

THE ANALYSIS OF A TANTALUM-THORIUM ALLOY  
BY X-RAY METHODS

by

HARRY STANLEY ADAMS

B. S., Kansas State College of  
Agriculture and Applied Science  
1945

---

A THESIS

submitted in partial fulfillment of the

requirements for the degree of

MASTER OF SCIENCE

Department of Physics

KANSAS STATE COLLEGE  
OF AGRICULTURE AND APPLIED SCIENCE

1947

Docu-  
ment  
LD  
2668  
.74  
1747  
A3  
c.2

11

## TABLE OF CONTENTS

INTRODUCTION	1
THEORY	4
Powder Method of Analysis	4
Miller Indices and Bragg Law	5
The Intensities of X-Ray Lines	8
Thomson Polarization Factor	8
Lorentz Factor	9
Structure Factor	9
Atomic Form Factor	10
Multiplicity Factor	10
Debye Temperature Factor	13
Absorption Factor	15
Superstructures	18
APPARATUS AND EQUIPMENT	20
X-Ray Machine and Tube	20
Powder Camera	21

## TABLE OF CONTENTS (cont.)

Micro-Comparator	- - - - -	21
Micro-Densitometer	- - - - -	26
The Vacuum Furnace	- - - - -	26
PREPARATION OF SAMPLE	- - - - -	27
DATA AND RESULTS	- - - - -	29
Calibration of the Camera	- - - - -	29
Measurements on Tantalum-Thorium Alloy	- - - - -	30
Sample Calculations	- - - - -	36
Calibration of Camera	- - - - -	36
Calculation of $a_0$	- - - - -	36
Calculation of I	- - - - -	36
DISCUSSION OF RESULTS	- - - - -	37
CONCLUSIONS	- - - - -	38
ACKNOWLEDGMENT	- - - - -	39
BIBLIOGRAPHY	- - - - -	40

## INTRODUCTION

Filaments for high voltage cathode ray tubes are usually made from tungsten. It would be desirable to use a thoriated tungsten filament because of its higher emissive properties when operated at the same temperature as a similar tungsten filament. The thoriated tungsten filaments are destroyed by positive ion bombardment, and so cannot be used at high voltages. A filament constructed from an alloy of tantalum and thorium might possess the high emissive properties of thorium and yet have the structural properties of tungsten. The components of this alloy form a homogeneous solid solution, while the thorium in the thoriated tungsten is present on the surface of the tungsten in a layer only a few atoms thick. A filament made of an alloy of 20 percent thorium and 80 percent tantalum was found to have good emissive properties and not be destroyed by positive ion bombardment as easily as thoriated tungsten.

Tantalum-thorium alloys have some unusual mechanical properties. Those alloys whose composition was not 20 percent thorium were found to be brittle and would not withstand the strain when pulled through the dies.<sup>1</sup> The 20 percent thorium alloy, however, did stand up under the strain. A possible explanation for this property might be that there exists a superstructure lattice at this composition. Therefore, the purpose

---

<sup>1</sup>This information obtained by the author from Dr. L. D. Ellsworth, Kansas State College.

of this investigation was to determine by x-ray analysis the structure of this alloy and the presence of any superstructure lattice.

Soon after the discovery of x-rays by Roentgen, investigators began to ponder the nature of these rays. It was proposed that the properties of these rays might be explained by Maxwell's electromagnetic wave theory. According to Maxwell's theory, if x-rays were of a wave nature, they would possess the property of being diffracted when passed through a slit. Experimenters, therefore, began to conduct many experiments to see whether they could detect this diffraction effect when radiation of x-ray frequencies were passed through a slit.

According to Compton and Allison (1, p. 20) Haga and Wind conducted a series of experiments to detect the diffraction of x-rays as they were passed through a wedge shaped slit. They calculated the wave-length of the rays used to be 1.3 angstroms. Later, Walter and Pohl repeated their experiment and came to the conclusion that the diffraction effects were smaller than Haga and Wind had estimated. Sommerfeld recalculated the results of Walter and Pohl's and found the wave-length to be 0.4 angstroms.

One of the first spectra produced by a grating was obtained by Compton and Doan (2). They used a grating of speculum metal and the K line of molybdenum, 0.71 Å.

Because of the difficulties encountered by these early investigators, their results were not too convincing; but they did inspire von Laue to carry out his experiments on crystal dif-

fraction. Sommerfeld had calculated the wave-length of ordinary x-rays to be between 0.1 Å and 1.0 Å. von Laue calculated, from the known number of molecules per unit volume, that the average distance between atoms in crystals to be about 1 to 10 angstroms. In order to build up a crystal it must be assumed that the molecules or atoms arrange themselves in a regular repeating pattern. Now this type of arrangement was very similar to that of an optical grating. It, therefore, occurred to von Laue that a crystal might act as a three dimensional grating for x-rays.

