A high-temperature x-ray diffraction camera and its application to copper and titanium

Robert R. Penman

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A HIGH-TEMPERATURE X-RAY DIFFRACTION CAMERA
AND ITS APPLICATION TO COPPER AND TITANIUM

BY

ROBERT R. PENMAN

A

THESIS

submitted to the faculty of the

SCHOOL OF MINES AND METALLURGY OF THE UNIVERSITY OF MISSOURI

in partial fulfillment of the work required for the

Degree of

MASTER OF SCIENCE IN METALLURGICAL ENGINEERING

Rolla, Missouri

1950

Approved by

D. S. Eppleisheimer
Professor of
Metallurgical Engineering
ACKNOWLEDGMENT

The writer is grateful to Dr. D. S. Eppelsheimer for suggesting the present investigation, and for the encouragement and guidance he received in carrying out this project. The writer is also indebted to Dr. A. W. Schlechten and other members of the Missouri School of Mines faculty who gave willingly and freely of their knowledge and time throughout this work.
CONTENTS

Acknowledgment ................................................................................... ii
List of Tables ................................................................. iv
List of Figures ................................................................. v
Introduction ................................................................. 1
Literature Review ............................................................. 3
Section I: A 19-cm High-Temperature X-Ray Diffraction Camera .......... 3
Section II: Thermal Dilation of Copper ........................................... 6
Section III: Accurate Determination of Lattice Parameter of Beta Titanium at 900 °C .............................................................. 7
Discussion ................................................................. 9
Section I: A 19-cm High-Temperature-Vacuum Camera and its Operation................................................................. 9
Section II: Thermal Dilation of Copper ........................................... 23
Section III: Accurate Determination of Lattice Parameter of Beta-Titanium at 900 °C .............................................................. 31
Conclusions ............................................................... 36
Summary ................................................................. 37
Appendix ................................................................. 38
Bibliography ............................................................... 43
Vita ................................................................. 44
### LIST OF TABLES

<table>
<thead>
<tr>
<th>Table</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>I.</td>
<td>Chemical Analysis of Copper Used</td>
<td>24</td>
</tr>
<tr>
<td>II.</td>
<td>List of Lattice Constants for Copper Between $18^\circ$ - $770^\circ$ C, Using Quadratic Form of Bragg's Law</td>
<td>26</td>
</tr>
<tr>
<td>III.</td>
<td>Data for Accurate Determination of Lattice Constant of Copper at Room Temperature, Using Method of Least Squares</td>
<td>29</td>
</tr>
<tr>
<td>IV.</td>
<td>List of Lattice Constants for Copper Between $18^\circ$ - $770^\circ$ C, Using Method of Least Squares</td>
<td>30</td>
</tr>
<tr>
<td>V.</td>
<td>Data for Accurate Determination of Lattice Constant of Titanium at $900^\circ$ C, Using Cobalt Radiation</td>
<td>32</td>
</tr>
<tr>
<td>VI.</td>
<td>Data for Accurate Determination of Lattice Constant of Titanium at $900^\circ$ C, Using Iron Radiation</td>
<td>33</td>
</tr>
<tr>
<td>VII.</td>
<td>Data for Accurate Determination of Lattice Constant of Titanium at $900^\circ$ C, Using Copper Radiation</td>
<td>34</td>
</tr>
</tbody>
</table>
**LIST OF FIGURES**

<table>
<thead>
<tr>
<th>Figure</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Schematic Diagram of 19-cm High-Temperature Powder Camera</td>
<td>14</td>
</tr>
<tr>
<td></td>
<td>(Key to Figure 1)</td>
<td>15</td>
</tr>
<tr>
<td>2.</td>
<td>Plan View of 19-cm High-Temperature Powder Camera</td>
<td>16</td>
</tr>
<tr>
<td></td>
<td>Showing Position of Furnace and Connections</td>
<td></td>
</tr>
<tr>
<td>3-A.</td>
<td>Photograph Showing Camera, X-Ray Tube, Rheostats and Direct Reading Temperature Indicator</td>
<td>17</td>
</tr>
<tr>
<td>3-B.</td>
<td>Photograph Showing Camera, X-Ray Tube and Vacuum Pump</td>
<td>17</td>
</tr>
<tr>
<td>4.</td>
<td>Schematic Diagram of Original Bradley-Jay 19-cm High-Temperature Powder Camera</td>
<td>18</td>
</tr>
<tr>
<td>5.</td>
<td>Schematic Diagram of Back Reflection Type High Temperature X-Ray Diffraction Camera</td>
<td>20</td>
</tr>
<tr>
<td>6-A.</td>
<td>Reproduction Showing Diffraction Pattern of Swaged Copper With Normally Collimated Beam</td>
<td>22</td>
</tr>
<tr>
<td>6-B.</td>
<td>Reproduction Showing Diffraction Pattern of Swaged Copper With Added Collimation</td>
<td>22</td>
</tr>
<tr>
<td>7.</td>
<td>Graph of Temperature Versus Lattice Constant of Copper Using Specific Planes in the Lattice</td>
<td>27</td>
</tr>
<tr>
<td>8.</td>
<td>Schematic Diagrams of Usual Film Arrangements in Debye-Scherrer Cameras</td>
<td>39</td>
</tr>
</tbody>
</table>
INTRODUCTION

The purpose of this research was threefold, and consists of the following sections:

(1) Effective use of a high temperature x-ray camera, capable of reaching temperatures of 1000 °C.

(2) Investigation of the thermal dilation of certain cubic metals.

(3) Accurate determination of the lattice constant (\(a_0\)) of Beta Titanium at 900 °C ± 5 °C.

Many high temperature cameras have been described in the literature, but the one used in these investigations was constructed by Unicam Limited of Cambridge, England, and was modeled after the original Bradley-Jay design which was described by A. J. C. Wilson \(^{(1)}\).

Reasons and advantages for using a 19-cm high-temperature camera are numerous. For instance, it is useful in phase determinations for phases that cannot be retained by ordinary quenching methods, e.g., gamma iron. A 19-cm camera is especially useful in particle size determinations where the particle size is greater than 30 \(\mu m\). Subtle differences in the x-ray spectra of macrocrystalline materials are easily missed in smaller cameras; the larger crystallites are more easily resolved in a camera of large diameter.

