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A HIGH-TEMPERATURE, CONTROLLED-ATMOSPHERE X-RAY DIFFRACTOMETER ATTACHMENT

by

J. H. Maurer and R. J. Ackermann

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Chemistry Division

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September 1967

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A HIGH-TEMPERATURE, CONTROLLED-ATMOSPHERE X-RAY DIFFRACTOMETER ATTACHMENT

by

J. H. Maurer* and R. J. Ackermann

I. INTRODUCTION

Many high-temperature X-ray diffractometers have been described in the literature.¹⁻⁹ All have in some way contributed to the elucidation and solution of various problems associated with X-ray measurements at high temperatures but many problems still exist, the most important of which are the maintenance of precise specimen alignment over broad temperature ranges and the simultaneous precise control of known temperatures and compositions of the specimens.

The diffractometer attachment described in this report was designed principally to cope more effectively with these problems. The instrument is designed to be used with a Norelco Wide Range Goniometer, specifically for X-ray investigations of materials at temperatures up to 2500°C in air, vacuum, or controlled atmospheres. Studies are accomplished by applying a thin powdered specimen to the resistance strip-heater, aligning the heater with the various positioning devices, and heating the sample while rotating it through a range of 2θ from 0. to 160°. The 2θ values characteristic of the crystalline specimen are detected by a scintillation counter, amplified, and recorded on a strip chart in conventional fashion.

To satisfy one of the geometrical requirements in this type of experimental approach, the entire specimen surface must lie precisely on a plane that passes through the goniometer's rotational axis. The elevation of the specimen is critical, since any misalignment perpendicular to the goniometer axis will result in 2θ values that will be too high or too low if the specimen lies, respectively, above or below the goniometer axis. The calibrated dial for elevation control designed for this attachment allows the vertical position of the sample to be precisely and reproducibly correlated to the 2θ values.

In addition to the elevation control that moves the specimen perpendicular to the goniometer axis, the present attachment is equipped with a eucentric goniometer which permits multiaxial movement of the specimen so the entire surface of the specimen will meet the necessary geometrical

*Present address: Polycor Plastics Corp., Box 246, Spring Grove, Ill.

conditions. Other geometrical relations are met by rotating the chamber about the goniometer axis, and by mechanical and X-ray alignment procedures explained in the MRC* X-86-N manual and Norelco's diffractometer instruction manual.

Other features incorporated in this new attachment include flexible water-cooled electrical leads, externally operated flexible shaft drives for the eucentric goniometer, and interchangeable connections for two thermocouples used for temperature control and measurement, respectively. Also added were external, water-cooled, electrical connections which allow the coolant-water tubing and high-current power leads to be easily disconnected from the internal water-cooled heater leads. A modified MRC chamber, a component of the MRC Model X-86-N Diffractometer Attachment, was used to mount the components described. In addition to the MRC chamber and its beryllium inlet and outlet windows, the mounting shaft and bearing shafts for the MRC stage assembly were retained. Two additional units essential to the operation of the diffractometer attachment are an electrical power supply, also purchased from MRC, and Leeds and Northrup components for controlling the specimen temperature.

II. APPARATUS

Figures 1 and 2 are drawings of the diffractometer attachment and heater-stage assembly, respectively. The heater is secured to the copper electrodes (30) with the two copper heater clamps (29). The electrodes are mounted on a quartz plate (45) screwed to the top of the eucentric goniometer (32). The quartz plate electrically insulates the electrodes from each other and from the eucentric goniometer. The nuts for the 4-40 electrode-mounting screws are sealed in an epoxy-filled groove on the underside of the quartz plate.

Two 90° copper elbows (31), nonconductively joined by a glass tube (44) sealed and strengthened with epoxy (E), connect the two coolant-water passages (46) in the electrodes. Copper sleeves (27) at the other end of the passage holes fit snugly into the ends of the polyethylene tubing (7). A brass clamp (26) secures the polyethylene tubing to the copper sleeves. The gap between the brass clamp and the electrodes is filled with epoxy, which produces a vacuum-tight seal between the ends of the plastic tubing and the sleeves.

Water is supplied from an external source through polyethylene tubing (11) to the Lucite junction block (8) and by section (7) to the electrode. Water-tight and vacuum-tight seals (12) at the junction block and where the

*MRC -- Materials Research Corporation, Orangeburg, New York, 10962.

tubing enters the chamber (12) are made with O-rings (0). The junction block is fastened to a metal support rod (5) which is screwed into the chamber.

