CANADA

# THERMOCOUPLE CALIBRATION IN A UNICAM S.150 HIGH-TEMPERATURE X-RAY DIFFRACTION CAMERA UP TO 1000°C

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# INTRODUCTION

The classic problem in high-temperature X-ray diffraction studies is to measure the sample temperature exactly. The difficulty is particularly aggravated by the physical situation present in the Unicam S.150 high-temperature X-ray diffraction camera, the instrument used by the writer of this report. In the first place, the thermocouple is not in direct contact with the sample, and, in the second place, the instrument is operated under high vacuum, so that heat transfer from the furnace to the sample and to the thermocouple is very largely by radiation, which can result in a considerable difference between the true mean specimen temperature and the temperature indicated by the thermocouple. This difference, when significant, was always in the sense that the indicated thermocouple temperature was lower than the true specimen temperature. For example, Ferguson et al.(1) report a negative error of 145 deg C at a sample temperature of 300°C in a Unicam camera. Various workers (2, 3, 4) have investigated this problem and it appears that the principal factors involved are:-

(i) differences in emissivity of the sample and of the thermocouple (emissivity is the ratio of radiation emitted by the surface of the specimen to the radiation emitted by a black body, i.e. a perfect radiator);

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(1) For references, see page 18.

(ii) heat conduction along the thermocouple leads;

and (iii) the spatial arrangement of the sample and the thermocouple.

Berry et al. (4) reported emissivity errors as high as 20 deg C at 500°C, whereas Brand et al. (3), using the prototype of the Unicam S.150 camera, found heat conduction to be more important, giving rise to an error of 42 deg C at 300°C. It is possible that these conclusions may have been reached on the basis of the considerable differences in the geometry and construction of the two cameras involved.

The problem of overcoming the error may be solved in two ways:-

(i) by modifying the camera and the thermocouple arrangement to reduce the error to negligible proportions (2, 3);

and (ii) by deriving a reliable calibration curve, i.e., by measuring the sample temperature independently the thermocouple can be calibrated.

The latter method was chosen in the present work, since the former course appeared uncertain and time-consuming.

The variation of the lattice parameter of a metal with temperature can be used as an accurate measure of that temperature. The method used to calibrate the thermocouple depends upon this fact. The desirable properties of such a standard metal have been discussed by Ferguson et al. (1), and their conclusions are summarized as follows:-

> In order to use a precise determination of the lattice constant as a measure of temperature, the metal chosen should preferably have
>
> (i) a high melting point,
>
> (ii) a high, precisely-determined thermal expansion,
>
> and (iii) cubic symmetry and high-angle X-ray diffraction.
>
> Points (i) and (ii) are generally reciprocal in operation, since substances with high melting points generally have low thermal expansions. Platinum, however, appears to be a reasonable compromise and is often used in this type of work.

The procedure used is to obtain a reliable reference curve expressing the variation of lattice constant of platinum as a function of temperature. Thus, at any given power input, the lattice constant of platinum is measured and, by reference to the standard graph, the mean sample temperature is obtained. This value is compared with the thermocouple temperature and the deviation noted. In this way, a calibration curve for the thermocouple can be constructed for any desired temperature range.

However, another difficulty arises here. There are considerable discrepancies noted among the various measurements of the lattice constant of platinum as a function of temperature. Some room-temperature values are given in Table 1.

TABLE 1

The Lattice Constant of Platinum at 25°C

| Lattice Constant (in A units) | Source of Data |
|---|---|
| 3.9240 | Owen and Yates (5) |
| 3.9226 | Moeller (6) |
| 3.9231 | Swanson and Tatge (7) |

The spread in these results is equivalent to a temperature interval of about 50 deg C, since the lattice constant of platinum varies by about $3 \times 10^{-5}$ A per deg C. It is suggested that, if there is such a discrepancy at room temperature, then the situation will most probably worsen with the added complications associated with high-temperature measurements.

Ferguson et al.(1) attempted to overcome the difficulty by averaging the results of several workers, thus obtaining a mean curve relating lattice constant to temperature. However, it is suggested by the writer that a more reliable reference curve could be constructed by inserting the lattice constant of platinum, measured with the particular instrument to be calibrated, into a thermal expansion equation, derived from macroscopic measurements on platinum bars. The point is that, whereas there is quite good agreement among various workers on the relative increase of lattice constant with temperature, there is a distinct lack of agreement on the absolute value of the lattice constant at any given temperature. This suggestion has been confirmed by Campbell et al.(8), who critically examined the thermal expansion data obtained by various workers using X-ray methods.

The success of the technique depends on whether one can compare thermal expansion equations of metals measured macroscopically with those obtained by X-ray diffraction. There is considerable evidence in the literature (9) to show that there is little or no discrepancy.

The validity of the calibration curve can easily be checked by an absolute measurement of temperature, such as the melting point of a metal or a known phase transition. Thus, it was hoped to be able to estimate a sample temperature to ± 10 deg C within the range from room temperature up to 1400°C, the upper temperature limit obtainable with the camera.

