 pharmaceutical companies affiliating with the Japan Pharmaceutical Manufacturers Association (JPMA). The exclusive beamline (Pharmaceutical Industry Beamline) for PCProt is now under construction at SPring-8. In Japan, this is the first exclusive beamline which is owned by pharmaceutical enterprises. Construction fee of this beamline is joint investment, so the ownership and right of usage of this beamline is equally shared with each company. Also annual maintenance expenses will be collected equally. Of course, it is possible to do experiments confidentially. By the way, the full-time operation of this beamline is scheduled to be started since this autumn. The specification of the Pharmaceutical Industry Beamline is almost same as that of RIKEN Structural Genomics Beamline I & II. It is very useful for PCProt to ensure the compatibility of the beamline to that of RIKEN. Because the Pharmaceutical Industry Beamline can be easily got technical supports by RIKEN and the Japan Synchrotron Radiation Research Institute (JASRI).

The Pharmaceutical Industry Beamline has the SPring-8 standard transport channel and optics for bending magnet. Energy of monochromatic X-rays is tunable from 5 to 18 keV using a double crystal monochromator. This could be useful for MAD experiments. The X-ray beam is focused on the position of a sample using a bent cylindrical mirror with typical glancing angle of 3.6 mrad.

Keywords: SYNCHROTRON BEAMLINE PROTEIN STRUCTURE ANALYSIS PHARMACEUTICAL CRYSTALLOGRAPHY