High-current power leads (13) from the MRC power supply are connected to fittings on the Lucite junction block with 1/4-in., brass compression unions (10). Banana plugs (9) soldered to one end of each stranded copper wire (6) plug into the ends of the fittings that terminate in the water passage of the junction block. The other end of the stranded wires pass inside the polyethylene coolant-water tubings (7) and are fastened inside the blind holes (47) in the electrodes with soft solder. The flexibility of the water-cooled current leads and the flexible shafts (33) permits unrestricted adjustment of the eucentric goniometer shafts and of the elevation control without distortion.

Reproducible positioning of the heater (28) is accomplished with shafts (A), (B), (C), and (D), on the eucentric goniometer, and the elevation control dial (23). Shafts (A) and (B) have no external control, being adjusted before the cover (38) is attached. The accessibility of shaft (A) is restricted on one side by the copper elbows and on the other side by the brass clamp. However, the heater can be moved easily along all axes parallel to the Y-axis when it is secured to the electrodes with the heater clamps. Shafts (C) and (D) are connected to the external rotation (34) and azimuth (35) control knobs by the flexible shafts.

The height adjustment for the heater is by an external control only and is provided by turning the elevation-control dial. If a little care is exercised when making the folds on the heater ribbon, the edges of the surface of the heater should be very closely parallel to the electrode edges. A press for forming the heaters, such as the one used by Smith,⁸ is an excellent accessory. The eucentric goniometer must be rotated by loosening the eucentric goniometer mounting unit (49) until shafts (A) and (C) are parallel to the Y-axis and shafts (B) and (D) are parallel to the Z-axis.

The heater surface is aligned when the arc controlled by shaft (C) is tangent to the Y-axis, the arc controlled by shaft (D) is tangent to the Z-axis, edge a of the heater surface lies on the YZ-plane and is bisected by the Z-axis, and edge b of the heater surface lies on the YZ-plane and is bisected by the Y-axis. The above conditions are met by placing the grooves in the alignment fixture (50) on the two bearing shafts (3) and the alignment pin (42). The heater height is adjusted with the elevation-control knob (23) until it is slightly below the alignment fixture. Edge b is set parallel to the fixture by turning the azimuth-control knob (35), and edge a is set parallel to the fixture by turning the rotation-control knob (34). The surface of the heater is bisected by the Z-axis by adjusting shaft (A) until the center of the alignment pin is in line with the center of the heater surface. The Y-axis will bisect the heater surface when shaft (B) is adjusted

so that the bearing shafts are in line with the center of the heater surface. A Lucite window in the center of the alignment fixture permits the transmission of light to the heater surface and thereby facilitates positioning of the heater edges with respect to the underside of the alignment fixture. When the above conditions are met, the heater is brought into contact with the bottom surface of the alignment fixture by adjustment of the elevation-control dial. The depth of the grooves in the alignment fixture is 0.0625 in., which is half the diameter of the bearing shafts and alignment pin. When the heater is positioned as described above, its bottom surface lies to close approximation in the YZ-plane.

The top surface of the elevation-control dial protrudes from the front of the elevation-mechanism housing (21) and is divided by scribed lines into 25 equal segments. Every fifth segment is numerically marked on the front edge of the dial. The elevation screw (19) has forty threads per inch; hence, one revolution of the dial represents 0.025 in. of travel in elevation, and every scribed line on the dial represents 0.001 in. travel. A vertical scribed line on the center of the housing directly above the dial serves as a reference. The scale (24) next to the reference line divides each segment into five equal parts; this allows the travel of the screw to be adjusted in increments of 0.0002 in. The final position of the dial should be consistently approached from decreasing to increasing values to eliminate the 0.001-in. lag of the screw.

The elevation shaft (22) is sealed with an O-ring and is prevented from rotating by pin (17), which protrudes into slot (18) in the shaft. The height-adjust lock (20) on the bottom of the elevation-mechanism housing (25) locks the elevation-control dial. The flange on the elevation-mechanism housing is secured to the chamber with four screws and sealed with an O-ring. Four vacuum-tight threaded caps (43) are sealed to the chamber with epoxy and extend the thread depth of the elevation-housing mounting screws. The front and back caps are not shown. The eucentric goniometer mounting nut (49) secures the eucentric goniometer to the mounting plate (15), which is kept from turning by a pin (16). A screw in the center of the mounting plate fastens it to the elevation shaft.