## EXPERIMENTAL

### A. Modification of the Unicam S.150 High-Temperature Powder Camera

The instrument, as supplied, was equipped with a Pt-Pt/13% Rh thermocouple. This was replaced by a Pt-Pt/10% Rh couple in order to make use of direct-reading potentiometers available in the Physical Chemistry Section. The hot junction was in the form of a small ring about 0.25 in. in diameter. In this way, the danger of breaking samples by contact with the tip of the thermocouple was eliminated, since the samples were always located in the centre of the ring. Also, the thermocouple error should be less, since the error is supposed to be inversely proportional to the area occupied by the hot junction(3).

## B. Temperature Control

The temperature of the furnace was controlled by the Variac Autotransformer supplied with the instrument. Power to the Variac was controlled to within ± 1% by the magnetic stabilizer of the Siemens Crystalloflex IV X-ray generator used in conjunction with this camera. This resulted in a temperature fluctuation in the camera at any given power input of about ± 5 deg C, as indicated by the thermocouple.

## C. Determination of the Lattice Constant of Platinum

A sample of commercially-pure platinum wire of 32 B and S gauge was cemented to the alumina sample holder by means of a paste made of alumina powder and polyvinyl-alcohol. Films were exposed for about 4 hours, using Ni-filtered CoK radiation. Duplicate measurements were made on the 331, 420 and 422 $\alpha_1\alpha_2$ doublets. The calculated lattice constants were then extrapolated to $\theta = 90°$, using the Nelson-Riley formula. These values were then corrected for refraction in the specimen.

## D. Construction of the Reference Curve of Lattice Constant of Platinum vs Temperature

Five determinations of the lattice constant of platinum were made at a measured room temperature. These were all corrected to 0°C in order to give a mean value from the readings obtained at the slightly variable room temperatures. This value was then inserted into the thermal expansion equation of Esser and Eusterbrook (10):

$$L_t = L_o\left[1 + 8.988 \times 10^{-6} t + 1.065 \times 10^{-9} t^2 + 1.26 \times 10^{-13} t^3\right] \quad \ldots 1)$$

where $L_t$ = length of specimen at t°C

and $L_o$ = length of specimen at 0°C.

This equation was used by Brand and Goldschmidt (3), and is considered by them to be reliable. Values of the lattice constant at intervals of 50 deg C were then calculated to give a plot of lattice constant as a function of temperature (see Figure 1). Details of the calculations are given in Appendix A (see page 20 ). The quantity K listed in column 6 of Appendix A is equal to the ratio $L_t/L_o$ at the temperature t°C.

## E. Verification of the Calibration Curve

The melting points of various metals (Zn, Ag and Au) were chosen as the most convenient fixed points for providing an absolute check of the calibration curve. Warlimont (11) has shown that this method is suitable for calibrating X-ray furnaces since satisfactory diffraction patterns can be obtained to within about 4 deg C of the melting point.

