ИНСТИТУТ ЯДЕРНОЙ ФИЗИКИ СО АН СССР

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EXPERIMENTAL STATION FOR THE STUDY OF THE DYNAMICS OF STRUCTURAL CHANGES USING SYNCHROTRON RADIATION

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НОВОСИБИРСК

EXPERIMENTAL STATION FOR THE STUDY OF THE DYNAMICS OF STRUCTURAL CHANGES USING SYNCHROTRON RADIATION

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A b s t r a c t

The study of the dynamics of processes accompanied by rapid changes in material structure is of great interest for many fields, in particular for physics of polymers, chemistry of solid states, etc. The studies of the structures of metastable states with small lifetimes are currently of special interest. The station (Fig.1) is designed for operation with synchrotron radiation (SR) within the 0.1-3 Å range of wave lengths at a high registration rate, 1000 diffraction curves per second.

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INTRODUCTION

X-ray diffraction station is a technique which can be used to study the dynamic experiments in which the microstructure of crystals is studied as an external parameter is varied. The opening of the station has made possible a wide range of new experiments which was fantastic at X-ray tube technique, because of radiation at the station has a unique combination of properties - high brightness, collimation, polarisation and time structure- as well as its broad wavelength span. This attracts experimental scientists in a wide variety of disciplines: physics, chemistry, biology and material science.

MONOCHROMATOR

The simplest design scheme is realised in the monochromator: a flat crystal mounted on the rorating axis. The axis rotation is produced by two step motors monitored with a computer. The first motor is for a rough adjustment of the monochromator with a scanning step of a crystal turn of 18 angular seconds; the second motor is for fine adjustment of the required wave length with a scanning step of 1 angular second (Fig.2). The monochromatization level is $\Delta\lambda/\lambda = 10^{-2} - 10^{-4}$, depending on the crystal used.

The adjustment to the required λ is performed automatically with a computer by a special program and calibration is made on the element K-edges. The elements are selected in such a way as to have their K-edges in the operational area of the forthcoming experiment. For example, if one needs to operate within the interval 1.5-2 A, the calibration is performed with Cu, Ni, Co, Fe filters introduced into the SR beam. Then, each step on the monochromator angle is normalized by these K-edges, completing the monochromator adjustment to the required λ .

For an appropriate operation with SR one should take into account that during operation, for example, with λ_o , high harmonics are reflected from the monochromator with a plane 220; $\lambda_o/2$ from 440, $\lambda_o/3$ from 660 etc. In order to minimize the influence of higher harmonics we used the following:

1) crystals cut by planes where the multiple harmonics are damped, for example, the plane III of Ge and Si;

2) absorption filters over a narrow area for suppression of harmonics;

3) careful selection of the optimum storage ring energy.

The crystal-monochromator is placed in the vacuum chamber connected with the SR channel. The rotation from the step motors is transmitted to the crystal through the plunger precision axis fixed to the vacuum chamber wall. Such a solution for transmitting the rotation into the vacuum provides the necessary accuracy of crystal rotation and also enables one to operate with vacuum down to 10^{-3}torr, or in the helium medium.

COLLIMATION SYSTEM

The collimator consists of two vacuum slits which (during alignment) can shift in two directions, and also can rotate around the SR beam axis.

Rotation around the beam axis is transferred through a special magnetic vacuum transmission (fig.3). The slit bending angle can be varied with an accuracy of up to 20 angular seconds.

The primary alignment of the slits is performed by a visual control of the beam positioning, by the luminophot applied to the slit. The final alignment of the slit is performed by optimising the detector signal in the beam path.

GONIOMETER

The vertical goniometer RW 1050/81 of Philips (Netherland) production is used in the station. The vertical version is selected for the following reasons:

1) the beam plane after the monochromator is parallel to the goniometer axis, enabling the use of all the beam aperture in a diffraction experiment;

2) the SR beam polarisation is used optimally.

