EMISSIVITY AND MICROSTRUCTURE:
A STUDY OF THE CONSISTENT IRREGULARITIES OF THE EMISSIVITY
OF CERTAIN METALS FROM A MICROSCOPIC BASIS

by

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A Thesis Submitted to the Faculty of the
DEPARTMENT OF MECHANICAL ENGINEERING
In Partial Fulfillment of the Requirements
For the Degree of
MASTER OF SCIENCE
In the Graduate College
THE UNIVERSITY OF ARIZONA

1962
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April 30, 1962
ACKNOWLEDGMENT

The author wishes to express his appreciation for the patience, understanding, and assistance given by Professor A. Ralph Yappel in the preparation of this thesis.

The author also wishes to express his gratitude to Dr. Benjamin S. Mesick for his valuable suggestions during the preparation of this thesis.
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<td>a</td>
<td>lattice parameter</td>
</tr>
<tr>
<td>b</td>
<td>subscript for black body</td>
</tr>
<tr>
<td>d</td>
<td>interplanar spacing, cm</td>
</tr>
<tr>
<td>f</td>
<td>total radiant flux, BTU/sec-ft²</td>
</tr>
<tr>
<td>h,k,l</td>
<td>Miller indices</td>
</tr>
<tr>
<td>o</td>
<td>subscript for normal flux</td>
</tr>
<tr>
<td>q</td>
<td>energy emitted per unit time, BTU/sec</td>
</tr>
<tr>
<td>r</td>
<td>radius of hemisphere of integration, ft</td>
</tr>
<tr>
<td>t</td>
<td>time, seconds</td>
</tr>
<tr>
<td>u</td>
<td>subscript for unknown surface</td>
</tr>
<tr>
<td>A</td>
<td>area emitting radiation energy, ft²</td>
</tr>
<tr>
<td>Q</td>
<td>total energy emitted by area, BTUs</td>
</tr>
<tr>
<td>T</td>
<td>absolute temperature, °Rankine</td>
</tr>
<tr>
<td>σ</td>
<td>Stefan-Boltzmann constant, BTU/sec-ft²-°R⁴</td>
</tr>
<tr>
<td>ε</td>
<td>total hemispherical emissivity</td>
</tr>
<tr>
<td>θ</td>
<td>azimuth angle of hemisphere, radians</td>
</tr>
<tr>
<td>λ</td>
<td>wavelength of X-ray radiation, cm</td>
</tr>
<tr>
<td>ϕ</td>
<td>elevation angle of hemisphere, radians</td>
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CHAPTER 1
INTRODUCTION

1.1 History. With the advent of the space age the speed of aircraft and pilotless vehicles increased into the high supersonic and hypersonic range. Because of this increase in speed, an increase in the heating of the vehicle was noted. This increase in the temperature of the vehicle had to be controlled to keep the temperature within the structurally tolerable limits.

Cooling by radiation, either alone or in combination with other cooling methods, is one of the most promising means of keeping the temperature within the required limits. Radiant cooling is attractive because of its simplicity and reliability as compared with other cooling methods. Materials which are poor radiators may be coated with a thin layer of a material of good radiation properties thus making the original material a good radiator. The rate of loss of heat by radiation is directly proportional to the total hemispherical emissivity of the surface. Therefore, in view of these considerations, the Pilotless Aircraft Research Division
of the Langley Aeronautical Laboratory (1)* began the
investigation of high-emissivity surfaces from the viewpoint
of their application to the cooling of supersonic and
hypersonic aircraft.

1.2 Problem. Part of Langley Aeronautical
Laboratory's general research program on the aerodynamic
heating of supersonic and hypersonic aircraft was a report
on the measurements of total hemispherical emissivity of
various oxidized metals at high temperatures. The tests
pertaining to this part of the research were performed by
William R. Wade (2). Wade tested eight different metals in
quiescent air for their emissivity at different temperatures.
The metals tested were stainless steel (AISI-303), mild steel
(AISI-C1020), titanium (TMCA-Ti-75A), titanium alloy (RS-120),
copper, aluminum (AA-3003), molybdenum, and tantalum.
Measurements of the radiant flux were made with a thermopile
which had been calibrated by use of a reference black body.
Temperatures were measured by use of thermocouples attached
to the specimens.

The results of this experiment were promising in
that a stable oxide coating of high emissivity was obtained
for all metals except molybdenum and tantalum. All of the
results of the experiment were explainable except for
a sudden decrease in emissivity at 1400°F for the titanium

*Numbers in parenthesis identify references.
alloy (RS-120). Numerous specimens of this titanium alloy (RS-120) were tested and all had the same sudden decrease in emissivity at 1400°F. This consistent irregularity of the emissivity of this metal was the basis for this thesis. It was felt that any consistent behavior had an explanation which may have numerous factors bearing on the solution. But, if a few or even one of the factors could be found, it is possible that the behavior may be explained.

This study approached the problem from a microstructural basis. This approach permitted a study of the microstructure and changes in emissivity in an endeavor to obtain some correlation. This approach was decided upon after a preliminary investigation did show a change in the microstructure of the titanium alloy (RS-120) at the temperature of 1400°F. Also, Wade did not appear to consider the microstructure of the metal.

