conclude, therefore, that the accuracy attained for z at these low temperatures may be estimated as ± 0.00002. The room temperature values of Table 2, on the other hand, are much poorer because of the large background corrections and diverge from the Table 1 value by 0·00031 (corresponding to about 0·003 Å). Considering all of the data, it may be concluded that by increasing the temperature from 4·2 to 78 °K z does not change by a measurable amount; and that increasing the temperature further to room temperature decreases z by an amount that is significantly greater than estimated errors when monochromatized Ag radiation is used (though this cannot be confirmed by the less accurate filtered Mo radiation data).

Table 3. *Lattice constants c and a (hexagonal axes) and nearest and second nearest neighbor distances A_1, A_2, in antimony*

Temperature (°K)	a (Å)	c (Å)	A_1 (Å)	A_2 (Å)
4·2	4·3007	11·222	2·902	3·343
78	4·3012	11·232	2·903	3·344
298	4·3084	11·274	2·908	3·355

Table 3 lists unit cell dimensions obtained with Cu $K\beta$ radiation ($\lambda = 1.39217$ Å) and the Bond (1960) technique, using 33·0 and 00·16 reflections respectively, for the a and c measurements. The equipment used in I and II was modified to provide a more highly collimated primary beam, and more care was taken in aligning the beam and the crystals. Crystals of high perfection were selected for these runs, the width at half height of 00·16 ($2\theta = 137°$) was 0·14°; and of 33·0 ($2\theta = 152°$) was 0·27°. No corrections for refraction and Lorentz-polarization effects were made. The standard error of the a and c values in Table 3, estimated from the several runs, was of the order of 1 part in 20,000. The interatomic distances in the table were computed from these a and c values and the average z values of Table 1. The thermal contractions indicated in Table 3 are in good agreement with Erfling's (1939) single-crystal measurements in the temperature range covered by him.

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A Device for Taking X-ray Photographs of Single Crystals at High Temperatures*

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The paper describes a device for taking single-crystal X-ray photographs at temperatures up to 1000 °C. The furnace mount fits on a standard single-crystal goniometer and is therefore interchangeable between oscillation, Weissenberg and precession diffractometers. A modification of a Weissenberg diffractometer to take it is described. No cooling of the film cassette is needed.

Introduction

Though techniques for taking high-temperature powder photographs are highly developed, comparatively little has been published on ways of obtaining high-temperature single-crystal photographs. We were interested in temperatures up to 900 °C. The design chosen (described in detail below) uses as a furnace a small platinum wire carrying a very heavy current, the whole device being mounted on standard goniometer arcs. Since a Weissenberg diffractometer can be used for taking oscillation and rotation photographs

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as well as moving-film photographs, we decided to concentrate on modifying this camera for our crystal mount.

The furnace mount

Fig. 1 is a photograph showing the crystal furnace mount on a single-crystal goniometer. The heater elements were made of platinum wire in the form of spiral coils (*A* and *C* Fig. 1) above and below the sample position (*B* Fig. 1). The diameters of the spiral coils were made smaller as they approached the sample position in order to keep the temperature gradient as small as possible in this region. The diameter of the platinum wire was 0·012″; finer wire burned out too rapidly and thicker wire produced excessive spot screening (discussed later). The crystal itself is in a fused-silica capillary and is held there by a high-temperature cement. (Emerson & Cumming Eccoceram *QC* quartz embedment compound and cement.) The proportions recommended by the makers produced too dry a mixture for our purpose and instead we used two parts solid to one part liquid with a 24-hour room-temperature cure; this gave a bond that held up to 1000 °C. The capillary was cemented with the same high-temperature cement in a hole drilled in the insulator; this was made of pyrophyllite, which acts as both an electrical and a thermal insulator for the furnace and has the useful property that it can be machined after it has been half fired. The metal side pieces are made from Kumium, a copper alloy chosen for its stability at high temperature, combined with excellent electrical conductivity (pure copper distorts under our experimental conditions).

One aspect of the design of any furnace is to avoid screening of the incident or diffracted rays by the furnace. Here the platinum wire inevitably produces some screening, but by suitable choice of wire thickness it is kept small, and it is restricted to an easily identified sector of the oscillation range. Screening of the parts of the reciprocal lattice below the equator is reduced by the cut-away shape of the assembly.

This furnace mount is interchangeable between Weissenberg, oscillation, and precession diffractometers.

Adaptation of the diffractometer

A photograph of a modified Weissenberg diffractometer is shown in Fig. 2. The cassette has been removed to show the leads attached to the Kumium elements. Power is fed into these through a modified spindle (Fig. 3); for this a standard spindle was drilled out, and two halves of a metal rod, split lengthwise and insulated from each other and from the sheath by silicon-fibre-glass tape, were fitted into the cavity. These two pieces are designed to carry the heavy alternating currents of up to 10 amp. The flexible leads connecting the spindle to the power supply were formed by sliding several braided copper sleeves together. A spiral arrangement of these leads between the spindle and terminals on the body of the diffractometer permits full 180° rotation of the spindle; another pair of such leads from the terminals to the power supply permits rotation of the body of the Weissenberg diffractometer.