The goniometer is mounted on an adjustment plate (Fig.4) which enables one to make the goniometer axis perpendicular to the SR beam.

DETECTOR

The coordinate sensitive detector developed at the INP, Novosibirsk,

is used in the station /1/.

The detector is a proportional chamber shose cathode performs as a delay line. Such a scheme enables one to detect the coordinate of an incident quantum by the time difference between the pulses from different ends of the delay line.

The detector resolution is 0.1 mm. The input window length is 100 mm. The maximum counting rate of the detector is 250 kHz.

THE ELECTRONICS FOR RAPID DATA COLLECTION FROM THE DETECTOR

Fast action time-to-digital converter (TDC) is used to determine the coordinate of a quantum reaching the detector. The TDC resolution is 400 picoseconds, its maximal counting rate is 500kHz.

The quantum coordinate is recorded in the memory device (MD) in the form of numerical code /3/. In order to increase the rate of the data transfer from TDC to MD, the operation mode through the front panel is used. Recording is performed in the increment regime.

The single MD capacity enables one to store up to 16 X-ray diagrams registered by the detector. Each X-ray diagram has its own individual zone in the MD - a "page". The change of the zone address for recording new X-ray diagrams is performed by a special program recorded in the crate-controller. In such a mode one can "turn pages" in one millisecond, with an interval of 10 microseconds between pages.

As a rule, 16 X-ray pictures recorded in one MD are not enough for the whole picture of the dynamics of the process under study. Therefore, the possibility of operation with a number of MD (up to ten) is envisaged. In this case, all MDs are connected in parallel with the TDC through the front panel.

COMPUTER CONTROL

The automation system /4/ developed at the Novosibirsk INP on the base of the computer ODRA-1325 is used to pick up information stored in terminal devices , and also for the operational control of the whole station. This system comprises both the hardware and the software of the computer-station communication.

Programming is performed with high level languages convenient to use, like BASIC and FORTRAN.

SUPPLEMENTARY EQUIPMENT OF THE STATION

To study the change dynamics of a material structure under various effects on the sample the station is supplied with:

1) a high temperature chamber, up to 1000°C,
2) a high pressure chamber up to 220 kBar,
3) a device for the stretching deformation of metals,
4) a chamber and system of microcollimation (5 x 5 μm) for the study of chemical reactions in a solid states,
5) a chamber for the study of selfpropagating high temperature synthesis,
6) a chamber for the study of phase transformations in polymers,
7) a chamber for the study of electrochemical reacitons,
8) a chamber for the study of metal reduction in the hydrogen medium.

All this auxiliary equipment has been made by various research groups specially for the station taking into account specific SR features.

EXPERIMENTS IN THE STATION

Various technological processes are studied with the aim of optimising their parameters and understanding the physics of different phenomena. This work was started in 1979 and up to eleven groups are now participating in the experiments.

A series of studies of the kinetics of structural changes in polymers during phase transitions Fig.6 and mechanical influences have been carried out by the staff of the Institute of Plastics,Hungary /5/.

The staff of the Institute of Solid State and Processing of Mineral Resources has obtained some interesting results in the study of the process of selfpropagating high temperature synthesis in the combustion regime for the systems:Hf+2B, Ta+B. In these systems the reactionkinetics were studied, and in interaction between Al and Ni the intermediate metastable phses /6/ have been discovered(Fig.7).

The staff of this Institute is successfuly studying the interaction processes of deformed metals with agressive media, and the kinetics of local structural changes in topochemical reactions.

The research group from the Polytechnic Institute, Leningrad, has been able to detect the development kinetics of structural distortions for various metals in the processes of their rapid deformation Fig.8 and also to record the effect of oscillations of the substructure parameters of the crystal lattice /7/.

Using the high pressure chamber the Institute of Geology and Geophysics is studying processes similar to those occuring in deep earth layers (Fig.9).

The Tula Polytechnic Institute is successfuly studying the influence of magnetic fields on the process of iron reduction in hydrogeneous media /8/.