1.3 Approach to the Problem. The exact approach taken during the study of this problem was, first, to review all literature having a bearing on the subject, second, to analyze any results obtained from experiments which were performed concerning the thesis subject, and third, to correspond personally with an authority in the field to determine if there was any information of value which had not been published.
With all of the above information a search and study was made to determine if there may be a correlation between the consistent irregular behavior noticed by Wade and the microstructure of the metal. Since a correlation was found, it should be confirmed by an extended experimental study. Such an experimental study would have to be quite extensive with requirements which are far beyond the endeavors of this study. However, a suggested experimental program has been included in this study. This suggested program includes theory, apparatus required, and procedure for experimentation, which should facilitate the initial phase of the experimentation. The experimentation should not be limited to just the metal used in Wade's experiment, titanium alloy (RS-120), but should be extended to include other material.
CHAPTER 2
REVIEW OF LITERATURE AND EXPERIMENTS

2.1 Literature Searched. In searching the literature available on the subject of emissivity, the lack of consideration of emissivity from a microstructural standpoint became evident. Experiments have been performed and charts have been prepared in an endeavor to give some values of emissivity at certain temperatures for certain materials. Generally, most of the information available has not considered the variation of emissivity with microstructure. However, there were some books which did present some facts which did assist in a microstructural analysis.

In 1914 the English translation of Planck's theory of heat radiation was made available (3). Planck commented that emission of radiation always take place at the expense of energy and that only material particles, not geometrical volumes or surfaces can emit heat rays. Planck also stated that the surface of a body never emits rays, but rather it allows part of the rays coming from the interior to pass through. Planck noted that the coefficient of emission,
called emissivity, depends not only on the frequency of the emitting substance, but also, in a very complicated way, on the physical and chemical processes that take place in the body. Planck's book was a most complete work and in most of the following literature one can see its influence.

In 1935, Sara and Srivastava commented that radiation was dependent on the physical properties of the body (4). Sara and Srivastava, stressed the macrostructure as most authors did and left the microstructure analysis to the metallurgist.

In 1942, McAdams in his book (5) and Brown and Marco (6) in their book stressed the point that the emission of radiation is dependent on the nature of the body, in addition to its temperature.

In 1949, Dusinberre gave the same conclusions as McAdams (7). In 1959, Jakob and Hawkins commented that the transfer of energy by radiation was composed of three distinct components (8). The first of these components is the conversion of the thermal energy into electro-magnetic wave motion, the second component is the passage of the wave motion through the intervening space to the sensing body, and the third component is the reconversion of the wave-motion energy into thermal energy by the sensing (cold) body. The microstructural analysis of these three components
concerns mainly the increase in energy of the electrons and then their release of this energy as electro-magnetic waves. These waves must find their way to the surface to be emitted as radiation to the cold body. Naturally, on their way to the surface some may be absorbed or reflected so the microstructure can have an effect on the radiation.

Also in 1959, Eckert and Drake noted that, although considerable knowledge can be obtained on emissivity from a macroscopic study, for a complete knowledge of the subject a study of the radiation and microstructure is necessary (9). Eckert and Drake were the only authors of those noted in this thesis that mentioned the need for a study on emissivity from the standpoint of microstructure.

This concludes the comments that were available from the books searched in the field of emissivity and microstructure. The lack of literature in this field is just further evidence of the need for more study and correlation between the engineers and scientists.

2.2 Previous Experimentations. Although the literature has been rather general in nature in reference to this study, there have been a few experiments performed which have a close relation to this study.

In 1943, Wahlin and Wright conducted an experiment on emissivities and temperatures for some metals of the iron
group (10). In their experiment they noted the sudden change of emissivity as Wade did in his experiment. However, Wahlin and Wright had their specimen in a vacuum during experimentation while Wade kept his specimen in the local quiescent air. There were a few other minor differences in apparatus and procedure which had little effect on the results.

Wahlin and Wright noticed that over the range of temperatures covered (1500°F - 2000°F) the emissivity was nearly constant for iron except where the microstructure changed from alpha, the body centered cubic, to gamma, the face centered cubic (11). The point where the microstructure makes this change in iron is the \( A_3 \) point (12). This was the first experiment in this field by these men and they commented that future experiments would be planned to investigate this change in emissivity. This initial report discussed the apparatus and procedure thoroughly and commented only briefly on the results of the experiment.

In 1948, Wahlin and Knop continued this research with an experiment on the emissivities and temperatures of iron and cobalt (13). This experiment utilized the same theory, apparatus, and procedure of the previously mentioned Wahlin and Wright experiment. The results of this experiment for iron showed the sudden decrease in emissivity at the \( A_3 \) point previously mentioned. This experiment also noted the
sudden increase in emissivity at the $A_4$ point when the temperature was increased to that point. The $A_4$ point ($2550^\circ F$) corresponds to that temperature at which the crystal structure of the iron changes from the gamma structure, the face centered cubic, to the delta structure, the body centered cubic. It should be noted here that the face centered cubic crystal structure is more dense than the body centered cubic crystal structure. Therefore, when the emissivity of the iron decreased at the $A_4$ point, the crystal structure was changing to the more dense crystal structure. And when the emissivity increased at the $A_4$ point, the crystal structure was changing to a less dense crystal structure. A possible correlation at this point would be that when the density of the crystal structure increases the emissivity decreases, and when the density of the crystal structure decreases the emissivity of the metal increases.

The second metal considered in Wahlin and Knop's experiment was cobalt. The emissivity of cobalt was rather consistent until the Curie Point was reached. The Curie Point corresponds with the temperature where ferromagnetism disappears from a ferromagnetic metal and what was once a well ordered metal becomes a disordered metal (12). In this experiment the emissivity increased when the Curie Point was reached. Cobalt, at the Curie Point, experiences a positive magnetostriction. This positive magnetostriction
caused a decrease in the density of the cobalt when the temperature was increased to the Curie Temperature.

In the conclusions to this experiment Wahlin and Knop suggested the possibility of noting structural changes in metals by plotting a graph with temperature and emissivity as abscissa and ordinate, respectively. To illustrate the marked increase or decrease of emissivity witnessed by Wahlin and Knop their results are presented on Figures 2.1 and 2.2 (13).

