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INSTRUMENTATION FOR MAPPING STRUCTURAL AND TEXTURAL CHANGES IN FIBRES WITH STRESS BY WIDE AND LOW ANGLE DIFFRACTION AT FRASCATI

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ABSTRACT

A versatile camera for low angle and wide angle diffraction of fibrous materials is described. It allows simultaneous recording of both patterns. It incorporates environmental chamber and a miniatured computerized tensometer/dynamometer to study the changes in diffraction patterns with application of stress. Diffraction patterns of stretched and unstretched tissues are presented to discuss the changes in crimped tissues on application of tensile stress. The camera and associated instrumentation has been used with the drift chamber detector at Frascati.

1. Introduction

Functional -mechanical- performance of many fibrous materials depend on their textural morphologies as well as their crystal structure or quasi crystalline structure. It is therefore important to study changes in textures and structures with application of stress within them. For this reason it is desirable to note how low angle diffraction/small angle scattering change at different degrees of stress. High intensity of synchrotron radiation allows one to do this in a dynamic or quasi-dynamic fashion. However in practice it is often difficult to perform these experiments for three reasons.

(1) Small angle scattering facility on synchrotron sources traditionally use a line focus to get a well defined narrow beam in one direction (usually along the meridional direction) to give a good resolution in that direction. As a consequence of this the resolution is worse in the equatorial direction. This essentially means that fine textural differences cannot be studied as the azimuthal resolution in diffraction data is very poor. This is exemplified by the diffraction pattern of a crimped collagen fibre taken at the SRS Daresbury (Fig.1a). Fig. 1b shows the diffraction pattern of the same fibre (human palmaris longus tendon) taken by Nicholls et al (1984) [1] with pinhole collimation. Comparison Fig. 1a and 1b clearly demonstrates that the presence of more than one meridional directions corresponding to the crimping (see discussion) in the fibre cannot be seen by using a line focus collimation system.

(2) Many materials, particularly biological materials change their mechanical properties due to loss of moisture while other materials require special environment to perform mechanically in a desirable way. Therefore they cannot be put in a vacuum diffraction camera and it is necessary to provide a special specimen environment while the diffraction pattern is being collected.

(3) Fibrous materials with interesting textures often show stress relaxation. It is therefore necessary to keep stress constant within the specimen during the collection of diffraction data.

We have developed a camera with pinhole collimation for collecting two dimensional diffraction and scattering data with a drift chamber area detector [2,3]. It can be

used for specimens which are required to be kept in a specific environment. There is a provision of a miniature computerized dynamometer/tensometer which can keep the specimen at a known constant degree of stress to overcome stress relaxation.

The camera and the associated instrumentation for stress/strain control can also be used for recording diffraction pattern with a photographic film using a low intensity "sealed tube" x-ray generator where long exposure times are required. This is because a specimen chamber is provided to maintain a specimen in appropriate environment, continuously while the camera is under vacuum or has helium atmosphere. Thus for example a fibre of biological origin can be maintained in 100% humidity in an environmental chamber while its diffraction pattern is being recorded. The environmental specimen chamber is suitable for any type of material because it is completely isolated from the camera. A computerized miniature dynamometer can be inserted in the camera to counteract stress relaxation in the specimen and to keep it at a known constant stress during data collection. It is possible to obtain data through a reasonable range of stresses.

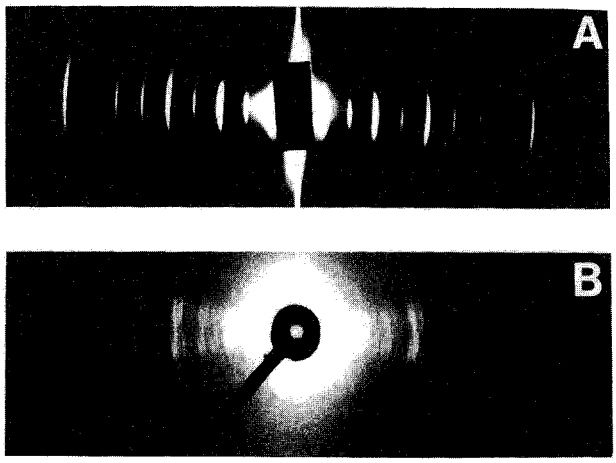

Fig. 1: Diffraction pattern of a human palmaris longus tendon: (a) taken at the SRS Daresbury without pinhole collimation (b) taken with a rotating anode X-ray generator and with pinhole collimation.

