3) Providing (e-e)-Patterson vectors and (e-n)-vectors (without (n-n)-vectors as in a normal Patterson function) in correct space group symmetry from 2 wavelengths, both vector sets being added together.

A study of the practicability of these symmetry constraints and their limitations by experimental errors will be presented. It is based on test computations using the program described in the abstract by Konz, Spilker, Schäfer and K. Fischer (Abstract, XIII IUCr Congress, 1984).

Thanks are due to the Deutsche Forschungsgemeinschaft for financial support.

15.4–9 STRUCTURAL STUDIES OF AMORPHOUS MATERIALS USING SYNCHROTRON RADIATION AND ANOMALOUS SCATTERING*. By A. Bienenstock, A. Fischer-Colbrie, J. Kortright, R. Lorentz, K. Ludwig, W. Warburton, L. Wilson, Stanford Synchrotron Radiation Laboratory, Stanford University, SLAC Bin 69, P.O. Box 4349, Stanford, CA, 94305, USA and P. Fuoss, AT&T Bell Laboratories, Holmdel, NJ 07733 USA.

In this talk, applications of synchrotron radiation and anomalous x-ray scattering to the determination of short-range atomic coordinations in polyatomic amorphous materials will be discussed. The advantages and limitations of differential anomalous scattering (DAS) techniques will be reviewed. It will be shown that the DAS technique provides information which is not obtainable in any other way and vastly increases our ability to determine the coordinations of specific elements in amorphous materials, particularly when combined with EXAFS analysis. The degree of success we have achieved in obtaining valid partial distribution functions will be described.

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15.5–1 CRYSTALLIZATION OF METALLIC GLASSES STUDIED BY SYNCHROTRON X-RAY RADIATION. By W.Minor, University of Warsaw, Poland, B. Schönfeld, Hamburger Synchrotronstrahlungslabor, DESY, F.R.G, B.Lebech, Risø National Laboratory, Denmark, B.Buras, University of Copenhagen, Denmark and W.Dmowski, Technical University, Warsaw, Poland.

Metallic glasses containing Fe are soft magnetic materials with potential technological applications. When crystallizing they become brittle and lose their magnetic properties. Therefore studies of the crystallization process in metallic glasses are of both scientific and technological interest. Studies of the crystallization process have been made by us by means of x-ray synchrotron radiation and the energy dispersive method, which enable the recording of a full diffraction pattern in a relatively short time. The amorphous to crystalline transition were investigated in $Fe_xSi_{90}B_{10}$ ($69<x<83$). We used the white spectrum of the synchrotron radiation at DORIS (Hasylab) in the energy range up to 50 keV which for the scattering angle 21 corresponds to 11 $Å^{-1}$. The crystallization was followed either by heating the sample stepwise from 20 °C to 1000 °C or by repeatedly recording the diffraction patterns obtained from a sample while annealing at a fixed temperature close to the crystallization temperature. Several time series of isothermal patterns have been obtained and used to study the kinetics of the crystallization. The crystallization of α-Fe in $Fe_{83}Si_7B_{10}$ at 350 °C is nearly complete after 700 minutes.

15.6–1 SMALL ANGLE SCATTERING ON SINGLE OSTEONS USING SYNCHROTRON RADIATION. By A. Ascenzi*, A. Bigi**, M.H.J. Koch ***, A. Ripamonti**, and N. Roveri**,
* Istituto di Anatomia Patologica, Policlinico Umberto I, Universita' di Roma,Italy. **Istituto Chimico "G.Ciamician", Universita' di Bologna, Italy. *** EMBL Outstation Hamburg, c/o Desy Hamburg.

Small angle X-ray diffraction patterns of single osteons have been recorded using synchrotron radiation at EMBL c/o Desy, Hamburg. The first six meridional reflections corresponding to the collagen axial periodicity have been measured, whereas using X-ray conventional sources the first three reflections could be recorded only for the most ordered samples.

The intensity distribution of the meridional reflections is in agreement with a model in which inorganic blocks at the level of the main band of collagen fibrils are arranged with the same axial periodicty of the collagen structure.

The intensity distribution of the meridional reflections is different from that of the native collagen fibers. However, the appearance of the strong first and third reflections indicates that the projected electron density is a step-function. The falling off of the intensity can be ascribed to the height of the step, representing the inorganic blocks at the level of the main band of collagen fibrils and much greater of any other possible density fluctuation.

The small angle diffraction pattern does not change passing from the initial to the final degree of calcification.