Research groups which started work recently are carrying out studies in the following areas:

1) Crystal structure of metals close to melting point;
2) The decay kinetics of amorphous magnetic alloys;
3) Dynamics of the crystal structure changes during chemo-mechanical reactions;
4) Dynamics of phase transformations in liquid crystal;
5) Kinetics of the de-salting of collagen;
6) Kinetics of the destruction of biological objects under the influence of cobra poison.

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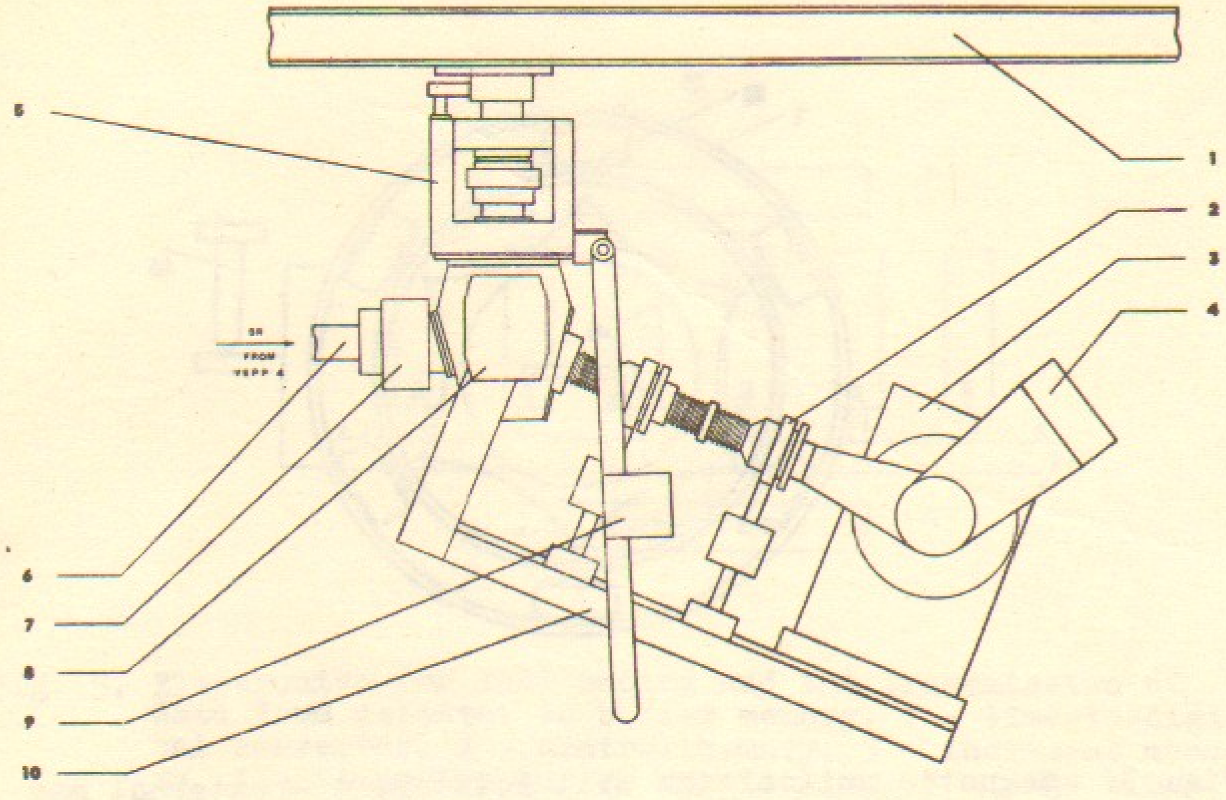

Fig. 1. Structural diagram of the mechanical part of the station: 1 - carrying beam, 2 - magnetic unit for slit rotation in vacuum, 3 - goniometer, 4 - one dimensional position sensitive detector (PSD), 5 - adjustment unit of the station, 6 - SR vacuum channel, 7 - entrance slit unit, 8 - monochromator rotating mechanism, 9 - collimator adjustment motor, 10 - optical bench.