In consideration of the thermal dilation of metals, it was observed that certain so called cubic metals exhibited the anomaly of anisotropy in their thermal expansion \(^{(2)}\).
It is a well known fact that certain metals crystallizing in the cubic system exhibit anisotropy of the modulus of elasticity (E). Because of the anisotropy of the elasticity modulus and possible departure from Hooke's law, there must be a relationship between the departure from Hooke's law and the linear thermal dilation. Any anisotropy in certain crystallographic directions with respect to Hooke's law, would also mean that there would be anisotropy in the thermal dilation. Therefore, the investigation of the thermal dilation of different crystallographic directions would serve a dual purpose. Copper was the cubic metal, (face-centered cubic) examined by the author since it was thought that it would be a truly representative and common cubic metal. In studying the thermal dilation of copper, the lattice constant (a₀) was calculated for two different back reflection planes and plotted against temperature.

The final part of this thesis was undertaken because of the great amount of interest in the alloying nature of titanium, and the increased purity of titanium metal over that available to earlier investigators. Also, the present method of x-ray analysis of the specimen at elevated temperatures over that of the original investigators is much improved.

In view of this interest in fundamental information, increased purity of material and improvements in the method of x-ray analysis, this investigation of Beta Titanium was considered worthwhile.
LITERATURE REVIEW

Section I:

Many high-temperature x-ray cameras have been designed by various investigators, but probably the most successful has been the one originally designed by Bradley and Jay. Their high-temperature camera was developed along the lines of the original 19-cm camera developed in Sir Lawrence Bragg's laboratories\(^1\). For rapid exposures and materials of poorly developed crystallinity, a small camera is favored, but for complicated patterns or conditions of highly developed crystallinity such as found in metals, it is better to use a camera of large diameter. For special problems, cameras as large as 35-cm in diameter have been used.

A camera of this general type was used by Hume-Rothery and Andrews to determine the lattice spacings and thermal coefficient of copper\(^2\).

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gradients. Temperatures were measured by means of a 3-mm diameter platinum ring welded to a platinum and platinum-rhodium thermocouple. This device was adjustable vertically in order to determine the temperature at various points in the furnace heating zone. This camera was one of the forerunners of the present improved model, with improvements as suggested by W. L. Bragg(4).


In his recommendations, Bragg stated that the following conditions must apply: "The temperature of the specimen must be uniform, or the lines will be diffuse because different parts of the specimen have undergone thermal expansion to a different extent. It must therefore be almost completely surrounded by a furnace so that it is effectively in a constant temperature enclosure, with only a narrow slit to allow the incident and diffracted x-rays to pass.

Although the furnace, which may be at a temperature of 1000 °C or more, is only a few centimetres distant from the film, the latter must be so efficiently water cooled that its temperature does not rise. The thermal insulation must therefore be very good".

When not being used as a high-temperature camera, but at room temperature, there are other advantages to a 19-cm camera. A larger camera gives greater accuracy and better resolution of the diffracted beams, due to its greater divergence of the cones of diffraction, the greater the distance from the origin of these cones. The exposure varies with the material being examined, but is approximately 4-5 times longer than the average smaller cameras (9-14 cms). It is possible
to obtain an accuracy in lattice constant determinations of the order of one part in 20,000 or even one part in 100,000 if the perfection of the crystalline structure permits (5).

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A high-temperature 19-cm camera is ideally suited to metallurgical investigations, because it is more suited to grain sizes above 30 Å. Grain sizes of metals seldom if ever get below 30 Å, except for colloidal metals and some electrodeposited metals such as antimony. For measurements below 30 Å, a 9-cm camera is preferred (6).

---


If it were possible to resolve the particle sizes below 30 Å in a 19-cm camera, it would necessarily require extremely fine collimation, and an inordinately long exposure time. The smaller camera achieves the same results in a much shorter time. It is a known fact that as the particle size gets smaller, the fineness of collimation must also increase in order to get better resolution (7).

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Goss states that W. A. Wood (8) indicates, "cold working cannot

---

fragment the grains of a metal beyond about $10^{-5}$ cm ($1000 \text{ Å}$), and if smaller fragment sizes are reached, they immediately recrystallize into regions of $10^{-5}$ cm or larger. If this is the case, an annealed metal which would have grains of at least $10^{-5}$ cm or larger, and a cold worked metal would recrystallize and show grain growth while at elevated temperatures. Therefore the use of a 19-cm camera is recommended since the grain size for metals would always be above the 30 Å limit.

**Section II:**

The thermal expansion of cubic and non-cubic metals has been of long standing interest, and in general the thermal dilation of cubic metals has been accepted as being isotropic. A departure from this accepted view was reported recently in the literature for iron throughout a range from 22-300 °C. Kochanovska reasoned that since some metals which crystallize in the cubic system exhibit anisotropy of the elasticity modulus for single crystals of iron, aluminum and alpha brass, there might be some question as to their behavior in the polycrystalline state with regards to their linear thermal dilation. Anisotropy in the thermal dilation, she reasoned, would also lead to departures from Hooke's law and the investigation would be of double interest.

In her investigations, Kochanovska used the back reflection method and obtained four photographs on the same film; different radiations were used for different planes in order to obtain each reflection at

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approximately the same Bragg angle. The furnace was around the specimen with an iron-constantan thermocouple attached to the back of the specimen. The temperature range, in which this original work was carried out, ranged from 22 - 366 °C ± 2°. Her calculations were carried out by using the quadratic form of Bragg's law for cubic crystals,

\[ a_0 = \frac{\lambda \sqrt{h^2 + k^2 + l^2}}{2 \sin \Theta} \]

where \( a_0 \) = lattice constant

\( \lambda \) = wave length of radiation used

\( \Theta \) = Bragg Angle

\( h^2 + k^2 + l^2 \) = Planes causing diffraction

For purposes of comparison, the same formula will be applied by the author in his calculations.

Section III:

The original determination of the accurate lattice parameter of Beta Titanium at 900 °C was carried out by Burgers and Jacobs(10).


In their determination, Burger and Jacobs used a 300 micron titanium wire, of unstated purity, as the filament in the center of a Lindemann-glass bulb. This bulb was then placed in the center of a Debye-Scherrer camera and rotated, with the titanium wire filament being heated to about 900 °C.