The power supply was designed to operate as a constant-current source and under load delivers about 3 volts to the furnace.

The furnace was made as small as possible so that no film cooling would be needed. The complete furnace was run for six hours at temperatures of about 1000 °C and produced no significant heating of the film cassette.

Measurement of temperature

It is difficult to measure the exact temperature when the sample has a very small thermal mass. Three

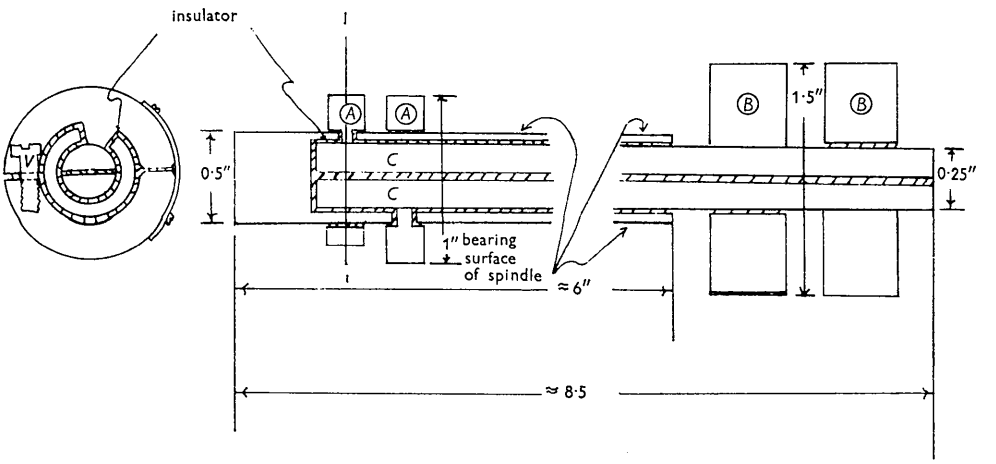

Fig. 3. Modification of Weissenberg spindle. Mechanically loading the contact between the split rod *C* and the contact elements (*A*) and (*B*) by means of the bolt *V* produces a good electrical contact. The electrical leads are hard soldered to these contact elements. Arcs are mounted on the spindle next to the contact elements marked *A*.

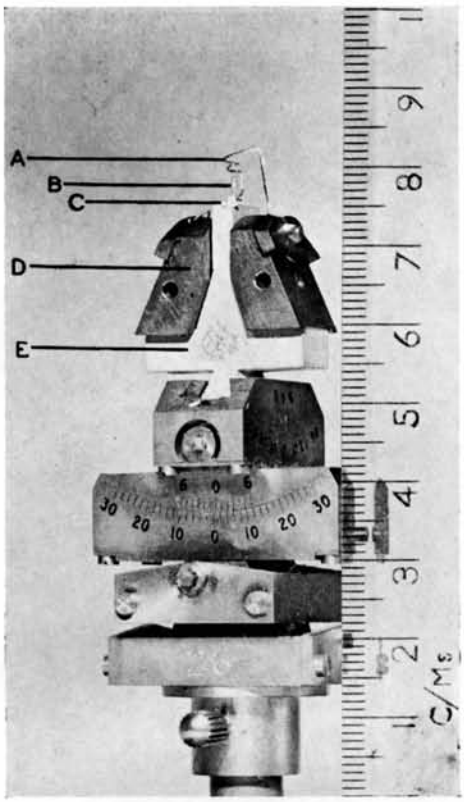

Fig. 1. Furnace mount in position on goniometer; the current leads are not shown. *A*, upper Pt. spiral coil (diameter ≈ 3 mm); *B*, crystal in silica glass capillary; *C*, lower Pt. spiral coil; *D*, Kumium side pieces; *E*. Pyrophyllite, thermal and electrical insulator.

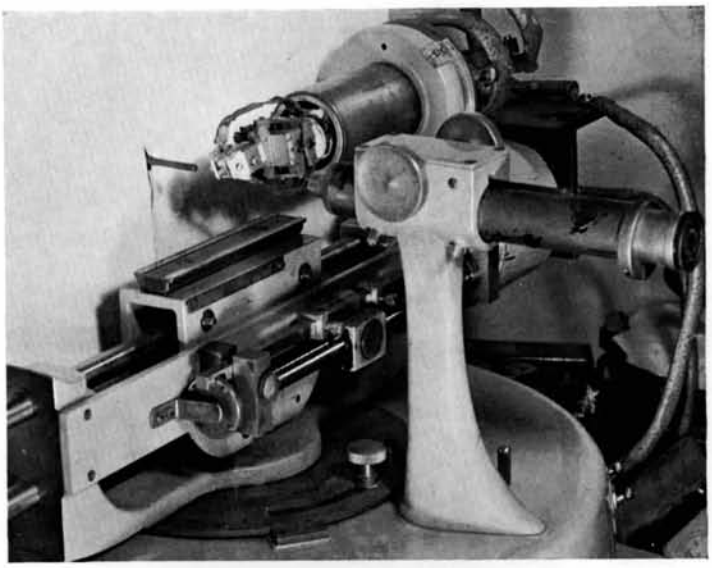

Fig. 2. Crystal furnace mount and arcs in position on the modified Weissenberg diffractometer.

