X-RAY DIFFRACTION ANALYSIS OF FABRICATION DEFORMITIES IN METALS

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MARTIN JOSEPH SKINNER

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Martin Joseph Skinner

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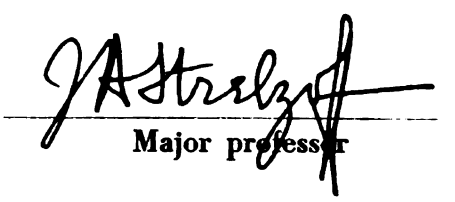

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THESIS

X-RAY DIFFRACTION ANALYSIS
OF
FABRICATION DEFORMITIES IN METALS

By

MARTIN JOSEPH SKINNER

A THESIS

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My special gratitude goes to Dr. R. B. Bowersox whose classes initiated the subject matter, and to Dr. J. A. Strelzoff who has given helpful suggestions and guided this thesis to completion.

INTRODUCTION

This thesis shall attempt to describe the methods of analysis of fabrication deformities in metals. Both the theoretical and practical descriptions will be given, and wherever possible sketches which show analyses of such deformities will be included.

Discovery of diffraction and interference effects associated with the passage of x-rays through matter very soon led to the recognition that studies of these effects are useful in determining the structure of matter. Work on the diffraction of x-rays by crystals was initiated in 1912 with the discovery made by von Laue and his associates. The result of their experiments will be dealt with later in the section on methods of analysis.

X-ray analysis provides a new tool for solving industrial problems. It found its earliest application in the study of crystalline structure, the pattern of the atomic arrangement in the perfect crystal. The results of these analyses have had profound influence on fundamental concepts of the nature of chemical combination. Such work ultimately influences technical application because it clarifies ideas that had previously been in question.

The progress of an x-ray analysis is far from being a direct method. Instead, the results are a series of clues which point to some possibilities and exclude others until finally enough evidence is accumulated to make one confident that he has arrived at the correct solution. Instead of logically deducing a solution, intelligent guesses have to

be made and tested against the observations. The same is true for x-ray analyses of all types. The clues afforded by the more or less diffuse spots, arcs, or lines on an x-ray photograph of some mysterious material must be combined with a wealth of other knowledge if they are to be followed up. Much of the fascination of x-ray analysis is due to its taking one into so many other fields of science. The x-ray expert must not only understand the optical principles on which his science is founded; he must at the same time be a chemist, a biochemist, or a metallurgist. The optical principles can be logically deduced, but imagination, common-sense, and a wide general knowledge lead him to make his intelligent guesses.

Each crystalline solid gives a characteristic diffraction pattern and can be recognized just as an element can be recognized by spectrum analysis. Extremely small samples measured in fractions of a milligram are sufficient, and the constituents are determined without destruction of the specimen. Applications are found in the structure of alloys, identification of intermediate products during processes, determination of crystal size and orientation, and submicroscopic changes within individual crystals such as occur in age-hardening. Such structures profoundly influence physical properties and hence are of great interest and importance.

THEORY OF DIFFRACTION PRINCIPLES

The diffraction principles are, in reality, a combination of two theories; the theory of crystal structure, and the theory of x-ray diffraction from such crystal structure. A fundamental understanding of these theories is necessary to thoroughly understand the working principles of x-ray diffraction analysis.

THEORY OF CRYSTAL STRUCTURE

Previous to the discovery of x-rays in 1897, investigations had shown that all crystalline bodies could be considered to be built up of molecules placed at the points of a space-lattice, such as that shown; the smallest component unit of this space-lattice having a regularity of structure being known as the unit cell. Different kinds of crystals have different types of space-lattice.

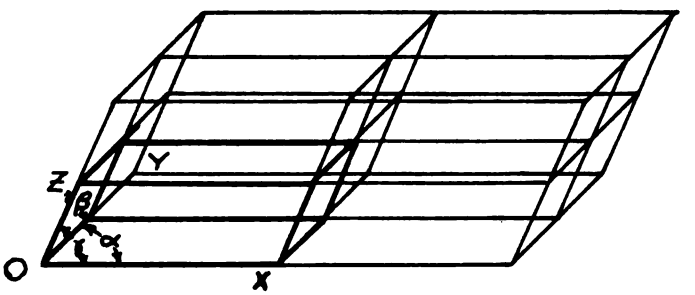


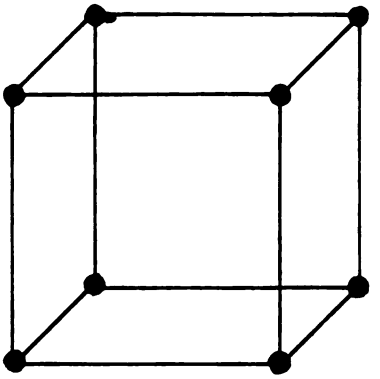
Fig. 1. The crystal space-lattice.

Since, in effect, the different types of space-lattices are determined by the differences between the axial lengths and the angles between the axes, it is necessary to know the lengths of the axes of the unit cell upon which the space-

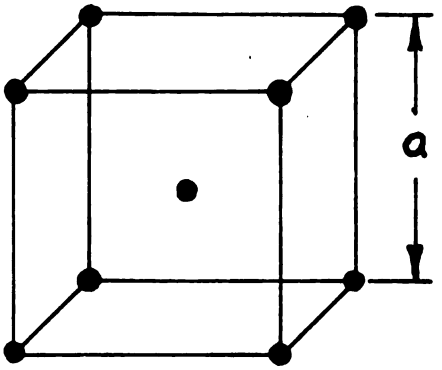
lattice is founded and the angles between these axes. Thus, in the space-lattice shown (Fig. 1.), the unit cell has the axes, X, Y, Z, and the origin being at O. The angles between these axes are α, β, γ, respectively. The possible number of combinations of axes and angles upon which a space-lattice can be built is limited, and although many thousands of crystals are known, they can be classified into only 14 types of space-lattices having these combinations of axes and angles, these space-lattices being based upon 7 different systems of crystal symmetry.

Although crystals of all these space-lattices are met with, by far the greatest majority of them have structures of the higher forms of symmetry, namely the cubic, tetragonal, and hexagonal. Among these three, the substances with which the metal industry most often deals, fall into the cubic and tetragonal classifications and it is these two which will be described more fully.

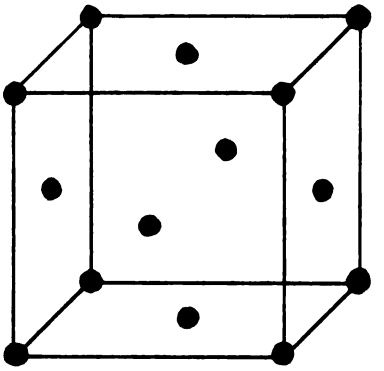
There are three forms of the cubic lattice, the simple cubic, the body centered cubic, and the face centered cubic. They derive their names from the location of the atoms in the lattice. These three are shown in Fig. 2a, b, and c. The body centered cubic has an atom at each corner and one at the center of the cell, and the face centered cubic has an atom at each corner and one in the center of each of the six faces. Because some of these atoms are shared by neighboring unit cells, the number of atoms per unit cell is one, two, and four respectively.



(a) Simple cubic

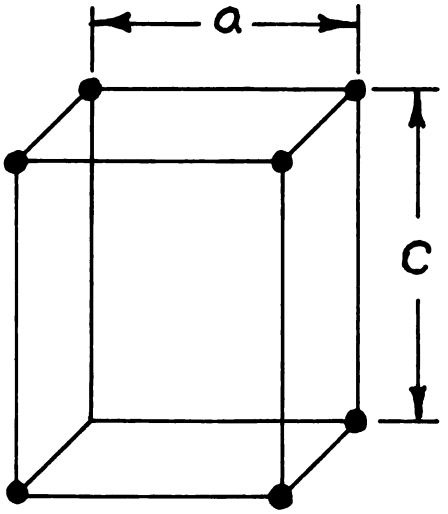


(b) B. C. cubic

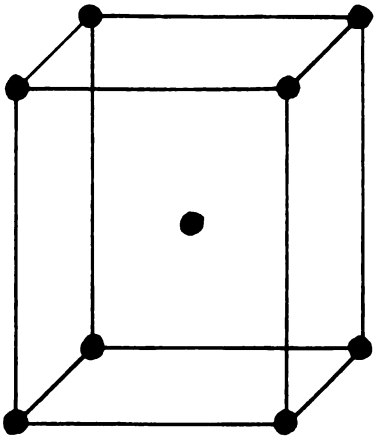


(c) F. C. cubic

Fig. 2. Arrangement of atoms in cubic lattices.



(a) Simple tetragonal



(b) B. C. tetragonal

Fig. 3. Arrangement of atoms in tetragonal lattices.

The tetragonal space-lattice has two forms, the simple and the body centered lattice. The differences between these and the corresponding cubic lattices in that one of the three axes is longer than the other two. See Fig. 3. If this axial length is designated as "c" and the length of the edge of the unit cell as "a", then the ratio $\frac{c}{a}$ is termed the axial ratio.

