NOTES AND NEWS

A HIGH TEMPERATURE STAGE FOR THE POLARIZING MICROSCOPE

Elizabeth A. Wood, *Bell Telephone Laboratories, Murray Hill, New Jersey.*

Introduction

In order to study polymorphic transformations in crystals at high temperatures we found it necessary to construct a microscope stage which would permit observation of crystals in crossed polarized light at carefully controlled and measured temperatures as high as 800° C.

The stage constructed for the purpose is of such simple design and has been found so satisfactory that it was thought that details of its construction might be of interest to others confronted with the same problem.

Design, Materials and Construction

Essentially the "stage" consists of a heated *metal mass* surrounded by a mass of *insulating material* with the crystals and thermocouple in the center of the hot metal mass. Figure 1 is a scale drawing of the stage and Figs. 2 and 3 show the stage, first, disassembled and, second, mounted for use.

The *metal mass* consists of two superposed right circular cylinders of INCONEL, an alloy available from the International Nickel Company, which is chemically and mechanically stable at high temperatures. Each of these has a central cylindrical hole of small diameter for the light path and a shallow cylindrical recess of larger diameter in its upper surface to accommodate a polished fused silica disc. The lower disc is the surface on which the specimen lies. The upper disc serves to reduce heat loss.

In the under surface of both pieces is an annular trench in which the coiled nichrome heating element lies, insulated from the metal cylinder by asbestos paper. Pieces cut from the heating element from an "Eagle Straight Glocoil" heater, available from the Eagle Electric Manufacturing Company, Inc., Long Island City, New York, were found to be satisfactory. The resistance of each of the pieces used was about 2 ohms. The cut ends of the heating element were allowed to extend at right angles to the axis of the cylinders for about an inch, the end being doubled back on to the standing part of the wire to decrease heating of the lead wires.

The upper metal cylinder also has in its lower surface a small straight radial trench which fits over the thermocouple housing when the stage is assembled. The chromel-alumel thermocouple, of .005″ wire is led in to the center of the stage through a double-barreled ceramic tube. It lies directly on the lower fused silica disc beside the specimen to be studied and is, therefore, in the field of view (see Fig. 4.)

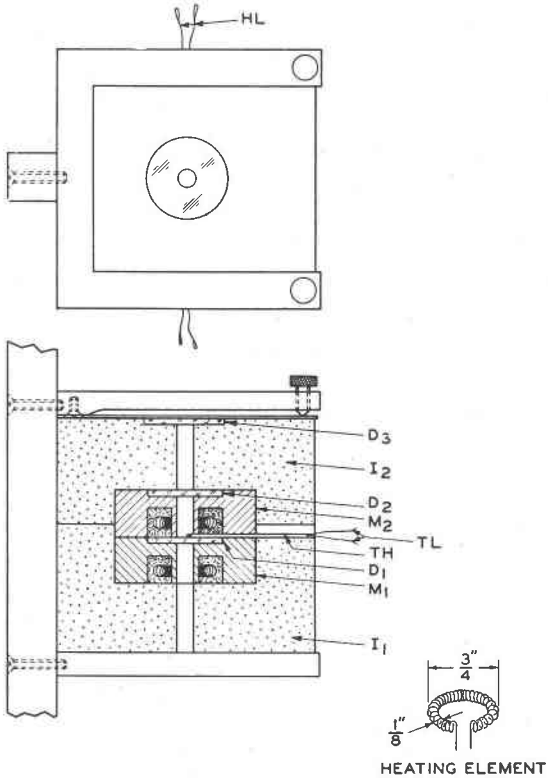

FIG. 1. Diagrammatic cross section of the heating stage

I_1 lower insulating block
I_2 upper insulating block
M_1 lower metal cylinder
M_2 upper metal cylinder
TH thermocouple housing
TL thermocouple leads
HL heating-element leads
D_1 fused silica disc on which the specimen lies
D_2 fused silica disc to reduce heat loss at top of M_2
D_3 pyrex disc to reduce heat loss at top of I_2

The *insulating material* surrounding the metal mass is a pair of rectangular blocks with central cylindrical recesses to accommodate the metal cylinders. Both blocks have a small central cylindrical hole for the light path. In the top surface of the lower block, in which the lower metal cylinder rests, are five small radial trenches, two for each pair of heating element leads and one for the thermocouple housing. In the photograph in Fig. 2, the lower block is shown with the lower metal

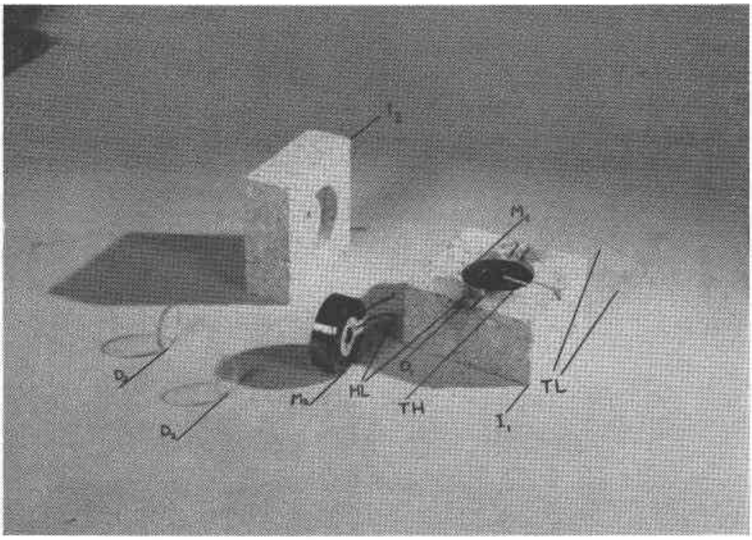

FIG. 2. Photograph of stage, partially disassembled

I_1 lower insulating block
I_2 upper insulating block
M_1 lower metal cylinder
M_2 upper metal cylinder
TH thermocouple housing
TL thermocouple leads
HL heating-element leads
D_1 fused silica disc on which the specimen lies
D_2 fused silica disc to reduce heat loss at top of M_2
D_3 pyrex disc to reduce heat loss at top of I_2

cylinder in place, its heating element leads lying in their trenches. The thermocouple trench is at right angles to these trenches. The top insulating block which fits down over the upper metal cylinder when it has been put in place has a shallow recess in its upper surface to accommodate a pyrex disc used to reduce heat loss. The material used for these blocks was SILOCELL obtainable from Johns Manville but other insulating brick might serve as well.