The phase transformation temperature for titanium has been determined very accurately by electrical conductivity methods to be 885 °C ± 2°(11); the Beta form being body-centered cubic is stable above 900 °C.
The lattice constant arrived at by these original investigators was 3.32 Å, and is the only determination that can be found for the Beta phase of titanium. Numerous values have been reported for the room temperature or Alpha phase of titanium.
DISCUSSION

I. A 19-cm High-Temperature-Vacuum Camera and its Operation.

The instrument used in this examination was a 19-cm high-temperature-vacuum type camera, constructed by Unicam of Cambridge, England. The camera is compact and easy to handle, and is capable of reaching temperatures up to 1000°C. It consists of four main parts; camera, evacuation chamber, furnace and main base. The camera can be removed from the instrument without disturbing the vacuum, the temperature of the furnace, the specimen or the cooling system. Several photographs can, therefore, be taken with one or more cameras.

The camera itself follows closely the construction of the Unicam model of the 19-cm Bradley-Jay Powder Camera. It is, however, somewhat stronger in its construction and provides protection of the film from thermal damage. There is absolutely no chance for the heat from the furnace chamber reaching the film and causing undue thermal expansion of the film during exposure. The steel knife edges are arranged to give a fiducial shadow at Bragg angles of 5° in the forward reflection region, and at 85° in the back reflection region. Two separate strips of film are used, one on each side. By using Cohen's(1) method of

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least squares, only one side of the camera need be used in a determination. It is simpler than the various analytical and graphical methods previously used, and in effect the powder camera becomes an absolute instrument independent of calibrating substances, and dependent only on the value of the x-ray wave length. Film used in the camera is
standard 35-mm film, with each strip being 1-3/8 x 11 inches, and identification niches are placed on the camera rings to distinguish the right and left hand film; also the top edge from the bottom. One collimating slit, 4 mm x 0.8 mm, is incorporated in the camera, and this system gives sharp, well defined lines. A simple labyrinth system has been devised to give necessary x-ray protection to the film, and prevents any fogging by scattered x-rays.

The evacuation chamber is necessary in order to prevent oxidation of the specimen at high temperatures, and prevent convection currents cooling the furnace. A cellophane window is supported by outer recesses which are water cooled to provide thermal protection to the cellophane window. A Cenco-Hyvac pump, capable of producing a vacuum of 0.3 micron, was coupled to the evacuation chamber, and run for at least one hour prior to a high-temperature analysis. No difficulty was experienced with the cellophane window, and it withstood the vacuum readily.

The furnace is so designed that the heat is concentrated at the center, and a constant temperature is maintained. It is wound with a platinum-rhodium alloy wire, and gives satisfactory performance up to 1000°C. It is readily removed from the instrument by disconnecting the current and thermocouple leads, and is so guided that it is impossible to damage a specimen in the process. Provision is made for reading the temperature by a standard indicator or by a potentiometer. The present standard indicator is not sufficiently accurate, and a Leeds and Northrup potentiometer was used for very accurate determinations.

Temperature readings are obtained from two points near the center of the furnace by means of two Pt-Pt/Rh thermocouples (13% rhodium). One couple (#1) coming down from the top of the furnace is adjustable
in position, and may be placed very near the specimen. The second couple (#2) comes in at the side and bottom of the heated specimen. The couples are connected to the indicating instrument by means of a long flexible compensating lead. A United States Bureau of Standards Pt-Pt/Rh thermocouple was used to check the thermocouples in the furnace, and they were found to be accurate within ±1°.

For determinations at room temperature, the furnace must still be left mounted in its normal position since it has the slit system and beam catcher in its assembly. The collimating slit and beam catcher are made up of a non-corrosive steel, and the whole of the metal work of the furnace is nickel plated so that it is little affected by high temperatures. The plates which form the top and bottom of the furnace are moulded from alumina cement, and are especially suitable for this work. The furnace contains no material which is volatile under 1000° C, and care must be exercised that no specimens which can be volatilized below this temperature be examined openly. Such materials can be analyzed by sealing the powder in thin walled silica tubes.

The main base consists of a strong circular casting which houses the magnetic drive to the centering eccentrics, the eccentrics themselves, the lower cooling jacket and control panel. The evacuation pipe connection is taken out of the rear of the instrument, and is centrally placed between the connecting pipes of the lower cooling system, and provides protection against any water coming near the electrical circuits. Control panel fittings on the front of the instrument consists of the three switches necessary for the two heaters and the motor, four protecting fuses, terminals for connecting the rheostats into the heating circuits, and the plug type key for the thermocouples.
All controls are readily accessible and easy to use when operating the instrument. The base of the casting is provided with three standard leveling screws, and a spirit level for aid in setting up the camera. The spirit level is of little or no practical value, however, since the x-ray beam from the diffraction unit comes out an angle of 5° to 8° from the horizontal. Adjustment was accomplished by observing the position of the specimen on a Patterson type "B" fluorescent screen pasted to the outside exit cover on the camera. The leveling screws were thus adjusted to obtain maximum efficiency from the beam.

Rheostats for controlling the current to the heating elements of the furnace are mounted on the wall behind the x-ray machine (see figure 3-A and 3-B), and are always started with full resistance in the circuit. Any attempts to heat the furnaces too rapidly results in blowing the one ampere fuses in the heater circuits. It normally took from three-quarters to one hour to heat the camera from room temperature to 900°C. The current at full load is about one ampere on each half of the furnace, and each half is separately fused.

The method of centering the specimen is accomplished through a series of three eccentrics, being the most compact system for the limited space available below the heating chamber. Constructed of Invar, the eccentrics are mounted directly on top of the armature shaft, and the specimen can be either in the form of a wire, or powdered and sealed in a fused silica tube. The armature is supported and pivotted on a specially designed sapphire mounting, and at the bottom it carries a follower which is rotated by a powerful magnet located under the evacuation chamber. This magnet is driven by an electric motor which is geared to give the specimen a rotation of
1 r.p. 7.5 m.; this speed was chosen to avoid frequency banding.