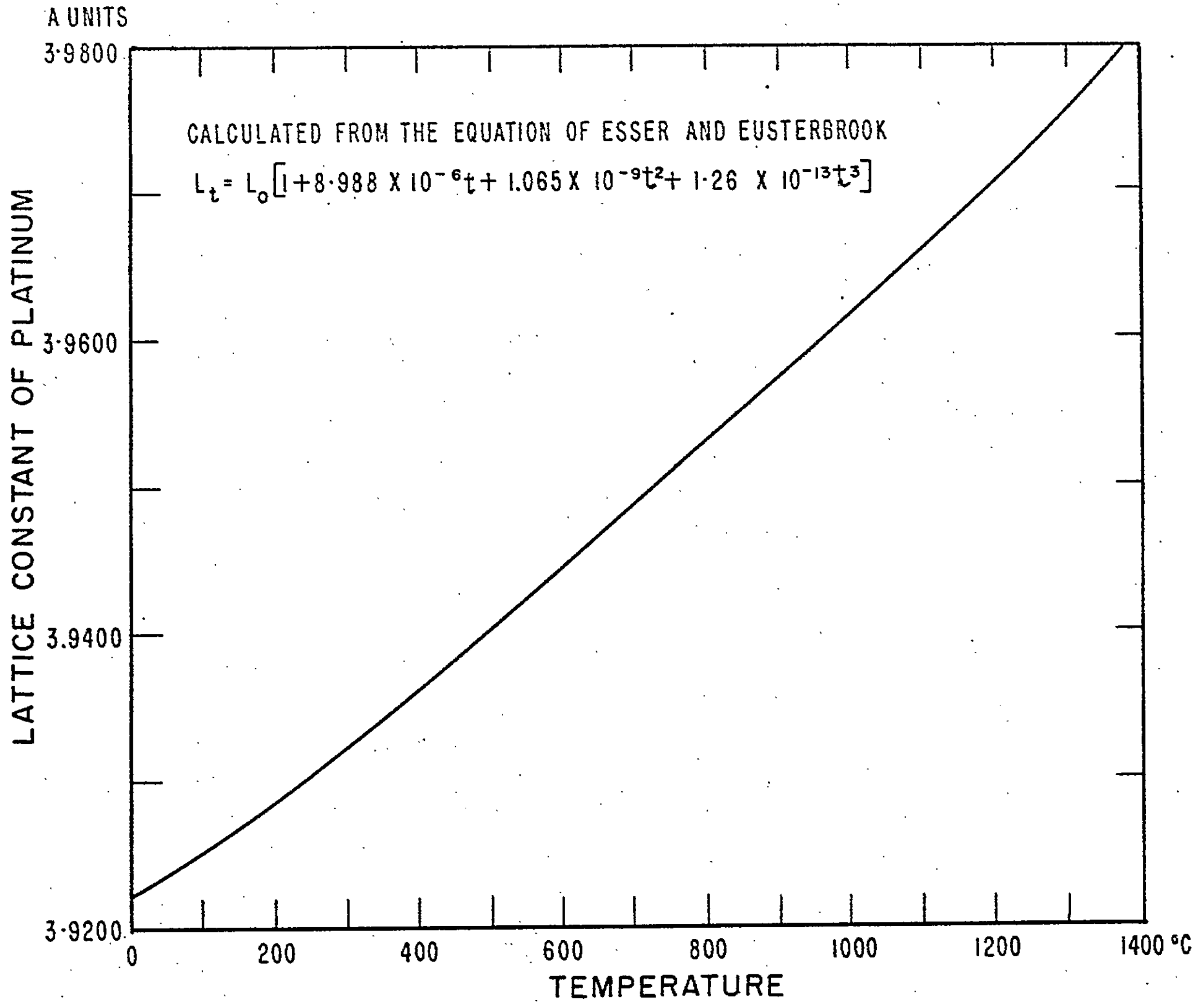

FIGURE I. STANDARD GRAPH OF LATTICE CONSTANT.

Melting-point determinations have certain advantages over phase transitions in that the quality of the films need not be very high. The detection of one or two of the strongest lines in the pattern is all that is required. Thus, one can use short exposures of the order of 1 hour with unfiltered radiation. On the other hand, to fix the $\alpha \longleftrightarrow \beta$ transition of quartz, one needs good-quality films requiring, perhaps, an 8-hour exposure, since the change is manifested by rather small alterations of comparatively weak lines in the back-reflection region of the pattern. In this work, the melting point was interpolated over a range of 10-15 deg C.

The metal sample, either in powder or wire form, was loaded in air into a 0.5 mm quartz capillary. A short piece of fine quartz rod was then placed on top of the sample to reduce the dead air space and to facilitate sealing. Next, the capillary was mounted on a vacuum manifold by means of Picein wax and then evacuated to 10-20 microns pressure. The capillary was then sealed by means of a micro-torch and the portion containing the sample was cut off at a suitable length. The sample was then fastened to the alumina sample-holder in the same way as for the platinum wire. During all runs, the thermocouple hot-junction was centred between the two hemispherical furnaces. Some observations on difficulties encountered in the operation of the equipment and on the means adopted of overcoming them are given in Appendix C (see page 22).

# RESULTS

In Table 2 are given the results for the measurement of the lattice constant of pure platinum at room temperature (25-27°C). In Table 3 are given the melting points of zinc, silver and gold, as obtained from the X-ray measurements.

TABLE 2

Measurement of Lattice Constant of Platinum at Room Temperature

| Run | Film No. | Lattice Constant (A) | Temperature of Measurement (°C) | Lattice Constant Extrapolated to 0°C* (A) | Mean Value (A) | Standard Deviation (A) |
|---|---|---|---|---|---|---|
| 1 | T 174 | $3.9225_4$ | 26 | $3.9216_2$ | 3.9217 | ± 0.00008 |
| 2 | T 175 | $3.9226_4$ | 25 | $3.9217_6$ | | |
| 3 | T 176 | $3.9226_2$ | 27 | $3.9216_7$ | | |
| 4 | T 177 | $3.9226_3$ | 27 | $3.9216_8$ | | |
| 5 | T 178 | $3.9227_6$ | 27 | $3.9218_1$ | | |

*Using Equation 1 - see page 7.

TABLE 3

Melting Point of Metals

| Metal | (A) Melting Point (Literature) (°C) | (B) Temperature indicated by Thermocouple at M Pt (°C) | (C) Corrected Temperature derived from Equation 1 (°C) | Deviation (C) - (A) |
|---|---|---|---|---|
| Zn | 419 | 329 | 415 | - 4* |
| Ag | 961 | 962 | 972 | + 11 |
| Au | 1064 | 1086 | 1098 | + 34 |

*A small negative deviation such as that quoted above is probably not significant since the precision cannot be claimed as any better than ± 10 deg C.