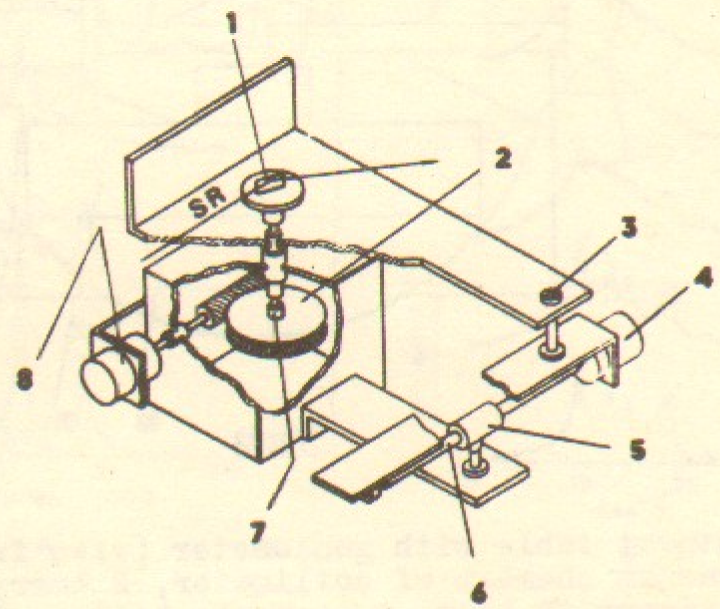

Fig. 2. Mechanism of monochromator rotation. 1 - crystal monochromator, 2 - worm screw, 3 - axis, 4 - fine adjustment motor, 5 - nut, 6 - microscrew, 7 - precision axis, 8 - rough alignment step motor.

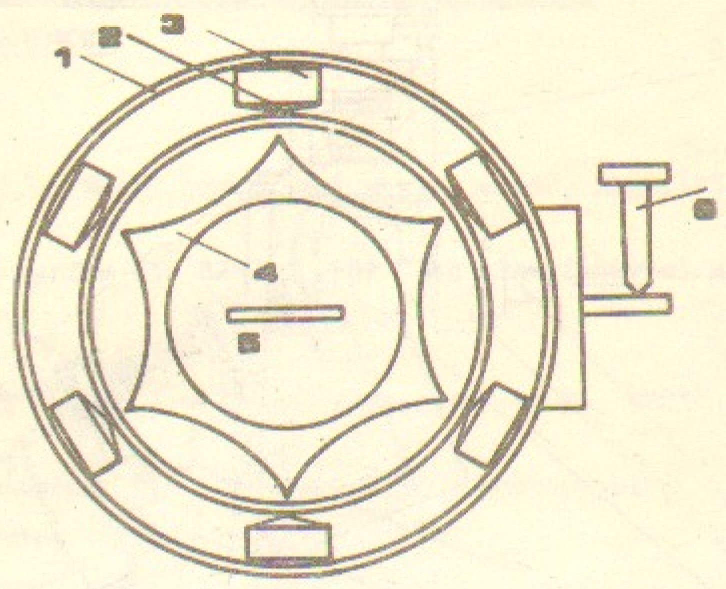

Fig. 3. Magnetic unit of the slit rotator. 1 - external magnetic guide, 2 - magnetic field concentrator, 3 - - magnet, 4 - armature, 5 - removable slit, 6 - microscrew.