Therefore he asked Friedrich and Knipping (1, p. 25) to try an experiment of passing a beam of x-rays through a crystal of zinc blende. They observed, on their photographic plate, spots which were symmetrically located around the central spot due to the primary beam. A simple explanation of the pattern was offered by W. L. Bragg (1, p. 27). He proposed that each of the images surrounding the central image could be explained as the reflection of the incident beam from some plane within the crystal. If one looks at a two dimensional array of points, he can observe that in some directions the density of the points is greater than that in other directions. The cleavage face of a crystal should be parallel to such a line or plane. An experiment of reflecting a beam of x-rays from the cleavage face of a crystal was conducted by W. H. Bragg (1, p. 27), and he found a spot on the photographic plate at the angle of reflection. He then replaced the plate with an ionization chamber and measured the ionization current as a function of the glancing angle and

obtained a smooth curve with sharp peaks at several points. From his results he derived his famous relation known as Bragg's law.

$$n\lambda = 2d \sin \theta \quad (1)$$

where:  $n$  = the order number

$\lambda$  = the wave-length of the incident radiation

$d$  = the interplanar distance between planes

$\theta$  = the glancing angle of the incident radiation

It can be readily seen that if the wave-length is known, one can calculate the distance between layers or planes of atoms.

## THEORY

### Powder Method of Analysis

Other methods of analysis have been developed, among which probably the most useful is the Debye-Scherrer-Hull or more commonly called the powder method. This type of analysis was developed by Debye and Scherrer, and independently by Hull.

It is sometimes difficult to obtain large enough crystals to be used in the Laue or in the rotating crystal method. However, if one has an aggregate of crystals in the form of a fine powder or a wire, so that the crystals have random orientation, then he may employ the powder method of analysis. In this type of analysis a beam of monochromatic x-rays is passed through a defining slit system,  $S_1S_2$ ; Plate I; and strikes the sample place



at O. The beam is diffracted through an angle of  $2\theta$  and intersects the film at radius R from the center O. Since the crystal is a three dimensional grating, the rays will be diffracted in a series of cones whose apexes will be at the diffracting centers. These cones will intersect the cylinder of film in concentric circles of varying radii depending upon the angle of diffraction, producing arcs in pairs symmetrically located with respect to the central beam.

From the geometry of the camera it is obvious that the angle  $\theta$ , in degrees, may be expressed as a function of the arc length L, and the radius R.

$$\theta = \frac{180}{2\pi} \frac{L}{R} \quad (2)$$

or

$$\theta = kL$$

### Miller Indices and Bragg Law

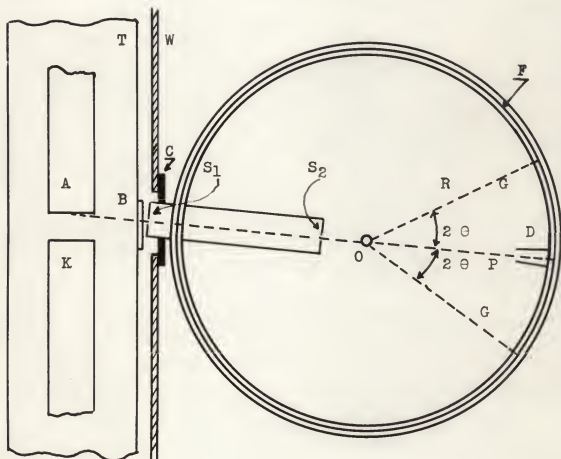
The planes of atoms within a crystal may be defined in terms of their intercepts upon the crystallographic axis. These may be given in terms of the unit lengths or the distances between like atoms along the unit cell edge. Three integers, called Miller indices, are used to represent these planes. These integers are the lowest three integers having the same ratios as the reciprocals of the intercepts expressed in terms of the axial lengths. If the intercepts of a plane upon the axis is, for instance,  $a/3$ ,  $2b/3$ , and  $c/2$ , then the Miller indices of this plane are (6 3 4).



# EXPLANATION OF PLATE I

- A. Target of x-ray tube.
- B. Beryllium Window.
- C. Lead rubber shield.
- D. Beam trap.
- F. Film bearing surface.
- G. Diffracted beam.
- K. Cathode of x-ray tube.
- P. Primary beam.
- R. Radius of camera.
- S<sub>1</sub>. Front pinhole insert.
- S<sub>2</sub>. Rear pinhole insert.
- T. X-ray tube.
- W. Tube casing of x-ray machine.
- O. Sample.
- $\Theta$ . Diffracting angle.

PLATE I



It can be shown that for a cubic crystal the interplaner distances may be expressed in terms of the Miller indices,  $hkl$ , and the unit cell size  $a_0$ . (3)

$$d = \frac{a_0}{\sqrt{h^2 + k^2 + l^2}} \quad (3)$$

Hence Bragg's law (Eq. 1) becomes

$$n\lambda = \frac{2 a_0 \sin \Theta}{\sqrt{h^2 + k^2 + l^2}} \quad (4)$$

The order number,  $n$ , may be absorbed in the Miller indices. A second order diffraction for the 100 plane may be expressed as the first order diffraction of a 200 plane.