## DISCUSSION OF RESULTS

Replicate measurements of the lattice constant of platinum (see Table 2) show good precision. The standard deviation of ± 0.00008 A implies a precision in temperature measurement of about ± 3 deg C. When this is combined with the temperature variation of ± 5 deg C caused by current fluctuation in the furnace windings, the overall precision in temperature measurement is ± 8 deg C. Thus, one may reasonably expect all results to carry a precision of ± 10 deg C.

Figure 2 shows a plot of the thermocouple deviation, $\triangle T$, as a function of temperature. The data of Brand and Goldschmidt (3) are included since their instrument was the Unicam S.150 prototype. The present work has a maximum $\triangle T$ value of 90 deg C, which is more than double the deviation of 42 deg C in the earlier work. There is no obvious reason for this large discrepancy, but it has been amply confirmed by other workers in this field (1, 12). Appendix B contains details of the measurements used in plotting Figure 2.

The main features of this thermocouple deviation curve have been accounted for by Brand and Goldschmidt (3), who made a semi-quantitative analysis of the problem. They state that the deviation can be represented by the following expression, derived from the Stefan-Boltzmann radiation law:-

$$\triangle T = \frac{C\,(T - T_o)}{4\,T^3} \qquad \ldots 2)$$

where $T$ = thermocouple temperature (°K),
$T_o$ = room temperature (°K),
and $C$ = constant.

Equation 2 tends to zero at high values of T and has a maximum value independent of C at T = 400°K approximately. These features are only generally preserved by the present experimental deviation curve, since $\triangle T$ is at a maximum at 643°K. Furthermore, $\triangle T$ rises again considerably at temperatures greater than 1000°C. No explanation is offered for this rise on the basis of Brand's analysis.

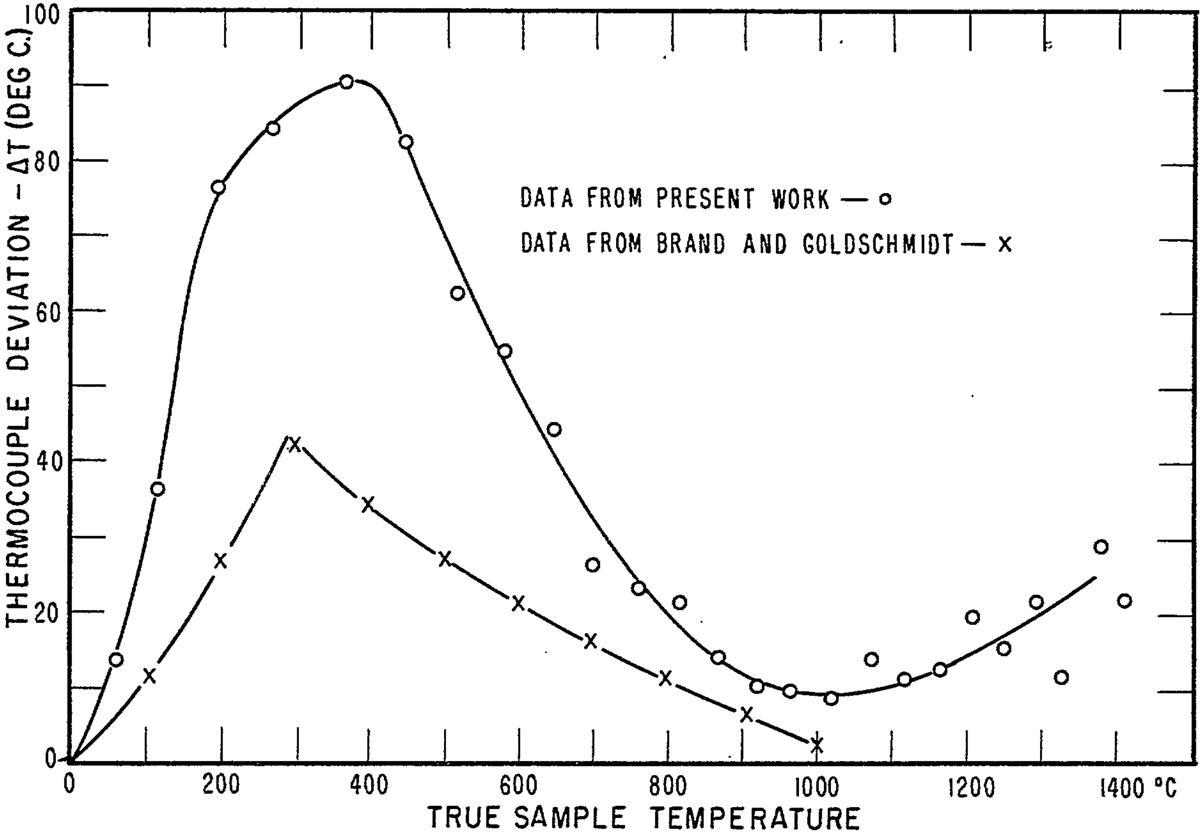

FIGURE 2. GRAPH OF THERMOCOUPLE DEVIATION (ΔT) VS. SAMPLE TEMPERATURE.