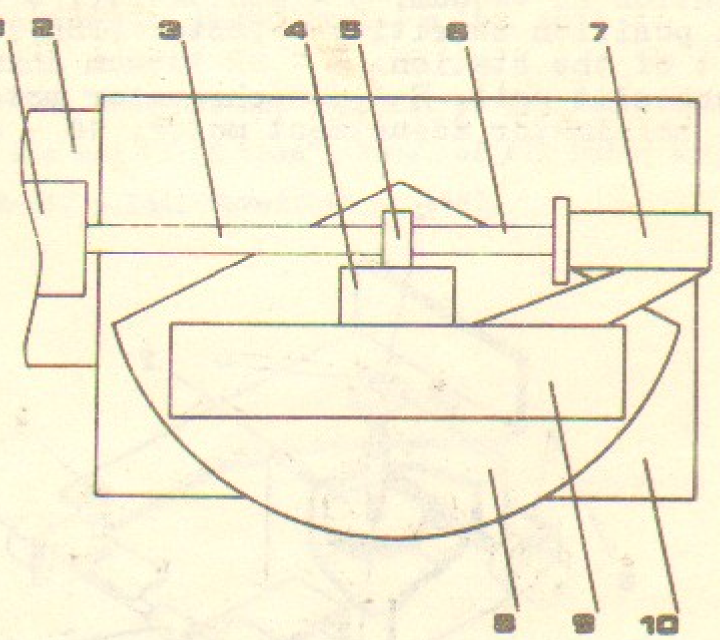

Fig. 4. Adjustment table with goniometer (view from above). 1 - vacuum chamber of collimator, 2 carrying frame, 3 - primary SR beam, 4 - sample holder, 5 - sample, 6 - diffracted beam, 7 - one-coordinate detector, 8 - bending plate of goniometer, 9 - vertical adjustment of goniometer.

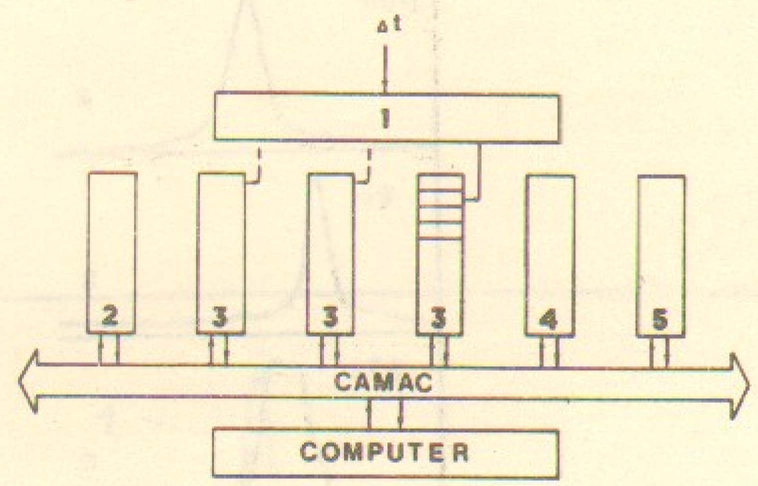

Fig. 5. Electronics for fast coding and the transmission of data from detector to buffer memory. 1 - time-to-digital converter, 2 - control memory, 3 - increment memory, 4 - timer for buffer reswitching, 5 - page timer.