In Fig. 4, it can be seen that on the planes marked ABC and ADEF passing through the unit cell, there lie certain atoms. These planes also pass through all the other unit cells making up the space-lattice of the crystal and on them lie the corresponding atoms of each cell. The number of such atomic planes that can be passed through the space-lattice in different directions is very great.

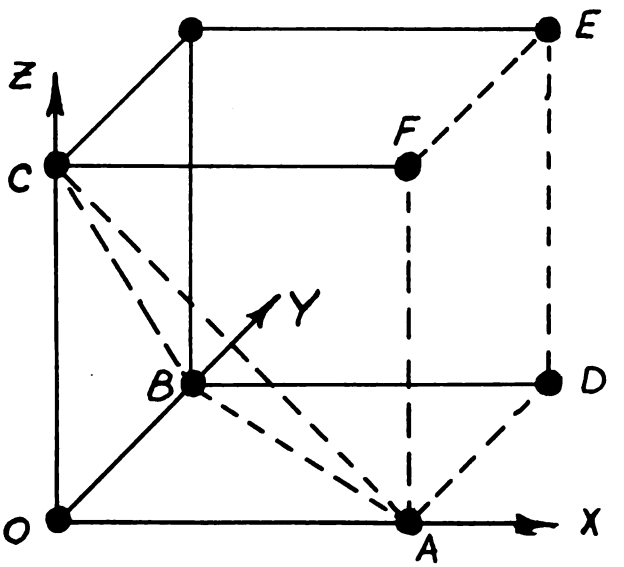


Fig. 4. Atomic Planes

To distinguish the different sets of planes from one another, they are disignated by the reciprocal of the intercepts made by each plane of each set on the axes of its

respective unit cell. Thus, in Fig. 4, the plane ABC cuts off a unit intercept on each of three axes and the reciprocals of these are 1, 1, 1. Similarily, the reciprocals of the intercepts made by the plane ADEF are 1, 0, 0, since the intercepts are $1, \infty, \infty$. The bigger the intercept, the smaller the index, and vice versa.

These reciprocals are termed Miller indices and are enclosed in round brackets, thus (111); (100).The general form is written (hkl), h being the reciprocal of the X axis intercept; k, the reciprocal of the Y axis intercept; and l, the reciprocal of the Z axis intercept. The Miller indices are always the least whole numbers, never fractions. If any of the intercepts are negative, the negative sign is shown above the indice as, $(2\bar{1}1)$.

THEORY OF X-RAY DIFFRACTION IN A CRYSTAL

The simplest form of x-ray diffraction is that arising from the impingement of x-rays upon a ruled grating. The theory of this diffraction is the same as von Laue proposed for the diffraction of x-rays from a single atomic plane. Fig. 5 is an example of such a diffraction.

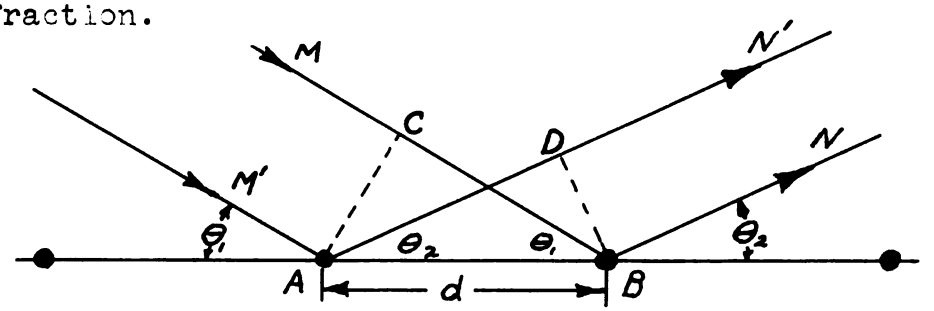


Fig. 5. X-ray diffraction from single atomic plane.

From this diagram, it may be seen that if the two diffracted beams are to reinforce each other at N'N, the difference in the length of path of the two beams must be a whole number of wavelengths of the impinging x-ray beam. In other words, CB - Ad = $n\lambda$; where "n" is designated as the order of reflection; "λ" is the wave length in cm. of the x-ray beam. From the figure, CB = d cos θ_1 ; AD = d cos θ_2 . Putting these identities into the above equation, von Laue's equation is established.

$$n\lambda = d(\cos\theta_1 - \cos\theta_2)$$

For the crystal von Laue really used three similiar equations; each dealing with a directional plane of the crystal.

W. H. and W. L. Bragg were the foremost physicists who turned their attention to x-ray diffraction after von Laue had published his discoveries. They soon proposed a simpler method of analysis that made the three-dimensional geometry involved easier to visualize. Instead of dealing with just the surface layer of atoms, or a single layer of atoms as von Laue did in his analysis, the Braggs based their analysis upon a number of successive parallel planes as is shown by Fig. 6.

This method is simplified by the fact that θ_1 must equal θ_2 in order that the scattered beams reinforce each other for all wavelengths. Then again set the difference in path length equal to a whole number of wavelengths. The difference in path length is BC + CD. BC = d sin θ_1,

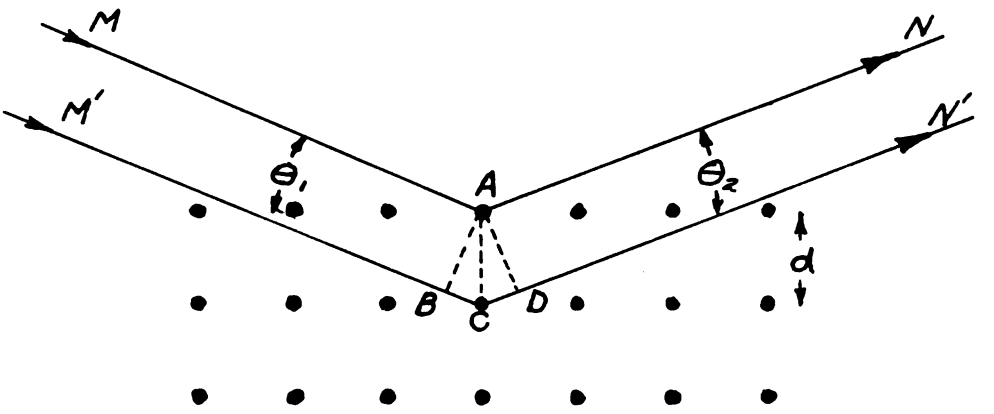


Fig. 6. X-ray diffraction from parallel atomic planes.

$CD = d \sin\theta_1$, $BC + CD = d(\sin\theta_1 + \sin\theta_2)$. But $\theta_1 = \theta_2$, therefore, Bragg's law is established.

$$n\lambda = 2d \sin$$

This expression is the fundamental equation of x-ray technique. The equation cannot be satisfied if n is greater than 2d and though several planes can reflect the beam in several orders, for a given wavelength and a certain lattice, the number of possible reflections is limited.

Fig. 7 shows the action of numerous atomic planes of a crystal upon the impinging x-ray beam. It is the basis for later discussion of x-ray diffraction analysis methods.

