An X-ray study of engineering materials.

James Edward Butler 1916-
University of Louisville

Follow this and additional works at: http://ir.library.louisville.edu/etd
Part of the Chemical Engineering Commons

Recommended Citation
https://doi.org/10.18297/etd/1697

This Master's Thesis is brought to you for free and open access by ThinkIR: The University of Louisville's Institutional Repository. It has been accepted for inclusion in Electronic Theses and Dissertations by an authorized administrator of ThinkIR: The University of Louisville's Institutional Repository. This title appears here courtesy of the author, who has retained all other copyrights. For more information, please contact thinkir@louisville.edu.
AN X-RAY STUDY OF ENGINEERING MATERIALS

A Thesis Submitted to the Faculty of the Graduate School of the University of Louisville in Partial Fulfillment of the Requirements for the Degree of

MASTER OF CHEMICAL ENGINEERING

Department of Chemical Engineering

James Edward Butler
1940
AN X-RAY STUDY OF ENGINEERING MATERIALS

James Edward Butler

Approved by the Examining Committee:

Director: P.C. Guest

R.D. Williams

E. H. Cheston

E. C. Pettit

J. C. Williams

May 25, 1940

13268
# TABLE OF CONTENTS

<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>List of Tables</td>
<td>ii</td>
</tr>
<tr>
<td>List of Figures</td>
<td>iii</td>
</tr>
<tr>
<td>Acknowledgement</td>
<td>vi</td>
</tr>
<tr>
<td>Abstract</td>
<td>vii</td>
</tr>
<tr>
<td>Introduction</td>
<td>1</td>
</tr>
<tr>
<td>Historical</td>
<td>4</td>
</tr>
<tr>
<td><strong>Part I: Radiography of Metals</strong></td>
<td>9</td>
</tr>
<tr>
<td>Theoretical</td>
<td>10</td>
</tr>
<tr>
<td>Apparatus</td>
<td>13</td>
</tr>
<tr>
<td>Experimental Work</td>
<td>20</td>
</tr>
<tr>
<td>Conclusions</td>
<td>36</td>
</tr>
<tr>
<td><strong>Part II: X-Ray Diffraction</strong></td>
<td>38</td>
</tr>
<tr>
<td>Theoretical</td>
<td>39</td>
</tr>
<tr>
<td>Experimental Work</td>
<td>46</td>
</tr>
<tr>
<td>Conclusions</td>
<td>51</td>
</tr>
<tr>
<td><strong>Part III: Study of Wood by Use of X-Ray</strong></td>
<td>53</td>
</tr>
<tr>
<td>Theoretical</td>
<td>54</td>
</tr>
<tr>
<td>Experimental Work</td>
<td>57</td>
</tr>
<tr>
<td>Conclusions</td>
<td>88</td>
</tr>
<tr>
<td>Literature Cited</td>
<td>90</td>
</tr>
<tr>
<td>Vita</td>
<td>91</td>
</tr>
</tbody>
</table>
LIST OF TABLES

Table I  Typical Radiographic Calibration Data  22
Table II  Types of Radiographic Studies of Wood  59
# LIST OF FIGURES