Also in the same year, 1948, Knop prepared another experiment (14). Knop's experiment was concerned with the emissivity of the iron-tungsten alloy and the iron-cobalt alloy. His results on this experiment were consistent with the previous experiments. He utilized the same theory, apparatus, and procedure as the two previous experiments. However, in the conclusions Knop mentioned the correspondence between the density and emissivity.

This concludes the experiments which have a relation to this thesis. These experiments present the consideration that there may be a correlation between the density of a metal and its emissivity. From all three of these experiments results were produced which showed the emissivity varying inversely with the density of the metal.

2.3 Personal Correspondence. After comparing the literature and experimental results, it was felt that if
FIGURE 2.1
WAHLIN'S AND KNOP'S EXPERIMENTAL RESULTS FOR IRON. (Reference 13)

FIGURE 2.2
WAHLIN'S AND KNOP'S EXPERIMENTAL RESULTS FOR COBALT. (Reference 13)
there were any other information available which had not been published one of the authorities would know of it.

A letter was sent to Professor Wahlin asking for any additional references on the subject. Professor Wahlin's reply contained no reference to additional publications other than a comment that he had done a similar study for the Manhattan Project on uranium, with the same results. His final paragraph was very conclusive, it follows: "In general, you can say that whenever the change is such as to cause an increase in the density of the material the emissive power goes down, and when the density is decreased the emissive power goes up." (15). This brief and clear summary of his work in this field should provide a desire to investigate the emissivity and density of materials.

This is not meant to be a complete solution to this problem but only a step in that direction. Planck's comment that the emissivity is dependent, in a very complicated way, upon the physical and chemical processes of the material now seems to be valid.
CHAPTER 3

CORRELATION OF WADE'S EXPERIMENT

3.1 Introduction. In consideration of the literature searched, the experiments mentioned, and the comment of Wahlin's on the inverse relation of density and emissivity, it appears that a correlation should be attempted in this area.

3.2 Area of Undetermined Behavior. The first step must be to define the area of Wade's experiment where the undetermined behavior was noted. The metal referred to when Wade commented on this undetermined behavior was the titanium alloy (RS-120). The plotted results which Wade obtained during experimentation with this alloy are reproduced on Figure 3.1 (2). Letters and numbers have been added on the graph to assist in this discussion. From point 2 to point 3 on the graph, also referred to as line C, was the area of undetermined behavior. All three of the lines, A, B, and C represent the measured results for Emissivity versus Time for three different but constant temperatures. Wade used a time and emissivity graph rather than a temperature and emissivity graph since he was interested in the time to establish an oxide coating on the specimen. Line A
FIGURE 3.1
WADE'S EXPERIMENTAL RESULTS FOR TITANIUM ALLOY RS-120 (Reference 2)
represents the measured results at a constant temperature of 1000°F, line B at 1200°F, and line C at 1400°F. The measured results along line C are listed as unexplained behavior because the expected change in emissivity with an increase in temperature and time of oxidation should have been an increase. This sudden decrease in emissivity, which was consistent for all of the titanium alloy (RS-120) specimens tested, could not be explained. Wade's experiment was an endeavor to establish a stable oxide coating on the surface of metals to increase their emissivity. With an increase in thickness of oxide coating the emissivity of the surface increased. The emissivity of an oxide is far greater than the emissivity of a bare metal. So, as the temperature of the metal was increased the thickness of the oxide coating increased, until finally the coating was thick enough to have a stable emissivity.

Although the measured results for emissivity along line B were as expected, there was a very sudden increase in emissivity while the alloy was being held at a constant temperature of 1200°F. This increase in emissivity could be attributed to an increase in the oxide layer but, as will be seen later, additional matters may have been involved.

3.3 Microstructure of the Metal. The results of the experiment near the area of undetermined behavior were studied. Now, a study of the microstructure of the alloy
The constitutional diagram for this titanium alloy (RS-120) is reproduced on Figure 3.2 (16). Additional letters have been placed on the diagram to facilitate cross-reference with the experimental results. Point A of Figure 3.2 indicates a temperature of 1000°F which is the same as the temperature for line A of Figure 3.1. Point B of Figure 3.2 indicates a temperature of 1200°F which is the same as line B of Figure 3.1, and Point C indicates a temperature of 1400°F which is the same as the temperature of line C of Figure 3.1.

The composition of the titanium alloy (RS-120) was seven per cent manganese. The temperature points of A, B, and C are on the seven per cent manganese composition line.

At point A of Figure 3.2 the microstructure is composed of all hexagonal close packed crystals. At point B the microstructure is composed of approximately one-half hexagonal close packed crystals as before, and one-half body centered cubic. Half of the hexagonal close packed crystals at A changed to the body centered cubic structure. At point C the microstructure is all body centered cubic, since the remainder of the hexagonal close packed crystals at B have transformed into the body centered crystals.

3.4 Explanation of Undetermined Behavior. With the sudden changes in emissivity as well as the microstructure...
FIGURE 3.2

CONSTITUTIONAL DIAGRAM: TITANIUM-MANGANESE
(Reference 16)
between the temperatures of 1000°F and 1400°F, there may be a correlation between the microstructure and the emissivity.

Both line A of Figure 3.1 and point A of Figure 3.2 refer to the condition of the alloy at the same temperature, 1000°F, and they express the emissivity and microstructure, respectively, at that temperature. The emissivity is almost constant and the microstructure is a one phase structure of hexagonal close packed crystals.