(NOTE: THE THERMOCOUPLE TEMPERATURE IS NORMALLY LOWER THAN THE TRUE SAMPLE TEMPERATURE.)

There is an increase in the scatter of the experimental points at temperatures above 1000°C, which is probably associated with grain growth in the platinum wire. This suggestion is confirmed by Figure 3, which shows the break-up of the diffraction lines into segments at higher temperatures making measurement of the lines considerably more difficult. For this reason, it is recommended that the thermal expansion of platinum be used as a calibration standard only up to 1000°C.

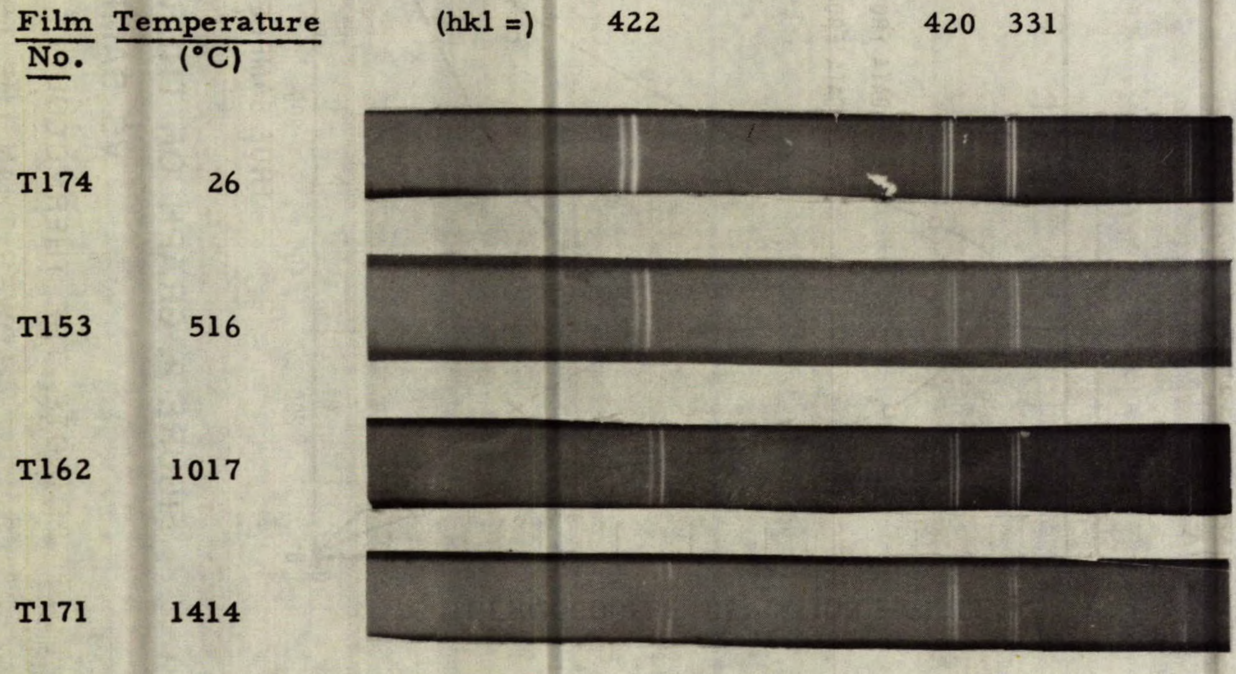

Figure 3. Diffraction Patterns of Platinum at Various Temperatures.

A calibration curve showing the observed thermocouple temperature plotted against the true sample temperature is shown in Figure 4. This curve was used in the estimation of temperature in all subsequent investigations involving the use of the high-temperature diffraction camera. This calibration curve should carry the same degree of precision, namely ± 10 deg C, as did the curve showing thermocouple deviation plotted as a function of true sample temperature (Figure 2).

This expected precision is, however, only partly confirmed by the results shown in Table 3. At temperatures in excess of 1000°C, the precision appears to have deteriorated by a factor of at least three compared with its value at lower temperatures. In addition, there appears to be a general trend towards decreasing precision at higher temperatures. It has not yet been found possible to show whether the above-mentioned grain-growth behaviour is the sole explanation for these unsatisfactory high-temperature results or whether additional causes are also operative.