<table>
<thead>
<tr>
<th>Figure</th>
<th>Title</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Figure 1</td>
<td>Diagram of High-Voltage Circuit</td>
<td>15</td>
</tr>
<tr>
<td>Figure 2</td>
<td>Rectifying Unit and Coolidge Transformer</td>
<td>18</td>
</tr>
<tr>
<td>Figure 3</td>
<td>Control Panel</td>
<td>19</td>
</tr>
<tr>
<td>Figure 4</td>
<td>Typical Calibration Radiograph</td>
<td>24</td>
</tr>
<tr>
<td>Figure 5</td>
<td>X-Ray Exposure Chart (for revealing blow-holes and inclusions)</td>
<td>26</td>
</tr>
<tr>
<td>Figure 6</td>
<td>X-Ray Exposure Chart (dense)</td>
<td>27</td>
</tr>
<tr>
<td>Figure 7</td>
<td>Optimum Current Curve</td>
<td>29</td>
</tr>
<tr>
<td>Figure 8</td>
<td>Radiograph of Small Casting, Showing Crack</td>
<td>31</td>
</tr>
<tr>
<td>Figure 9</td>
<td>Radiograph of Small Casting, Disclosing Blow-Holes</td>
<td>32</td>
</tr>
<tr>
<td>Figure 10</td>
<td>Radiograph of Section of Large Casting by Immersion Technique</td>
<td>34</td>
</tr>
<tr>
<td>Figure 11</td>
<td>Laue Pattern of Oxalic Acid Crystal</td>
<td>41</td>
</tr>
<tr>
<td>Figure 12</td>
<td>Laue Pattern of Citric Acid Crystal</td>
<td>42</td>
</tr>
<tr>
<td>Figure 13</td>
<td>Gnomonic Projection Curve</td>
<td>43</td>
</tr>
<tr>
<td>Figure 14</td>
<td>Laue Pattern of Oxalic Acid Crystal (gas-filled tube)</td>
<td>48</td>
</tr>
<tr>
<td>Figure 15</td>
<td>Laue Pattern of Oxalic Acid Crystal (same as Fig. 14 except unmasked)</td>
<td>49</td>
</tr>
<tr>
<td>Figure 16</td>
<td>Radiograph of Poplar, Showing Knot</td>
<td>61</td>
</tr>
</tbody>
</table>
Figure 17  Radiograph of Beech, Showing Knot .... 62
Figure 18  Radiograph of Indiana Soft Maple,
           Showing Knot .................................. 63
Figure 19  Radiograph of Hard Maple, Showing
           Knot ............................................. 64
Figure 20  Radiograph of Beech and Walnut,
           Showing Relative Absorption ............... 66
Figure 21  Radiograph of Oak and Magnolia,
           Showing Relative Absorption ............... 67
Figure 22  Radiograph of Black Gum and Magnolia,
           Showing Relative Absorption ............... 68
Figure 23  Radiograph of Soft Maple and Cedar,
           Showing Relative Absorption ............... 69
Figure 24  Radiograph of Hard Maple and Oak,
           Showing Relative Absorption ............... 70
Figure 25  Radiograph of Cedar and Soft Maple,
           Showing Relative Absorption ............... 71
Figure 26  Radiograph of Poplar Pieces of
           Different Thickness, Showing
           Relative Absorption .......................... 72
Figure 27  Radiograph of Two Pieces of Sound
           Oak Taken Across the Grain .................. 73
Figure 28  Radiograph of Sound Oak ............... 74
<table>
<thead>
<tr>
<th>Figure</th>
<th>Radiograph of Sap Gum, With and Across the Grain</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>29</td>
<td></td>
<td>75</td>
</tr>
<tr>
<td>30</td>
<td>Radiograph of Poplar, With and Across the Grain</td>
<td>76</td>
</tr>
<tr>
<td>31</td>
<td>Radiograph of Poplar Pieces That Have Been Subjected to Compression</td>
<td>77</td>
</tr>
<tr>
<td>32</td>
<td>Radiograph of Oak Piece Containing Internal Cracks</td>
<td>79</td>
</tr>
<tr>
<td>33</td>
<td>Radiograph of Oak, 1&quot; Thick, Containing Cracks Invisible to Both Surfaces</td>
<td>80</td>
</tr>
<tr>
<td>34</td>
<td>Radiograph of Oak, Showing Grub-Holes</td>
<td>81</td>
</tr>
<tr>
<td>35</td>
<td>Radiograph of Poplar, Containing Nails and Screws</td>
<td>83</td>
</tr>
<tr>
<td>36</td>
<td>Radiograph of Cedar, Containing Nails</td>
<td>84</td>
</tr>
<tr>
<td>37</td>
<td>Radiograph of Plywood, Consisting of 5 Laminated Woods With Flaw in Only the Top Piece</td>
<td>85</td>
</tr>
<tr>
<td>38</td>
<td>Radiograph of Plywood in Steps of 1/2 inch each</td>
<td>86</td>
</tr>
<tr>
<td>39</td>
<td>Radiograph of Plywood Composed of Different Woods</td>
<td>87</td>
</tr>
</tbody>
</table>
ACKNOWLEDGEMENT

The Author wishes to acknowledge the kind assistance and helpful guidance of Dr. R. C. Ernst who directed this research.
ABSTRACT

A mechanically rectified, 120 K. V. X-ray unit using a Coolidge tube was calibrated for producing radiographs of castings. Typical castings were radiographed by simple and immersion techniques, disclosing cracks, blow-holes, and inclusions.

A simple pin-hole camera was used in conjunction with both the Coolidge tube and a gas-filled tube for the production of several Laue diffraction patterns.

A study of typical flaws in wood materials was conducted by use of the same X-ray unit. Pictures of representative specimens are included.
INTRODUCTION
This investigation consisted of calibrating the unit for the study of castings, of the use of a small pin-hole camera for producing Laue patterns, and of a study of cellulosic material.

Various techniques were employed for producing radiographs of castings primarily for the purpose of inspecting the specimens for flaws such as blow-holes, inclusions, and internal cracks. The main advantage in the use of the X-ray for these inspections was that the specimen was not damaged in any way.

The Laue pattern is the only diffraction pattern employing polychromatic radiation. By operating at a sufficiently low voltage (approximately 30 kilovolts) radiation of the proper wave-length for the examination of crystals was produced. Several Laue patterns were produced with organic crystals.