### The Intensities of X-ray Lines

An important aid in performing a complete structural analysis is the measurement and comparison between observed and calculated intensities of the x-ray lines. The intensity of the lines depend upon seven factors. (4) These are:

1. Thomson polarization factor
2. Lorentz factor
3. Structure factor
4. Atomic form factor
5. Multiplicity factor
6. Debye temperature factor
7. Absorption factor

Thomson Polarization Factor. The question of the polar-

ization of x-rays emitted by the target of an x-ray tube has never been fully investigated. Bearden and Wollen have shown that the characteristic rays are unpolarized. (4) It is, therefore, assumed that when discussing the scattering of x-rays that the primary rays are unpolarized. However, the diffracted beam will be partially or completely polarized, depending upon the angle of diffraction. Various investigators have shown good agreement between their results and Thomson's classical theory of scattering which states the intensity of the scattered radiation is proportional to  $\frac{(1 + \cos^2 2\theta)}{2}$ . Consequently this term is known as the Thomson polarization factor.

Lorentz Factor. The intensity also depends upon another factor called the Lorentz factor. This accounts for the fact that the primary beam is not a pure monochromatic beam and also that it is not parallel, but in general is slightly diverging. Blake shows that it is proportional to  $\frac{(\csc^2 \theta \sec \theta)}{2}$ . (4) Darwin and also Compton have shown, that for single crystals, the Lorentz factor to be proportional to  $\csc 2\theta \sec \theta$ . (4) Compton has also shown, that for a thick plate, it is proportional to  $\csc 2\theta \sec \theta$ . (5, p. 125) This last expression is also true for cylinders of powdered crystals.

Structure Factor. The structure factor may be defined as the ratio of the amplitude of the waves scattered by the atom to those of a free classically scattering electron. This is represented by the following equation:

$$S_{hkl} = \sum_j f_j \exp \left[ 2\pi i \left( \frac{hx_j}{a} + \frac{ky_j}{b} + \frac{lz_j}{c} \right) \right] \quad (5)$$

where  $x$ ,  $y$ , and  $z$  are the coordinates of the atoms of the unit cell, and  $h$ ,  $k$ , and  $l$  are the Miller indices.  $f$  is the atomic form factor which is discussed below.  $a$ ,  $b$ , and  $c$  are the dimension of the unit cell.

Atomic Form Factor. The results of the structure factor imply that the waves are all reflected in phase from the electrons, which is not true. Since the atom is not a point, but has dimensions of the order of  $10^{-13}$  cm,  $S$  is a function of the atomic number,  $Z$ , and the glancing angle,  $\theta$ . The waves reflected by an electron in one part of the atom will not necessarily be in phase with those reflected by another electron, so that in certain directions there will be constructive interference, and in other directions destructive interference. Therefore  $S$  is a function of  $\frac{\sin \theta}{\lambda}$  and is expressed by:

$$f = \frac{\int U(r) \sin(4\pi r \sin \theta / \lambda) dr}{4\pi r \sin \theta / \lambda}$$

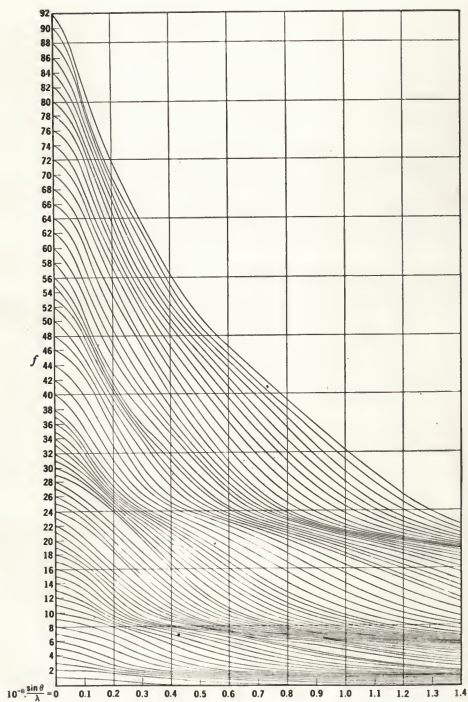
where  $U(r)$  is the density of electrons between  $r$  and  $r + dr$ .  $f$  is called the atomic form factor and is generally combined with the structure factor. Values for  $f$  have been calculated with considerable accuracy, based on the work of Hartree, Pauling and Sherman, and Thomas and Fermi. (3, p. 314) Values of  $f$  are shown by the curves in Plate II.