Along line B of Figure 3.1 and at point B of Figure 3.2, which depict the emissivity and microstructure at 1200°F, the emissivity increases with time. The microstructure, in the meantime, changed from the one phase hexagonal close packed crystals to the two phase structure of half hexagonal close packed and half body centered cubic crystals. If we accept the conclusions that the density of the microstructure and emissivity of the metal vary inversely, the density at point B should be less than the density at point A since the emissivity increased along line B of Figure 3.1. This conclusion is verified since the hexagonal close packed crystals are more dense than the body centered cubic crystals and the microstructure changed from a one phase structure to a two phase structure. A one phase structure should be more uniform and have less imperfections than a two phase structure.
Along line C of Figure 3.1 and point C of Figure 3.2 the emissivity and microstructure of the titanium alloy (RS-120) at the temperature of 1400°F are revealed. The emissivity decreased and the microstructure changed from the two phase crystal structure of half hexagonal close packed and half body centered cubic to the one phase crystal structure of all body centered cubic. The decrease in emissivity should signify an increase in density if the inverse relation of density and emissivity is correct. The change of the remaining hexagonal close packed crystals to body centered cubic crystals would seem to have the effect of decreasing the density which would be opposite to what is desired. However, the effect of the change from a two phase microstructure to a single phase microstructure would have been accompanied by a decrease in the imperfections in the microstructure which would present a more dense microstructure. Therefore, if the decrease in imperfections more than compensated for the transformation of the hexagonal close packed crystals to the body centered cubic crystals, an increase in the density would have been the result.

3.5 Results. The results of this correlation are mixed. There seems to be a correlation between the microstructure and the emissivity of the titanium alloy (RS-120). The explanation of the increase or decrease in the
density of the microstructure, although fairly adequate for point B, Figure 3.2, was rather weak for point C. There were more items involved than just the phases present and imperfections. The density of the alloy, if it were a single crystal, may have been easier to define. However, the titanium alloy (RS-120) was not a single crystal.

There are numerous items which may have an effect on the density of the alloy. In a report from Frankford Arsenal, D. J. Murphy noted many of the items which have an effect on the density, some of which are voids, lattice vacancies, grain boundaries, dislocations and the presence of foreign atoms, both substitutional and interstitial (16). Wade commented on the absorption of nitrogen as a possible cause of the decrease in emissivity along line C, Figure 3.1. Nitrogen is absorbed by the titanium alloy as an interstitial atom. The nitrogen atom slips in between the alloy atoms and therefore increases the density of the microstructure (17).

With the correlation obtained between the emissivity and the microstructure, it is felt that experimental studies should be undertaken to attempt to experimentally check the correlation between the emissivity and the microstructure.
CHAPTER 4

SUGGESTED EXPERIMENTAL APPROACH

4.1 Introduction. This study of the emissivity and microstructure of certain metals has suggested a correlation between the density of the metal and the sudden unexpected changes in emissivity. This sudden change in emissivity was noted in Wade's experiment for the titanium alloy (RS-120). Wade called this sudden change "unexpected behavior" and could not account for it being present in each specimen of the titanium alloy (RS-120) that was tested. This study has attempted to show some correlation, by use of the constitutional diagram of titanium-manganese and other experimental results. This attempted correlation should be substantiated with an experiment designed for this purpose. Such an experimental arrangement would not be limited to the study of this one metal but could be utilized to investigate any other metal desired.

Since the experiment is an attempt to provide information on the correlation of a metal's emissivity and the metal's density, a plot of Emissivity versus Temperature would reveal any sudden changes in the emissivity. At those temperatures where there is a sudden change in the emissivity
of the metal a comparison of the crystal structure which was present immediately prior to the sudden change in emissivity and the crystal structure following the sudden change should give some evidence of a sudden change in the crystal structure.

A possible means of determining the density of the crystal structure would be by X-ray diffraction. The equipment and procedure for accomplishing this will be discussed later in this chapter.

4.2 Theory. To determine the emissivity of a metallic surface, the energy emitted from the surface must be compared to the energy emitted from a reference black body surface. With a few simplifying considerations the calculations may be restricted to a simple comparison of the radiant flux of the two surfaces.

The radiant energy emitted by a surface is:

\[ Q = \varepsilon \sigma T^4 At \]  

where \( \varepsilon \) is the total hemispherical emissivity of the surface, \( \sigma \) is the Stefan-Boltzmann radiation constant, \( T \) is the absolute temperature of the surface, \( A \) is the area of the surface emitting energy, and \( t \) is the length of time during which radiation occurs.
From Equation (1), the rate of emission, $q$, is:

$$q = \frac{Q}{t} = \varepsilon \int T^4_A \quad (2)$$

and from Equation (2) the emissivity is:

$$\varepsilon = \frac{q}{\int T^4_A} \quad (3)$$

The ratio of the emissivity of a surface (subscript $u$) to the emissivity of a black body surface (subscript $b$) is:

$$\frac{\varepsilon_u}{\varepsilon_b} = \frac{q_u T_b^4 A_b}{q_b T_u^4 A_u} \quad (4)$$

but the emissivity of a black body surface is unity, therefore if the temperatures and the areas of the two surfaces are made equal, Equation (4) reduces to:

$$\varepsilon_u = \frac{q_u}{q_b} \quad (5)$$

There exists a relationship between the rate of emission, $q$, and the radiant flux, $f$, such that the integral of the radiant flux (radiant energy per unit of area per unit of time) over the surface of a hemisphere surrounding the emitting area (see Figure 4.1) must equal the rate of emission, $q$. The differential area is equal to the product of $(r d\phi)$ and $(r \sin \phi d\theta)$. Then the relationship between the rate of emission, $q$, and the radiant flux, $f$, is:
FIGURE 4.1
HEMISPHERE OF INTEGRATION TO DETERMINE THE RADIANT ENERGY EMITTED FROM AREA "A"
(Reference 1)
\[ q = \int_0^{\frac{\pi}{2}} \int_0^{2\pi} r^2 f \sin\theta \, d\theta \, d\phi \]  

(6)