Simple radiographic technique was employed in the study of wood. Pictures were included to show how easily various flaws can be identified without harm to wood. Grain structure was studied with a view to devising a possible means for identifying the various kinds of wood, especially when in a manufactured article.
either covered by veneer or a coat of pigmented paint.
Abosption differences of various woods were noticed.
HISTORICAL
Probably the first person actually to produce X-rays was William Morgan in 1785. He was unaware of having produced radiation that would penetrate substances opaque to ordinary light, and it remained for Wilhelm Konrad Von Rontgen to make the actual discovery in the autumn of 1895. While experimenting with a highly exhausted vacuum tube on the conduction of electricity through gases, Rontgen (2) observed the fluorescence of a barium platinocyanide screen lying near. When he enclosed the discharge tube with heavy black paper he observed that the crystals continued to fluoresce. By interposing obstacles Rontgen traced back the unknown or "X" rays to their source which proved to be the region of impact of the cathode rays on the walls of the tube. The feature of the new rays was their uncanny ability to penetrate many substances opaque to light. Rontgen saw at once the immense importance of his discovery to surgery, and communicated his results to the Physico-Medical Society of Wurzburg, Bavaria, in November, 1895.

During the first few weeks after Rontgen's discovery reports were received from all over the world recording the great value of the new radiation. About four days after his discovery was known in this country, X-rays were successfully used to locate a bullet in a patient's thigh (2).
Two months after Rontgen's discovery Sir Herbert Jackson introduced the focus tube having a metal target, thus paving the way for further research in this field.

The true nature of the new radiation was not known until Prof. Max von Laue discovered that a crystal could serve as a diffraction grating (1). He suggested, early in 1912, that the experiment be tried. This was then verified by Friedrich and Knipping, who worked under the direction of von Laue. This discovery aroused immediate interest, and in England it was taken up by Sir William Bragg and his son, who in the same year (1912) determined the first crystal structures, those of rock salt and zinc blende. At the same time they developed a method of analysis which was to be the basis of all further work.

The vacuum tube with which Rontgen made his discovery had a flat cathode, the cathode rays impinging on the glass walls. Experience soon showed the way to improvements. Campbell-Swinton inserted a Platinum target obliquely in the path of the rays, and later Sir Herbert Jackson (2) replaced the flat cathode by a concave one thus bringing the rays to a focus on the target. This new cathode was a great step forward, for the exposures were enormously shortened; and, owing to the small area of emission, the resulting photographs were improved in sharpness and detail.
The next great step forward was the discovery of the electron type tube in 1913 by Dr. W. D. Coolidge (7) of the Research Laboratory of the General Electric Company, Schenectady, N. Y. As basis for his work he turned to the work of Richardson, who had made quantitative measurement of the electrons produced by heating a negatively charged metal.

After these basic discoveries others continued to make their appearance even more rapidly. As a theoretical basis, the Danish physicist, Niels Bohr in 1913, by a few bold yet simple assumptions showed how the quantum theory could be applied to the problem of the structure of the atom. The great success of the Bohr theory in explaining X-ray and optical theory inspired many workers.

In 1923 Dr. Arthur H. Compton discovered the change in the wave-length of X-rays when scattered and also the total reflection of the rays. Later he effected the complete polarization of X-rays, and was first to obtain X-ray spectra from ruled gratings.

Debye, Sherrer, and Hull (3) have all contributed toward the perfection of what is now one of the most promising of all methods of analysis: The use of monochromatic radiation for making powder diagrams. In 1919 Hull (4) described a new method of chemical analysis by
means of X-ray diffraction. He gave illustrations of
the fact that the diffraction method tells the state of
chemical combination of the elements present in the un-
known, and stated the basis for the method: "that every
crystalline substance gives a pattern; that the same
substance always gives the same pattern; and that in
an admixture of substances, each produces its pattern
independently of the other, so that the photograph ob-
tained with a mixture is the superimposed sum of photo-
graphs that would be obtained by exposing each of the
components separately for the same length of time.
This law applies quantitatively to the intensities of
the lines, as well as to their positions, so that the
method is capable of development as a quantitative
analysis." Hanawalt, Rinn, and Prevost (3) have devel-
oped this method into a complete, new, workable system
of analysis. The great step made by these men was not
so much that they did a great bit of diffraction research
(which, however, they did) but was their development of
a classification system which makes possible the use of
tables of data for the identification of any chemical
substance that can be submitted in a powdered crystalline
form.
PART I

RADIOGRAPHY OF METALS
THEORETICAL
The X-rays used in radiography consist of an aggregate of radiation of different wave-lengths. The rays having the longer wave-lengths are absorbed much more readily by materials such as the steel castings studied in this section. The shorter rays penetrate farther into the casting and many of the very shortest pass completely through the steel, and strike the film, thus providing a record of the relative intensities of rays that have passed through the different portions of the casting.