Multiplicity Factor. A careful inspection of equation (4) will show that reflections from more than one plane will con-

EXPLANATION OF PLATE II

The atomic form factor vs  $\frac{\sin \theta}{\lambda}$

## PLATE II





tribute energy to the same line in the powder spectrum. This is because the position of the line on the film depends only upon the sum of the squares of the indices when restricted to the cubic system. The multiplicity factor, usually designated by  $n$ ,  $j$ ,  $p$ , or  $z$ , is the total number of combinations of  $h$ ,  $k$ , and  $l$  which when squared and added will give the same sum. If one considers the planes for the values of  $h = k = l = \pm 1$ , he will find that there are eight possible combinations. These are:  $(1\ 1\ 1)$ ,  $(\bar{1}\ 1\ \bar{1})$ ,  $(1\ 1\ \bar{1})$ ,  $(\bar{1}\ \bar{1}\ 1)$ ,  $(\bar{1}\ 1\ 1)$ ,  $(1\ \bar{1}\ 1)$ ,  $(\bar{1}\ \bar{1}\ \bar{1})$ , and  $(1\ \bar{1}\ \bar{1})$ . Since there are eight possible combinations of 1 and 1, taking three integers at a time, the multiplicity factor is 8. Multiplicity factor values are listed in Wyckoff's Structure of Crystals, 2nd edition, page 177. The values for a part of the cubic system are listed below.

Table 1. Multiplicity Factors for  $T_d$ ,  $O$ , and  $O_h$  structures of the cubic system.

hkl	n
hkl	48
100	6
111	8
110	12
0kl or hhl	24

Debye Temperature Factor. The atoms which compose the structure of a crystal are not at rest but vibrate about their equilibrium position because of heat energy. As the amplitude

of vibration increases with rise in temperature, the possibilities of x-ray interference is reduced, thus reducing the intensities of the x-ray lines. Blake (4) states that theoretical work concerning this temperature effect has been done by v. Laue, Darwin, Schrodinger, Faxen, Brillion, Waller, and Debye. Comparison between experiment and theory has largely been made using the theory as formulated by Debye.

In 1918 Born and Karman showed that the heat motion of atoms in crystals may be considered as a series of elastic waves. (4) Upon the basis of this theory and the quantum theory Debye calculated the effect of thermal vibration on interference maxima and he showed, assuming zero point energy, that the term  $\exp(-2W)$  must be introduced into the intensity formula.  $W$  is given by the equation:

$$W = \frac{6}{mk} \frac{h^2}{\lambda} \left[ \frac{\psi(X)}{X} + \frac{1}{4} \right] \left[ \frac{\sin \theta}{\lambda} \right]^2 \quad (6)$$

Where  $h$  is Planck's constant;  $k$  is Boltzman's constant;  $\theta$  is the characteristic temperature, which equals  $h \nu_m/k$  where  $\nu_m$  is the maximum frequency of the elastic spectrum;  $m$  is the mass of the atom;  $\lambda$  is the wave-length; and  $\theta$  is the glancing angle in degrees.

$$\psi(X)/X = \frac{1}{X^2} \int_0^X \frac{t dt}{e^t - 1}$$

where  $X = \theta/T$  and  $T$  is the absolute temperature of the crystal.

It has been shown by Debye that  $\psi(X)$  may be expressed in terms of a converging infinite series, and he has calculated

values of  $\psi(X)$  for the corresponding values of  $X$ . These values are listed in the following table.

Table 2. Values of  $\psi(X)$  for corresponding values of  $X$ .

$X$	$\psi(X)$	$X$	$\psi(X)$	$X$	$\psi(X)$	$X$	$\psi(X)$
0.0	1.000	1.2	0.740	3	0.483	9	0.183
0.2	0.951	1.4	0.704	4	0.388	10	0.164
0.4	0.904	1.6	0.669	5	0.321	12	0.137
0.6	0.860	1.8	0.637	6	0.271	14	0.114
0.8	0.818	2.0	0.607	7	0.234	16	0.103
1.0	0.778	2.5	0.540	8	0.205	20	0.0822

Absorption Factor. The last factor effecting the intensity is the absorption factor. This may be defined as the ratio of the intensity of the diffracted beam to the same beam without absorption. It has been shown by Claassen (6) that the absorption factor is:

$$A = \frac{1}{\pi r^2} \int_0^{\pi} e^{-\mu d \sin^2 \theta} d\theta$$

where  $d$  is the path length of the ray through the powder sample. This integral cannot be evaluated by the usual methods, and Claassen has evaluated it by graphical means.

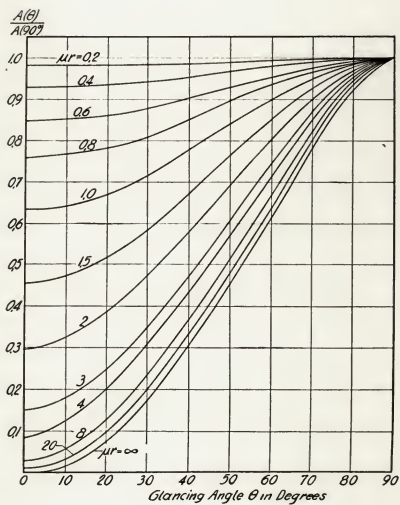
The results of his work are shown in Plate III which is a plot of  $A(\theta)/A(90)$  as the ordinate and  $\theta$  as the abscissa.  $A(\theta)$  is the absorption at the glancing angle  $\theta$ , and  $A(90)$  at 90 degrees.  $\mu$  is the linear absorption coefficient and  $r$  is the radius of the sample.