Furthermore the only difference in the diffraction pattern of longitudinal and alternate single osteons is an increased arcing of the reflections clearly due to a higher spread of the inorganic blocks with respect to the osteon axis. Thus the orientation of the inorganic blocks, that is the course of the collagen fibrils in single osteons and in osteonic hemisections, has been deduced from the arcing of the small angle meridional reflections. These results are used for a more detailed description of the structural organisation of collagen fibrils and inorganic particles in osteonic lamellae.

15.6–2 X-RAY DIFFRACTION STUDIES OF VERY SMALL CRYSTALS WITH SYNCHROTRON RADIATION. By Janet E. Hails and Marjorie M. Harding, I.P.I. Chemistry Department, University of Liverpool, Liverpool, U.K.

The high intensity of the synchrotron radiation source should allow the recording of diffraction data for structure determination from crystals substantially smaller than those which can be studied with conventional X-ray sources and equipment. We are using an Arndt-Wonacott oscillation camera and other protein crystallography equipment, set up at SERC Daresbury Laboratory by Dr. J.R. Helliwell, to study crystals including nucleotides and oligosaccharides. Diffraction patterns have already been recorded for a crystal of dimensions 0.03 x 0.03 x 0.05 mm, using radiation of wavelength 1.488 Å; further progress will be reported.

15.6–3 STRUCTURE INVESTIGATION OF A 6 µm CaF_2 CRYSTAL: FIRST EXPERIENCES WITH SYNCHROTRON RADIATION. By R. Bachmann, H. Kohler, Heinz Schulz and H.-P. Weber, Max-Planck-Institut für Festkörperforschung, D-7 Stuttgart F.R.G.

Two sets of Bragg reflections have been collected from a CaF_2 crystal with an average edge length of 6 µm (Fig. 1). Crystal orientation and data collection were carried out with synchrotron radiation at the storage ring DORIS II, HASYLAB, DESY, Hamburg in cooperation with the Institute of Crystallography of the University of Göttingen (Bachmann, Kohler, Schulz, Weber, Kupcik, Wendschuh, Wolf, Wulf, Angew. Chemie 95 (1983) 1013). The scattering power S of this crystal is equal to

$$S = \left(\frac{F_{000}}{V_e}\right)^2 V_c \lambda^3 = 1.3 \cdot 10^{14}$$

V_e V_c: volume of unit cell and crystal, respectively. This crystal scattering power S is the smallest one ever used for an X-ray diffraction experiment. A typical rocking curve is shown in Fig. 2. 131 Bragg intensities were collected, which were averaged to 16 (data set I) respect. 17 (II) unique and observed ($I>3\sigma(I)$) reflections with $\sin\theta/\lambda \leq$ 0.58 (I) resp. 0.78 (II) $Å^{-1}$. In addition data were measured on a 90 µm ø CaF_2 sphere with synchrotron radiation (data set III) and with a conventional MoK_α X-ray tube (IV). Due to technical reasons the experiments with synchrotron radiation had to be carried out at the wave length λ = 0.91 A and with horizontal diffraction geometry. For this configuration the polarization correction is strongly θ dependent. As the polarization K of the incident beam could not be determined experimentally, we carried out structure refinements for several values of K. The temperature factors are very sensitive to the values of K assumed, which, in any case, are only average values. At $R_w(F)$ = 0.005(K=0.93,I), 0.031(K=0.94,II), 0.017 (K=0.5,III), 0.011(K=0,IV) we have B(Ca)=0.38(5)(I+II), 0.64(3)(III), 0.610(6)(IV), B(F)=0.83(4)(I+II), 0.92(3)(III), 0,812(8)(IV). The intensities of 6 µm crystal were almost not affected by extinction, in contrast to the 90 µm sphere, where the strongest reflection had to be increased by a factor of about two.

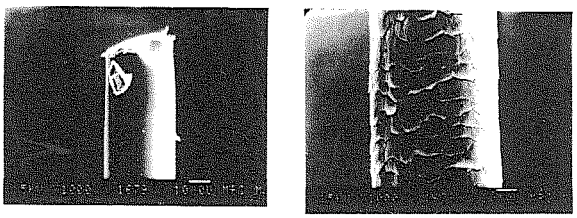

Fig. 1 Comparison of the 6 µm CaF_2 crystal fixed to glass pin in comparison with a human hair. Bar at the lower edge corresponds to 10 µm

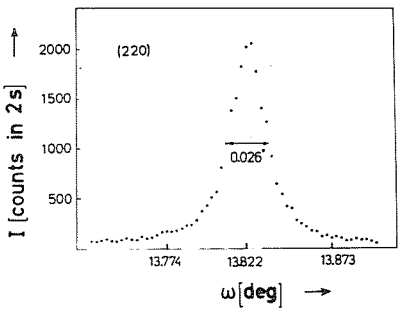

Fig. 2 ω-scan of the (220) reflection