The ribbon resistance heaters used are similar to the designs described by Intrater and Hurwitt¹ and by Smith.⁸ Intrater and Hurwitt state that they used thin heaters, and Smith used 0.005-in. material. Platinum heaters fabricated from 0.010-in. sheet have been used most successfully with this attachment. The increase in structural strength of thicker heaters reduces the extent of heater distortion at high temperatures. With 0.010-in. platinum a temperature of approximately 1400°C at 80 A is obtained with the MRC power supply. Since the current required for any specific heater temperature is inversely proportional to the resistance of the heater material, heaters made of materials that have lower resistance than platinum at high temperatures will require a power supply with a larger current rating than the MRC unit.

Two 0.003-in. Pt-Pt 10% Rh thermocouples (48) spot-welded to the underside or topside of the heater surface are used for temperature control and measurement, respectively. Because the type of heater used has a low thermal capacity, small-diameter thermocouple wire is essential. Any thermocouple junction in contact with the heater will function as a heat sink and create a cold spot at the weld. One thermocouple is connected directly to the internal ends of the modified BNC connectors (14). The other thermocouple terminates at connections on the opposite side of the eucentric goniometer and is connected to the BNC connectors with copper jumper wires (40). The connections at the ends of both thermocouple wires are approximately the same distance from the heater, which equalizes the effects of radiant and conductive heat at the connections and minimizes any difference in emfs that may be produced at these points.

The BNC connectors were used to expedite the switching of thermocouples and their external connections to the controller and potentiometer. The difference in emf between many pairs of thermocouples spot-welded to various central areas ($\sim 1/4 \times 1/4$ in.) of the heaters has never indicated temperature differences greater than 6° . One thermocouple is used for temperature measurement and is connected to a potentiometer, while the other furnishes the input signal to a Leeds and Northrup (L&N) Set Point Unit. The Set Point is a reference emf source; the difference between the emf of the control thermocouple and the preset reference is amplified by a L&N 9834-2 Electronic Null Detector. As used here, the Null Detector is simply an error amplifier whose output is connected to a L&N Series 60 control, which contains proportional, reset, and rate-signal circuits, and in turn controls the magnetic amplifier. The magnetic amplifier is one stage of the L&N single-phase, two-stage reactor package. The other stage is a saturable reactor which regulates the AC supply for the ribbon heater. The AC current is fed to the heater by the MRC power supply, which consists of a powerstat, a step-down transformer, and an ammeter. The units described provide a very reliable temperature-control system. All temperatures between room temperature and 1400°C for a platinum heater can be quickly and continuously stabilized to within at least $\pm 1^\circ\text{C}$ and usually within $\pm 0.5^\circ\text{C}$. Stability is maintained equally well during overnight heating and shorter periods.

A port (36) was attached to the original MRC cover (38). A metal bellows connects the port with either a vacuum or controlled atmosphere system, and is sealed to the port by an O-ring on the outer circumference of the port. This seal also allows the unit to rotate independently of either system.

A 6-V lamp (4) inside the chamber provides illumination for inspecting the various components when operating with the cover attached. The specimen can be observed through the sight port (37) in the cover. The flexible shaft which connects shaft (D) on the eucentric goniometer

to the shaft on the rotation-control knob is detachable, and can be slipped onto shaft (D) before the cover is attached. Clearance groove (39) on the cover allows the cover to clear the front vacuum-tight threaded cap.

The chamber end of the original MRC hollow stainless steel mounting shaft (not shown) is sealed with epoxy, and the alignment pin (42) is embedded in the epoxy at the center of the shaft. A container (not shown) filled with the required amount of lead shot is attached to an arm at the outer end of the shaft to counterbalance the elevation-mechanism housing, Lucite junction block, high-current power leads, and the external coolant-water leads. The counterweight permits the diffractometer attachment to be rotated freely when not locked to the Norelco goniometer and reduces unnecessary torque on the goniometer mechanism.