One may now combine these factors into a single formula

EXPLANATION OF PLATE III

$\frac{A(\Theta)}{A(90)}$  vs the glancing angle  $\Theta$  in degrees.

## PLATE III



formula which gives the relative intensities of the coherent radiation on a diffraction photograph by the powder method.

This is:

$$I = \frac{n(1 + \cos^2 2\theta)}{2} \frac{1}{\sin^2 \theta \cos \theta} \left[ \text{Sf. exp } (-M) \right]^2 \frac{A(\theta)}{A(90)} \quad (7)$$

This may be shortened in form to

$$I = n \phi S^2 A \quad (8)$$

where  $\phi$  is the combination of the Thomson and Lorentz factors, S. the structure, atomic form, and Debye temperature factors.

### Superstructures

One of the types of alloys is the "substitutional solid solution". This is sometimes made by adding quantities of metal B to metal A. The crystals of the alloy are very much like those of the pure metal except that on some lattice sites B atoms have been substituted for A atoms. Usually this substitution took place in a purely random manner, but it has been determined by x-ray diffraction methods that at certain concentrations in some alloy systems the atoms arrange themselves in a definite order to form a superlattice.

The first discovery of such a mechanism was made by Tammann (8) and was not done by x-ray techniques. He noticed the differences in chemical activity of Cu-Au alloys when attacked by nitric acid. He explained this difference by the presence of a superstructure.

More conclusive evidence of the presence of superstructures

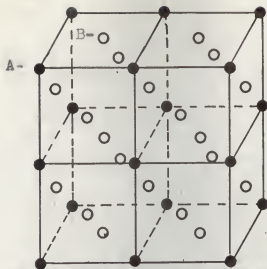


Fig. 1. The lattice structure of an ordered alloy.

or superlattice is furnished by the presence of the so-called superstructure lines on x-ray diffraction pictures. The intensities of these lines is, to some extent, a measure of the amount of order or disorder present in the alloy.

Figure 1 shows the lattice system of a substitutional alloy if it were in the ordered state. It is a face centered cubic lattice with A atoms at the corners and B atoms in the faces of the unit cell. This system may be thought of as two interpenetrating simple cubic lattices. One being a cube of A atoms, the other a cube of B atoms.

From structure factor considerations it can be shown that for a simple cubic lattice, lines should appear for all values of  $hkl$ . For a face centered cubic system, only those lines appear whose indices are all even or all odd.

If the alloy were in a dis-ordered state, we would have only a face centered cubic lattice. Consequently certain lines would



be absent. If the alloy were in a completely ordered state, we could consider the system as two interpenetrating simple cubes and the missing lines should appear. This is because of the different scattering power of the A and B atoms. If the atoms had equal scattering power, then certain lines would be missing because we have effectively only a face centered cube.

## APPARATUS AND EQUIPMENT

### X-Ray Machine and Tube

The diffraction pictures were obtained using the General Electric XRD crystal diffraction unit. The x-ray tube employed is a General Electric type CA-6. The x-rays are emitted from a molybdenum target and pass through either of two beryllium windows located opposite each other in line with the target. The maximum radiation occurs at an angle of minus six degrees with the normal to the tube. The XRD unit is shown in Plate IV.

The current is turned on or off by means of the circuit breaker located in the center of the panel door. The high potential is controlled by a Variac (auto-transformer) located to the left of the clock. The meter directly above measures the primary voltage applied to the high voltage transformer. The tube current is varied by means of the control on the right of the clock, which is a variable resistance in the filament circuit of the x-ray tube. The current is rectified by a half-wave

rectifier using a General Electric kenotron, type KR-6. The tube current is read on the milliammeter which is in the grounded side of the high voltage circuit. This meter is above the current control. The clock operates only when the unit is operating. Utility outlets are provided on the sides of the unit to provide current for the auxiliary equipment used on the camera.

#### Powder Camera

The powder camera is shown in position in Plate IV and a larger view is shown in Plate V. The camera consists essentially of a round circular drum upon which the film is placed, the slip system, chuck to hold the sample, motor to rotate it, and the beam trap to prevent scattered rays and the direct beam from fogging the film. The radius of the camera is approximately  $22.5/\pi$  or 7.17 cm. so that the angle of diffraction in degrees is  $\theta = 4 L$ . Accurate values of the coefficient of  $L$  are obtained through calibration of the camera.

#### Micro-Comparator

The micro-comparator is an instrument for measuring the positions of the lines on the photographic film. The one used for this investigation was constructed by Mr. E. V. Floyd<sup>2</sup> using a

---

<sup>2</sup>Professor of Physics at Kansas State College, Manhattan, Kansas.