In the operation of the present camera, probably the greatest difficulty arises from the existing method of centering the specimen. The process of centering the specimen is accomplished by using a horizontal microscope, and lining the specimen up on a set of cross hairs by means of the three aforementioned eccentrics. The process is extremely tedious and time consuming, since any minute eccentricity in the specimen will be aggravated at high temperatures. The author attempted to improve upon the present system by replacing the uppermost eccentric by means of a three-screw fixture and adjusting the specimen by use of these screws. This method was a decided improvement over the original method, and a permanent fixture was machined out of pure nickel. It showed no effects of oxidation at temperatures up to 900°C.

Figure 1, courtesy of Unicam Limited, shows a cross sectional view of the 19-cm camera used in this investigation, and is explained by the Key to Figure 1 on page 15. Figure 2 is a plan view of the camera, illustrating the relative position of the furnace, furnace connections and the camera (cassette). Figure 3 illustrates the camera in its operating position on the x-ray machine, and the related operating parts.

Other high-temperature x-ray cameras have been devised, but the original 19-cm high-temperature camera designed by Bradley and Jay(2)


is illustrated by Figure 4. It is readily apparent that the present
19 CM. HIGH TEMPERATURE POWDER CAMERA.
KEY TO FIGURE 1.

A - Complete Camera
B - Evacuation Chamber
C - Locating Ring
D - Locating Pin
E - Labyrinth Pieces
F - Rubber Vacuum Seat
G - Locating Pin for Evacuation Chamber
H - Cellophane Window
I - Heater and Thermocouple Terminals
J - High Temperature Furnace
K - Furnace Guide and Locator
L - Screws Holding Furnace Guide (K)
M - Furnace Locating Pin
N - Spider Clamp - Thermocouple #1
S - Projections of Labyrinth System
T - Top Edge Furnace Guide (K)
U - Vacuum Pump Connection
V - Cooling Water Connections
W - Base Casting
X - Compensating Leads From Thermocouple Switch
Y - Thermocouple Switch
Z - Filter Holder
AA - Square channel for camera cover
BB - Step forming light trap
CC - Light tight black paper
DD - Screws holding black paper
EE - Exposure ring blocks
E' - Armature shaft
FF - X-Ray entrance slit
GG - Locating pin for entrance slit
Figure 3-A. Showing Camera, X-Ray Tube, Rheostats and Temperature Indicator

Figure 3-B. Showing Camera, X-Ray Tube and Vacuum Pump
Unicam model has many improvements over this older, original model, although it still incorporates the good principles of this earlier camera.

Figure 5 shows the details of construction of a back-reflection high temperature x-ray camera, used in analyzing beryllium\(^3\). While undoubtedly an excellent camera, it does not incorporate the recommendations for a high temperature type of camera as stated by W. L. Bragg\(^4\).


\(^4\) Bragg, W.L., op. cit., p. 4.

It has the possible disadvantage that the temperature might not be uniform, since the furnace is just around the sides, and a thermal gradient could exist within the specimen.

Specimens for use in the camera used by the author were produced by swaging, and were taken down to a final diameter of 0.050 inches. Further reductions were either obtained by acid etching or sanding down on a belt sander. The etching method is preferred, since it removes any stressed metal from the surface and does a more uniform job. Etching and final sanding are done only after the specimens have been annealed.

Since a 19-cm camera is so useful in metallurgical grain size determinations, an investigation of the effect of collimation and grain size was undertaken\(^5\). An x-ray diffraction pattern was ob-

\(^5\) Goss, N.P., op. cit., p. 5.
Figure 5.
tained from swaged copper at room temperature with the standard col-
limating system, 4mm x 0.8mm. A second x-ray diffraction pattern was
run, using a circular collimating system of 0.16 mm diameter. This
gave a finely collimated pencil of x-rays, and decided improvement
in the resolution. Figure 6-A shows the diffraction pattern using
the original collimating system, and Figure 6-B shows the effects of
increased collimation. It is interesting to note also, that there
is little or no difference in the intensities of the lines, except
that Figure 6-B has its lines more concentrated and Figure 6-A is
more diffuse and over a greater area. Both examinations were for
4 hours at 35 KVP and 15 ma. To achieve the ultimate results by
fine collimation, a longer exposure would undoubtedly be required.
It has been previously stated that actual lattice distortion does
not take place during cold working, but that the crystallites are
so finely broken down that a large beam of x-rays cannot be resolved
as readily as a finely collimated pencil of x-rays\(^6\).

\(^6\) Goss, N.P., Ibid.

Method of calculations, and film arrangements (Figure 8) are
shown in the Appendix.
Figure 6-A  Swaged Copper Without Added Collimation

Cu - Swaged - e.i. Collimation

Figure 6-B  Swaged Copper With Added Collimation
II. **Thermal Dilation of Copper.**

It was previously stated by Kochanovska\(^{(7)}\) that certain cubic metals, although considered cubic, might not be precisely cubic throughout a range of temperatures below their melting points. In the event that this is the case, such departures along with the departure from Hooke's law, would indicate that the thermal dilation is not isotropic, and that the thermal dilation is anisotropic.

For the purpose of studying the thermal dilation of cubic metals, Kochanovska investigated the thermal dilation of iron, choosing the (013) and (112) planes between 22-366 °C. Although iron exhibits the phenomena of allotropy, her examinations were well below the observed allotropic transformation temperature of 909 °C for pure iron. In her investigation a definite departure from isotropic dilation was observed when the lattice constant for each set of planes was plotted versus temperature. It was this departure, where departure was not normally expected, that prompted the present investigation by the author.