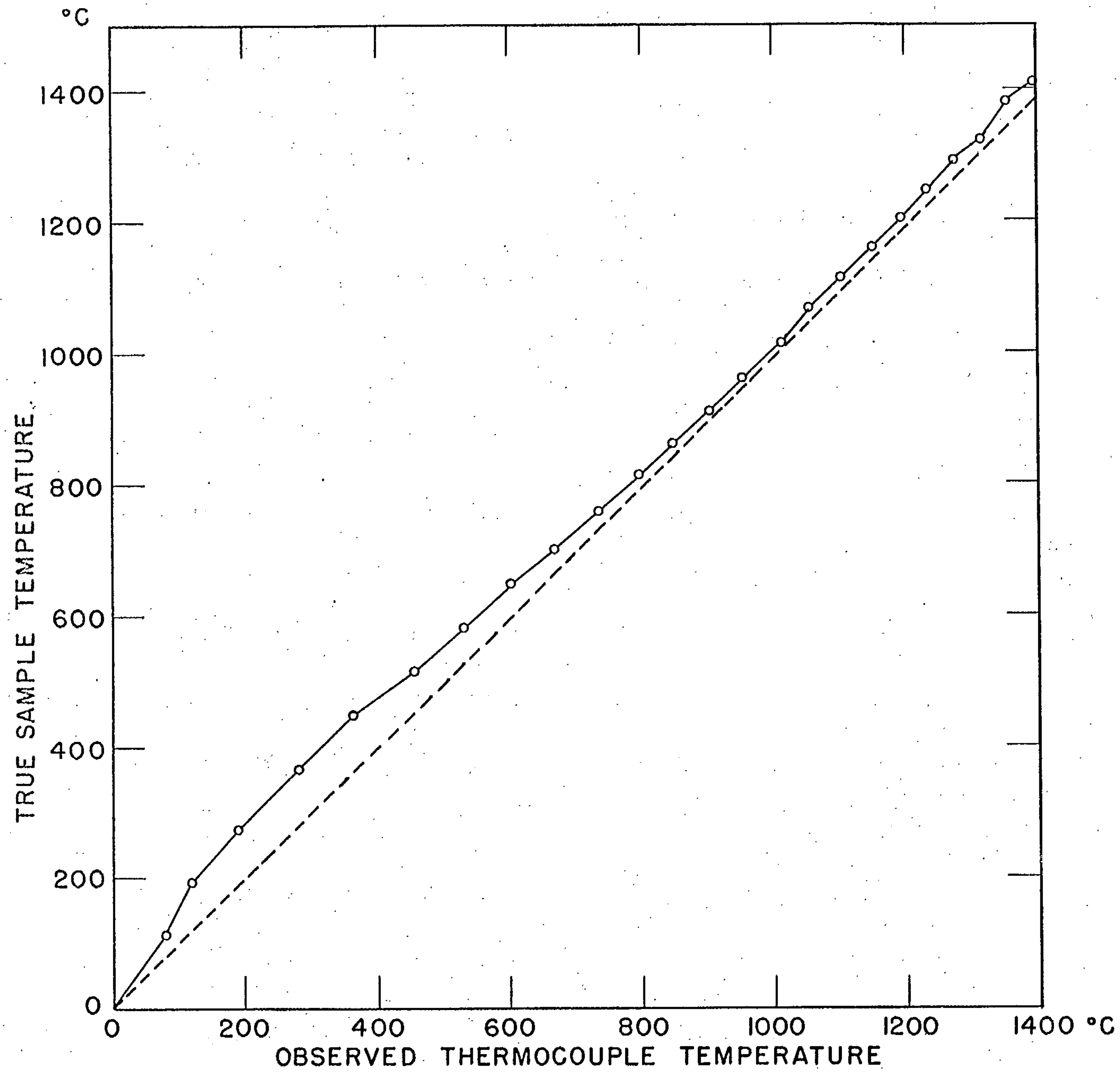

FIGURE 4. THERMOCOUPLE CALIBRATION CURVE OF THE S.150 HIGH-TEMPERATURE X-RAY CAMERA.

Further work is required on suitable refractory materials as standards which might yield more reproducible results. There are two possible kinds of material which may be used as calibration standards, namely, a refractory metal, such as tungsten, which has been used by Brand and Goldschmidt (3), and refractory oxides such as alumina or magnesia, which have been used as secondary standards by Campbell et al. (13). It is realized that alumina has a hexagonal structure, whereas it has already been stated (page 3) that a cubic structure is desirable for this purpose. However, alumina is a chemically stable material having accurately known crystallographic constants and reliable stoichiometry. It can readily be obtained well-crystallized in a state of high purity, and should prove to be a satisfactory calibration material.

It is intended to explore the possibility of the use of tungsten metal and of alumina in order to establish a reliable calibration curve for use in the temperature range 1000°C to 1400°C.

## CONCLUSIONS

1. The thermocouple of the Unicam S.150 high-temperature X-ray camera may be calibrated to within ± 10°C up to about 1000°C by the method outlined in this report, namely the thermal expansion of platinum.

2. It is recommended that calibration above 1000°C

should not be attempted with platinum, because of a marked deterioration in precision, probably as a result of grain growth and of other causes as yet undetermined.

## REFERENCES

1. F. Ferguson, R.S. Street and R.W.M. D'Eye, "High-Temperature X-ray Diffraction Studies: Part I", AERE (Harwell) Report R3344 (1960).

2. Z.S. Basinski, W.B. Pearson and J.W. Christian, "Furnace Construction and Thermocouple Arrangements for a High-Temperature X-ray Camera", J. of Sci. Instr. 29, 154 (1951).

3. J.A.Brand and H.J. Goldschmidt, "The Temperature Calibration of a High-Temperature X-ray Diffraction Camera", J. of Sci. Instr. 33, 41 (1956).