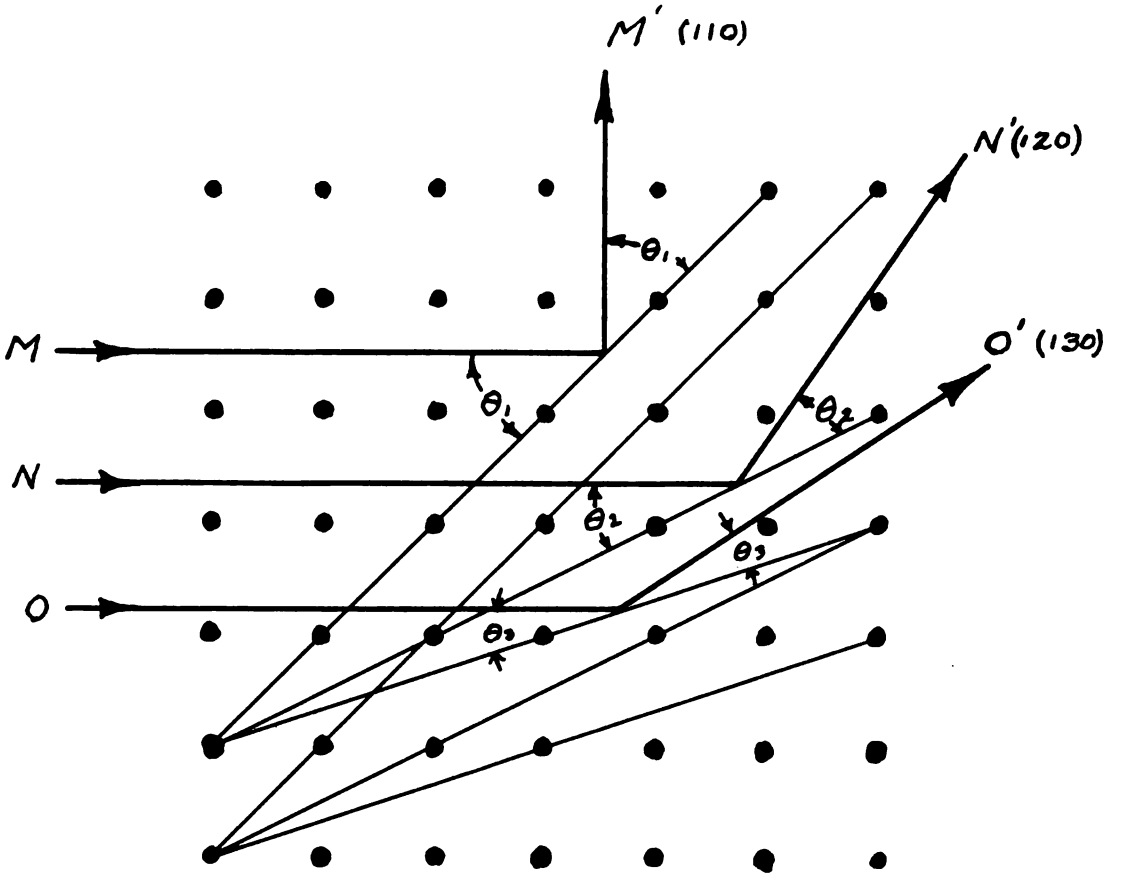


Fig. 7. Reflected x-ray beams from numerous atomic planes in a crystal.

Impinging x-ray beam - polychromatic
Reflected x-ray beam - monochromatic

X-RAY DIFFRACTION METHODS

A logical division of diffraction methods is by the three different ways in which the diffraction patterns may be photographed. Some authors have separated the methods into the work of various experimentors, but under each of these divisions come the three basic methods of photography; direct transmission, reflection, and powder methods. It is this division that will be followed in this thesis.

No matter which method of diffraction is used, each crystal has a certain diffraction pattern and that crystal may be identified and analysed to ascertain its nature, whether it is perfectly formed or deformed in some manner. Thus, it may be seen that the various methods for x-ray diffraction lead to a study of crystal size, stress and strain, and deformation.

DIRECT TRANSMISSION METHODS OF X-RAY DIFFRACTION

This method of x-ray diffraction is most commonly referred to as the Laue method for it follows the manner in which his investigations were carried out. This is especially true for the stationery crystal method. Although this is the oldest method of crystal analysis by x-rays, it is still used today by some of the foremost crystal analysts. It differs from other methods in that continuous radiation, or polychromatic x-ray beam is used.

Fig. 8 illustrates a simple type of "Laue camera". The beam of x-rays, usually from a tungsten target, passes through a slit system onto a fixed specimen which consists of a small single crystal. From the x-ray beam, the different sets of atomic planes pick out the appropriate wavelength according to Bragg's law and give rise to reflection spots on the photographic film. The two main features of the Laue diagram are, (1) the reflection spots lie on ellipses, the spots on each ellipse being due to reflection of the x-ray beam by the planes belonging to one particular zone, one end of the major axis of the ellipse being at the central spot of the film, and (2) all the spots produced by reflection from one wavelength lie on a circle with its center at the central spot.

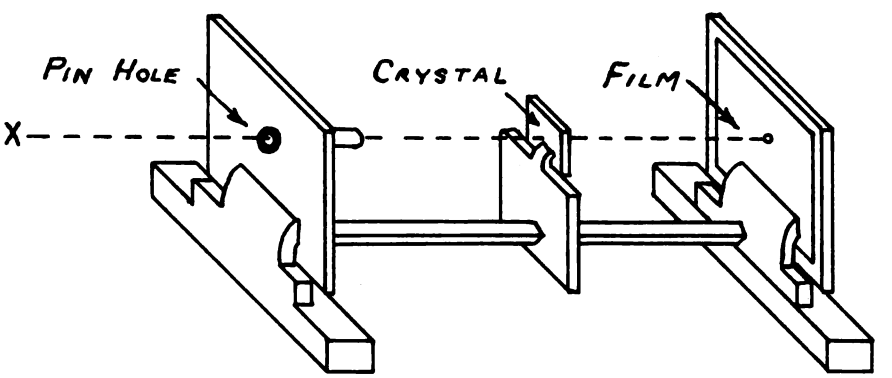


Fig. 8. Simple Laue camera.

As an example of just what happens to create a Laue photograph, Fig. 9 is included in the hope that the process may be made more clear. The x-ray action is like that shown in Fig. 7.

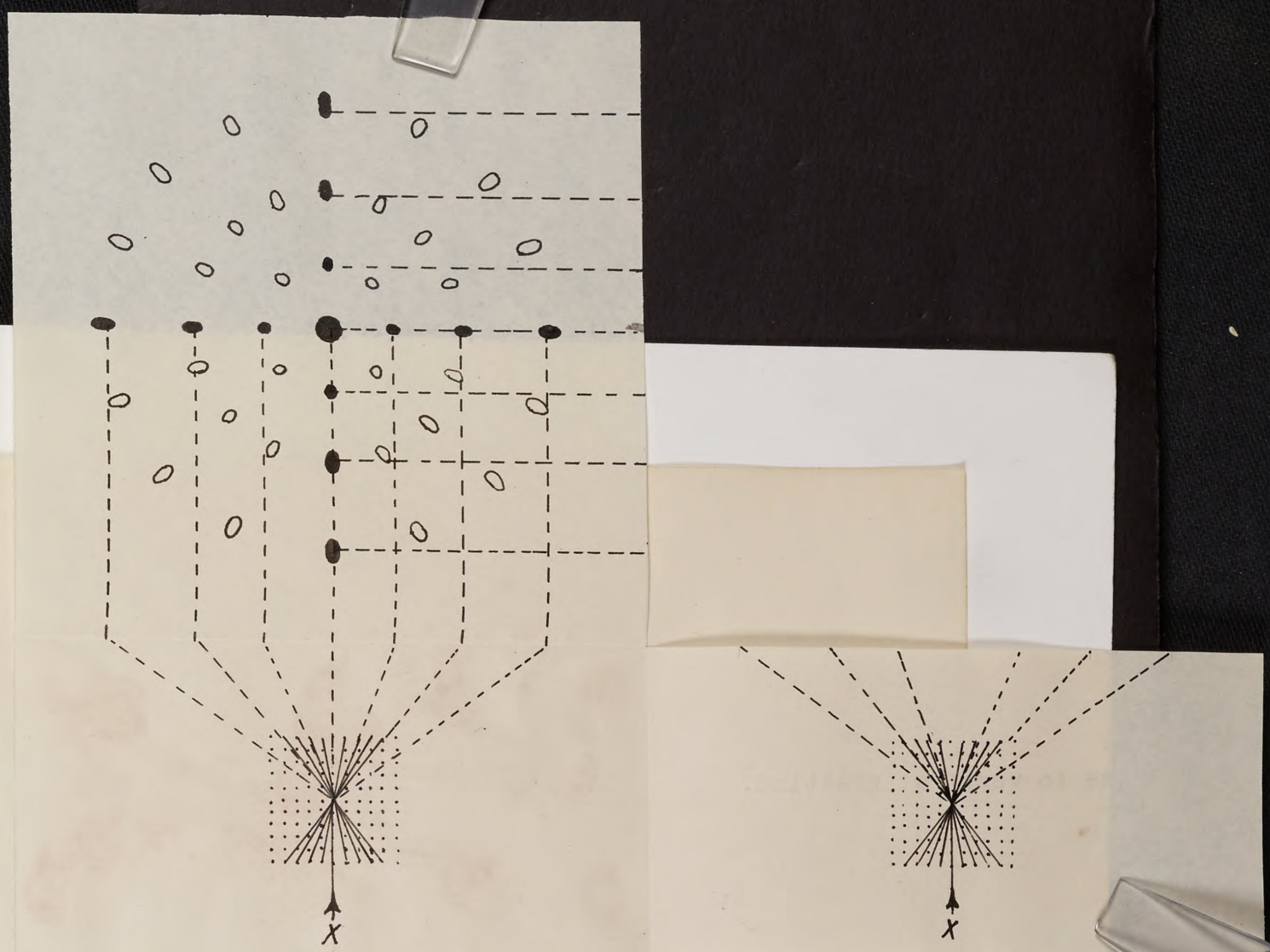


Fig. 9. The manner in which a Laue photograph is made.

The usual dimensions used in the Laue system are a target-specimen distance between 15-20 cms., the specimen to film distance being 5 cms. A pin hole slit diameter of 1 mm. is generally employed, while the thickness of the specimen crystal varies from 0.1 to 2 mm. according to the density.

The specimen-film distance is extremely important, and is measured usually on a graduated scale on which the film holder slides. Alternatively, it can be determined by taking a preliminary photograph of a crystal whose structure is known. As an example of exposure time, at 60 KV., 10 ma., with a 1 mm. slit, the time is 15-30 minutes with an intensifying screen.

Unless required for some special purpose, Laue photographs are taken with the x-ray beam traveling in simple crystallographic directions. In such circumstances the photograph will be symmetrical. Several methods can be used to obtain this condition. They are:

(1) With well-developed crystals, the positions of the axes can be determined by measuring the angles between the crystal faces with a goniometer.