Letting the radius of the hemisphere of the unknown surface and the black body surface equal each other the emissivity of Equation (5) reduces to:

\[ \varepsilon_u = \int_0^{\frac{\pi}{2}} \int_0^{2\pi} \frac{f_u \sin\theta \, d\theta \, d\phi}{f_b \sin\theta \, d\theta \, d\phi} \]  

(7)

The flux for a black body is equal to the product of the normal flux, \( f_{b.o} \), (the subscript "o" signifies the normal flux) and the cosine of the elevation angle, \( \phi \). This is possible since the black body follows Lambert's Law. Substituting in Equation (7) for the black body flux:

\[ \varepsilon_u = \frac{\int_0^{\frac{\pi}{2}} \int_0^{2\pi} f_u \sin\theta \, d\theta \, d\phi}{f_{b,o}} \]  

(8)

and when the unknown surface emits in accordance with Lambert's Law, Equation (8) reduces to:

\[ \varepsilon_u = \frac{f_{u,o}}{f_{b,o}} \]  

(9)

Thus, the total hemispherical emissivity of surfaces which emit in accordance with Lambert's Law may be determined
by calculating the ratio of the total normal flux from a non-black surface to the total normal flux from a black body surface.

4.3 General Procedure. Although this experimental approach has been designed to evaluate experimentally the conclusions suggested for the sudden change in the emissivity in Wade's experiment for the titanium alloy (RS-120), it need not be so limited. Since two independent calculations will be required, one for the emissivity and one for the density, a small sample of the titanium alloy (RS-120) must be prepared for the density calculations and a larger sample for the emissivity calculations. The density sample size requirements preclude its use for adequate emissive calculations. Both experiments, one for emissivity and one for density, should use the same stock material.

The general procedure of the experiment should be first to attempt to determine at what temperatures the emissivity of the metal has sudden changes. At these sudden changes in emissivity the temperatures should be noted. Once the temperatures are determined at which the sudden changes in emissivity are noticed, the first phase of the experiment would be complete. In the second phase of the experiment, an attempt should be made to determine the lattice parameters (atomic dimensions of
the crystal structure) of the metal. These parameters can be determined by use of the X-ray camera at the temperature of the metal just prior to the sudden change in emissivity and at the temperature just following the change. With the lattice parameters and the crystal structure known, the density of the microstructure just prior to and just following the sudden change in emissivity can be determined. In the third phase of the experiment the degree of change in density may possibly be correlated. If the degree of change in emissivity can be correlated with the degree of change of density then with any future change in emissivity the change of density could be calculated. This completes the general approach to the procedure. A more specific consideration of each of the three phases follows.

4.3.1 Emissivity Calculation. The purpose of this phase of the experiment is to develop a graph of the emissivity as it changes with an increase in temperature.

A specimen of the metal to be tested having no larger than a 1/8 sq ft surface area (so as not to exceed heating capacity of power supply) should be placed in a bell jar vacuum apparatus. A thermocouple should be attached to the specimen to record its temperature.
The thermocouple should be located opposite the area of the specimen that will emit the radiation to be measured by the sensing device. Power leads to the specimen must be attached to provide a controllable heat source to the specimen. The bell jar with the test specimen installed should then be attached to the vacuum equipment and sealed. This completes the test specimen procedure and it is now ready for the vacuum equipment to establish a vacuum of approximately $10^{-6}$ mm mercury.

The sensing device (see Figure 4.4) must be calibrated by use of a reference black body before it may be used to measure the radiation emitted from the test specimen. A cylinder with a high-emissivity surface should be installed in a high-temperature furnace. A thermocouple must be attached to the cylinder to measure its temperature. With the cylinder installed in the high-temperature furnace, the radiation emitted through a peephole of the furnace by the cylinder will approximate black body radiation. This radiation when sensed by the sensing device will produce a voltage. This voltage should be measured by a potentiometer. The values of voltage at varying temperatures will produce a graph of black body temperature and voltage developed. On this graph the normal flux may be noted since it is a function
of absolute temperature to the fourth power.

With this graph and the sensing device calibrated, measurements of the radiation being emitted by the test specimen may be made. The radiation emitted by the test specimen will develop a voltage which should be measured by the potentiometer. This voltage may be referred to the graph obtained in black body calibration and by comparison of normal flux values obtained, an emissivity may be calculated for a specific temperature.

A graph of Emissivity versus Temperature may be produced from the results of the above measurements. This graph should show any sudden changes in the emissivity, and at the temperatures of these sudden changes in emissivity, the density should be determined. The next section will discuss the procedure for the density calculations.

4.3.2 Density Calculation. At those temperatures where the emissivity had changed abruptly, the density of the microstructure just prior to and following this sudden change must be determined. With the use of a X-ray camera the density at high temperature may be accurately determined.

A small specimen (1 cm x 0.5 cm x 0.1 mm) of the same test metal as used in the emissivity phase should be
installed in the apparatus of a vacuum container (see Figure 4.3). The small size of the specimen is required to limit the heating requirement to a minimum. A thermocouple is attached to the apparatus to measure the temperature of the specimen.

With the specimen installed and the bell jar sealed the system may be evacuated to approximately $10^{-6}$ mm mercury. Then the X-ray generating unit may be employed to radiate the specimen by directing the radiation through the AlSi (Aluminum-Silicate) window at the specimen. The AlSi windows are utilized because of their high transmissivity for X-rays. This radiation is absorbed by the specimen and the specimen re-radiates X-rays at the same wavelength and at the same angle of incidence as the incoming radiation. The re-radiated X-rays will pass through an exit window of AlSi and be sensed by a geiger counter at the exit window.

The geiger counter may be moved on an arc to measure the angle deviation where the maximum intensity of radiation is sensed. This angle is utilized to determine the lattice parameters of the specimen. With the lattice parameters of the test specimen known, the density of the specimen may then be determined (Appendix B).
Electro-magnetic waves 1 and 2 are in phase, maximum intensity, when the distance A-B-C is equal to the wavelength, $\lambda$.