The heating elements were energized through a 10 volt, 12 amp. transformer connected to a variac drawing current from the 110 volt 60 cycle line. The two heaters were connected in parallel.

Mounting

The photograph in Fig. 3 shows the manner in which the stage was mounted for use. The simple microscope used on the brass rod mount

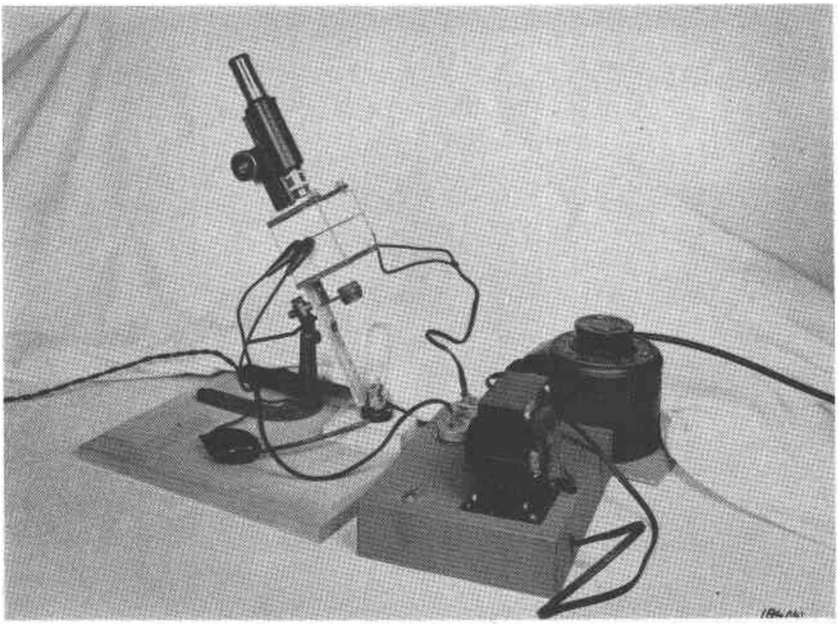

FIG. 3. Photograph of stage, mounted for use.

was one previously assembled by Mr. W. L. Bond for other purposes. The details of the spring holder designed for the heating stage are shown in the diagram in Fig. 1. A 150 watt projection lamp was used as a light source and pieces of polaroid were mounted below the stage and on top of the ocular of the microscope. The cheapest grade of polaroid was used with the expectation that it would have to be frequently discarded because of deterioration when heated. It has, however, shown no signs of deterioration after nearly 100 hours of use. The image was found to be greatly improved by the insertion of a diffusing screen between the light source and the stage. A piece of cellulose acetate with a frosted surface was used. This was mounted with the lower polaroid in a collar shown in Fig. 3 which could be easily rotated. The upper polaroid is rotated by rotating the ocular.

Performance and Maintenance

Figure 4 is a photomicrograph of sodium niobate taken with this microscope at 560° C. The lighting of the field is uniform and the image satisfactorily sharp. The fact that surrounding light does not reach the field of view is an advantage. The field of view has an area of approximately 8 square millimeters. Exploration of this area with the thermocouple showed no measurable temperature variation to exist.

Although the stage was designed for slow heating and cooling, heating rates as high as 1° per second have been used without damage to the equipment. Cooling rates are slow because of the large heat capacity of the metal mass and the low conductivity of the insulation. A typical average cooling rate is 10° per minute when the power has been turned off. The highest temperature to which the stage has been heated to date

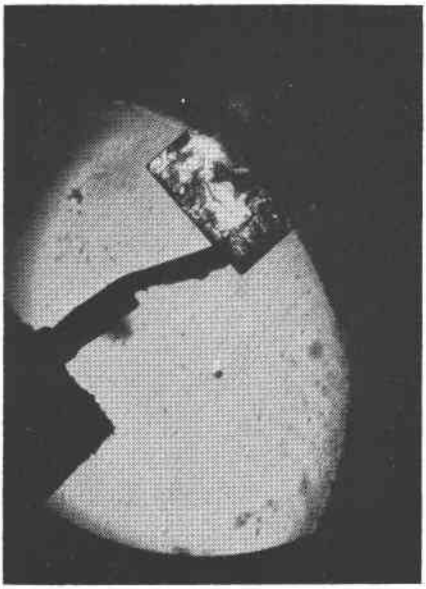

FIG. 4. Photomicrograph of sodium niobate at 560° C. Length of crystal: 1.05 mm.

is 800° C but it could probably be heated to 1,000° C without damage to the heating elements. Prolonged heating above this temperature will result in the devitrification of the fused silica discs.

Since the parts of the stage are simply fitted together and not clamped or cemented in any way, the whole can be disassembled and reassembled in a few minutes. Thus, the crystals under observation or the heating elements, in case of burn out, can be easily replaced.

The writer is grateful to W. L. Bond for helpful advice with respect to materials used in the stage, to Mr. J. A. deFeo for its construction.