(2) Photographs are taken with the crystal at different settings in order to obtain those with the highest symmetry.

(3) With good reflecting crystals, the specimen can be adjusted by observing the Laue pattern on a fluorescent screen.

There are various other methods which are correlated with the Laue x-ray diffraction. One of these is the crystal rotation method in which the crystal is rotated at a constant angular velocity. This creates a different diffraction pattern that, for some study, is more adaptable than the stationary method. Three such photographs around the principal axes make it possible to obtain almost complete crystal information. Usually the film is placed differently also. It consists of a cylindrical film, enclosing the specimen as a center. Fig. 10 is a sketch of such a Laue camera which allows for sample rotation. For this method a monochromatic beam of x-rays is used. Crystal analysis by the rotation method was not used extensively

until 1922, but since has been used for the analysis of more different substances than any other method.

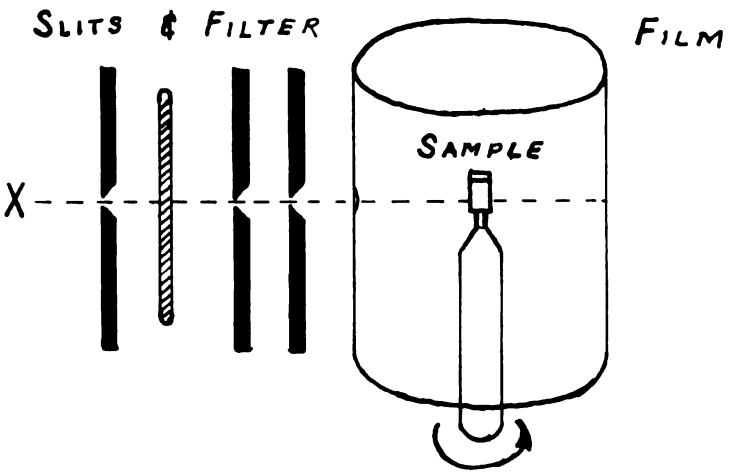


Fig. 10. Crystal Rotation Camera.

Still another popular variation of the Laue method of x-ray diffraction analysis is the use of a specimen consisting of more than a single crystal in the form of a powder. This is more practical for a large number of substances since the single crystals are so minute. This gives a diffraction pattern that is completely different from that of the single crystal. As the number of grains increase, the spots become more numerous and smaller, all traces of the zone ellipses for one grain being obscured. At the same time the intensity decreases, and longer times of exposure are required. The condition of minute size is finally reached where on a Laue diagram there appears an almost continuous darkened ring. Further discussion of such continuous rings will be found in the section on the powder method of x-ray diffraction.

REFLECTION METHODS OF X-RAY DIFFRACTION

Reflection from the surface of samples too thick for penetration by x-rays can be used, as illustrated in Fig. 11.

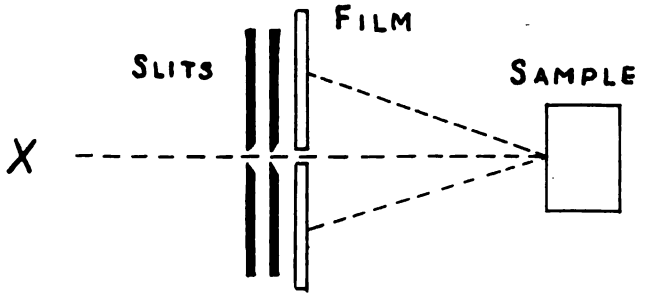


Fig. 11. Diagram of surface reflection method.

In industrial practice it is frequently desired to know the ultimate crystalline condition of a finished product or of a large specimen that cannot be sampled. For example, in very large steel structures, such an examination of a finished unit before installation would be invaluable.

This is the method that is commonly used in a study of surface stress and strain deformities in the metal under consideration. The reasons for its use will be described with the methods for stress analysis.

Photographs of the relections may be taken at almost any position with respect to the sample, but the two most common positions are at the pin hole as shown in Fig. 11, or at a position where the reflections arise from grazing incidence. The choice of positions depends entirely upon the study that is desired.

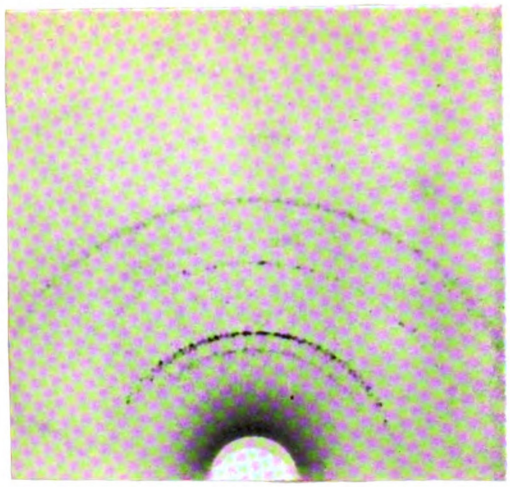


Fig. 12. Diffraction pattern; Grazing incident method. See Fig. 16c for back-reflection method.

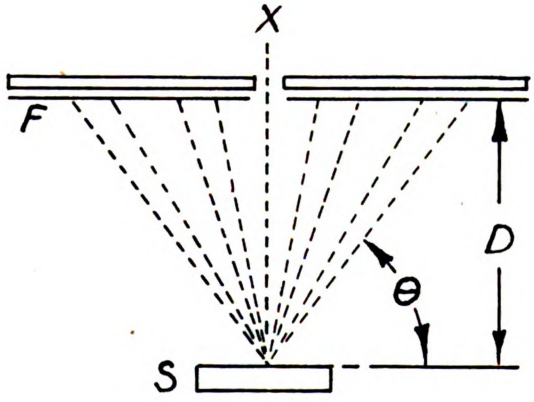


Fig. 13. Sketch showing the formation of pattern.

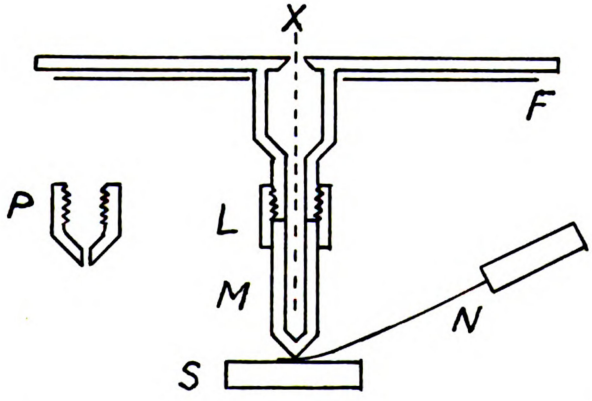


Fig. 14. Method of accurate determination of film to specimen distance.

Fig. 13 is almost self explanatory. The primary source of x-rays is located at X. The specimen is designated as S, and the film as F. The specimen to film distance is D, and θ is the usual Bragg angle of reflection. This figure shows that the distance D must be obtained very accurately in order that the correct values of reflection are ascribed to the various rings on the diffraction pattern.

There are two common methods for accurate measurement of the film to specimen distance. One common method is the use of a very thin film of either gold or silver on the surface of the specimen. This produces the diffraction pattern of the gold or silver along with that of the specimen and knowing the values for the standard, the correct values of the unknown may be determined. The second method is one of mechanical measurement. This is shown in Fig. 14. The pointer M is held to the slit system by the lock-nut sleeve L and the whole unit is moved until M just presses the feeler N to the specimen. The thickness of N is of the order 0.0015 in. When the adjustment has been made, the pin-hole P is exchanged for M and L before the photograph is made.

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POWDER METHODS OF X-RAY DIFFRACTION

Such diffraction methods are commonly termed the Hull-Debye-Scherrer powder methods for it was these men who did the initial work in establishing this method of practical x-ray diffraction analysis. These men worked independently but their work was so alike that all three are referred to in such work. This new method, first used successfully in about 1916, made it possible to deduce the crystal structure of metals and other solids of commercial importance.

To the person interested in the practical applications of x-rays, the powder method is far more than a method of crystal analysis, like the Bragg method, the Laue method, or the rotation method. Since the sample may be in its ordinary natural polycrystalline condition, the x-ray pattern is able to reveal information regarding three characteristics of the solid that have as much practical importance as the structure of the individual crystals that compose it. These three important characteristics are (1) the average size of the crystals, commonly called the "grain size"; (2) the absence or presence of any tendency for the crystals to orient themselves in a preferred manner; (3) actual bending, twisting, or similar mechanical distortion of the crystals, commonly called "grain distortion" or strain.

Figs. 15a, b, and c show various ways in which powder-sample photographs may be taken.