The absorption of this radiation follows a definite law, known as the standard law of absorption. Each of the curves in Figures 5 and 6 is a graphical application of this law which may be written as: $I_x = I_0 e^{-mx}$, where $I_x$ is the intensity of a particular radiation, whose initial intensity was $I_0$, after absorption in a layer (of steel) of thickness, $x$ (7). The value $m$ is a constant (the linear absorption coefficient) for a given material and a given wave-length. Since $I_x/I_0 = e^{-mx}$ and $mx = -\log(I_x/I_0)$, and since $I_x$ must be a constant for a given film and given developing conditions, a constant times $x$ equals $\log I_0$ plus another constant. The other constant is $-\log I_x$. This absorption equation becomes a straight-line curve.
of the form, \( y = mx + b \), when plotted on semi-logarithmic paper. Since \( m \) does not remain constant when the voltage is varied, a separate curve must be plotted for each voltage.

In the equation, \( I_x \) equals \( I_0 \) times \( e^{-mx} \). \( I_0 \) represents the intensity of the rays before passage through a material of thickness, \( x \). \( I_x \) represents the intensity after the passage of the radiation through the material. In order to have the same amount of exposure \( I_x \) must always be the same.

This theory was verified in the study of the square-distance law as discussed in the experimental section.

These laws of absorption, and consequently similar exposure charts, apply to all radiographic work. But, because of the greater length of exposure, the charts are much more essential in radiographing metals.
APPARATUS
The X-ray unit used in this research was a three-milliamperes, 120-kilovolt outfit employing a tungsten-target Coolidge tube. Figure 1 is a diagram of the high-tension equipment showing all the principal connections. A synchronous motor was employed for mechanically rectifying the high-voltage current. The entire unit, when operating, required about 25 amperes at 220 volts. The power supplied was 60 cycle alternating single phase current at 220 volts. This voltage was stepped up by means of the Coolidge transformer to the voltage applied to the tube and was controlled by means of a rheostat and a step-watt auto-transformer, both operated from the switch board. The voltmeter was on the primary and was calibrated according to the primary-secondary ratio. This method of voltage measurement introduced a certain amount of unavoidable error in the voltmeter reading especially when operating over long periods of time at rather high currents. The Coolidge transformer heated up and increased the resistance in the secondary side.

The synchronization of the motor with the alternating current was automatic, but the direction of flow of the direct current thus produced was not automatic and had to be controlled by a polarity switch.
The rectifying device (Figure 2) consisting of the synchronous motor and the four-pole rectifying disc) served to reverse the direction of every second half-cycle and at the same time to apply only the peak of the waveform to the tube. This device produced a pulsating direct current of practically uniform voltage.

The operation of the unit consisted of a few simple steps after all preliminary precautions had been taken. The first of these precautions was to be certain that all other switches were off, especially those in the center of the control panel (Figure 3), before turning on the main switch. The high-voltage wires were carefully connected to the terminals of the tube, the double wire being attached to the filament end of the tube. Care was necessary in keeping the high-tension wires a safe distance from the framework of the unit.

The main switch was the first one to throw, then the small filament switch on the front of the panel (it was seldom necessary to have the filament switch in an "off" position). The motor was started by turning the motor knob left to the "start" position, holding the switch in this position only until the motor attains full speed, then throwing it to the "run" position. The polarity switch was turned in the direction of the
deflection of the polarity needle, and the transformer switch was turned to the "full" position unless it was desired to operate at voltages lower than 30 kilovolts in which case it was turned to the "limited" position. When this switch was connected the voltmeter reading appeared and this was adjusted by means of the step-wise rheostat and auto-transformer control knobs on the left side of the panel. For steady flow of high-tension current, then, the push-button switch on the panel was used. For intermittent flow and for short exposures the timer switch was used.

A sensitive milliammeter was connected in the high-tension line, and the reading on this meter was controlled by the Coolidge regulator (Figure 1). By means of a small switch on the milliammeter, the dial was made to read from 0 to 10 or from 0 to 100 milliampereas as desired.
Fig. 2 Rectifying Device and Coolidge Transformer
Fig. 3 Control Panel
EXPERIMENTAL WORK
In order to determine the optimum current-intensity, voltage, time of exposure, and focus-film distance for a given thickness of sample, a means had to be devised whereby samples of the material (steel, brass, or aluminum) of varying thicknesses could be radiographed without danger of fogging the film by secondary rays or because of the excessive fluorescence of the intensifying screens. Nine 3/4-inch holes were drilled in a 10" by 10" lead plate having a minimum thickness of 1/8 inch. These holes were arranged irregularly so that the corresponding spots could be easily distinguished on the developed film, and so that the proper side of the film would be viewed. The samples for calibrating the instrument for steel were formed by cutting a 7/8-inch steel rod into discs having thicknesses varying from 1/8-inch to 3 inches.

