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.

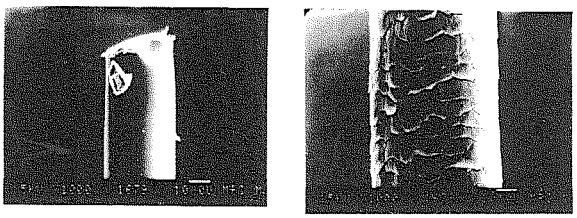

Fig. 1 Comparison of the 6 µm CaF_2 crystal fixed to glass pin in comparison with a human hair. Bar at the lower edge corresponds to 10 µm

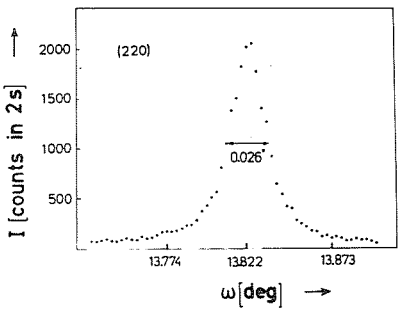

Fig. 2 ω-scan of the (220) reflection