From the diagram:

$AB = d \sin \psi$  
$BC = d \sin \psi$  
$A-B-C = 2d \sin \psi$

and when $2d \sin \psi = n\lambda$, where "$n$" is a positive integer, waves are in phase and at maximum intensity.

**FIGURE 4.2**

**TWO DIMENSIONAL PORTRAYAL OF INTERPLANAR DISTANCE**
The determination of the lattice parameters from the angle measured by the geiger counter is based on the geometry of the crystal structure. Since the angle measured by the geiger counter is located by sensing the maximum radiation near a predetermined angle, the angle represents the location where the radiation from first few layers of the specimen is in phase and therefore at maximum intensity (see Figure 4.2). The predetermined angle is selected from a knowledge of the crystal structure present in the specimen at that temperature and the approximate angle of maximum intensity for planes in the crystal. A constitutional diagram will provide the required crystal structure information. This approach is possible since the wavelengths of X-ray radiation are of the same order of magnitude as the lattice parameters of the crystal structures.

The distance between planes is obtained by use of the following formula:

\[ \lambda = 2d \sin \varphi \]  \hspace{1cm} (10)

where "d" is the interplanar spacing, \( \varphi \) is the angle measured by the geiger counter, and \( \lambda \) is the wavelength of the X-ray radiation. Once the angle \( \varphi \) is known, and \( \lambda \) is predetermined, d may be determined. With the crystal
structure known, an equation for the lattice parameters is available (20). For a cubic crystal structure the equation is:

\[ d = \frac{a}{(h^2 + k^2 + l^2)^{\frac{1}{2}}} \]

(11)

where "d" is the interplanar spacing for a specific plane, "a" is the lattice parameter (edge of cube), and "h", "k", and "l" are the Miller Indices of the specific plane. Once the interplanar spacing, "d", for a specific plane has been determined, the only unknown in equation (11) is the lattice parameter, "a".

4.3.3 Correlation of the Results. If, in addition to providing experimental proof that the inverse relation between the emissivity and density of a metal is valid, there exists a correspondence between the degree of change of both the emissivity and density, then more definite conclusions may be drawn. A check should be made to determine by what per cent the former emissivity changed. This per cent of change should be correlated with the per cent of change in density of the metal, if possible. The results should be tabulated and perhaps plotted. The temperature range at which this behavior occurs should also be recorded.
4.4 Apparatus Required for Experimentation.

In the experiments mentioned in this thesis the apparatus was designed to measure the changes in the emissivity. The correlation of the changes in the emissivity to the density of the specimen was dependent upon the constitutional diagram. The utilization of the constitutional diagrams to correlate the microstructure and the sudden changes in emissivity was adequate since the changes in emissivity occurred at the phase changes and Curie points. However, this correlation would only permit the conclusion that if the emissivity did change then the density of the microstructure would change inversely. There was no consideration of the degree of change for the correlation. A certain change in emissivity may correspondingly signify a certain amount of change in density. An attempt to accomplish such a correlation demands that both the emissivity and density of the microstructure be carefully calculated at the temperatures of interest. The apparatus to calculate the emissivity and density are available and will be discussed in this chapter. An estimate of the cost of the apparatus will be included as an appendix to this thesis (Appendix A).

4.4.1 Apparatus Required for Density Calculations. In calculating the density of metals at
high temperatures, the utilization of an X-ray camera to assist in determining the density would be convenient to use. The determination of density changes from the data obtained by use of an X-ray camera would be a more accurate and simpler method than the gravimetric method. This conclusion has been strengthened with the reporting of the development of a high temperature, high vacuum, X-ray camera by the Journal of Scientific Instruments. Hatt, Kent, and Williams reported the development of a camera which can be used to overcome the difficulties of specimen contamination and the elimination of gases in the camera interior (18).

Since the high vacuum, high temperature, X-ray camera was just developed, it is not known to be available commercially. However, since the article in the Journal of Scientific Instruments is so complete the X-ray camera could be fabricated locally. Fabrication of this apparatus is recommended, even if it were available, to provide an adaptability of the high vacuum apparatus to both the X-ray camera apparatus and the test specimen requirement for high vacuum equipment which follows. Such an adaptation would save the expense of duplicating the expensive high vacuum apparatus. A recommended fabrication is given on Figure 4.3.
In reference to Figure 4.3, there are two sections shown, the vertical Section A-A and the horizontal section. The location of the exit window is dictated by the planes of the crystal structure that must be sensed by the geiger counter, as discussed earlier. The energy to heat the specimen to the required temperature is supplied by a 1000 watt, projection bulb with ellipsoidal reflector as shown. If more heat is required an additional heater may be utilized. One heater is capable of heating a specimen to 1000°C. Notice on the horizontal section the degree scale to determine the angle which is required to calculate the interplanar spacing, d, mentioned earlier. The specimen is held by the specimen holder and rotated by the magnetic drive. The AlSi windows are utilized for their high transmissivity of X-rays.

4.4.2 Apparatus Required for Emissivity Calculations. The apparatus required for the emissivity calculations should include a reference black body with furnace, a container for the test specimen, a temperature sensing device to record the temperatures of the test specimen and black body, a power supply to heat the test specimen, and a device to sense and measure the radiant normal flux emitted by the test specimen and the black
FIGURE 4.3

HIGH TEMPERATURE HIGH VACUUM X-RAY CAMERA, SHOWING VERTICAL SECTION AND HORIZONTAL SECTION
body. These items will constitute a minimum requirement for satisfactory calculations.