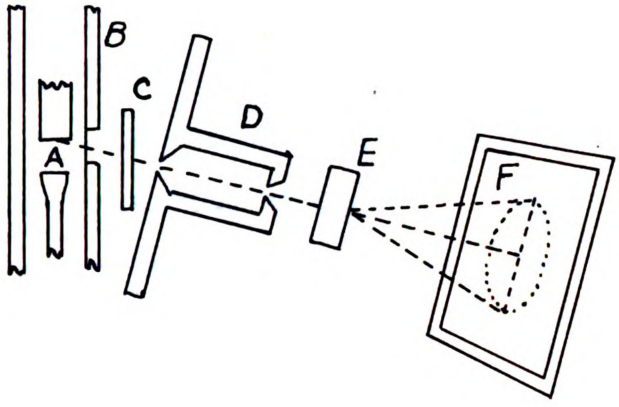


Fig. 15a. Flat cassette powder camera.

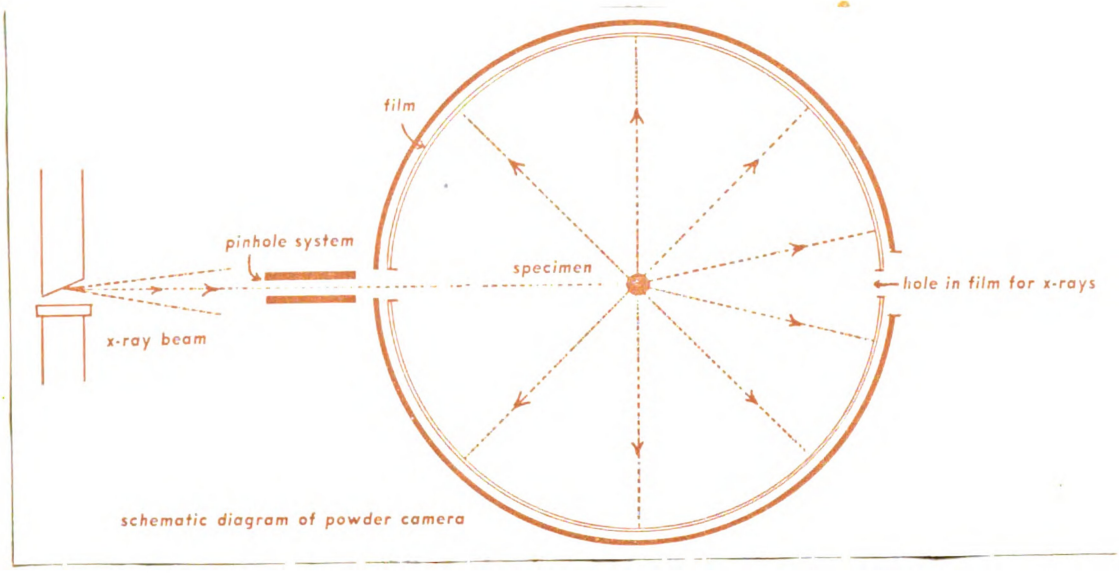


Fig. 15b. Cylindrical powder camera.

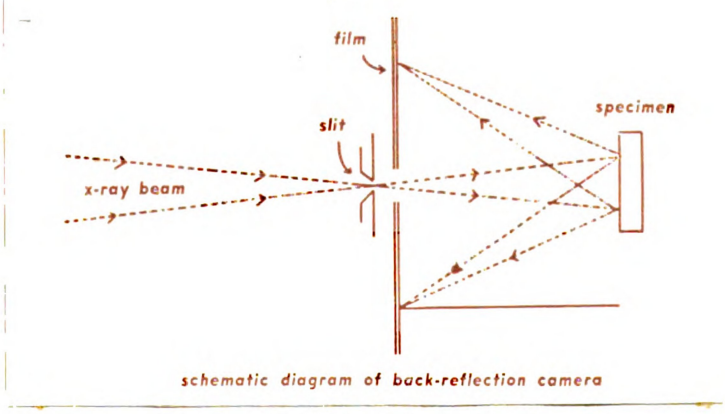


Fig. 15c. Back-reflection powder camera.

The preparation and mounting of a powder sample is an exacting task. Metals may be reduced to a fine powder by using light pressure on a fine, clean file. The powder or filings should be sieved through a 200-mesh screen or cloth. Care must be used in obtaining these filings so that no unwanted working of the metal takes place to give incorrect results in the following analysis. Having obtained the powder, one may coat it with Canada balsam to produce a thick paste, which is coated onto a human hair; this is then mounted taut in a line perpendicular to the primary beam. For this type of sample, a camera having a vertical axis is preferable, so that one may suspend the hair and hang a small weight on it to keep it taut and vertical. If the axis of the camera is not vertical, a glass tube or rod drawn down to a diameter of about 0.01 in. may be substituted for the hair. Still another manner of preparing the specimen is especially useful for substances that are hygroscopic. For this method the powder is loaded into a thin-walled glass tube having an inside diameter of $\frac{1}{4}$ mm. If a comparison test is to be taken, one half of the tube may be filled with a known substance and the remaining half with the unknown. The tube is then rotated while the analysis is being carried out and from the resultant patterns, the properties of the unknown may be discerned.

The powder method of x-ray diffraction analysis is most useful when the analysis is a study of grain size. In cases where the study is one of stress or distortion

analysis, this method falters for it is practically impossible to produce a powder specimen without adding more stress or distortion to the material, thus giving rise to erroroneous results. For such studies, the two previous methods of analysis are more satisfactory.

Figs. 16a, b, and c are, respectively, the type of photograph as obtained with the three types of powder diffraction cameras as shown in Figs. 12a, b, and c. Each circle is due to the diffraction of the x-ray beam by one certain series of atomic planes.

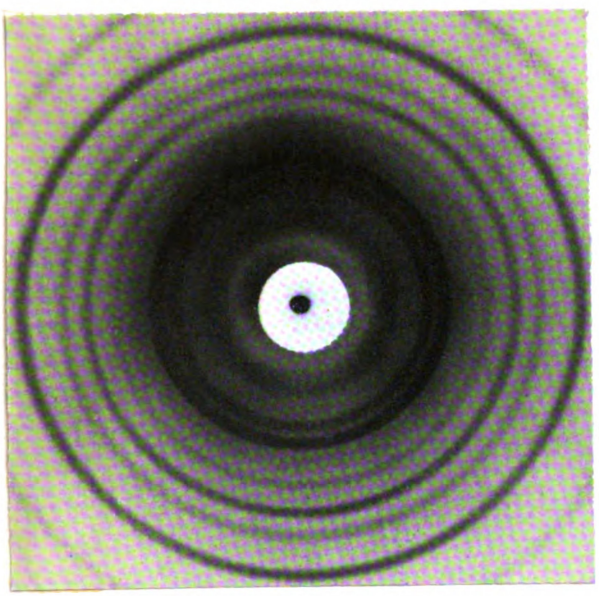


Fig. 16a. Diffraction rings of transmission photograph.

Fig. 16c. Diffraction rings of back reflection photograph.

Fig. 16b. Diffraction rings of cylindrical photograph.

X-RAY STUDY OF GRAIN SIZE AND PREFERRED ORIENTATION

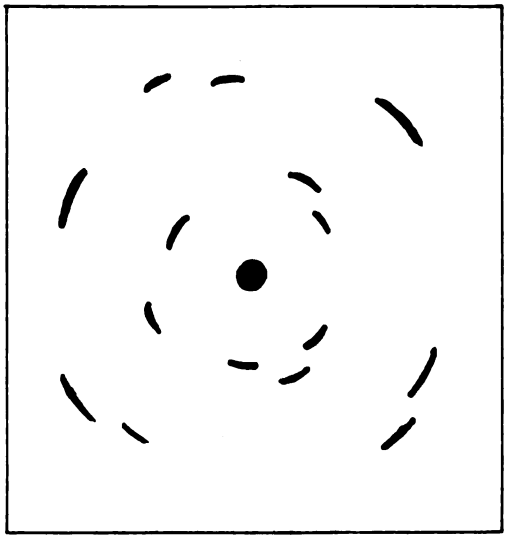
The objective of this thesis, diffraction analysis of fabrication deformities, splits itself into two natural groupings. They are; a study of grain size and preferred orientation, and a study of stress and strain. Since these often take different methods of x-ray diffraction for analysis and the techniques developed at different times, they will be dealt with separately.

The matter of x-ray diffraction studies of grain size has little significance until the size of the crystal falls into the region of 10^{-4} cm. All grain or crystal sizes larger than this figure are measured more conveniently and more accurately with standard microscopic methods of etching and magnifying. However, at this point the x-ray method becomes an important factor in such measurements for as the crystal size becomes smaller, the diffraction pattern rings become progressively broadened, especially at 10^{-5} cm. or smaller. A measurement of this broadening is a moderate indication of the grain size. (See Fig. 17)

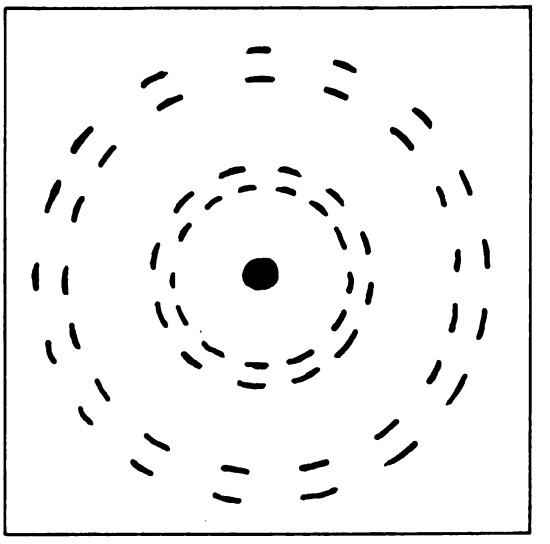
In the x-ray examination of these particles, the powder method is usually adopted. As well as being dependent upon the particle size, the width of the diffraction lines is also influenced by the following factors.