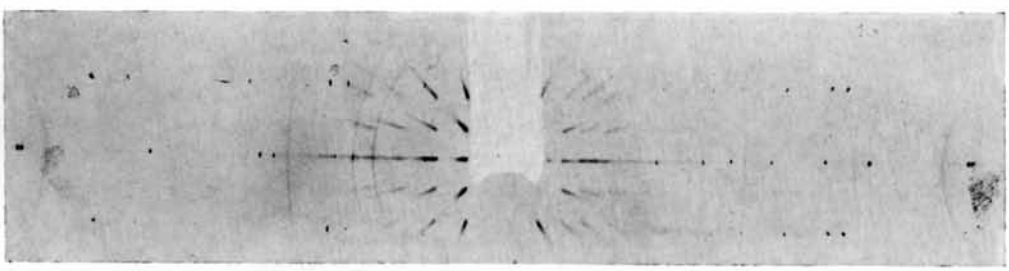

Fig. 4. Oscillation photograph of $NaNbO_3$ at about 480 °C. The $\alpha_1\alpha_2$ splitting can be seen in both the powder lines and in the diffracted spots. The effect of screening by the furnace mount of parts of the reciprocal lattice below the equatorial layer line can be seen.

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types of thermometer were used: (i) thermally sensitive colour crayons, (ii) high-temperature-stabilized thermistors, (iii) micro-miniature thermocouples. These thermometers were small enough to be placed in direct contact with the sample position.

(i) Thermally sensitive colour crayons and paints are a quick and effective means of estimating sample temperature. The change of colour gives an estimate of the temperature with no direct thermal loading of the sample. Because of the small sample size and bright background of the glowing furnace elements it is difficult to tell the exact point of colour change, which may be overestimated by up to 20 °C. This technique is useful to estimate whether the sample is above a threshold temperature, but gives no further information.

(ii) and (iii). Both the micro-miniature thermocouple and thermistor* are very small. The thermistor bead is barely visible (actually invisible to the naked eye, only its glass coating being seen) and its leads are 0·001″ PtIr wires. The active elements of the micro-miniature thermocouple are 0·001″ in diameter. The additional heat dissipated at the crystal position owing to the contact of these devices is very small. A plot† of radiation error as a function of wire diameter indicates that the wires used here produce an error of about 20 °C at 1000 °C; at 500 °C it will be much smaller. Another source of error is lack of thermal contact between crystal and temperature probe; a rough test suggested that this contributed a 10 °C uncertainty to our measurement. The use of measuring devices of small thermal mass has the difficulty that the thermal time constant of these devices is very small (*e.g.* the time constant for micro-miniature thermocouples is 1·3 milliseconds for a 60% step change in temperature), and fluctuations of short time constant obscure the steady-state temperature. Hence to obtain the mean temperature a damped meter was used in the measuring circuit.

Alternatively, the diffraction photograph itself can be used to determine the temperature, in one of two ways: either a powder of known linear expansion can be used in contact with the sample; or, when the linear expansion of the sample itself is known from other investigations, accurate measurement of the position of high-angle reflections allows the temperature to be deduced. The latter method was used in studies of $NaNbO_3$, taking as standard the powder measurements of Solov'ev, Venevtsev & Zhdanov.* The temperatures thus deduced agreed with those determined by the thermistors and thermocouples.

Stability and operation

The long-term temperature stability is primarily dependent on the constancy of the power supply. That this is adequate is shown by Fig. 3, an oscillation photograph of $NaNbO_3$ at 480 °C, in which the $\alpha_1\alpha_2$ doublet is clearly seen. The estimate of the half width of the spot in comparison with the $\alpha_1\alpha_2$ separation gives an upper limit of 14 °C for the temperature variation.

The complete instrument has been used for a sequence of long exposures totalling five hundred hours and has required no special care. It is reliable and does not reduce the flexibility of the Weissenberg diffractometer, even when samples are at the maximum temperature.

We wish to thank Dr W. H. Taylor for provision of facilities. Our thanks are also due to Mr Broad for helpful discussions. We are indebted to Mr C. Chapman and Mr D. Wollard who were responsible for making parts of the instrument described.

* These thermistors were stabilized at 500 °C.

† Supplied by the makers, Baldwin Lima Hamilton: Data Sheet 4334.

* Solov'ev, S. P., Venevtsev, Yu. N. & Zhdanov, G. S. *Kristallografiga*, (1961), **6**, 218; English translation, *Soviet Physics, Crystallography*, (1961), **6**, 171.