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Apparatus for continuous expansion measurement and uniaxial stress application during neutron diffraction experiments

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Résumé. — Nous décrivons deux appareils qui ont été utilisés pour l'étude, par diffraction des neutrons, de la transition α - β du quartz (573 °C). Le premier est un support prévu pour être placé dans un four à vide standard de l'I.L.L. Il permet la mesure continue et simultanée ($\lesssim 950$ K) de la dilatation thermique du cristal dans le but d'en contrôler les gradients thermiques. Le deuxième est un appareil de compression uniaxiale (jusqu'à 1 kbar sur un échantillon de $7,5 \times 7,5 \times 30$ mm³) qui fonctionne ($\lesssim 900$ K) à l'air ambiant et peut montrer aussi le gradient thermique sur l'échantillon grâce à la dilatation.

Abstract. — Two devices are described which were used for the study of the α - β transition (573 °C) of quartz by neutron diffraction. The first one is a support designed for operation in a standard I.L.L. vacuum furnace. It enables continuous and simultaneous measurement of the thermal expansion to be made ($\lesssim 950$ K) in order to control thermal gradients on the sample. The second is a uniaxial compressive stress apparatus (up to 1 kbar on a $7.5 \times 7.5 \times 30$ mm³ sample) which works in open air ($\lesssim 900$ K) and can also give an estimate of the thermal gradient from dilatation.

1. Introduction.

We describe two devices which were designed for studying the α - β first order transition of quartz (573 °C) by neutron diffraction. For the first apparatus the problem was to identify the nature of a new phase which was found by dilatometric and birefringence measurements [1] in a 1.3 K range around the well-known α - β transition (Fig. 1). The thermal homogeneity of the sample appeared as a crucial parameter which could affect the meaning of the results. So to control this point and in addition to know at each temperature the phase under study, we built a special dilatometric support designed for operation in a standard I.L.L. vacuum furnace. Indeed the dilatation of quartz shows a sharp discontinuity at the transition (Fig. 1) within good temperature conditions. Thus by comparison with this « ideal » curve we can estimate the temperature gradient ⁽¹⁾ over the whole sample and take measures to reduce it. This dilatometric support enabled us to show by means of neutrons

Fig. 1. — Dilatation of quartz during its first order phase transition [1]. The new phase which was to be identified by neutron diffraction is in the A-B region.

the incommensurate nature of the new phase [2] (satellite peaks around Bragg reflections) and also to study the coexistence state by controlling the position of the interface during the transition.

The second apparatus works in open air and has a rougher construction with respect to thermal gradients.

⁽¹⁾ This simple and cheap method seems more reliable for the desired accuracy of a tenth of K than multiplying the number of differential thermocouples which requires very careful « *in situ* » calibration to determine the e.m.f. corresponding to a 0 K temperature difference (see for instance [3, 4]).

It was designed to begin the study of the influence of a uniaxial stress (up to 1 kbar along a $7.5 \times 7.5 \times 30 \text{ mm}^3$ sample) on the incommensurate phase proved before. It includes a lever-press, plunged into a home-built furnace, whose deformation also gives the thermal expansion of the sample and therefore enables the thermal homogeneity in the vicinity of the transition to be estimated.

In both apparatus the sample temperature is measured with better than 0.02 K resolution with a 50Ω platinum sensor coupled to the sample by a metallic foil using an a.c. bridge ⁽²⁾. Expansion and Bragg peak neutron intensity values are recorded on a $X-Y_1-Y_2$ recorder with the temperature on the X -axis.

2. Dilatometric support.

A diagram of this set up is given in figure 2. The maximum diameter of the support is imposed by the 50 mm diameter port of the furnace. The useful volume of the sample 1 was chosen to be $\varnothing 12$ with 12 mm height. Its vertical position is imposed as shown by the figure. The platinum sensor 2 and the sample are both wrapped with a copper foil for good thermal coupling. This foil is cut in the middle to leave a passage for the neutron beam. The sample is 30 mm high with flat parallel ends. It is placed on parts 3 and 4 which also provide centring and temperature. The base 4 is held in position by means of three springs 5 which pull it against three silica rods 6 via Nichrome wire 7. The sample expansion is transferred outside the furnace to the L.V.D.T. transducer 12 ($2.5 \times 10^{-5} \text{ mm}$ resolution) by means of three silica rods 10 and parts 8, 9 which also provide the temperature (by heat exchange with the furnace). Atmospheric pressure through welded bellows 11 provides a small force which ensures correct mechanical stability of the mechanism. Clearances at silica rod level are suppressed by folded stainless steel foil wedges. The furnace is controlled with a thermocouple (chromel-alumel) 13 associated with standard I.L.L. temperature control equipment (i.e. Drusch regulator-ice point reference and magnetic amplifier to supply the niobium resistor). The long term drift as read at the sample was found to be about 0.1 K/K ambient temperature. A differential thermocouple 14 and two extra heaters 15-15' enable the vertical temperature gradient to be changed. To improve short term stability and reduce thermal inhomogeneity in the sample the support is built relatively heavy with large block surfaces in front of the inner furnace element. Shields 16 (niobium 50 μm) and 17 reduce thermal

Fig. 2. — Diagram of the dilatometric support. (----) I.L.L. furnace; hatched parts : stainless steel (310).

radiative losses around the sample and through the ends.