To provide a systematic record of all the data obtained the author compiled a set of tables from which Table I is an excerpt. The first of each pair of numbers in this table under the heading "Arrangement" refers to a certain hole in the lead plate, the holes having been numbered from one to nine, spirally, in a clockwise direction. The second of this pair of numbers refers to
TABLE I

Typical Radiographic Calibration Data

<table>
<thead>
<tr>
<th>Date</th>
<th>Plate</th>
<th>Focus-Film Distance (min.)</th>
<th>Exposure (K.V.)</th>
<th>Voltmeter Reading</th>
<th>Milliamperes-Minutes</th>
<th>Depth Proper Exposure</th>
<th>Arrangement</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dec. 9</td>
<td>10</td>
<td>18</td>
<td>10</td>
<td>86.5</td>
<td>12.5</td>
<td>1/&quot;  3&quot;</td>
<td>Steel Brass</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Steel</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Brass</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Results

Nos. 2 and 7—Weak but sharp (ideal for disclosing inclusions or blow-holes).
No. 4—Excellent contrast.
Nos. 5 and 6—Too much.
Nos. 8 and 9—Barely visible.
the thickness of the specimen, usually in inches.

Figure 4 is an example of the type of picture obtained in this calibration. The values for the "Depth Proper Exposure" were obtained by mere inspection of the film itself. Under the heading, "Results" is given a brief word-picture of the film itself. Contrast was excellent on the specimen covering hole No. 4, which, according to the "Arrangement," was 3/4 inches of steel. This is the ideal exposure for showing up any inclusions in steel having a greater atomic weight than iron, such as lead. But most defects in steel are inclusions having a lower density than iron or are blow-holes, cracks, etc. Good but rather faint penetration of the solid, pure steel is ideal for showing defects of this type. Thus the ideal exposure in the above case was approximately 1 1/4 inches of steel, as determined by extrapolation between holes No. 3 and No. 8.

The X-ray exposure chart shown in Figure 5 is the one to use in revealing the most common defects in steel and brass. The chart shown in Figure 6 is the proper one to use in disclosing any substance of high atomic weight with a background of iron or steel. Figure 6 can also be used when it is desired to provide maximum definition at the discontinuity between a thick section
Fig. 4  Typical Calibration Radiograph  
(Operating conditions were 86 K.V., 1.2 M. A., 20 Min. Spots in center represent steel of thickness, 3" and 2.5", respectively.)
and a thin section, in which case the thickness of the thin section must be used for reference.

The abscissas of the points were located merely by inspection of the films, leaving much to the judgment of the operator.

The charts (Figures 5 and 6) were drawn up according to the method given by St. John and Isenburger (8).

Numerous attempts were made to get the tube to operate above 100,000 volts. The factor which prevented exceeding this voltage was the ionization of the air both by the sparks from the rectifier and by the high-voltage rays themselves. The conductivity of the air was increased to such an extent that sparks shorted the rectifier so that the overload was blown.

The first few runs were made with the voltage low enough but with the current too intense. The tube had a great tendency to overheat. It is obvious that if a single run were long enough, an equilibrium would be reached between the heat radiated and that added by the bombardment of the target. At low voltages the optimum current was determined by a reasonable equilibrium target temperature, which was judged to the temperature at which the target maintained a bright cherry-red. At high
Fig. 5  X-Ray Exposure Chart
(for disclosing blow-holes)
Fig. 6  X-Ray Exposure Chart (dense)
voltages the optimum current was determined by the
maximum current that did not cause the peculiar "silenc-
ing effect" which served as an indication that excessive
sparking was about to occur.

Enclosed is a curve (Figure 7), milliamperes
versus kilovolts, which can be used to determine the
best operating conditions for the tube used in this
work.

When current and voltage are held constant
while time is varied as the square of the distance,
the resulting intensities should, according to theory,
be the same. When the first attempt was made to verify
this condition it was found that at close range the
final intensity (after passage of 86 kilovolt rays
through one inch of steel) was higher than that de-
manded by theory as compared with a focus-film dis-
tance of 1.5 times that at close range. The proof was
that the close range film was darkened more under the
one inch specimen than was the long-range one under the
same specimen. During another similar experiment the
time of exposure was not nearly as long as before, and
consequently the thickness of steel receiving the proper
exposure was much thinner. Therefore, in the latter case,
the difference between the focus-film distance and the
Fig. 7  Optimum Current Curve
distance from the focus to the top of the specimen was only about 1/4 inch, which was negligible in comparison with a total distance of 12 to 18 inches. In the earlier case if the time had been varied as the square of the distance from the top of the specimen to the focus it would have been impossible to see any difference in the resulting intensities of the spots representing the samples receiving the proper amount of exposure.

Figures 8 and 9 are radiographs, in each case, of a small steel casting 3/8 inch thick. The time of exposure was taking from the 60 kilovolt curve on the exposure chart, Figure 6, creating a dense negative. Nevertheless, excellent prints were produced.

A popular misconception is that a radiograph of steel can be produced merely by placing the casting on the film holder and exposing it. The effect of doing this is pictured in Figure 4. The opening represented by the upper-right spot had the same diameter as those in the upper-left. The fogging around the openings in the upper-left would have been even worse had they not been covered by 1/2 inch of steel. Fogging would take place if the film were wrapped in black paper during exposure, but the effect in these pictures is accentuated by the use of intensifying
Fig. 8 Radiograph of Small Casting, Showing Crack
Fig. 9  Radiograph of Small Casting, Dis-closing Blow-Holes
screens in the holders. The screens glow with a light-frequency producing the greatest effect on the photographic emulsion when struck by X-rays.