EXPLANATION OF PLATE IV

The General Electric XRD Unit.

PLATE IV



EXPLANATION OF PLATE V

The diffraction camera.

## PLATE V



24 inch vernier caliper as the scale. Readings may be taken to 0.02 mm.

#### Micro-Densitometer

The intensities of the lines were measured by the Knorr and Albers (9) recording micro-photometer. A fixed beam of light is passed through the film to be analyzed. The transmitted light is received by an emission type photoelectric cell producing a current which is passed through a fixed resistance. The difference in potential developed across this resistance is applied to the grid of an amplifier, the output of which is applied to a Leeds and Northrup Speedmax recording galvanometer. This gives a continuous, inked trace of the transmitted light or intensity of the film as a function of the distance along the film.

#### The Vacuum Furnace

The furnace used to anneal the powder specimen itself was constructed from a thermocouple insulator 1.5 inches long, approximately  $1/8 \times 1/4$  inches in cross-section. The insulator had two holes through its longitudinal axis. One end of one of the two holes was sealed with some refractory cement. The heating coil was wound with No. 22 nichrome wire around the insulator. The winding was one inch in length per layer with three layers. Each layer was insulated from its neighbor with sheet mica. The ends



of the nichrome wires were spot welded to two wires of a press seal. A thermocouple was made of No. 22 Alumel and Chromel wire and spot welded to the other two wires of the press seal. After the furnace was filled with the sample it was sealed into a one liter flask which was sealed to a vacuum system. The vacuum system consisted of a mercury vapor diffusion pump, mechanical forepump, and McLeod gauge. The potential of the thermocouple was measured using a Leeds and Northrup Portable Potentiometer Indicator (8657-c).

#### PREPARATION OF SAMPLE

The wire sample was obtained from Fansteel Corporation at Chicago. The wire was filed on a fine file and the filings were screened by passing them through a 325 mesh sieve. They were then passed under a strong alnico magnet to remove any particles of iron that may have been chipped from the file. The magnet may not remove all the iron because of the adhesive forces between small particles. One sample was then placed in a vacuum furnace at a pressure of  $10^{-5}$  mm Hg and heated to a temperature of  $800^{\circ}$  C. ( $1472^{\circ}$  F.) for 12 hours. It was then slowly cooled to room temperature over a period of 12 hours. It was desirable to anneal the sample in this manner for two reasons. First, the small crystals may have become distorted during the filing process or during the wire forming process when the wire was pulled through the dies. Second, if there is any ordering process

present, the atoms assume their ordered positions only over a long period of time. If the sample were cooled too quickly, it would tend to freeze the atoms in the disordered state which prevails at the high temperatures.

The sample is then placed in a small capillary tube which is drawn from soft glass. A piece about 15 mm long and not more than 0.6 mm in diameter is filled with the sample by the following technique. One end of the tube is sealed by a flame. A piece of paper is folded and then opened. The sample is placed in the vee of the fold and the open end of the tube is scraped along the vee, holding the tube between the thumb and forefinger. The tube is then held in a vertical position and tapped lightly on the table. Since the powder adhering to the open end did not fall down the tube and pack well, the following technique was tried. A file was drawn lightly across the side of the tube when held in the vertical position. The vibrations caused by the file moving across the tube jarred the particles into position. After the tube was completely filled, it was sealed with some soft wax ready to mount in the camera. The chuck on the camera consists of a small flat cylinder of steel with a small hole in the center. This was held to the motor by a small magnet mounted on the armature. The chuck was centered on the magnet and then the sample was placed in the hole, it being held in position by some soft tacky wax. It was then necessary to center the free end of the tube. This was accomplished by aid of a low power microscope. The microscope was focused on the free end of the

tube, and while the chuck was rotated by hand, the sample was adjusted until it was centered. This could be checked by observing the shadow of the sample on a fluorescent screen when the x-ray machine was operating. The film was then loaded in the camera and the picture taken.

## DATA AND RESULTS

### Calibration of the Camera

The camera was calibrated using a sample of sodium chloride of known cell size. The positions of the lines were measured and the constant  $k$  in equation (2) was calculated. The values of this constant are listed in Table 3.

Table 3. Calibration constant for Powder camera using NaCl.

Plane	$2L$ (cm)	$\theta$ (degrees)	$k/2$ (deg./cm.)
111	3.151	6.271	1.990
200	3.632	7.247	1.995
220	5.156	10.272	1.994
311	Not observed		
222	6.328	12.075	1.994
400	7.318	14.615	1.997
420	8.209	16.385	1.996
422	9.020	18.000	1.995

The average value for  $k/2$  was 1.994. This gives the value of  $k$  as:

$$k = 3.989 \pm .001$$

#### Measurements on Tantalum-Thorium Alloy

The film was exposed for eight hours at a potential of 45 kilovolts, and a current of 16 ma. A filter of zirconium 0.004 inches thick was used to absorb the beta radiation. The films were developed in Eastman D-11 developer for eight minutes at 18° C. (65° F.). The results for the annealed sample are shown in Table 4. Those for the unannealed sample are listed in Table 5.