4. R.L. Berry, W.G. Henry and G.V. Raynor, "The Manipulation and Temperature Calibration of High-Temperature Debye-Scherrer X-ray Cameras", J. Inst. Metals 78, 643 (1951).

5. E.A. Owen and E.L. Yates, "Precision Measurements of Crystal Parameters", Phil. Mag. 15, 472 (1933).

6. K. Moeller, "Über Präzisionbestimmungen von Gitterkonstanten nach der Methode von Debye-Scherrer", Zeit. f. Krist. 97, 170 (1937).

7. H.E. Swanson and E. Tatge, "Standard X-ray Diffraction Powder Patterns", National Bureau of Standards Circular 539, Vol. I (1953), p. 31.

8. W.J. Campbell, S. Secura and C. Grain, "High-Temperature Furnaces for X-ray Diffractometers", U.S. Bureau of Mines Report of Investigations RI 5738 (1961).

9. W.B. Pearson, "A Handbook of Lattice Spacings and Structures of Metals and Alloys", Pergamon Press, 1st edition, p. 68 (1958).

10. H. Esser and H. Eusterbrook, "Thermal Expansion of Some Metals and Alloys", Arch. Eisenhüttenwesen <u>14</u>, 341 (1941).

11. H. Warlimont, "Eine Versuchsanordnung und Beispiele zur Kontinuierlichen Strukturanalyse mit Röntgenstrahlen", Zeit. f. Metallkunde <u>50</u>, 708 (1959).

12. L. Calvert, Division of Applied Chemistry, National Research Council, Ottawa, Ontario, Canada, Private communication.

13. W.J. Campbell and C. Grain, "Thermal Expansion of Alpha Alumina", U.S. Bureau of Mines Report of Investigations RI 5757 (1961).

## ACKNOWLEDGMENT

The author wishes to thank Dr. L.D. Calvert of the Applied Chemistry Division, National Research Council, Ottawa, for his advice on the operation of the Unicam S.150 high-temperature camera.

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(Appendices A, B and C )
(follow on pages 20 to 23)

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# APPENDIX A

## CALCULATION OF STANDARD CURVE OF LATTICE PARAMETER OF PLATINUM AS A FUNCTION OF TEMPERATURE

$$a_t = a_o \left[ 1 + 8.988 \times 10^{-6} t + 1.065 \times 10^{-9} t^2 + 1.26 \times 10^{-13} t^3 \right]$$

| t (°C) | $8.988 \times 10^{-6} t$ | $1.065 \times 10^{-9} t^2$ | $1.26 \times 10^{-13} t^3$ | $\sum$ | K | $a_t = a_o \times K$ |
|---|---|---|---|---|---|---|
| 50 | $4.494 \times 10^{-4}$ | $0.027 \times 10^{-4}$ | $0.0002 \times 10^{-4}$ | $4.5212 \times 10^{-4}$ | 1.000452 | 3.9235 |
| 100 | 8.988 | 0.1065 | 0.00126 | 9.0958 | 1.000910 | 3.9253 |
| 150 | 13.482 | 0.2396 | 0.0043 | 13.7259 | 1.001373 | 3.9271 |
| 200 | 17.976 | 0.4260 | 0.0101 | 18.4121 | 1.001841 | 3.9289 |
| 250 | 22.470 | 0.6656 | 0.0197 | 23.1553 | 1.002316 | 3.9308 |
| 300 | 26.964 | 0.9585 | 0.0340 | 27.9565 | 1.002796 | 3.9327 |
| 350 | 31.458 | 1.305 | 0.054 | 32.817 | 1.003282 | 3.9346 |
| 400 | 35.952 | 1.704 | 0.081 | 37.737 | 1.003774 | 3.9365 |
| 450 | 40.446 | 2.157 | 0.115 | 42.718 | 1.004272 | 3.9385 |
| 500 | 44.940 | 2.663 | 0.158 | 47.761 | 1.004776 | 3.9404 |
| 550 | 49.434 | 3.222 | 0.210 | 52.866 | 1.005287 | 3.9424 |
| 600 | 53.928 | 3.834 | 0.272 | 58.088 | 1.005809 | 3.9445 |
| 650 | 58.422 | 4.500 | 0.346 | 63.268 | 1.006327 | 3.9465 |
| 700 | 62.916 | 5.219 | 0.432 | 68.567 | 1.006857 | 3.9486 |
| 750 | 67.410 | 5.991 | 0.532 | 73.933 | 1.007393 | 3.9507 |
| 800 | 71.904 | 6.816 | 0.645 | 79.365 | 1.007937 | 3.9528 |
| 850 | 76.398 | 7.695 | 0.774 | 84.867 | 1.008487 | 3.9550 |
| 900 | 80.892 | 8.627 | 0.919 | 90.438 | 1.009044 | 3.9572 |
| 950 | 85.386 | 9.612 | 1.080 | 96.078 | 1.009608 | 3.9594 |
| 1000 | 89.880 | 10.650 | 1.260 | 101.79 | 1.010179 | 3.9616 |
| 1050 | 94.374 | 11.742 | 1.459 | 107.58 | 1.010758 | 3.9639 |
| 1100 | 98.868 | 12.887 | 1.677 | 113.43 | 1.011343 | 3.9662 |
| 1150 | 103.362 | 14.085 | 1.916 | 119.36 | 1.011936 | 3.9685 |
| 1200 | 107.851 | 15.336 | 2.177 | 125.37 | 1.012537 | 3.9709 |
| 1250 | 112.350 | 16.641 | 2.461 | 131.45 | 1.013145 | 3.9733 |
| 1300 | 116.844 | 17.999 | 2.768 | 137.61 | 1.013761 | 3.9757 |
| 1350 | 121.338 | 19.410 | 3.100 | 143.85 | 1.014385 | 3.9781 |
| 1400 | 125.832 | 20.874 | 3.457 | 150.16 | 1.015016 | 3.9806 |