(1) The diameter of the camera and of the specimen.

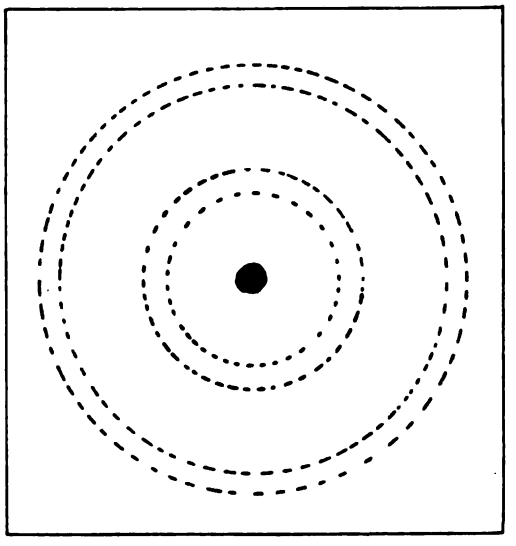
(2) The size and shape of the slit system of the camera.



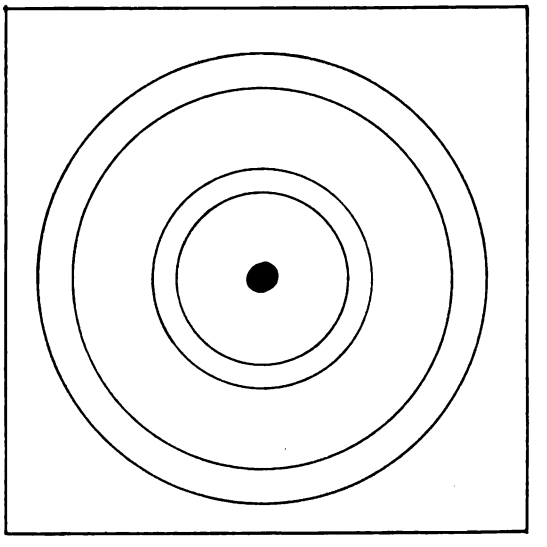
Very large grain size



Grain size approximately 0.2 - 0.5 mm.



Grain size of the order of 10^{-3} cm.



Grain size in the range of 10^{-4} to 10^{-5} cm.

Fig. 17. Diffraction patterns of various grain sizes of a metal.

(3) The absorbsion of x-rays by the specimen.

(4) The size of the focal spot of the x-ray tube.

In view of the variation in these factors from one camera to another, it might appear to be extremely difficult to determine the particle size. However, some success has been achieved in this direction, although in some cases, the values obtained are to be regarded more as relative than absolute.

For crystals of cubic shape belonging to the cubic system, Scherrer, Bragg, and Seljakow give the following expression connecting the line breadth and the particle size:

$$\beta = \frac{K\lambda}{a \cos \frac{X}{2}}$$

where β is the breadth in radians of the intensity peak measured at the point where the intensity falls to half its maximum value (see Fig. 18), λ the wavelength of the x-radiation, $\frac{X}{2} = \theta$, the Bragg reflection angle of the line that is being measured, and a is the cube length of the crystal.

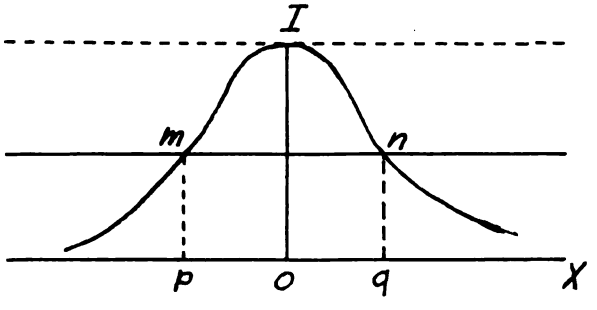


Fig. 18. The "half-value width" (pq) of a diffraction maximum.

The values of K range between 0.9 and 1.0 so that for most problems the value of unity is used in the previous equation. The value of β is easily calculated when a cylindrical camera is used, for in this case the subtended angle of pq is equal to $\frac{pq}{r}$, where r is the radius of the cylinder. The half-value points of intensity are found by use of an ionization chamber or by densitometry of the photographed pattern.

In using the previous formula to calculate grain size from a pattern, there is some doubt whether β should be called "breadth" or "broadening", for β refers only to the extra breadth of the lines due to the grain size being less than 2×10^{-4} mm. When the grain size is of the order of 10^{-5} or 10^{-6} mm., this point is not important, but if the size is of the order of 10^{-4} mm., so that the line broadening is only just detectable, allowance must be made for the breadth of the line due to causes other than small partical size. This can be accomplished experimentally by photographing on the same film the pattern of a substance having a grain size known to be of the order of 10^{-3} mm. For accurate work, a simple subtraction of the breadth of one of these reference lines from the observed breadth of one of the lines of the unknown is not permissible. For a fair estimate, one may use the relation $\beta^2 = \beta_m^2 - \beta_o^2$ where β_m is the measured breadth and β_o is the breadth of the reference line.

The accuracy to be expected in the measurement of the

size by the above expression is in the order of 20 per cent.

As well as particle size, strain that may be present in a metal will produce broadening of the diffraction lines. For this reason, the strain must be removed from the specimen either by annealing or other means. Any of the above methods, on the other hand, can apparently be applied to samples in which some degree of preferred orientation is present.

Another manner in which the line broadening may be attributed to grain size instead of deformation or strain is accomplished by studying the effects of λ and θ upon this line broadening. Since a broadening due to grain size is proportional to λ and sec θ, and, due to strain is proportional to only tan θ, the true source of the broadening can be determined.

The aggreement between the apparent particle size as given by the different lines is as good as can be expected, considering that generally the particles will not all be of the same size. It has been suggested that the discrepancy between the particle size as given by the different diffraction lines is tied up with the non-uniform shape of the particles. Because of the effect of this factor on the shape of the intensity curves, the optical dimensions must be variable. For example, if the particle size is large, x-rays of long wavelength should be employed and the ratio of the specimen diameter to that of the camera should be small.

In processes involving the plastic deformation of a metal, such as drawing a rod down to a wire, rolling a billet down to a sheet, or "deep drawing" a sheet to form a cup, the metal crystals cleave and glide or slip along certain crystallographic planes parallel to certain crystallographic axes in those planes. Such planes of slip are usually among the most densely populated sets, that is, sets of planes having more atoms per square angstrom unit than other crystallographic planes.

These processes obviously will reduce the grain size by fragmentation; in addition, the special direction of the fragmentation and slip results in the fragments acquiring preferred orientations with respect to the drawing or rolling axis. In a few instances such a preferred orientation is desirable or beneficial. For example, it has been found that the electrical properties of silicon sheet steel for transformer laminations are improved by allowing it to retain its preferential orientation. Such steel has a considerably higher magnetic permeability in the direction of roll than in other directions. Also, it has been found that the resistance of nickel steel to corrosion improves when the orientation is preferential. In general, however, preferential orientation is objectionable because it weakens a metal and reduces its ability to withstand further deformation. The forming operations that cause preferential orientation are usually performed in several stages. Each forming step

increases the preferential orientation, thus reducing the capacity of the metal to undergo additional forming steps. Therefore it is often necessary to anneal the metal between some of the stages to relieve the preferential orientation by permitting the grains to "recrystallize" before proceeding to the next stage. In most cases, annealing causes a recrystallization in which new crystals form of a size comparable with the size of those originally present, and these new crystals should have a nearly random orientation.

The most usual effect of preferred orientation on the diffraction pattern is the lumping of spots in symmetrically located positions instead of the ordinary distributation about the ring. Fig. 19a and b show such an effect.

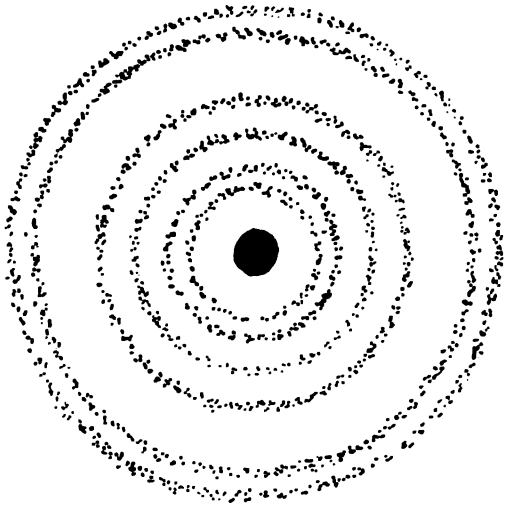


Fig. 19a. Transmission pattern of aluminum wire.