The reference black body with furnace could utilize a Laboratory and Heat Treating, Automatically Controlled, 2000°F, Lindberg type B-2, Furnace, (Central Scientific Company #13703, 115 volts, 60 cycle), and a high-emissivity, cylindrical body small enough to fit into the furnace. The cylindrical body, which must be fabricated, may be supported in the furnace by simple supports which permit the sensing device to view the emitting area through a peephole. A thermocouple must be attached to the cylindrical body, opposite the area emitting radiation, to record its temperature.

The container or chamber for the test specimen must have a high vacuum capability. A High Vacuum Pump Outfit, Cenco Mercury Diffusion Type, (Central Scientific Company #93265, 110 volts, 60 cycle) and a Bell Jar, High Straight Form, Glass Stoppered Top, (Central Scientific Company #14315) would provide an adequate chamber and vacuum system for the test specimen. A calcium fluoride plate should be installed in the bell jar to provide a greater transmissivity for the radiation of the test specimen, since the transmissivity of glass is limited.
An adequate temperature sensing device to show the temperatures of the test specimen and the black body would be an Indicator Pyrometer, 0-2000°F, Veritell Model I, (Central Scientific Company #13672). This pyrometer would be used to record the temperature of the body emitting radiation and its accuracy of one to five per cent of its scale would be adequate for this comparison.

A specimen heater power supply of capable performance should be available with variable controls to provide fine temperature control of the test specimen. A possible commercial combination is a variable auto-transformer (Superior Electric Co., Bristol, Conn., powerstat, model #1256L-25-B) feeding a stepdown transformer (Central Transformer Co., Chicago 7, Ill., model GI-117). The input to the autotransformer is 440 volts, 60 cycle, single-phase current, and it is adjustable from zero to 440 volts at a maximum current of 28 amperes. The stepdown transformer has an output of 4 volts at 440 volts input and a maximum output current of 600 amperes. The output of the stepdown transformer is attached to the test specimen. This energy is sufficient to raise the temperature of the specimen to 2000°F if its area is less than 1/8 ft².
The requirement for a sensing device to measure the radiant energy emitted by the test specimen and the black body should include the following as a minimum. A calcium fluoride lens to transmit and focus the radiation on a thermopile of at least ten thermocouples. The thermocouples should be blackened to increase their absorptivity. An optical stop should be present to permit a definite area of radiation to be viewed by the thermopile. A water-cooled stop should be included to prevent excessive heating of the sensing device. A shutter arrangement should be included to permit an exact time exposure of the thermopile. A diagram of a suitable arrangement of these items is given on Figure 4.4.

The final item required is an instrument to measure the voltage sensed by the thermopile of the sensing device. A Leeds and Northrup Company Potentiometer #8692 with an accuracy of 0.05 millivolts would be sufficient to establish a relative radiant normal flux.

4.4.3 Interconnection of Apparatus. The interconnections between the apparatus mentioned in the emissivity calculation phase will be noted first. The pyrometer will be connected to either the test specimen
FIGURE 4.4

SENSING DEVICE TO MEASURE RADIANT ENERGY EMITTED FROM TEST SPECIMEN AND REFERENCE BLACK BODY (Reference 1)
or the black body, which ever is being checked at the time. The sensing device will be connected to the potentiometer to record the relative values of voltage developed by the thermopile. The sensing device will be so placed to observe the radiation from either the black body cylinder or the test specimen. The heater supply will be connected to the test specimen. The vacuum equipment will be connected to the test specimen chamber. The only piece of equipment which will require movement is the sensing device, all other equipment may be permanently located. A recommended layout of equipment is included in Figure 4.5.

The density measurement equipment will not require movement and other than attaching the vacuum equipment to the X-ray camera no other interconnection is required.

4.5 Alternate Experimental Approach. This experiment is actually two separate experiments. The emissivity calculation is one separate experiment and the density calculation is another separate experiment. The purpose for keeping both experiments together as one experiment was to save the expense of duplicating equipment. The high-vacuum equipment and the temperature sensing pyrometer were required for use in both experiments.
FIGURE 4.5
SUGGESTED EXPERIMENTAL LAYOUT
(Reference 1)
It may be desired to separate the experiments because of an availability of equipment in different buildings from which the equipment may not be moved. This division of the experiments would be recommended at the University of Arizona. The X-ray equipment of the Metallurgy Laboratory could be used in conjunction with the high vacuum equipment in that laboratory to calculate the density of the material. The high vacuum equipment and sensing equipment of the Mechanical Engineering Department could be utilized for the emissivity calculation of the material.

The apparatus recommended for the experiment is believed to be about the most economical that is available. There is one major improvement that would be recommended if sufficient money were available. The utilization of a high-vacuum equipment with all associated equipment completely installed would simplify and provide a more efficient experiment. Central Scientific Company has such an apparatus available for $3950.00. The item is a Laboratory Vacuum System #94700.
CHAPTER 5
CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions. The sudden changes in emissivity of the titanium alloy (RS-120) did have some correlation with the changes in density of the microstructure of the alloy. This correlation between the emissivity and density of a metal is believed to be of an inverse nature. An increase in emissivity is believed to be accompanied by a decrease in the density of a metal.

The possibility exists that the degree of change in emissivity of a metal may be accompanied by a corresponding degree of change in the density of the metal. If this correspondence does exist the change in density of a metal may be predicted from a study of the emissivity, or the change in emissivity may be predicted from a study of the density.

The lack of literature relating to the correspondence between the emissivity and microstructure is evident. The need for extended experimentation in this area is also evident.

The expense of the proposed experiment as mentioned in Appendix A assumes that none of the equipment
is available. Normally, most of this equipment or comparable equipment is available at most universities. The availability of equipment at the University of Arizona for this experiment is noted in Appendix C.