In Figures 8 and 9 this fogging was prevented by outlining the casting with small pieces of lead. Because of the shape of many articles it is not always possible to radiograph a casting in this manner. Furthermore, if it were possible to do this, two exposures would be necessary to disclose the defects in a casting varying in thickness from, say, one inch to two inches.

These difficulties are overcome by the "immersion technique." A solution of lead acetate was made so concentrated that it had slightly less the absorptive power of metallic iron or steel.

The necessary concentration was calculated in this manner:

1 cc. of steel is equivalent in absorption to a piece of lead 1 cm. X 1 cm. X .15 cm.

\[
.15 \times \frac{11.35}{7.6} \text{ equals } .25 \text{ grams of lead}
\]

Therefore .25 grams of lead per cc. of solution would have the absorptive power of metallic steel.

Figure 10 was produced by covering the casting with a solution of lead acetate containing .15 grams of lead per cc. of solution. The fogging produced around the edges of the specimen indicates that a concentration
Fig. 10 Radiograph of Section of Large Casting
by Immersion Technique
of lead of .20 grams per cc. would have been more ideal. With this concentration and a slightly longer exposure, any flaws that might have been present in the hub of the wheel (Figure 10) would have been visible.
CONCLUSIONS
The unit used in this research is adaptable to studying metallic engineering materials. By employing the various techniques described one can easily study for internal structure and defects any ferrous material up to two inches thick. Aluminum castings up to a thickness of five or six inches can be successfully investigated, while brass materials of thicknesses up to 3/4 inch can be successfully studied.
PART II

X-RAY DIFFRACTION
THEORETICAL
Several diffraction techniques are employed in research methods. The only one of these to utilize the "white" radiation of the tube is the Laue method for crystal study. In this method, the crystal is kept fixed, and a heterogeneous beam of rays is passed through it, usually parallel to a crystallographic axis.

None of the pictures enclosed were produced by passage of the beam parallel to an axis, however, and therefore the arrangement of the spots is not symmetrical. Several symmetrical patterns were made of crystals of calcite and citric acid. The general fogging of the film in these cases was so great that prints were not made.

Figures 11 and 12 were projected by use of a graticule scale, but space does not permit enclosing the projected pattern. The curve (Figure 13) used in constructing the scale is included, however.
Fig. 11  Laue Pattern of Oxalic Acid Crystal
Fig. 12 Laue Pattern of Citric Acid Crystal
The classical formula developed by Sir William Bragg (7), \( n\lambda = 2d \sin \theta \), has been the basis for most work done in the field of diffraction. The value, \( n \), represents the order of the diffraction, which is usually first order; \( \lambda \) represents the wave length of the incident radiation; \( d \), the distance between the planes; and \( \theta \), the angle of diffraction. In the Laue method \( \lambda \) is not constant, while in all other methods this wave-length is constant and usually a known value.

When projected by the gnomonic method, the Laue method of analysis gives a good picture of the symmetry of the crystal (1) and the method can be used for determining the angles between the planes. Also the relative intensities of a great number of reflecting planes is often indicated. But even these relative intensities cannot be relied upon, because if a plane within the crystal happened to have the right combination of angle to the incident beam and distance between the next parallel plane for reflection of one of the characteristic wave-lengths then the intensity recorded on the film would be out of all proportion to that representing the plane.

The "White" radiation necessary for production of the Laue patterns is produced by the electrons being
gradually brought to a stop within the massiveness of the target. The wave-length produced is dependent upon the speed with which the electrons strike a target, which, in turn, is dependent upon the voltage applied to the tube. Nicholas (23) has approached the production of monochromatic radiation by the use of very thin targets in a specially designed tube.
EXPERIMENTAL WORK
Diffraction work with this unit was possible only after enlargement of the pin-hole of the camera to about 100 times the conventional area. This made possible the production of successful Laue patterns with exposures ranging from about 30 minutes to two hours at a current of only three milliamperes. In the usual diffraction technique exposures of six to 20 hours at 20 to 30 milliamperes are common.

Figures 11 and 12 are examples of the type pattern produced with the Coolidge tube operating at about three milliamperes and 30 kilovolts for two hours.

Figures 14 and 15 were produced with a gas-filled tungsten tube operating at about 10 milliamperes and 30 to 40 kilovolts for two hours. The fogging on one end of the film was created by a discharge taking place on the portion of the anode immediately behind the target. Figure 14 is exactly the same as Figure 15 except in making the print the lighter end of the negative was partially masked to bring out the spots otherwise invisible.