# APPENDIX B

## EXPERIMENTAL DATA FOR THE ERROR CURVE

| Run No. | Current (amp) | Film No. | Lattice Spacing (A) | Equivalent Temperature (°C) | Thermocouple Temperature (°C) | Difference (deg. C) |
|---|---|---|---|---|---|---|
| 1 | 1.0 | T144 | 3.9237 | 59 | 45 | 14 |
| 2 | 1.5 | T145 | 3.9256 | 113 | 77 | 36 |
| 3 | 2.0 | T146 | 3.9287 | 196 | 120 | 76 |
| 4 | 2.5 | T150 | 3.9316 | 274 | 190 | 84 |
| 5 | 3.0 | T151 | 3.9353 | 369 | 279 | 90 |
| 6 | 3.5 | T152 | 3.9384 | 448 | 366 | 82 |
| 7 | 4.0 | T153 | 3.9410 | 516 | 454 | 62 |
| 8 | 4.5 | T154 | 3.9437 | 584 | 530 | 54 |
| 9 | 5.0 | T155 | 3.9464 | 650 | 606 | 44 |
| 10 | 5.5 | T156 | 3.9484 | 698 | 672 | 26 |
| 11 | 6.0 | T157 | 3.9509 | 757 | 734 | 23 |
| 12 | 6.5 | T158 | 3.9534 | 816 | 795 | 21 |
| 13 | 7.0 | T159 | 3.9556 | 866 | 852 | 14 |
| 14 | 7.5 | T160 | 3.9587 | 916 | 906 | 10 |
| 15 | 8.0 | T161 | 3.9599 | 964 | 955 | 9 |
| 16 | 8.5 | T162 | 3.9623 | 1017 | 1009 | 8 |
| 17 | 9.0 | T163 | 3.9647 | 1068 | 1055 | 13 |
| 18 | 9.5 | T164 | 3.9668 | 1114 | 1103 | 11 |
| 19 | 10.0 | T165 | 3.9691 | 1163 | 1151 | 12 |
| 20 | 10.5 | T166 | 3.9712 | 1209 | 1190 | 19 |
| 21 | 11.0 | T167 | 3.9732 | 1251 | 1236 | 15 |
| 22 | 11.5 | T168 | 3.9753 | 1294 | 1273 | 21 |
| 23 | 12.0 | T169 | 3.9768 | 1324 | 1313 | 11 |
| 24 | 12.5 | T170 | 3.9796 | 1382 | 1354 | 28 |
| 25 | 13.0 | T171 | 3.9813 | 1414 | 1393 | 21 |

# APPENDIX C

## NOTES ON THE OPERATION OF THE UNICAM S.150 HIGH-TEMPERATURE X-RAY POWDER DIFFRACTION CAMERA

### 1. Maintaining a Vacuum

The Unicam S.150 camera is operated under high vacuum (0.1-0.01 micron), which is difficult to maintain. The two principal sources of trouble are leakage of the aluminum window used to allow the diffracted rays to pass from the furnace chamber into the film cassette, and failure of the Wilson seal, which permits entry of the thermocouple leads. Pin-holes often appeared in the aluminum window, which could be effectively sealed with shellac. About every 2-3 months, the Wilson seal at the base of the instrument failed, probably because of deformation of the rubber gasket under pressure. New gaskets were easily cut, with suitably sized cork borers, from rubber sheeting of the correct size.

Since the Unicam S.150 is an all-metal instrument, leak detection is difficult. The instrument should be constantly monitored with a wide-range pressure gauge (1000-0.01 microns), preferably equipped with a leak detector of the hydrogen type.

### 2. Cooling Water System

The self-sealing valve assembly is subject to corrosion and to blockage from dirt if ordinary tap water is used. Also, tap water temperature ranges from 4°C to 28°C in Ottawa, which causes fluctuations in the performance of the furnace. It is highly recommended that a "closed-circuit" water-cooling system be used, controlled to 25 ± 5°C. The water should be distilled, oxygen-free, and should contain a corrosion inhibitor.

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