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A four-coordinate H atom has been unambiguously located, by single-crystal neutron diffraction for the first time, in the centre of the tetrahedral metal complex $Y_4H_8(Cp')_4(THF)$ [Cp'=$C_5Me_4(SiMe_3)$]. The core of the molecule consists of a tetranuclear cluster with one interstitial, one face-bridging and six edge-bridging hydride ligands. At the present stage of structural refinement, the four individual Y–H distances to the unique interstitial hydride ligand are 2.184(16), 2.189(16), 2.221(13) and 2.168(12)Å. The compound was prepared via the reaction of $YCp'(CH_2SiMe_3)_2(THF)$ with $PhSiH_3$ and gaseous H_2, and an initial x-ray analysis suggested the present geometry. [1]

The existence of 4-coordinate hydrogen now completes the series of high-connectivity hydride ligands located in the interstitial cavities of molecular cluster complexes. We had previously reported the existence of 6-coordinate H in the octahedral cavity of $[HCo_6(CO)_{15}]-$ in 1979, [2] and 5-coordinate H in the square pyramidal cavities of $[H_2Rh_{13}(CO)_{24}]_3-$ in 1997, [3] via single-crystal neutron analyses.

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Keywords: 4-coord-H, neutron-diffraction, interstitial-hydride

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Neutron Cryocrystallography of Proteins

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Neutron diffraction can directly determine the hydrogen isotope positions of a protein and its bound solvent. By collecting the data at cryo-temperatures the quality of the resulting maps can be improved. It has proved possible to cryo-cool very large concanavalin A protein crystals (> 1.5 mm^3) suitable for high resolution neutron and X-ray structure analysis. We can thereby report the neutron crystal structure of the saccharide-free form of concanavalin A to 2.5 Å resolution [1]. This is the first cryo- neutron protein crystal structure ever to be reported and the first 15K to 293K neutron protein crystal structure comparison. Comparison with the 293K neutron structure [2] shows that the bound water molecules are better ordered and have lower average B-factors than those at room temperature. Overall, twice as many bound waters (as D_2O) are identified at 15K than at 293K.

Methodologically, this successful neutron cryo protein structure refinement opens up new categories of neutron protein crystallography, including freeze trapped structures. Other large crystals of proteins have also proved amenable to cryo-cooling and examples of these will be presented too (Blakeley, Meilleur, Myles, Bau *et al* to be published).

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Keywords: cryocrystallography, neutron diffraction, bound solvent structure

MS60 MICROSTRUCTURAL PROPERTIES FROM POWDER DIFFRACTION DATA

Chairpersons: Davor Balzar, Paolo Scardi

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Subgrain Size-Distributions, Dislocation Structures, Stacking- and Twin Faults and Vacancy Concentrations in Crystalline Materials Determined by X-ray Line Profile Analysis

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X-ray diffraction patterns, especially when measured with high precision, are a detailed fingerprint of the microstructure of crystalline materials. Up to date computer assisted numerical methods enable to describe whole diffraction patterns by physically well established profile functions based on specific microstructural models. Size profiles are modelled by assuming log-normal size-distribution of grains of subgrains. Distortion or strain profiles are based on the dislocation model developed by Wilkens. Stacking- and twin faults are incorporated by the method developed by Treacy et al. and are parametrized for the density of intrinsic-, extrinsic and twin faults, respectively. The diffuse background scattering is interpreted in terms of point defects, especially vacancies. It is shown that subgrains can be separated either (i) by tilt- or twist angles produced by geometrically necessary dislocations, or (ii) by dipolar dislocation walls whith no tilt or twist between the adjacent subgrains. Both types of subgrain boundaries break down the coherent scattering of X-rays, thus delineate undistorted crystalline regions in terms of X-ray scattering. Stacking- and twin faults are analysed in terms of splitting of dislocations, and are discussed as a function of grain- or subgrain size. Diffuse background scattering or background scattering, which is usually discarded as a disturbing part of diffraction patterns, is discussed in terms of vacancy concentrations within the grain interior and grain boundary regions, respectively. In plastically deformed copper it is shown that, when the deformation and the diffraction measurements are carried out at temperatures lower than the annealing temperatures of vacancies, relatively large vacancy concentrations, of the order of 10^{-7} - 10^{-6} are accumulating within the grain interior regions. More surprisingly, it is found that, within the grain-boundary or subgrain-boundary regions the vacancy concentration values can reach values corresponding to the melting remperature of copper, i.e. $5x10^{-5}$ - 10^{-4}. The microstructural parameters provided by X-ray line profile analysis will be discussed in specific case studies.

Keywords: microstructure, line profile analysis, subgrain size-distribution dislocations

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Microstructural Studies of Nanocrystalline Materials Using WPPM

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Whole Powder Pattern Modelling (WPPM) is a recently proposed and continuously evolving procedure for the microstructural analysis of materials using X-ray powder diffraction [1-4]. The procedure is based on the analysis of the whole information contained in a powder pattern, without relying on an *a-priori* arbitrarily imposed peak profile function. Through the use of WPPM, microstructural features of nanocrystalline materials such as domain size distribution, quantity of line & plane defects can be obtained non destructively in a matter of minutes. Even if it can be used for the analysis of any sample, WPPM performs at best for nanocrystalline materials.

Ball milling is an easy and cost effective technique for the production of nanocrystalline powders. The deformation energy introduced in the powders during the milling causes both a reduction of the domain size and an increase in the defect content, features that can be easily monitored using WPPM. In this contribution, the application of WPPM for the analysis of nanocrystalline materials produced by ball milling will be shown and features/drawbacks with respect to traditional techniques, discussed

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