In projecting the seven spots forming the ring in Figure 11 by the gnomonic projection method a straight line was formed. Each spot in the pattern represents a given set of parallel planes in the crystal.
Fig. 14 Laue Pattern of Oxalic Acid Crystal

(gas-filled tube)
Fig. 15  Laue Pattern of Oxalic Acid Crystal  
(same as Fig. 14 except unmasked)
The gnomonic projection curve is merely a plot of the formula:

\[ x = 3 \cot \left( \frac{1}{2} \arctan \frac{a}{3} \right) \]

By using the curve a straight-edge scale was constructed for the purpose of producing the gnomonic projections. The method can be demonstrated by reference to Figure 13. For example, if a spot is 2½ inches from the center spot on the negative the projected spot would fall 4½ inches from the center spot exactly opposite the diffracted spot.
CONCLUSIONS
Experience in operating a gas-filled tube at currents up to 15 milliamperes at the usual diffraction voltage of about 30 kilovolts indicates that the high-voltage equipment herein described can be used successfully with a water-cooled research diffraction tube.

Today there are two practical uses of the Laue method. One is in the estimation of the grain size of metals; the other is in the determination of strain in some crystals. In grain-size estimation the Laue method is of value only in supplementing data obtained by micro-metallographic methods.

The value of the polychromatic diffraction done here is that it lays a foundation for research methods employing homogenous radiation.
PART III

STUDY OF WOOD BY USE OF X-RAY
THEORETICAL
The study of flaws in materials such as those of wood is a good example of the versatility of X-rays. The illustrations are pictured here to demonstrate the ease with which studies of this nature may be made, and to give typical examples of the way most defects appear in woods.

Any variation of the material under examination in absorptive power relative to X-rays created a corresponding variation in exposure on a film. This variation may be caused by an opening (that is, a crack, grub-hole, or the like) in the wood, by a foreign substance (such as a nail), by a variation in mineral content, or by a variation in moisture content. The dense knots and annual growth-rings contain deposits of salts of higher atomic weight than the material comprising the rest of the wood.

According to Glazebrook (2): "In the case of timber, the different varieties absorb X-rays to different degrees. The sap wood is differentiated from the denser heart wood, the summer and spring growths of the annual rings are readily identified." Glazebrook states further that because of the submarine in the world war high-grade timber could not be shipped from America to England and laminated structures had to
be used in the manufacture of aircraft. The X-ray became an indispensable aid in preventing the use of bad materials, and in studying the finished structures.

The laws of absorption that apply to the radiography of metals also apply to the study of materials that are much more transparent to X-radiation. Wood is so transparent to X-rays that pieces up to two feet can be easily studied for defects. The method is also useful for watching the behaviour of the various hidden members and joints of a composite wooden structure while it is being subjected to test.
EXPERIMENTAL
All the illustrations included in this section were produced by the usual radiographic technique. Film holders containing intensifying screens were used in conjunction with special X-ray film. In all cases presented here the time of exposure varied from 1/8 second to 1/2 second.

The fact that special X-ray film is so expensive, and that the time of exposure is so short when making radiographs of wood, makes it more economical to use printing paper for this type of work, producing the negatives directly on the paper. In using an intensifying screen the time of exposure necessary, as in this case, is 80 times that necessary for special X-ray film. Several paper-negatives were produced proving that clarity is sufficient to disclose all defects revealed by the conventional method.

Table II provides a summary of the types of wood-studies that were conducted.
<table>
<thead>
<tr>
<th>Figures</th>
<th>Type of Study</th>
</tr>
</thead>
<tbody>
<tr>
<td>16 to 19</td>
<td>Appearance of Knots</td>
</tr>
<tr>
<td>20 to 25</td>
<td>Relative Absorption of Different Varieties of Wood</td>
</tr>
<tr>
<td>26 to 30</td>
<td>Appearance of Sound Specimens of Wood</td>
</tr>
<tr>
<td>31</td>
<td>Distortion of Wood</td>
</tr>
<tr>
<td>32 to 34</td>
<td>Flaws Offering no Absorption of X-Rays</td>
</tr>
<tr>
<td>35 to 36</td>
<td>Foreign Substances Offering Great Absorption of X-Rays</td>
</tr>
<tr>
<td>37 to 39</td>
<td>Appearance of Laminated Woods</td>
</tr>
</tbody>
</table>
On the pages immediately following are illustrations showing the clarity with which knots were disclosed. The blackened areas of the prints represent greater absorption of radiation than occurred in the surrounding material. Figures 18 and 19 both contained very small knots that gave good contrast. In Figure 20 a knot with the center removed is pictured in the beech specimen. The X-ray proves that deposition of salts occurs around the knot as well as in it.
Fig. 16 Radiograph of Poplar, Showing Knot
Fig. 17  Radiograph of Beech, Showing Knot
Fig. 18  Radiograph of Indiana Soft Maple, Showing Knot
Fig. 19 Radiograph of Hard Maple, Showing Knot
Greatest contrast between different varieties of wood of the same thickness is produced in Figure 21. Part of this difference in absorption may be attributed to the fact that oak is the heavier wood. But in Figure 24 the two woods illustrated, oak and hard maple, are of approximately of the same density, but oak offers greater absorption to the radiation.