The intensities were measured by the Knorr and Albers recording micro-densitometer. Two centimeters on trace is equivalent to one centimeter on the film. A curve was drawn by hand to represent the background. The areas between the densitometer curve and background were used as a measure of the observed intensities. The areas were measured by projecting the curves on a grid screen and counting the squares enclosed by each line. The magnification of projection was 2.5. The theoretical intensities were calculated using equation (8). Each of the values calculated had to be multiplied by the concentration of the respective components to give the true intensities. The calculated and observed intensities are shown in Table 6.

Table 4. The lattice constant for 20 percent Th - 80 percent Ta Alloy Heat treated at 800° C.

Line	Plane	L (cm)	$\theta$ (degrees)	a. (Å)	
				Th	Ta
Th	111	1.423	7.213	4.936	
Th	200	Not observed			
Ta	110	2.180	8.693		3.319
Th	220	2.834	11.305	5.122	
Ta	200	3.015	12.029		3.407
Th	311	3.303	13.176	5.165	
Th	222	Not observed			
Ta	211	3.820	15.237		3.308
Th	400	4.002	15.965	5.163	
Ta	220	4.419	17.663		3.308
Th	331	Not observed			
Th	420	Not observed			
Ta	310	4.978	19.860		3.304
Th	422	5.234	20.880	4.969	
Th	511	5.335	21.282	5.082	
Ta	222	5.698	22.730		3.182
Ta	321	5.926	23.640		3.312
Ta	411	6.781	27.050		3.293
Ta	420	7.225	28.324		3.124

A picture of 20 percent Th - 80 percent Ta is shown in Plate VI.

Table 5. The lattice constant for 20 percent Th - 80 percent Ta Alloy (Not heat treated).

Line	Plane	L (cm)	$\theta$ (degrees)	a- (Å)	
				Th	Ta
Th	111	Not observed			
Th	200	Not observed			
Ta	110	2.209	8.836		3.268
Th	220	2.828	11.284	5.131	
Ta	200	3.135	12.509		3.278
Th	311	3.380	13.484	5.050	
Th	222	Not observed			
Ta	211	3.852	15.365		3.281
Th	400	Not observed			
Ta	220	Not observed			
Th	331	Not observed			
Th	420	4.472	18.169	5.091	
Ta	310	Not observed			
Th	422	5.018	20.018	5.081	
Th	511	Not observed			
Ta	222	5.525	22.042		3.277
Ta	321	5.996	23.922		3.276
Ta	411	6.872	27.416		3.271
Ta	420	7.247	28.909		3.284
Ta	332	7.898	31.507		3.186

Table 6. The intensities of the diffraction lines for various percentages of thorium.

Line	Plane	I <sub>obs.</sub>	I <sub>cal.</sub>		
			15 percent	20 percent	25 percent
Th	111	4.4	13.4	19.	25.7
Th	200	0.0	6.2	6.9	19.
Ta	110	100.0		100.	
Th	220	12.4	7.8	11.	15.
Ta	200	50.5		32.7	
Th	311	15.9	12.7	18.1	24.5
Th	222	0.0	0.8	1.0	1.2
Ta	211	90.0		120.0	
Th	400	13.3	2.3	3.1	3.9
Ta	220	32.7		41.8	
Th	331	0.0	1.3	1.7	2.1
Th	420	0.0	1.6	2.1	2.6
Ta	310	40.7		59.	
Th	422	4.9	2.7	3.6	4.5
Th	511	4.3	2.7	3.6	4.5
Ta	222	23.4		23.3	
Ta	321	61.0		60.2	
Ta	411	48.6		54.8	
Ta	420	10.6		39.6	



EXPLANATION OF PLATE VI

The diffraction spectrum of 20 percent Th - 80 percent Ta.

## PLATE VI



## Sample Calculations

Calibration of Camera.  $2L = 3.151 \text{ cm.}$ 

$$\theta = 6.271 \text{ degs.}$$

From equation (2)