2. Apparatus

The configuration of the apparatus and its mode of control is shown schematically in fig. 2. Briefly, the stress signal is derived from a pair of high sensitivity semiconductor transducers mounted on an elastic spring which deforms linearly, in proportion to the stress experienced by the specimen. Electrically and physically the transducers are configured in such a way that there is no voltage due to temperature differences between the transducers is present in the "error signal" which controls th tensometer stepping motor (see below). It mainly consists of the voltage generated due to the actual deformation of both the transducers.

The specimen itself is mounted on to the specimen carrier, containing the instrumented spring, and coupled to a shaft of a stepping motor. Thus the specimen is stretched by a motion of the stepping motor in an appropriate direction. The initial position at the start of the experiment and the current position of the stepping motor enables one to calculate the strain in the specimen continuously at all the time during the diffraction data acquisition. The signal from the deforming transducers, which are arranged in a "half bridge" configuration, is used as a feed back signal to control the movement of the stepper motor through an interface card resident on the "Nubus" in the Macintosh computer and incorporating a medium speed A to D converter and sixteen digital input output facilities.

The camera (Fig. 3) can be used to obtain both low angle and wide angle data simultaneously. For recording a wide angle pattern a specially designed a film cassette is provided. It has a hole in its center to pass the radiation with in a cone of sufficient semi

angle so that low angle diffracted intensities can be recorded on another film cassette placed beyond the wide angle film cassette or on to an area detector through a thin mylar window at the back of the camera.

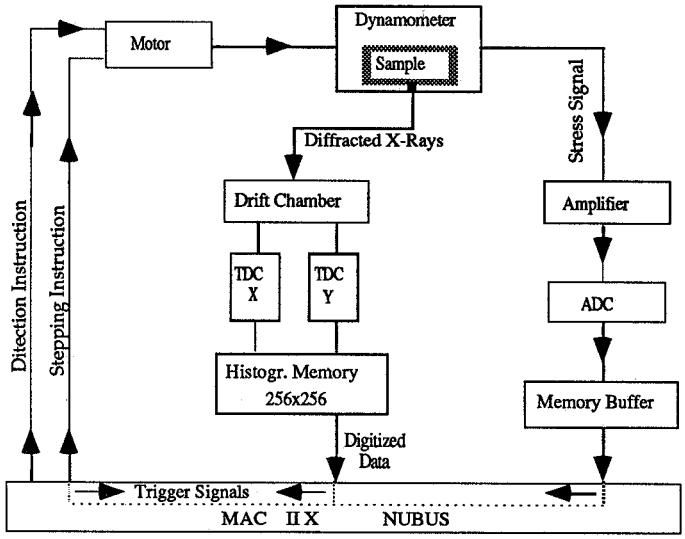

Fig. 2 : Schematic diagram of the control system for the dynamometer and the data acquisition system.

The collimation system for the camera is flexible in that it can be very easily dismounted from the camera body. Any combination of collimation and guard aperture sizes can be easily assembled and mounted on the camera within a matter of minutes.

We have fabricated a number of such assemblies for rotating anode generators, sealed tube generators and synchrotron radiation sources so that the intensities of desired diffraction pattern is maximum on the detecting plane and the pattern can be recorded in shortest possible time whatever the source of X-rays. The usefulness of the system is confirmed by the results obtained for collagenous fibres from uncalcified and calcified tissues of turkey tendons both using the drift chamber area detector at Frascati and using a sealed tube x-ray generator.