Even different samples of the same variety of wood offer different absorption to the radiation. This effect is illustrated in Figure 20 at the point where the two pieces of walnut are glued together. The effect is again illustrated in Figure 27, in which case, however, the difference may be attributed to difference in moisture content.
Fig. 20 Radiograph of Beech (top) and Walnut (bottom), Showing Relative Absorption
Fig. 21 Radiograph of Oak (top) and Magnolia (bottom), Showing Relative Absorption
Fig. 22 Radiograph of Black Gum (top) and Magnolia (bottom), Showing Relative Absorption
Fig. 23  Radiograph of Cedar (top) and Soft Maple (bottom), Showing Relative Absorption
Fig. 24 Radiograph of Hard Maple (left) and Oak (right), Showing Relative Absorption
Fig. 25 Radiograph of Cedar (top) and Soft Maple (bottom), Showing Relative Absorption
Fig. 26  Radiograph of Poplar Pieces of Different Thickness, Showing Relative Absorption

Left: 1" Thick    Right: 2" Thick
Fig. 27 Radiograph of Two Pieces of Sound Oak Taken Across the Grain
Fig. 28 Radiograph of Sound Oak, 2" Thick
Fig. 29 Radiograph of Sap Gum, (top) Across and (bottom) With the Grain
Fig. 30 Radiograph of Poplar, (top) Across and (bottom) With the Grain
Fig. 31 Radiograph of Poplar Pieces That Have Been Subjected to Compression

Piece at Right: Pressure Applied to Sides
Piece at Left: Pressure Applied to Ends
The most outstanding example of the flaws offering no absorption of X-rays is that shown in Figure 33. This radiograph is of a piece of oak one inch thick which contained several cracks, all being invisible to both surfaces of the wood. Another excellent example of this type of flaw is that shown in Figure 32.

Figure 34 is an excellent example for the demonstration of the sensitivity of the X-ray method. The grubholes were all nearly one millimeter in diameter and were clearly distinguished in this two-inch piece of oak.
Fig. 32 Radiograph of Oak Piece Containing Internal Cracks
Fig. 33  Radiograph of Oak, 1" Thick, Containing
Cracks Invisible to Both Surfaces
Fig. 34 Radiograph of Oak, Showing Grub-Holes
Nails are the most common foreign particles found in wood materials and they offer such great absorption that they can never fail to be detected. Even large nail and screw holes can be disclosed. (See Figure 35 where the opening left by the removal of the large screw is clearly visible.)

The sensitivity of the method is again illustrated in Figure 38 where a defect in only one-tenth of an inch of wood is visible through a background of two inches of wood.
Fig. 35 Radiograph of Poplar, Containing Nails and Screws
Fig. 36 Radiograph of Cedar, Containing Nails
Fig. 37 Radiograph of Plywood, Consisting of 5 Laminated Woods With Maw in Only the Top Piece
Fig. 38 Radiograph of Plywood in Steps of 1/2 inch Each
Fig. 39 Radiograph of plywood composed of different woods glued together
CONCLUSIONS
Radiographic methods are applicable to the study of the various defects in structural materials having low absorption of X-rays.

The sensitivity of the method to the slightest changes in absorbing material is best demonstrated by Figure 34 in which a change in thickness of one millimeter (caused by grub) is clearly defined in a piece of oak two inches thick. This sensitivity ratio will be very nearly maintained in pieces of wood up to two feet thick.

Because of their greater absorption, knots always show up as light spots on the negative, and even the smallest spots are revealed.

Further experiment in this field supplemented by micro-photographic technique can easily lead to a new method for identifying the different species of wood and proving where the wood was grown.
LITERATURE CITED


(2) Glazebrook, Richard, "Dictionary of Applied

(3) Harawalt, J. D., Rinn, H. W., and Frel, L. K.,
1938).


(5) Miller, W. D., A textbook written at Purdue Univ.
for all students taking Physics 146, "X-Ray"
(1938).

(6) Nicholas, Warren W., Bureau of Standards Journal

(7) Terrill, K. M. and Ulrey, C. T., "X-Ray Technology,"

(8) St. John, A., and Ischburger, H. R., "Industrial
Radiography," New York, John Wiley and Sons, Inc.
(1937).
VITA

James Edward Butler was born at Woodville, Alabama, March 31, 1916, to Almira Bottoms Butler and Robert L. Butler, both of Scotch-Irish descent.

He graduated from New Hope High School of New Hope, Alabama, May, 1933. In September of the same year he entered Central State Teachers College at Edmond, Oklahoma, which school he attended until June, 1935. In September, 1935, he entered the Alabama Polytechnic Institute, at Auburn, Alabama, where he received the degree of Bachelor of Science in Chemical Engineering in August, 1938.

In September, 1938, he enrolled in the Graduate School of the University of Louisville as a candidate for the degree of Master of Chemical Engineering, at which time he also became employed by the Girdler Corporation of Louisville, Kentucky.