$$\theta = kL$$

$$k/2 = 6.271/3.151 = 1.990$$

$$k = 3.980 \text{ deg./cm.}$$

Calculation of  $a$ .  $L = 2.180 \text{ cm.}$ 

$$\theta = 3.980 \times 2.180 = 8.698 \text{ deg.}$$

$$\sin \theta = 0.15159$$

$$hkl = 110$$

$$a. = .710 \times 1/2 \times 2 \times 1/.1516$$

$$a. = 3.319 \text{ angstroms}$$

Calculation of  $I$ .  $hkl = 110$ 

$$n = 12$$

$$\theta = 8.698 \text{ deg.}$$

$$\frac{1 + \cos^2 8.698 \text{ deg.}}{2} = .955$$

$$\frac{1}{\sin^2 8.698 \times \cos 8.698 \text{ deg.}} = 43$$

$$\sin^2 8.698 \times \cos 8.698 \text{ deg.}$$

$$\sin 8.698/710 = .2155$$

$$f = 58 \text{ (from Plate II)}$$

$$SF = 4 f = 116$$

$$\frac{a(\theta)}{\Lambda(00)} = .02 \text{ (from Plate III)}$$

$$h = 6.624 \times 10^{-27} \text{ erg-sec.}$$

$$m_{ta} = 3.0 \times 10^{-22} \text{ gm}$$

$$k = 1.3708 \times 10^{-16} \text{ erg/deg.}$$

$$\phi = 245 \text{ deg.}$$

$$\frac{6 h^2}{mk \phi} [\psi(X) + .25] = .262 \times 10^{-2}$$

$$\text{where } X = 245/300$$

$$\psi(X) = .798 \text{ (from Table 2)}$$

$$M = B \left[ \frac{\sin \theta}{\lambda} \right]^2$$

$$\text{and } B = \frac{6 h^2}{mk \phi} [\psi(X) + .25] = .00322$$

$$2M = .2155 \times .00322 \times 2 = .0298$$

$$\exp(-M) = .471$$

$$I' = .955 \times 43 \times .02 \times (116 \times .0298)^2 = 1110$$

$$I = .8 \times 1110 = 888$$

#### DISCUSSION OF RESULTS

The errors involved in the powder, or any other, method of analysis may be grouped into two classes; systematic errors, and random errors. Errors due to film shrinkage and to the eccentricity of rotation of the sample are classified under systematic errors. Errors in measurement are classified under random errors. These were treated by using the probable error equations. (10)

The background of the films was quite intense and this made it difficult to measure the position of the weak lines.

The errors in the intensities of the lines may be attributed to the uncertainty of the exact location of the background curve on the densitometer trace. Also, for small areas, there may be considerable error because the smallest unit of area counted on

the screen is one square centimeter.

### CONCLUSIONS

An analysis of Tables 5 and 6 shows that the alloy consists of two phases. One phase being the crystals of pure tantalum and the other being pure thorium. The lattice constants, dimensions of the unit cell, are:

Heat treated sample:

Thorium  $a.$  =  $5.065 \pm .051 \text{ \AA}$

Tantalum  $a.$  =  $3.234 \pm .017 \text{ \AA}$

Raw sample:

Thorium  $a.$  =  $5.088 \pm .010 \text{ \AA}$

Tantalum  $a.$  =  $3.273 \pm .001 \text{ \AA}$

Accepted values:

Thorium  $a.$  =  $5.074 \text{ \AA}$

Tantalum  $a.$  =  $3.281 \text{ \AA}$

Within the limits of error there is no change in the lattice constant when the alloy is annealed.

There is no superstructure lattice present because the components of the alloy exist in separate phases. Further evidence for the absence of superstructure is that none of the so-called "forbidden" lines appeared in the spectrum of the alloy.

## ACKNOWLEDGMENT

The author wishes to thank his major professor, Dr. L. D. Ellsworth, also Dr. A. B. Cardwell, Head of the Department of Physics, for their many helpful suggestions and criticisms during the tenure of this problem. The author also wishes to thank Fansteel Corporation of Chicago for supplying the sample.

## BIBLIOGRAPHY

- (1) Compton, A. H. and S. K. Allison.  
X-Rays in theory and experiment. New York.  
D. Van Nostrand. 827 p. 1934.
- (2) Compton, A. H. and R. L. Doan.  
X-Ray spectra from a ruled reflection grating.  
Natl. Acad. Sci. Proc. 11: 598. 1925.
- (3) Clark, G. L.  
Applied X-Rays. New York. McGraw-Hill. 673 p.  
1926.
- (4) Blake, P. C.  
On the factors affecting the reflection intensities  
by the several methods of X-Ray analysis of crystal  
structures. Rev. Mod. Phys. 5: 170-192. 1933.
- (5) Compton, A. H.  
X-Rays and electrons. New York. D. Van Nostrand.  
403 p. 1926.
- (6) Classen, A.  
The calculation of absorption in X-Ray powder-  
photographs and the scattering power of tungsten.  
Phil. Mag. 9: 57. 1930.
- (7) Nix, F. C. and W. Shockley.  
Order-disorder transformations in alloys. Rev. Mod.  
Phys. 10: 2. 1938.
- (8) Tammann, G.  
"Die chemischen und galvanischen Eigenschaften von  
Mischkristallreihen und ihre Atomverteilung."  
(In German) Ziets. f. anorg. allgem. Chem.  
107: 1. 1919.
- (9) Knorr, H. V. and V. M. Albers.  
A new recording microphotometer. Rev. Sci. Instru-  
ments. 8: 183. 1937.
- (10) Miller, D. C.  
Laboratory physics. New York. Ginn. 438 p. 1932.

## Date Due

[illegible]