Water cooling for the chamber and cover is not necessary when operating with platinum heaters. If more elevated temperatures from tantalum or tungsten heaters cause the chamber to heat excessively, two loops of copper tubing, one attached on the circumference of the cover and the other on the circumference of the chamber, will adequately cool the unit.

III. SAMPLE PREPARATION

Powder specimens that are stable in air are mixed with a lacquer binder composed of thirty parts amylacetate and one part nitrocellulose, thinned with acetone to the proper consistency for spraying. This binder burns off completely at 200-300°C in air and does not react with most inorganic compounds. The powder specimens are sprayed onto the heater surface with a Binks Manufacturing Company* Model 59-10002B "Wren" Air-Brush. Specimens that are not stable when heated in air can be mixed with a solvent vehicle that can be slowly, but completely, evaporated under vacuum. Application of the powdered specimen by spraying is preferred to all other techniques because of the excellent adherence and uniformity of the very thin layers (≤ 0.001 in.) obtained after vaporization and/or combustion of the binder. Good physical bonding between the heater and specimen are obtained, and temperature gradients are considerably reduced from those obtained with thin slabs and wafers, or capillaries filled with sample.⁸ By syphoning only the upper portion of the lacquer-specimen mixture into the Air-Brush, the grain sizes are kept fairly constant in size and the turbulence of the air stream causes them generally to be randomly oriented on the heater surface.

*Binks Manufacturing Company, 3114-44 Carroll Avenue, Chicago 12, Ill.

IV. PERFORMANCE DATA

The effect of the variation in sample height on the observed values of $2\theta_{hkl}$ is clearly seen in Fig. 3. The platinum heater itself has been chosen as the specimen. The 200 and 420 reflections produced by copper $K\alpha_1$ radiation and scanned at a rate of $1/8^\circ$ in 2θ per minute demonstrate that the observed position of the 200 reflection is nearly twice as sensitive as that of the 420 reflection to the change in elevation (Δh) of the heater, i.e., displacement of the heater along the X-axis (see Fig. 1) by the adjustment of the elevation-control dial (23) in 0.001-in. increments. The maximum scatter of individual points from the curve never exceeds 0.01° . The values of $2\theta_{hkl}$ observed in Fig. 3 were obtained from a random choice of previous settings, by consistently making the final approach from a decreasing to increasing value of Δh . In this way the effect of the small lag of the elevation screw is eliminated.

The adjustment of the azimuth angle by means of shaft D [control knob (35)] of the eucentric goniometer is necessary to position correctly the heater in the Y-Z plane. The effect of this adjustment on both the intensity and the observed values of 2θ for the 200 reflection is shown in Fig. 4. This adjustment does not simply rotate the heater about the Z-axis, which axis must bisect the heater, but also translates it to some extent along the Z-axis, which causes unequal areas of the heater to be located above and below the Y-Z plane. As a consequence of the behavior shown in Fig. 3 there is a shift in the observed value of $2\theta_{200}$ as well as a diminution of the intensity resulting from loss of focus as the azimuth angle is varied from the horizontal.

The expansion behavior of typical platinum heaters from 25 to 1200°C is shown in Fig. 5. The observed values of 2θ for the 200 reflection obtained with copper $K\alpha_1$ radiation at a scan rate of $1/8^\circ$ in 2θ per minute are given for two heaters by the circular and triangular points. The calculated curve was obtained from the known lattice parameter of platinum, $a = 3.9231 \text{ \AA}$ (Ref. 10) and the linear coefficient of thermal expansion of platinum, $\alpha = 10.2 \times 10^{-6}/\text{deg.}^{11}$. In both cases the expansion of the heater causes an increasing displacement of the diffracting surface above the goniometer axis with increasing temperature up to 1000°C . An increase of 0.05° in $2\theta_{200}$ amounts to an increase in elevation of 0.003 in. as is seen from Fig. 3. Above 1000°C the observed values of 2θ appear to approach the calculated values. This behavior suggests that the tension from thermal expansion in the central portion of the heater is somewhat relieved at higher temperatures.

The problem of temperature measurement in high-temperature X-ray diffraction studies is well-known and is generally one of attempting to associate a measurable temperature near a crystalline sample with that of the actual surface from which X-ray reflections are emanating.