As copper is a common and representative cubic metal, it was chosen for purposes of examining its thermal dilation. The (024) and (331) planes were chosen as the planes to be investigated, and the temperature ranged between 18-770 °C. The copper used was produced by the National Research Corporation, Cambridge, Mass., and the analysis is given in Table I. Specimens for each examination were prepared by swaging down to 0.050 inches, annealed at 200 °C for 4 hours in cast iron chips, and further reduced down by etching in concentrated \(\text{HNO}_3\).
<table>
<thead>
<tr>
<th>Element</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe</td>
<td>0.0015 %</td>
</tr>
<tr>
<td>Sb</td>
<td>0.0009</td>
</tr>
<tr>
<td>Pb</td>
<td>0.0008</td>
</tr>
<tr>
<td>Sn</td>
<td>0.0004</td>
</tr>
<tr>
<td>Ni</td>
<td>0.0004</td>
</tr>
<tr>
<td>Bi</td>
<td>0.0002</td>
</tr>
<tr>
<td>As</td>
<td>0.0010</td>
</tr>
<tr>
<td>Te</td>
<td>0.0003</td>
</tr>
<tr>
<td>Ag</td>
<td>0.0031</td>
</tr>
<tr>
<td>S</td>
<td>0.0001</td>
</tr>
<tr>
<td>O</td>
<td>0.00004</td>
</tr>
<tr>
<td>H</td>
<td>0.0000085</td>
</tr>
</tbody>
</table>

Total Impurity = 0.0087485 %

Purity of Copper = 99.9912515 %
Copper radiation was used and the quadratic form of Bragg's law was applied.

\[ a_0 = \sqrt{\frac{h^2 + k^2 + l^2}{2 \sin \theta}} \]

The data obtained by using the above equation was uncorrected, and is listed in Table II. No attempts were made to correct the data for errors due to eccentricity of the specimen, film shrinkage, absorption in the specimen, or operator errors. All such errors would be incorporated in each film, and would be approximately the same for each line being compared on individual films. A direct reading temperature indicator was used for convenience, in lieu of a potentiometer. While not as accurate as a potentiometer, it gave a constant temperature reading which was desired and achieved. The lines on the film were clear and distinct with no indication of fuzziness, indicating that the temperature was constant in the furnace \( \pm 5^\circ \).

From the data in Table II, a plot was made of the lattice constant "a₀" versus temperatures in °C, Figure 7. It is readily seen from the data, and plot of the data, that copper does not exhibit any anisotropy in its thermal dilation, and is precisely cubic throughout the temperature range investigated.

In view of the fact that no attempt was made to correct the data in Table II for the purpose of investigating the thermal dilation of copper, corrections were applied to demonstrate the actual accuracy of the data. Cohen's\(^{(8)}\) method of least squares was applied to the data

\(^{(8)}\) Cohen, M.U., *op. cit.*, p. 9
TABLE II
A LIST OF LATTICE CONSTANTS FOR VARIOUS TEMPERATURES
USING SPECIFIC PLANES IN THE LATTICE OF COPPER

<table>
<thead>
<tr>
<th>Temperature °C</th>
<th>( \Theta )°</th>
<th>Plane</th>
<th>&quot;a₀&quot; in Å</th>
</tr>
</thead>
<tbody>
<tr>
<td>18</td>
<td>72.43</td>
<td>(024) ( \alpha ), (331) ( \alpha )</td>
<td>3.6059 = 3.61</td>
</tr>
<tr>
<td>260</td>
<td>71.77</td>
<td>(024) ( \alpha ), (331) ( \alpha )</td>
<td>3.6194 = 3.62</td>
</tr>
<tr>
<td>450</td>
<td>71.17</td>
<td>(024) ( \alpha ), (331) ( \alpha )</td>
<td>3.6321 = 3.63</td>
</tr>
<tr>
<td>770</td>
<td>70.33</td>
<td>(024) ( \alpha ), (331) ( \alpha )</td>
<td>3.6507 = 3.65</td>
</tr>
</tbody>
</table>
Temperature °C vs. Lattice Constant "a₀" Å for Copper

x-(024) Plane
0-(331) Plane

FIGURE 7
in Table III for the room temperature x-ray determination on copper. Actually, the temperature of the specimen was that of the circulating water which flows through the cooling channels of the camera even when the heaters are not being used. The temperature during such determinations was $18^\circ \pm 2^\circ C$.

The deviation of the experimental lattice constant $a_0$, which was found to be $3.60790 \text{ Å}$, is 1:36,000 from the accepted value which is $3.6080 \text{ Å}$. The original lattice constant has been determined accurately to only four decimal places, being uncertain in the fourth place. Cohen's method is accurate to five places being uncertain in the fifth place. It is possible that the accuracy is even better than indicated.

Table IV lists the data for the separate temperatures of investigation, and the method of least squares was used in their calculation.
TABLE III
ACCURATE DETERMINATION OF THE LATTICE CONSTANT "$a_0" FOR COPPER USING METHOD OF LEAST SQUARES

<table>
<thead>
<tr>
<th>$\theta^\circ$</th>
<th>d</th>
<th>hk1</th>
<th>$\alpha$</th>
<th>$\alpha^2$</th>
<th>$\sin \theta$</th>
<th>$\sin^2 \theta$</th>
<th>$\sin^2 \varphi$</th>
</tr>
</thead>
<tbody>
<tr>
<td>72.91</td>
<td>0.8062</td>
<td>(024)$\alpha$, 20</td>
<td>400</td>
<td>0.95584</td>
<td>0.90909</td>
<td>0.56179</td>
<td></td>
</tr>
<tr>
<td>72.43</td>
<td>0.8064</td>
<td>(024)$\alpha$, 20</td>
<td>400</td>
<td>0.95335</td>
<td>0.90888</td>
<td>0.57558</td>
<td></td>
</tr>
<tr>
<td>68.71</td>
<td>0.8271</td>
<td>(331)$\alpha$, 19</td>
<td>361</td>
<td>0.93176</td>
<td>0.86387</td>
<td>0.67664</td>
<td></td>
</tr>
<tr>
<td>68.38</td>
<td>0.8269</td>
<td>(331)$\alpha$, 19</td>
<td>361</td>
<td>0.92965</td>
<td>0.86425</td>
<td>0.68505</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>$\sin^2 \varphi$</th>
<th>$\gamma$</th>
<th>$\gamma^2$</th>
<th>$\alpha \sin^2 \theta$</th>
<th>$\gamma \sin^2 \theta$</th>
<th>$\alpha \gamma$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.314039</td>
<td>3.14039</td>
<td>9.86205</td>
<td>18.18180</td>
<td>2.85490</td>
<td>62.80780</td>
</tr>
<tr>
<td>0.331292</td>
<td>3.31292</td>
<td>10.97544</td>
<td>18.17760</td>
<td>3.01105</td>
<td>66.25840</td>
</tr>
<tr>
<td>0.455571</td>
<td>4.55571</td>
<td>20.75450</td>
<td>16.41353</td>
<td>3.93554</td>
<td>86.59494</td>
</tr>
<tr>
<td>0.469294</td>
<td>4.69294</td>
<td>22.02369</td>
<td>16.42075</td>
<td>4.05587</td>
<td>89.16586</td>
</tr>
</tbody>
</table>

| 63.61568 | 69.19368 | 13.85736 | 304.79055 |

$\gamma = 1522 \quad A_0 \not= 304.79055 \quad D = 69.19368$

$\gamma = 304.79055 \quad A_0 \not= 63.61568 \quad D = 13.85736$

Solving the above two simultaneous equations:

$D = 0.00033678$

$A_0 = 0.045395$

"$a_0" = 3.60790 \text{ Å}""
ACCURATE DETERMINATION OF THE LATTICE CONSTANT \(a_o\) FOR COPPER AT VARIOUS TEMPERATURES USING THE METHOD OF LEAST SQUARES

<table>
<thead>
<tr>
<th>Temperature</th>
<th>18(^\circ) C</th>
<th>260(^\circ)</th>
<th>450(^\circ)</th>
<th>770(^\circ)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(a_o) in (\AA)</td>
<td>3.6079(_0)</td>
<td>3.6228(_7)</td>
<td>3.6360(_6)</td>
<td>3.6538(_6)</td>
</tr>
</tbody>
</table>
III. Accurate Determination of the Lattice Constant "\(a_0\)" of Beta Titanium at 900°C ± 5°C.

Although the original\(^{(9)}\) method of determining the high temper-

\(\text{(9) Burger, W.G., and Jacobs, F.M., op. cit., p. 7.}\)

ature lattice constant of titanium was ingenious, it is believed that the present determination will prove to be more accurate. The purity of the titanium wire used was not stated, and the temperature was stated to be about 900 °C.

Titanium used in this determination was supplied by the Remington Arma Corporation, and was reported to be 99.0\% Ti, 0.3\% C, O\(_2\), N\(_2\), Fe, a few hundredths to a few tenths percent each, and other elements a trace. The nature of the alloying elements and impurities were such as to have little effect on the high temperature modification, since their atomic diameters are such as to permit interstitial solid solutions. Titanium wire specimens were prepared by swaging down to 0.050 inches, and were then annealed at 1000 °C for one hour. The specimens were placed in cast iron chips and covered with a carbon plug during the annealing. Further reductions in size were achieved by grinding on a belt sander.

Many attempts were made to obtain a satisfactory x-ray diffraction pattern from titanium using various types of radiation, and a titanium target itself was tried. Due to the large radius of the camera and the weakness of the titanium radiation, a satisfactory diffraction pattern was not obtained. The three following radiations were used with success; cobalt, iron and copper, with the latter giving the best results of the three. Data for the three successful radiations at
TABLE V
TITANIUM (900 °C ± 5°) COBALT RADIATION

<table>
<thead>
<tr>
<th>θ°</th>
<th>d</th>
<th>hkl</th>
<th>λ</th>
<th>λ²</th>
<th>sin θ</th>
<th>sin²θ</th>
<th>sin 2θ</th>
</tr>
</thead>
<tbody>
<tr>
<td>41.40</td>
<td>1.3568</td>
<td>(211)γ 6</td>
<td>36</td>
<td>0.65790</td>
<td>0.43283</td>
<td>0.99093</td>
<td></td>
</tr>
<tr>
<td>58.75</td>
<td>1.0441</td>
<td>(310)γ 10</td>
<td>100</td>
<td>0.85491</td>
<td>0.73087</td>
<td>0.88701</td>
<td></td>
</tr>
<tr>
<td>58.90</td>
<td>1.0448</td>
<td>(310)γ 10</td>
<td>100</td>
<td>0.85627</td>
<td>0.73000</td>
<td>0.88458</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>sin²θ</th>
<th>ρ</th>
<th>λ²</th>
<th>λ sin²θ</th>
<th>ρ sin²θ</th>
<th>λρ</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.981942</td>
<td>9.31942</td>
<td>96.42101</td>
<td>2.59698</td>
<td>4.25014</td>
<td>58.91652</td>
</tr>
<tr>
<td>0.786787</td>
<td>7.86787</td>
<td>61.90338</td>
<td>7.30870</td>
<td>5.75039</td>
<td>78.67870</td>
</tr>
<tr>
<td>0.779070</td>
<td>7.79070</td>
<td>60.69501</td>
<td>7.30000</td>
<td>5.68721</td>
<td>77.90700</td>
</tr>
</tbody>
</table>

219.01940 17.20568 15.68774 215.50222

\[
\left(\frac{K \lambda_1}{K \lambda_2}\right)^2 = \left(\frac{1.78522}{1.78919}\right)^2 = 0.99564
\]

(215.50222)x 236 A₀ ≠ 215.50222 D = 17.20568

(236)x 215.50222 A₀ ≠ 219.01940 D = 15.68774

D = -0.0010587

A₀ = 0.073872

\[\sqrt{A₀} = 0.27179\]

σ₀ = 3.28432 Å
TABLE VI
TITANIUM (900 °C ± 5°) IRON RADIATION

<table>
<thead>
<tr>
<th>θ°</th>
<th>d</th>
<th>hkl</th>
<th>α</th>
<th>α²</th>
<th>sin θ</th>
<th>sin²θ</th>
<th>sin 2θ</th>
</tr>
</thead>
<tbody>
<tr>
<td>45.31</td>
<td>1.3589</td>
<td>(211)</td>
<td>6</td>
<td>36</td>
<td>0.71092</td>
<td>0.50541</td>
<td>0.99996</td>
</tr>
<tr>
<td>67.45</td>
<td>1.0460</td>
<td>(310)</td>
<td>10</td>
<td>100</td>
<td>0.92355</td>
<td>0.85294</td>
<td>0.70834</td>
</tr>
<tr>
<td>67.72</td>
<td>1.0461</td>
<td>(310)</td>
<td>10</td>
<td>100</td>
<td>0.92534</td>
<td>0.85277</td>
<td>0.70166</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>sin²θ</th>
<th>γ</th>
<th>γ²</th>
<th>α sin²θ</th>
<th>√sin²θ</th>
<th>α γ</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.999920</td>
<td>9.99920</td>
<td>99.98400</td>
<td>3.03246</td>
<td>5.05370</td>
<td>59.99520</td>
</tr>
<tr>
<td>0.501746</td>
<td>5.01746</td>
<td>25.17490</td>
<td>8.52940</td>
<td>4.27960</td>
<td>50.17460</td>
</tr>
<tr>
<td>0.490328</td>
<td>4.90328</td>
<td>24.04215</td>
<td>8.52770</td>
<td>4.18137</td>
<td>49.03280</td>
</tr>
</tbody>
</table>