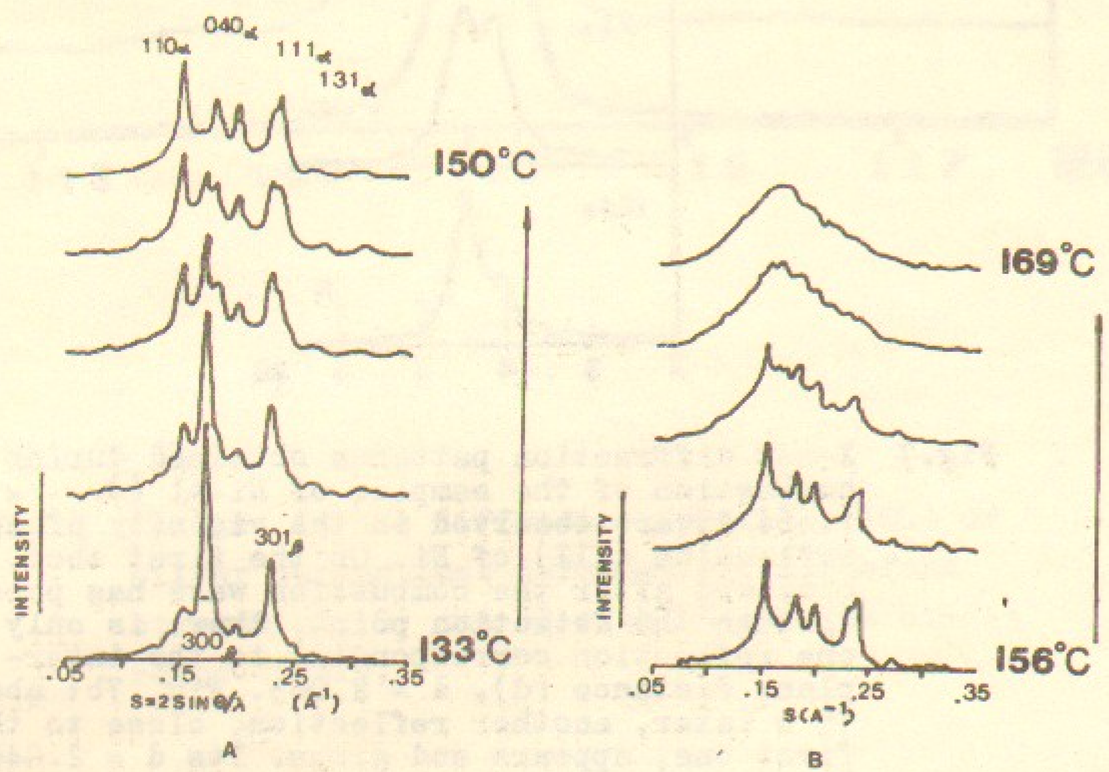

Fig. 6. Changes in the wide-angle X-ray diffraction behaviour during heating the polypropylene: a) β-α transition, b) final melting.