The thermal constant time measured at the sample is about 25' at 573 °C. The work of the dilatometric support is illustrated by figures 3 and 4. Figure 3 shows a typical sequence of gradient adjustment operations. With zero extra power the transition is spread over about 5°. 3C (3a) with a vertical gradient of 7.5° as read from the differential thermocouple. When we increase the power in 15 (a few watts) we get the hysteresis curve b, c.

By comparing with figure 1 we retain the value of 1.5° for the differential thermocouple indication for the best thermal condition corresponding to a residual gradient of about 0.3° on the sample.

⁽²⁾ The fluctuation ($\Delta I = 2 \text{ A}$) of the intense ($I = 100 \text{ A}$) heating current of the furnace, due to regulator action, generates a low frequency modulation of the readout when we used a 5 1/2 digital multimeter followed by a D.A. convertor.

Fig. 3. — Influence of a temperature gradient on the dilatation of a X-quartz during the α - β transition as shown by the dilatometric support. ($\times$) data from the differential thermocouple. ---- « ideal » curve. Insert : dilatation from the ambient temperature to the β phase. $\times$ Mayer (rapport C.E.A. (France) n° 1330 (1960)). — rough values from dilatometric support.

Figure 4 shows how we can follow the state of the sample during a neutron experiment [5], in particular when the interfacial region progressively sweeps the central scattering volume (curve defgh). It is even possible to interrupt the cooling before completion into α phase and heat again (curve jklm).

3. Compressive uniaxial stress apparatus.

The essential features of both the lever-press and home built furnace are shown in figure 5. The requirements were to obtain the largest solid angle possible and to put the quartz sample 1 ($7.5 \times 7.5 \times 30 \text{ mm}^3$) in horizontal position for neutron diffraction reasons. Because the thermal neutron diffusion of aluminum was found to be negligible in previous experiments we used it extensively for its high thermal conductivity. An aluminium sheet (2) 0.5 mm thick wraps the 50Ω sensor 3 and the sample together. The stress is applied by the action of stainless steel (310) arms 4 through axis 5 and gold foil. A notch in part 6 gives a virtual (0) elastic axis of articulation. A magnification of 12 is obtained when we applied a force F. Thermal expansion of the sample is measured by conductive plastic linear potentiometer 7 with a magnification of fourteen. The arms are held in vertical position by the axis 8 and two ball-bearings 9 which allow the free deformation

Fig. 4. — Simultaneous measurements of thermal expansion (dilatometric support) and neutron scattering. The central curve shows the dilatation of a Z-quartz while the surrounding curves show the shapes of the satellites (incommensurate) peaks ($\langle \zeta 3 0 \rangle$ scans) at different stages. The full lines correspond to homogeneous phases, the dashed lines to phase co-existence (for more details see [5]).

Fig. 5. — Diagram of the compressive uniaxial stress apparatus (lever-press).

of the arms⁽³⁾. F was first created with a spring system but because of friction problems we found that the best way for a narrow temperature range is to fit elastic rubbers. The lever-press is heated by means of air thermal conduction and thermal radiation by the stainless steel casing 10 on which an asbestos insulated Nichrome wire is wound. Suitable windows are cut

⁽³⁾ The elastic deformation of both arms is $\Delta L = 2$ mm with $F = 24$ kg at $T \sim 570$ °C.

out corresponding to a solid angle of 90° in the horizontal plane and 60° in the vertical plane. A 0.5 mm aluminium foil is partly screwed on arms 4 while three extra 0.5 mm aluminium foils act as a heat shield. The $50\ \Omega$ sensor is fixed on the wall of 10 to operate the Eurotherm regulator. The regulator controls a d.c. power supply. 400 W is necessary to bring the sample up to ~ 580 °C (with a ~ 4 cm thick Fiberfrax paper insulation). Figure 6 shows that a thermal inhomogeneity of about 2.8 K is present along the whole sample. We think we can reduce it by suitable reduction of the thermal losses around the sample.

Fig. 6. — Dilatation of a Y-quartz during its $\alpha \rightarrow \beta$ transition as shown by the lever-press (0.1 kbar). (....) reconstructed from discontinuous data.

Nevertheless, with neutrons, the satellite peaks from incommensurate phase (broadened by temperature gradients) were clearly observed and their changes with temperature and stresses could be followed in three runs (0-0.23-0.7 kbar at which the sample broke). At the same time we also check the well-known ~ 10 °/kbar α - β temperature transition displacement. Thus this lever-press apparatus appears to work satisfactorily as far as the initial objective is concerned.

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