In the design and preliminary performance of the present apparatus no claim is made to the ultimate solution of this problem, although several precautions and techniques are proposed which tend to reduce the errors associated with the accuracy of the temperature. One of the prerequisites of accuracy is precision. The control units described in Sect. II enable the experimenter to achieve a precise control of the temperature (± 0.5 to 1.0°), at least at the points of contact between the thermocouples and the heater. Previous investigators^{8,12} have recommended the use of known transition and melting temperatures. In the present case we have observed the melting of KBr, NaCl, and K_2SO_4 on the platinum heater to occur respectively at 4, 5, and 7 degrees lower than the respective reported values, 730, 801, and $1076^\circ C$.

In the initial stages of the evaluation of the performance of the diffractometer attachment, it became apparent that the orientation of the heater with respect to the incident X-ray beam was such that the beam impinged on the pleated ends of the heater. Diffraction contributions from these poorly focused areas were not insignificant; consequently, the diffraction peaks were broad, unsymmetrically shaped, and even exhibited multiplicity at certain angles. Therefore, the lateral shields shown in Fig. 6 were constructed in order to mask off these areas from the X-ray beam. The lateral shields have the additional function of collimating the X-ray beam to a narrow strip ($\approx 1/8$ in. wide) in the center of the heater, over which area the temperature gradient is undoubtedly smaller than over the entire flat section of the heater. The lateral shields in the present case were fabricated from 0.010-in.-thick gold sheet.

Since high-angle reflections are less sensitive to variation in the displacement of the specimen from the goniometer axis than are low-angle reflections as seen in Fig. 3, it is possible to determine reliable unit-cell dimensions by extrapolating to $2\theta = 180^\circ$ via methods discussed by previous investigators.^{2,13} In addition to these extrapolation methods the present apparatus and associated techniques permit the use of an internal standard in the form of the platinum (or other metal) heater whose diffraction peaks can be seen through the extremely thin specimens prepared by the spraying technique. It is a simple matter to set the goniometer on the proper 2θ value at a given temperature, as given in Fig. 5, and maximize the peak height by adjustment of the elevation-control dial (23) and the azimuth-control knob (35). The heater containing the thin specimen is thereby quickly and reproducibly aligned. The three most intense reflections of orthorhombic tungsten trioxide at $700^\circ C$ are given in Fig. 7 along with the 200 reflection of platinum, which is seen through the thin oxide layer prepared by the spraying technique. The $\alpha_1\alpha_2$ doublet is seen to be resolved in the case of platinum. The $2\theta_{200}$ value for copper $K\alpha_1$ radiation has been normalized to the value calculated from thermal expansion data shown in Fig. 5. The three reflections of tungsten trioxide: 001, 020, and 200 are nearly equally intense in accordance with single-crystal¹⁴ and powder¹⁵ data; hence, the sample appears to possess random orientation on the heater.

V. REFERENCES

1. J. Intrater and S. Hurwitt, *Rev. Sci. Instr.* 32, 905 (1961).
2. P. Chiotti, *Rev. Sci. Instr.* 25, 683 (1954).
3. *Advances in X-ray Analysis*, Vol. 5, Edited by William M. Mueller, Plenum Press, New York (1961), paper by H. J. Goldschmidt, pp. 191-212.
4. J. A. Perri, E. Banks, and B. Post, *J. Appl. Phys.* 28, 1272 (1957).
5. *Advances in X-ray Analysis*, *op. cit.*, paper by I. Corvin, F. Schosberger, and F. Ticulka, pp. 221-228.
6. L. S. Birks, *Rev. Sci. Instr.* 25, 963 (1954).
7. *Advances in X-ray Analysis*, *op. cit.* paper by F. A. Mauer and L. H. Bolz, pp. 229-237.
8. D. K. Smith, *Norelco Reporter*, Vol. X., No. 1, p. 19 (1963).
9. *Advances in X-ray Analysis*, *op. cit.*, paper by W. J. Campbell, S. Stecura, and C. Grain, pp. 169-190.
10. *X-ray Powder Data File*, Sets 1-5 (Revised), American Society for Testing Materials, Philadelphia (1960).
11. *American Institute of Physics Handbook*, McGraw-Hill Book Company, Inc., New York (1957).
12. J. A. Brand and H. J. Goldschmidt, *J. Scientific Instr.* 33, 41, (1956).
13. H. Ebel, *für angew. Physik* 16, 490 (1964).
14. S. Tanisaki, *J. Phys. Soc. Japan* 15, 573 (1960).
15. Arne Magneli, Georg Andersson, Birgitta Blomberg, and Lars Kihlberg, *Anal. Chem.* 24, 1998 (1952).