5.2 Recommendations. The first recommendation is that the experimentation or one of comparable nature be undertaken at the earliest opportunity to provide the experimental conclusions to the suggested correspondence between the emissivity and density of a metal.

It is recommended that any experimental study in this field not limit the experiment to the titanium alloy (RS-120) but include as many other metals as possible.

It is also recommended that controlled atmospheres be introduced into the vacuum chamber to observe the effect of these atmospheres on the emissivity and density of the metals. Such an approach should provide some correlation as to the effects oxygen and nitrogen have on the emissivity and density of the metal.
### APPENDIX A

**Cost of Experimental Apparatus**

<table>
<thead>
<tr>
<th>ITEM</th>
<th>COST</th>
</tr>
</thead>
<tbody>
<tr>
<td>Laboratory and Heat Treating, Automatic Control, Control, 2000°F, Furnace, Lindberg Type B-2 (Central Scientific Company #13703), 115-230 volts, 50/60 cycle.</td>
<td>$758.00</td>
</tr>
<tr>
<td>High Vacuum Pump Outfit, Cenco Mercury Diffusion Type, 10⁻⁵mm Hg. (Central Scientific Company #93265) 120 volts, 60 cycle.</td>
<td>$222.50</td>
</tr>
<tr>
<td>Bell Jar, High Straight Form, Glass Stoppered Top, Size #3, (Central Scientific Company #14315),</td>
<td>$32.50*</td>
</tr>
<tr>
<td>Potentiometer, single-range, portable, temperature, Leeds and Northrup Co.</td>
<td>$400.00</td>
</tr>
<tr>
<td>Indicator Pyrometer, 0-2000°F, Veritell Model I, (Central Scientific Company #13672).</td>
<td>$95.70</td>
</tr>
<tr>
<td>High Vacuum High Temperature X-Ray Camera X-ray generator</td>
<td>$2000.00**</td>
</tr>
<tr>
<td>Geiger counter</td>
<td>$200.00**</td>
</tr>
<tr>
<td>Bell Jar, same as above</td>
<td>$32.50*</td>
</tr>
<tr>
<td>Autotransformer Superior Electric Co., #1256L-25-B 440 volts, 60 cycle, single phase.</td>
<td>$261.00</td>
</tr>
<tr>
<td>Stepdown transformer Central Transformer Co., #61-117.</td>
<td>$105.00</td>
</tr>
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</table>

**TOTAL** $4107.20

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*Equipment requires slight modifications as mentioned in thesis.*

**Prices courtesy of Blair Surgical Supply, Tucson, Arizona.**
APPENDIX B

Sample Density Calculations

The crystal structure and the lattice parameters must be known for these calculations. The crystal structure and the lattice parameters may be determined by use of the X-ray diffraction approach (19).

Example: Given: Crystal structure = BCC
Lattice parameter = 2.86 Å
Substance = Iron

\[
\text{Density} = \frac{\text{weight/unit cell}}{\text{volume/unit cell}} = \frac{\text{number of atoms/unit cell} \times \text{weight per atom}}{\text{lattice parameter (edge)}^3}
\]

Since there are 2 atoms/unit cell in the BCC structure, and the weight per atom is 55.84 gm/6.02x10^{23} atoms, the weight per unit cell is:

\[
\text{weight/unit cell} = 2(55.84/6.02x10^{23}).
\]

The volume/unit cell is just the cube of the lattice parameter or:

\[
\text{volume/unit cell} = (2.86x10^{-8} \text{cm})^3.
\]

Therefore:

\[
\text{Density} = 2(55.84/6.02x10^{23})/(2.86x10^{-8})^3 = 7.9 \text{gm/cm}^3
\]
APPENDIX C
Availability of Experimental Apparatus at the
University of Arizona

<table>
<thead>
<tr>
<th>Equipment</th>
<th>Location</th>
<th>Availability</th>
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</thead>
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<tr>
<td>Furnace Assembly</td>
<td>Metallurgy</td>
<td>Fair</td>
</tr>
<tr>
<td>High Vacuum Equipment</td>
<td>Mechanical Eng.</td>
<td>Poor (in use)</td>
</tr>
<tr>
<td></td>
<td>Metallurgy</td>
<td>Fair</td>
</tr>
<tr>
<td></td>
<td>Physics</td>
<td>Fair</td>
</tr>
<tr>
<td>Bell Jar and associated glass requirements</td>
<td>Chemistry</td>
<td>Fair</td>
</tr>
<tr>
<td></td>
<td>Physics</td>
<td>Fair</td>
</tr>
<tr>
<td>Sensing Equipment</td>
<td>Mechanical Eng.</td>
<td>Good</td>
</tr>
<tr>
<td></td>
<td>Physics</td>
<td>Fair</td>
</tr>
<tr>
<td>X-ray generating unit.</td>
<td>Geology</td>
<td>Poor (in use)</td>
</tr>
<tr>
<td></td>
<td>Metallurgy</td>
<td>Fair</td>
</tr>
<tr>
<td></td>
<td>Physics</td>
<td>Fair</td>
</tr>
<tr>
<td>Geiger counter</td>
<td>Geology</td>
<td>Poor (in use)</td>
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<tr>
<td></td>
<td>Physics</td>
<td>Fair</td>
</tr>
<tr>
<td>Transformers</td>
<td>Electrical Eng.</td>
<td>Poor (only 220)</td>
</tr>
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</table>
REFERENCES


BACKGROUND REFERENCES

Holladay, J. W., "References to Research on High-Emissivity Surfaces," Defense Metals Information Center, Memorandum 57, Columbus, Ohio, 27 Jun 60.


Wood, W. D., Deem, H. W., and Lucks, C. F., "Emissivity and Emittance—What Are They?" Defense Metals Information Center, Memorandum 72, Columbus, Ohio, 10 Nov 60.