\[
\left(\frac{K}{K'}\right)^2 = 0.99594
\]

\[
(159.20260) \times 236 \times A_0 \neq 159.20260 \ D = 20.08956
\]

\[
(236) \times 159.20260 \times A_0 \neq 149.20105 \ D = 13.51467
\]

\[
D = -0.00089683
\]

\[
A_0 = 0.085730
\]

\[
a_0 = 3.29934 \AA
\]
TABLE VII
TITANIUM (900° C ± 5°) COPPER RADIATION

<table>
<thead>
<tr>
<th>θ°</th>
<th>d</th>
<th>hkl</th>
<th>α</th>
<th>α²</th>
<th>sin θ</th>
<th>sin²θ</th>
<th>sin 2θ</th>
</tr>
</thead>
<tbody>
<tr>
<td>61.03</td>
<td>0.8786</td>
<td>(321)α₁</td>
<td>196</td>
<td>0.87487</td>
<td>0.76540</td>
<td>0.847493</td>
<td></td>
</tr>
<tr>
<td>61.39</td>
<td>0.8772</td>
<td>(321)α₁</td>
<td>196</td>
<td>0.87790</td>
<td>0.76688</td>
<td>0.840756</td>
<td></td>
</tr>
<tr>
<td>69.25</td>
<td>0.8220</td>
<td>(400)α₁</td>
<td>256</td>
<td>0.93514</td>
<td>0.87449</td>
<td>0.662620</td>
<td></td>
</tr>
<tr>
<td>69.67</td>
<td>0.8218</td>
<td>(400)α₁</td>
<td>256</td>
<td>0.93771</td>
<td>0.87493</td>
<td>0.651569</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>θ°</th>
<th>d</th>
<th>hkl</th>
<th>α</th>
<th>α²</th>
<th>sin θ</th>
<th>sin²θ</th>
<th>sin 2θ</th>
</tr>
</thead>
<tbody>
<tr>
<td>69.25</td>
<td>0.8220</td>
<td>(400)α₁</td>
<td>256</td>
<td>0.93514</td>
<td>0.87449</td>
<td>0.662620</td>
<td></td>
</tr>
<tr>
<td>69.67</td>
<td>0.8218</td>
<td>(400)α₁</td>
<td>256</td>
<td>0.93771</td>
<td>0.87493</td>
<td>0.651569</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>sin²θ</th>
<th>ρ</th>
<th>ρ²</th>
<th>α sin²θ</th>
<th>ρ sin²θ</th>
<th>α ρ²</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.718244</td>
<td>51.58744</td>
<td>10.71560</td>
<td>5.49744</td>
<td>100.55416</td>
<td></td>
</tr>
<tr>
<td>0.703358</td>
<td>49.47125</td>
<td>10.73632</td>
<td>5.39591</td>
<td>98.47012</td>
<td></td>
</tr>
<tr>
<td>0.439065</td>
<td>4.39065</td>
<td>13.99184</td>
<td>3.83958</td>
<td>70.25040</td>
<td></td>
</tr>
<tr>
<td>0.422432</td>
<td>4.22432</td>
<td>17.84468</td>
<td>3.99388</td>
<td>67.58912</td>
<td></td>
</tr>
</tbody>
</table>

138.18138 49.44264 18.42691 336.86380

(336.86380) x 904 A₀ / 336.86380 D = 49.44264
(904) x 336.86380 A₀ / 138.18138 D = 18.42691

D = 0.0017314
A₀ = 0.054047
a₀ = 3.30651 A
900 °C are listed in Tables V - VII inclusive. Theoretically, a vanadium target would give the best results since the K-alpha radiation from such a target lies just over the absorption edge of titanium, and the beta radiation should be largely absorbed by the specimen.

Attempts to obtain x-ray diffraction patterns of the alpha form of titanium at room temperature met with little or no success. Lines in the back reflection region were below Bragg angles of 60°, and too diffuse and weak to read. Apparently the structure factor for the high temperature form was more favorable than that at room temperature.

From an examination of the data, it is readily recognized that the accuracy increases as the angles approach the back reflection region of the film. Although the structure factor of Beta Titanium is more favorable than the low temperature form, the x-ray reflections were still weak and films were difficult to read. Exposures were for a duration of 4 hours at 40 KVP and 15 ma; it was not deemed advisable to expose the specimen longer than this, since the background became more dense and the diffraction lines did not increase in intensity proportionately.

The diffractions from the exposure using copper radiation gave the highest Bragg angles, and is, therefore, considered the most accurate. The lattice constant for Beta Titanium, body-centered-cubic, at 900 °C ± 5°, was determined to be 3.3065 ± 0.00001 Å, and the corresponding atomic diameter is 2.86 Å.
CONCLUSIONS

1. A 19-cm high-temperature-vacuum camera was successfully used in several metallurgical investigations. It was found to be a versatile and effective addition to the ever increasing apparatus and equipment required by the physical metallurgist.

2. The thermal dilation of polycrystalline copper was found to be isotropic in the range of temperatures, 18-770 °C.

3. The accurate determination of the lattice of Beta Titanium (body-centered-cubic) was found to be \( 3.3065 \pm 0.00001 \, \text{Å} \), at 900 °C ± 5°, and the corresponding atomic diameter to be 2.86 Å.
SUMMARY

A 19-cm high-temperature-vacuum camera was used in a series of metallurgical investigations to determine its versatility and applicability to problems of a metallurgical nature. It was found that, with a little extra skill in its operation, it is applicable and necessary for many metallurgical problems. The effect of collimation on grain size was investigated, and it was readily determined that increased collimation gave added resolution to the x-ray beam with a decrease in grain size.