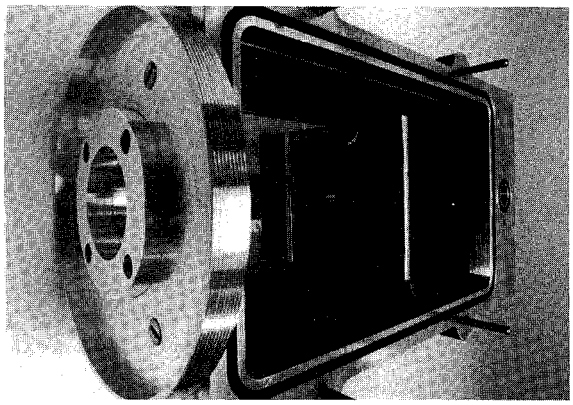

Fig. 3: Camera body showing wide angle cassette and exit window for collecting low angle diffraction pattern outside the camera body

3. Results and discussions

Frascati drift chamber area detector produces an intensity map within 256 x 128 pixels corresponding to 17mmx20mm. Fig. 4a shows a black and white photograph of the pseudo colour map of the intensities of diffracted X-rays as detected by the drift chamber detector from an uncalcified tendon from a 20 week old turkey in its natural relaxed state. It is clear therein that the peak intensities corresponding to the reflections is not confined within a single pixel but is spread in a fashion which clearly suggests that the reflections are not

confined to one single meridian. Thus the two dimensional map of the diffracted intensities map is indicative of multiple reflections corresponding to more than one meridional directions present in the specimen. In this respect Fig. 1b is very similar to Fig. 4a even though the tissues they represent are from two very different animal species.

The multiplicity of the meridional directions is due to the crimp morphology of the fibre [1]. Crimping comprises a predominant arrangement of the fibrils in the tissues whereby the axes of the fibrils undulate in a well known zig-zag pattern. It is due to this that in the low angle diffraction pattern one should find reflections corresponding to at least two different directions. Fig. 4a also clearly demonstrates that the azimuthal resolution of the drift chamber detector is adequate to detect crimp angle of 10 degrees or less. The actual sensitivity of the detector for the specimen to film distance of 500 mm is estimated to be $\Delta d/d = 1.47 \times 10^{-2}$. Fig. 4b shows the diffraction pattern of a stretched uncalcified fibre from a 20 week old turkey tendon. It is evident therein that there are no multiplicities of meridional directions. This is because the applied tensile stress to the specimen was sufficient to stretch out crimping and consequently all the fibres/fibrils within the specimen are well aligned to give only one meridional direction for the whole specimen.

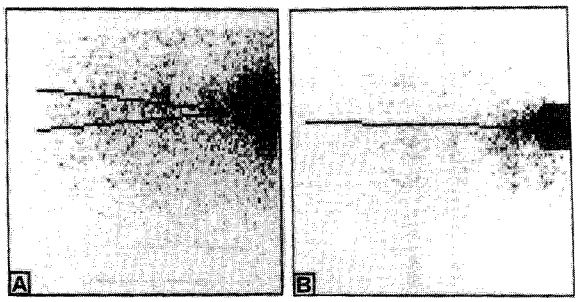

Fig. 4: Low angle diffraction patterns of 20 week old uncalcified turkey tendon. (a) unstretched tendon showing multiplicity of meridional directions corresponding to crimping (zig-zag type undulations) (b) Stretched specimen within which the crimps are pulled out due to the application of a tensile load.

4. Conclusions

It has been demonstrated that the newly developed apparatus is versatile for conducting experiments which will give important new information on the interdependence of structural morphology and functional properties of many different type of materials and particularly of biological fibres. Crimping in collagenous tissues is of course fundamental to the biomechanical studies of connective tissues [4] and for designing soft tissue prostheses [5]. It is intended to use the apparatus for further dynamic studies of mechanical behavior of crimping and other small angle scattering experiments.

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References

[1] S. P. Nicholls et al., Annals Rheum. Dis., 43, (1984), 477.
[2] A. L a Monaca et al.,"Improved Performance of the Drift-Chamber Detector at Adone Wiggler ". These Proc.(1989).
[3] A. La Monaca et al., BIOPHYSICS AND SYNCHROTRON RADIATION. Springer Series in Biophysics vol. 2 p.303. Ed. by A. Bianconi and A. Congiu-Castellano. Springer and Verlag, Berlin 1987.
[4] J. S. Shah et al., Engineering in Med., 8, (1979), 95.
[5] J. S. Shah, "Connective Tissue Prosthesis", U.S. Patent No 462,119 (1987). Also U.K. & European patents.