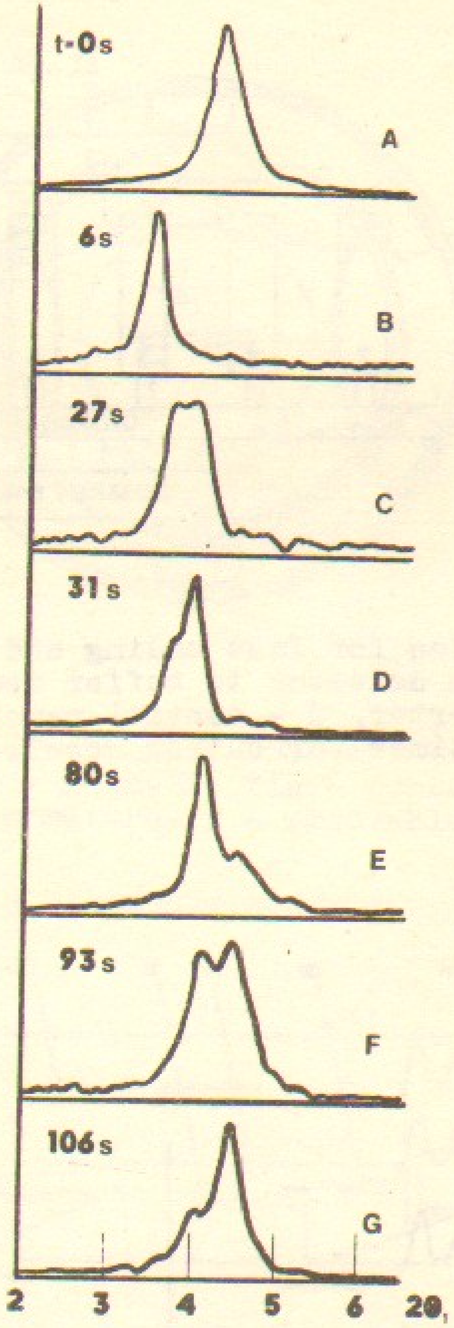

Fig.7 X-ray diffraction patterns obtained during combustion of the samples of Ni+Al (λ = 1.664 Å) are observed in the vicinity of the reflection (III) of Ni. On the first shot, obtained after the combustion wave has passed through the detection point, there is only one reflection corresponding to the inter-plane distance (d), d = 2.065. Fig. 7b: about 15 s later, another reflection, close to the first one, appears and grows. Its d = 2.046 Å. Fig. 7c shows the moment when the intensities for both reflections became equal. The intensity of the first line then decreases until it disappears (Fig. 7e). At t = 70 s the transformation starts (Figs. 7f,g), which leads to the formation of the final product - NiAl (d = 2.022 Å).

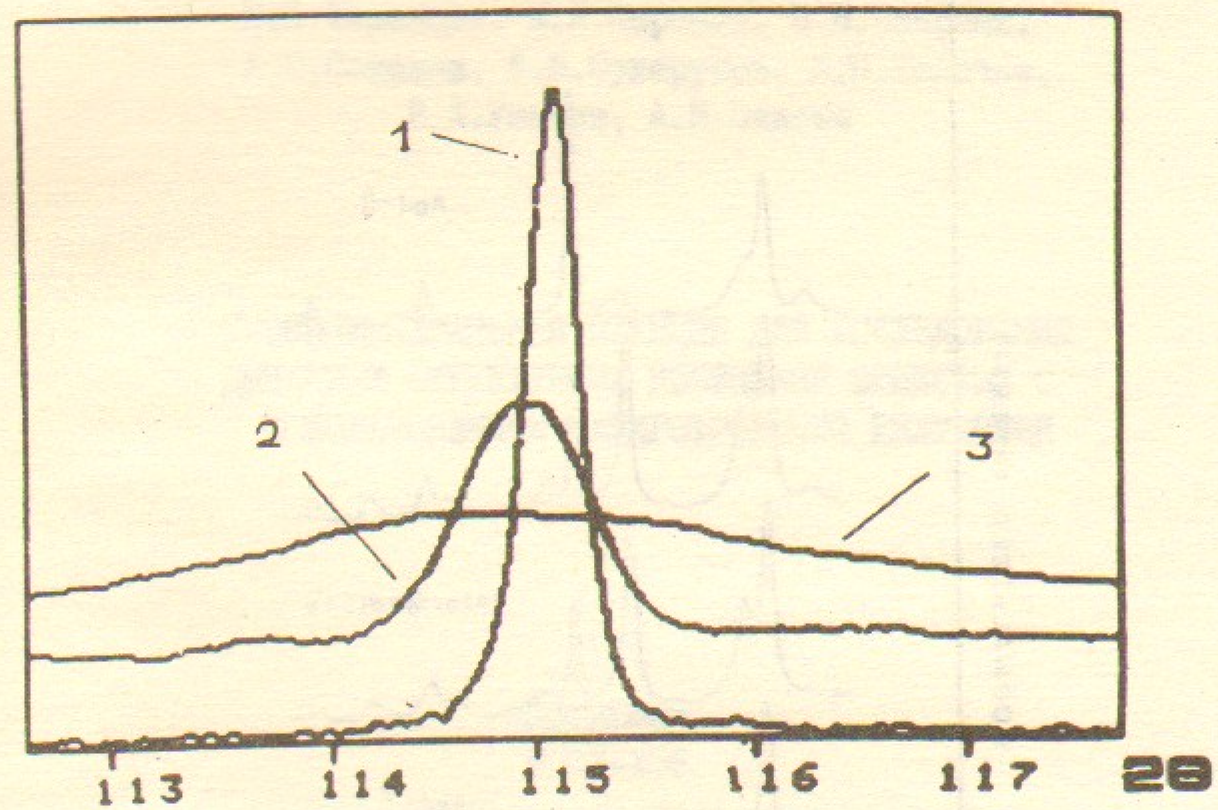

Fig.8 Changes in the diffraction profile (400) of silver during fast deformation: 1) before stretching, 2) and 3) with 5% and 15% strain respectively.