KEY TO FIGURES 1, 2, AND 6

<p>A. }
 B. }
 C. } Eucentric Goniometer Shafts
 D. }</p> <p>E. Epoxy--"Torr Seal"--Model No. 953-0001
 (Varian Associates, Vacuum Products
 Division, 661 Hansen Way, Palo Alto,
 California)</p> <p>0. Neoprene O-ring</p> <p>1. MRC Beryllium Window Assembly</p> <p>2. MRC Chamber</p> <p>3. Bearing Shaft</p> <p>4. 6-V Lamp and Socket</p> <p>5. Metal Support Rod</p> <p>6. Stranded Copper Wire (0.125-in. OD)</p> <p>7. Polyethylene Tubing (1/4-in. OD;
 0.040-in. Wall)</p> <p>8. Lucite Junction Block*</p> <p>9. Banana Plug</p> <p>10. 1/4-in. Compression Union</p> <p>11. Polyethylene Tubing</p> <p>12. Vacuum Fitting</p> <p>13. Power Lead</p> <p>14. Amphenol BNC Connector</p> <p>15. Mounting Plate</p> <p>16. Antirotation Pin</p> <p>17. 1/8-in. Dowel Pin</p> <p>18. 1/8-in. Slot</p> <p>19. Elevation Screw</p> <p>20. Height-adjust Lock</p> <p>21. Elevation-mechanism Housing--SS
 (Stainless Steel)</p> <p>22. Elevation Shaft--SS</p> <p>23. Elevation-control Dial--Brass</p> <p>24. Scale</p> | <p>25. Bottom-elevation Mechanism
 Housing--SS</p> <p>26. Brass Clamp</p> <p>27. Copper Sleeve</p> <p>28. Ribbon Heater</p> <p>29. Heater Clamp</p> <p>30. Copper Electrode</p> <p>31. Copper Elbow</p> <p>32. Eucentric Goniometer (Electronics and
 Alloys Inc., 435 Victoria Terrace,
 Ridgefield, New Jersey)</p> <p>33. Flexible Shaft</p> <p>34. Rotation-control Knob</p> <p>35. Azimuth-control Knob</p> <p>36. Port</p> <p>37. Pyrex Window</p> <p>38. MRC Cover</p> <p>39. Clearance Groove</p> <p>40. Jumper Wires (rear wire not shown)</p> <p>41. O-ring Groove</p> <p>42. Alignment Pin</p> <p>43. Vacuum-tight Threaded Cap (4 caps--
 located 90° apart)</p> <p>44. Glass Tube</p> <p>45. Quartz Plate</p> <p>46. Passage</p> <p>47. Blind Hole</p> <p>48. Thermocouple</p> <p>49. Eucentric Goniometer Mounting Nut</p> <p>50. Alignment Fixture</p> <p>51. Lucite Window</p> <p>52. Front Lateral Shield Mounting Block</p> <p>53. Back Lateral Shield Mounting Block</p> <p>54. Lateral Shields</p> |
|--|--|

*All fittings attached to the Lucite junction block were made from standard brass fittings.

Fig. 1. Schematic Drawing of the High-temperature Diffractometer Attachment

Fig. 2. Schematic Drawing of the Heater-stage Assembly

Fig. 3

The Effect of the Variation of Sample Height on the Observed Values of $2\theta_{hkl}$ (Copper $K\alpha_1$) for the 200 and 420 Reflections of Platinum

Fig. 4
The Effect of the Variation of the Azimuth Angle of the Sample on the Intensity and Observed Values of 2θ (Copper $K\alpha_1$) for the 200 Reflection of Platinum

Fig. 5

The Effect of Thermal Expansion of Two Platinum Heaters on the Observed Values of 2θ (Copper $K\alpha_1$) for the 200 Reflection Compared with Those Calculated from Thermal-expansion Data

Fig. 6
Schematic Diagram of Lateral Heater Shields

Fig. 7. Relative Intensities for Copper $K\alpha_1\alpha_2$ Radiation of the 001, 020, and 200 Reflections of Orthorhombic Tungsten Trioxide at 700°C and the 200 Reflection of Platinum Observed through the Oxide Sample