The prediction that certain cubic metals may not be precisely cubic throughout a range of temperatures below their melting points led to the examination of pure copper. By means of x-ray analysis, and the use of the 19-cm camera mentioned above, specific planes in the lattice were examined for anisotropy. Temperatures of examination ranged between 18-770 °C. Plotting the lattice constant of the two planes chosen, (024) and (331), versus temperature, the thermal dilation of copper was isotropic in nature, and the lattice was precisely cubic throughout the temperature range.

The accurate determination of the lattice constant \(a_0\) for Beta Titanium at 900 °C ± 5° was determined to be 3.3065 ± 0.00001 Å. Copper radiation gave the best results of the several radiations used, and the method of least squares was used in the calculations. The corresponding atomic diameter at the above mentioned temperature and lattice constant is 2.86 Å.
Fig. 41.—The usual film-arrangement in Debye-Scherrer cameras and the types of film resulting from each. The dotted lines join corresponding points on the film rolled and flat.

(a) Van Arkel arrangement.
(b) Bradley arrangement.
(c) Ievins and Stavroudis' asymmetric arrangement.
(d) Arrangement of two symmetrical films in Bradley 18-cm. diameter camera.

(H. Lippert and A. J. C. Wilson, Journal of Scientific Instruments, 18, 144, 1941.)

Figure 8.
ACCURATE MEASURE OF UNIT CELL DIMENSION USING DEBYE-SCHERRER CAMERAS FOR CUBIC CRYSTALS

All precision lattice parameter measurements are based on the use of back reflections or x-rays that have been diffracted through angles of $2\theta$ nearly equal to $180^\circ$. For values of $\theta$ near $90^\circ$ the value of $\sin \theta$ is known to high precision even if the angle $\theta$ is known only roughly.

In any calculation there are two types of error, random and systematic. The random errors will average out. The sources of systematic error are:

(a) Shrinkage of the film.
(b) Error in the radius of the camera.
(c) Displacement of the sample from the center (eccentricity).
(d) Absorption

DEBYE CAMERAS: * It can be shown that the systematic errors give values of $\sin^2 \theta$ in error by the amount $D \sin^2 2\theta$, where $D$ is different from film to film but is a constant for any one film.

For cubic crystals the formula for each reflection when no error is present is:

$$A_o (h^2 + k^2 + l^2) = \sin^2 \theta$$

where $A_o = \frac{\lambda^2}{4a_0^2}$

When errors are present the formula becomes:

$$A_o (h^2 + k^2 + l^2) \neq D \sin^2 2\theta = \sin^2 \theta$$

Given two or more reflections (at least three for accuracy) it is then possible to calculate both $A_0$ and $D$. The planar spacing "$d" was first calculated,

\[ \frac{2\theta}{360} = \frac{S}{2\pi R} \]

\[ \theta = \frac{90 S}{\pi R} \]

\[ \theta^0 = 85^\circ - \frac{90 S}{\pi R} = K \cdot \theta^0 \approx 85^\circ - KS \]

where $S$ equals the distance from the $85^\circ$ fiducial knife edge to a diffraction line (cm), and $R$ is the radius of the camera in cm.

The angle $\theta$ obtained by the above method is the angle $\theta$ required by the Bragg Law formula, and angles of $\theta$ less than $60^\circ$ are not accurate for calculation by this method.

From the "$d" values, the Miller indices were calculated using a rough figure for "$a_0$", \( h^2 + k^2 + l^2 \) = \( \frac{a_0^2}{d^2} \)

The following equations were set up and solved for $A_0$ and $D$:

\[ A_\sigma \sum \lambda \sum \varphi = \sum \alpha \sin^2 \theta \]

\[ A_\sigma \sum \alpha \sum \varphi = \sum \varphi \sin^2 \theta \]

where $\alpha = (h^2 + k^2 + l^2)$

\[ \varphi = 10 \times \sin^2 2\theta \]

The $\sin^2 \theta$ can be calculated back from

\[ \sin^2 \theta = \frac{\lambda^2 (h^2 + k^2 + l^2)}{4 a_0^2} \]

and the deviation from the measured $\sin^2 \theta$ noted.

The error in the measurement of the arc, or "$S" from the original equation can be determined by simply using the calculated $\sin^2 \theta$ value as found above, and back calculating.
Using copper as an example, the following are actual measurements and results on annealed copper at 18°C ± 2°C.

**DATA**

<table>
<thead>
<tr>
<th>θ°</th>
<th>d</th>
<th>hkl</th>
<th>α</th>
<th>α²</th>
<th>sin θ</th>
<th>sin²θ</th>
<th>sin 2θ</th>
</tr>
</thead>
<tbody>
<tr>
<td>72.91</td>
<td>0.8062</td>
<td>(024)α₀</td>
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<td>(331)α₀</td>
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<td>0.93176</td>
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<td>0.92965</td>
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<td>0.68505</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>sin² 2θ</th>
<th>γ</th>
<th>γ²</th>
<th>α sin² θ</th>
<th>γ sin² θ</th>
<th>α γ</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.314039</td>
<td>3.14039</td>
<td>9.86205</td>
<td>18.18180</td>
<td>2.85490</td>
<td>62.80780</td>
</tr>
<tr>
<td>0.331292</td>
<td>3.31292</td>
<td>10.97544</td>
<td>18.17760</td>
<td>3.01105</td>
<td>66.25840</td>
</tr>
<tr>
<td>0.455571</td>
<td>4.55571</td>
<td>20.75450</td>
<td>16.41353</td>
<td>3.93554</td>
<td>86.55849</td>
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<td>0.469294</td>
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<td>22.02369</td>
<td>16.42075</td>
<td>4.05587</td>
<td>89.16586</td>
</tr>
</tbody>
</table>

| 63.61568 | 69.19368 | 13.85736 | 304.79055 |

1522  \( A_0 \neq 304.79055 \)  \( D = 69.19368 \)

304.79055  \( A_0 \neq 63.61568 \)  \( D = 13.85736 \)

\( D = 0.00033678 \)

\( A_0 = 0.045395 \)

\( a_0 = 3.6079 \) Å

Accepted  \( a_0 = 3.608 \) Å

Accuracy: 1:36,000
BIBLIOGRAPHY


U.S. Dept. of Commerce, National Bureau of Standards, Table of Sines and Cosines to Fifteen Decimal Places at Hundredths of a Degree.


VITA

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