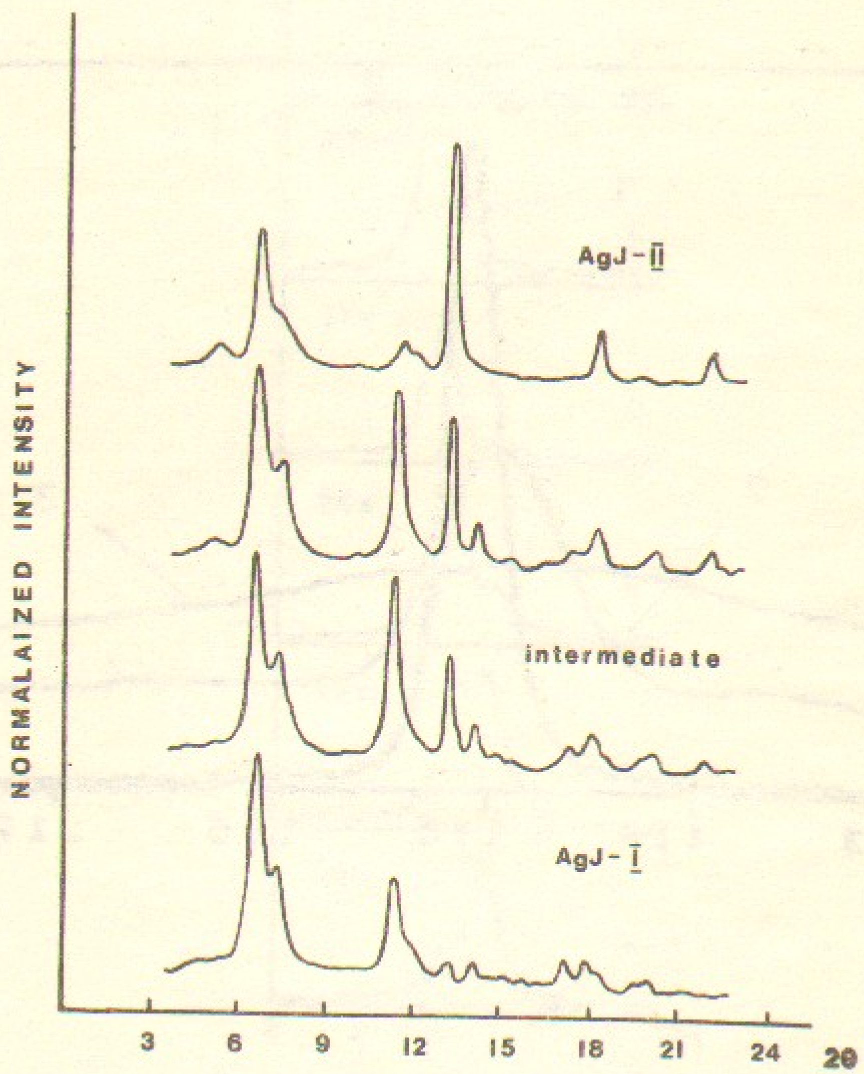

Fig.9 High-pressure induced changes in the wide-angle X-ray diffraction behaviour during the transition from AgJ-II (2.9 kbar) to AgJ-I (2.5 kbar) through the intermediate state

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ЭКСПЕРИМЕНТАЛЬНАЯ СТАНЦИЯ ДЛЯ ИССЛЕДОВАНИЯ ДИНАМИКИ СТРУКТУРНЫХ ИЗМЕНЕНИЙ ВЕЩЕСТВА С ИСПОЛЬЗОВАНИЕМ СИНХРОТРОННОГО ИЗЛУЧЕНИЯ

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