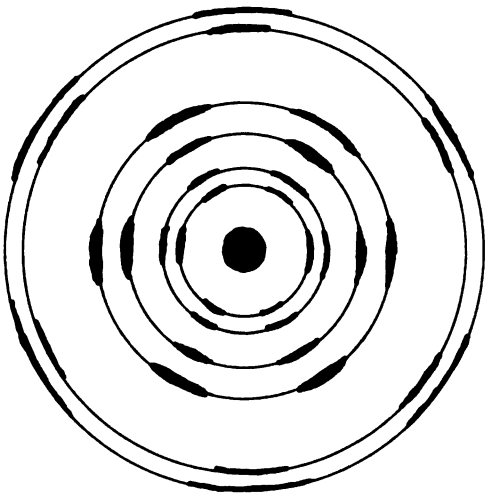


Fig. 19b. Pattern of same wire after stretching beyond elastic limit.

The broadening of the rings in Fig. 19b point to the fact that the grain size has been greatly reduced by this plastic deformation, and the "lumpiness" of the rings indicates that the crystals have acquired a preferential orientation as a result of the stretching.

A method of analyzing such a preferential orientation diffraction pattern may be accomplished with use of Fig. 20. This figure is similar to that showing how a transmission photograph is made. The difference is that a preferential orientation of crystals is considered instead of the previous random orientation.

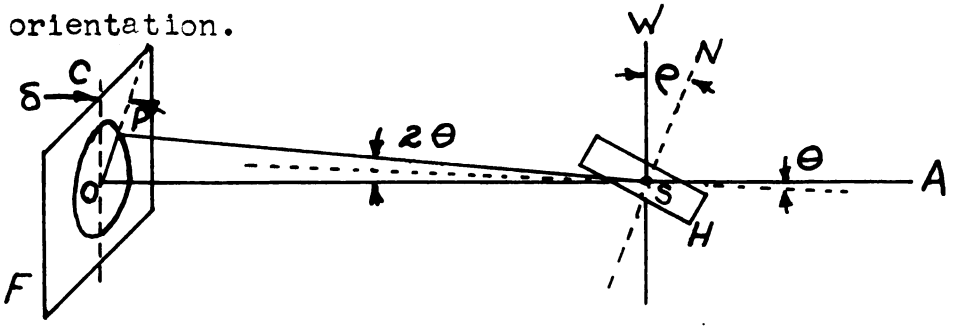


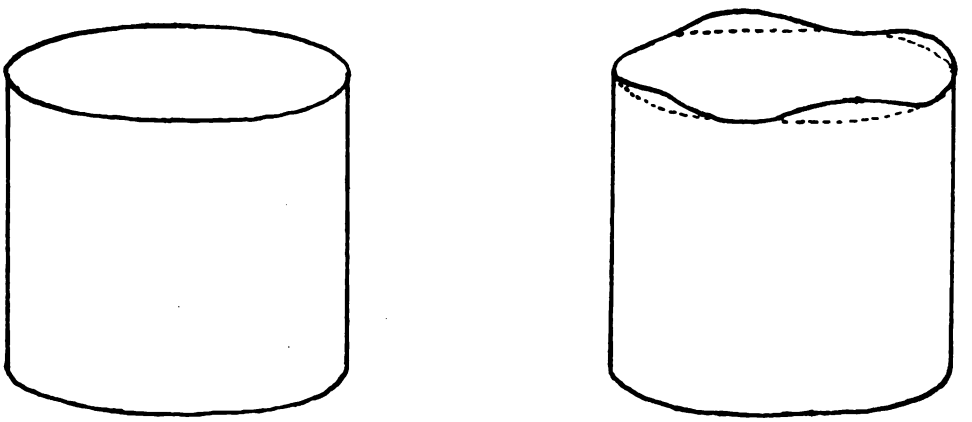
Fig. 20. Analysis of lumped pattern due to preferred orientation.

In Fig. 20, AS represents the primary x-ray beam striking the wire WS perpendicularly at S, the undiffracted beam then striking the film F at O. The diffracted ray SP strikes the film at a point P on the circumference of one of the rings R in the resulting pattern like Fig. 19b. Suppose, now, that P is one of the abnormally intense lumps on the ring R. CO is a line drawn on the film parallel to the wire WS. From the analogy with the case of a single crystal rotation pattern, such spots P will

occur in groups of four, symmetrically located on opposite sides of CO and on opposite sides of a line on the film through O perpendicular to CO. This may be seen, also, by imagining the reflecting plane H to rotate about NS, maintaining its fixed inclination ρ to it (NS is the normal to plane H). If δ is the angle between OP and OC and θ is the usual Bragg angle for the set of planes H from which ray SP is "reflected",

$$\cos \delta = \frac{\cos \rho}{\cos \theta}$$

The angle δ is an indication of the shift associated with the preferential orientation of the crystals when deformed. Fig. 21 shows the effect of preferred orientation upon deep-drawn brass.



Correctly treated brass shows no preferred orientation.

Brass still showing a tendancy for preferred orientation.

Fig. 21. The effect of preferential orientation in deep-drawn brass cups.

X-RAY STUDY OF STRESS AND STRAIN

Earlier it has been stated that a broadening of diffraction pattern rings may be attributed to grain distortion as well as to a reduction of grain size. This distortion is associated with strains, designated by the letter ϵ, which may be introduced by such processes as cold working. Strains of this type have been called "microstrains" by x-ray diffraction workers. Microstrains may be defined as strains that vary widely and erratically in magnitude and direction between neighboring grains, or within the individual grains, in a crystalline solid. They occur when the elastic limit or yield point is exceeded, as in cold working. On the other hand, the strains resulting from elastic distortion of a solid, such as a spring, when the elastic limit is not exceeded, may be called "macrostrains." Such distortion effects the crystals in such a way that the strains introduced vary from crystal to crystal and from point to point within each crystal in a systematic manner.

The fact that x-ray diffraction is capable of measuring strains in the surface layers of any solid crystalline object, having such a grain size that it will yield sharp rings or lines (about 2×10^{-4} to 10^{-2} mm. grain size), was pointed out by G. Sachs and J. Weerts in 1930.

In some respects, x-ray diffraction is inferior to other methods of analysis; in others, superior. Its most important advantages, perhaps, are that (1) it can measure

the strain without any necessity for comparing the strained specimen with a supposedly unstrained specimen and (2) it can measure the strain at a spot 1 mm. in diameter or less without any interpolation or extrapolation obtained from measurements outside that spot. Its disadvantages are that it is capable of measuring the macrostrain only when microstrains are not unduly severe and over the limited grain size range given above. In ferrous specimens, for example, this limits one to material having a hardness below 40 Rockwell C. The method is slow and requires an experienced operator and rather expensive equipment. It is useless for measuring rapidly changing strains, as in the parts of an engine while it is operating.

The strains introduced in a bar when it is stretched, compressed, bent, or twisted without exceeding the elastic limit are macrostrains, whereas quenching and cold work introduce both macrostrains and microstrains. A process like shot peening also introduces both macrostrains associated with the compressive stress set up in the surface layer, balanced by forces of tension underneath, and microstrains in the surface layer, which is stressed beyond the elastic limit by the peening. As a result of plastic distortion, the grains are broken, compressed, stretched, bent, and twisted slightly, and the fragments slip along the cleavage planes of the crystal.

Both macrostrains and microstrains cause variation in

the interplaner d spacings in the crystals composing the solid. The grain fracture resulting from plastic deformation soon reduces the grain size below 5×10^{-3} mm., so that a continuous ring pattern is obtained. The variation in d spacing associated with the microstrain then causes a broadening due to ultrafine grain, as mentioned previously, and it will be independent of the direction of the x-ray beam in the sample. The broadening follows directly from the Bragg equation; slight variations of d correspond to slight variations in θ for a given λ in a powder sample where all possible crystal orientations exist.

When a force of 20,000 pounds is applied to a steel bar 1 in. square and 1 ft. long, tending to stretch it, the bar becomes more than 1 ft. long and less than 1 in. square. This elongates the grains in a direction parallel to the bar and causes them to shrink in directions perpendicular to it. This elongation in one direction and shrinkage in another appears in the x-ray pattern as a slight shift in the lines or rings corresponding to the increase or decrease in the various crystal lattice spacings d and depending upon the direction of the primary and diffracted x-ray beams in the sample. This dependence of the line shift on the beam direction readily distinguishes such line shifts due to strain from the otherwise similar line shifts resulting from thermal expansion (which increases d uniformly in all directions), or from the shifts caused

by change in composition. Composition changes also produce a greater amount of line shift.

To summarize these theories, then, ordinary macrostrains manifest themselves in the x-ray pattern as a line shift, whereas microstrains manifest themselves as a line broadening which may be difficult to distinguish from the similar broadening due to ultrafine grain. In spite of this possible ambiguity in some cases, x-ray diffraction yields more information on microstrain than any other known method. It is obvious that severe microstrains will subtract from the ability of a structural member to withstand the macrostresses for which it was designed. When a large casting is heat-treated to "relieve stress", the thermal stresses due to unequal rates of cooling in thick and thin portions (which are macrostresses) are relieved, but the relief of the microstresses in such an operation may be equally important.

Both the slight line shift due to macrostrain and the slight line broadening due to microstrains are much easier to detect and measure in a back-reflection pattern than in a transmission pattern. In the first instance, the reason may be seen from the relation

$$d\theta = -\frac{dd}{d} \tan \theta \qquad *$$

This shows that the slight shift $d\theta$ resulting from a strain $\frac{dd}{d}$ varies as $\tan \theta$, the minus sign indicating merely that

* See appendix for derivation.

d decreases for positive strains (tensions), and increases for negative strains (compressions). Since $\tan\theta$ becomes large as θ approaches 90°, the line shift due to small changes in d becomes greater, and hence easier to detect and measure, when θ approaches 90°. This occurs only in back-reflection patterns.

The reason why a slight line broadening due to micro-strains is easier to detect by back-reflection is also partly explainable from the above equation when $\frac{dd}{d}$ is regarded as a continuous range of strain and $d\theta$ the corresponding continous range of θ causing the broadening. Another reason appears upon differentiation Bragg's equation where d is regarded as a constant and λ as a variable.

$$d\lambda = 2d \cos\theta \, d\theta$$

$$\frac{d\theta}{d\lambda} = \frac{1}{2d} \sec\theta$$

These equations show that a wavelength doublet will be resolved much more readily in the Bragg reflection from any given set of planes with spacing d for large values of than for small ones because the angular separation $d\theta$ for any given wavelength separation $d\lambda$ and interplanar spacing d varies as $\sec\theta$. When θ becomes large enough so that $\sec\theta$ equals 5 or 10 or more, as in a back-reflection pattern, then $d\theta$ becomes a degree or two and the doublet reflections are separated by an angle larger than the usual line breadth, that is, they are resolved. Obviously, if the

usual line width is $\frac{1}{2}°$ in a doublet separated by $1°$, the line appears double; but as this line breadth increases to $1°$ owing to microstrains or extremely fine grains, or both, the doublet merges into a single line in the pattern. Therefore doublet resolution in a back-reflection pattern may be conveniently used as a quick and easy index of the presence and magnitude of microstrains when the grain size is known to be above 2×10^{-4} mm. or as an index of grain size below 2×10^{-4} mm. when microstrains are absent. Fig. 22 illustrates this point.

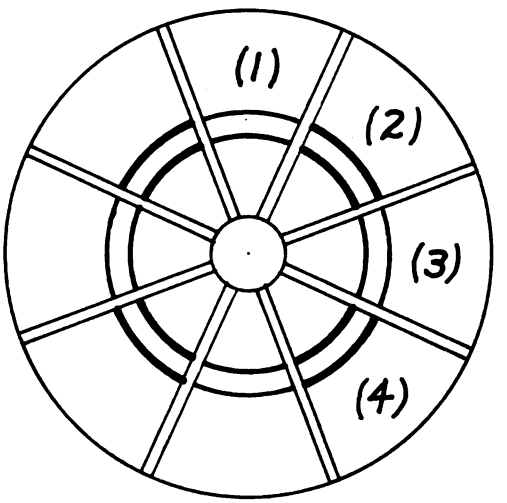


Fig. 22. Back-reflection patterns of mild steel bar, taken perpendicular to side. (1) and (3) before, (2) and (4) after shot peening.

It has been seen that back-reflection is the best technique for measuring strain, and the choice of the proper radiation now requires consideration. For back-reflection, $\sin\theta$ is nearly 1, so that Bragg's equation becomes $n\lambda = 2d$ approximately. In Fig. 23 this means

that $n\lambda =$ BC + CD (exactly) = $2d$ or 2CF(approximately). In accordance with usual practice in calculating Bragg reflections, fictitious planes permit one to set n = 1, and on this basis it is seen that the selection of the proper radiation involves a choice of λ slightly less than $2d$.

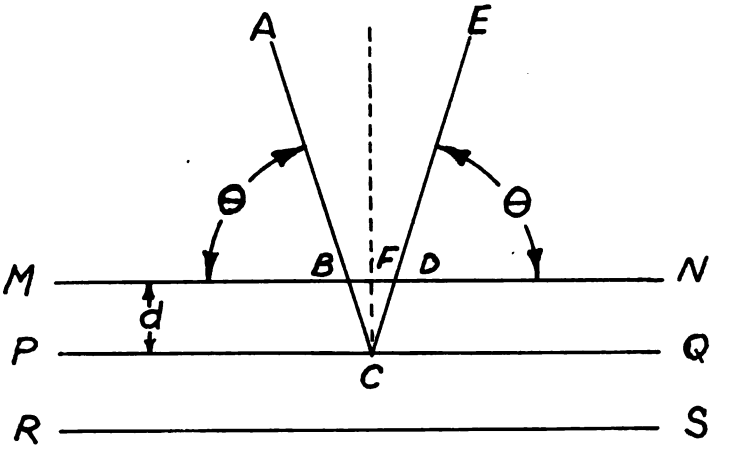


Fig. 23. Showing why the wavelength should be slightly less than $2d$ for back-reflection work.

For the sake of discussion, suppose that iron, copper, and aluminum and their alloys are regarded as the most important and the commonest materials in which one usually tries to measure stress. Table 1 lists the d values and corresponding intensities for the most prominent reflections from these metals, together with the indices of the reflections. Table 2 lists the mean wavelength of the K_α doublet for the common target elements for diffraction work.

Table 1

Aluminum, face-centered cubic

indices	d	2d	percent I
(111)	2.330	4.660	100
(200)	2.020	4.040	40
(220)	1.430	2.860	30
(311)	1.219	2.438	30
(222)	1.168	2.336	7
(400)	1.011	2.022	8
(331)	0.928	1.856	4
(420)	0.905	1.810	4

Iron, body-centered cubic

indices	d	2d	percent I
(110)	2.010	4.020	100
(200)	1.423	2.856	15
(211)	1.166	2.332	33
(220)	1.010	2.020	10
(310)	0.904	1.808	3
(222)	0.825	1.650	3
(321)	0.764	1.528	10

Copper, face-centered cubic

indices	d	2d	percent I
(111)	2.080	4.160	100
(200)	1.810	3.620	53
(220)	1.277	2.554	33
(311)	1.089	2.178	33
(222)	1.043	2.086	9
(400)	0.905	1.810	3

Table 2

target	atomic number	mean wavelength of K_α doublet, A°
Cr	24	2.290
Fe	26	1.932
Co	27	1.785
Ni	28	1.655
Cu	29	1.540
Zn	30	1.435
Mo	42	0.710

It is evident from these tables that the most prominent reflections, like (111) for aluminum, have a value of $2d$ which would make λ much too great for practical work. Even iron K_α radiation (1.932 A.) is so soft that it is absorbed to a serious degree by the air in a large camera. Thus it is seen that, in general, back-reflection work requires a soft radiation. The radiation chosen should, however, be hard enough to eliminate the need for vacuum cameras for general industrial application. Iron K_α is about the softest radiation suitable for ordinary cameras; in case of necessity, chromium K_α can be used in small precision cameras, although with this radiation it is advisable to find some means other than black paper to protect the film from the light.

Considering the 1.785 A° cobalt K_α it is seen that this radiation is suitable for the (331) and (420) aluminum, the (400) copper, and the (310) iron reflections. Since this radiation is hard enough to penetrate a thin piece of paper and a few inches of air, it is now clear why cobalt is the most widely used target material for strain measurement by back-reflection. This radiation, however, is so soft that it is easily absorbed by the metal under investigation and so the reflected pattern is characteristic of only the surface of the material, perhaps to a depth of 0.002 or 0.003 in. into the metal. Thus the back-reflection method is limited to a study of a thin surface layer as previously mentioned.

CONCLUSION

From the previous discussions it may now be noticed that x-ray diffraction methods play an important role in the analysis of fabrication deformities in metals. These analyses are not only of detrimental deformities but also of structural changes that are beneficial. One may also use such diffraction analysis to study the rate of change of deformities in order that the most economical method of fabrication is used to prepare a product that will be satisfactory.

For an example of a fabrication process, let us take the case of structual changes due to successive reductions of a low-carbon steel by cold working. The following data was obtained during the process, to a final point of 97 percent reduction. The accompanying reproductions in Fig. 24 show the change of diffraction pattern throughout the progressive reduction.

Fig.	pass	gage, inches	percent reduction	pattern
	0	0.1580 (orginal)	0	
a	1	0.1475	7	Fragmentation below 35μ and appearance of rings.
b	14	0.0460	71	Appearance of four-point fiber pattern characteristic of drawing.
c	21	0.0165	90	Typical rolling pattern. (six point)
d	30	0.005	97	Perfected orientation. (six point)

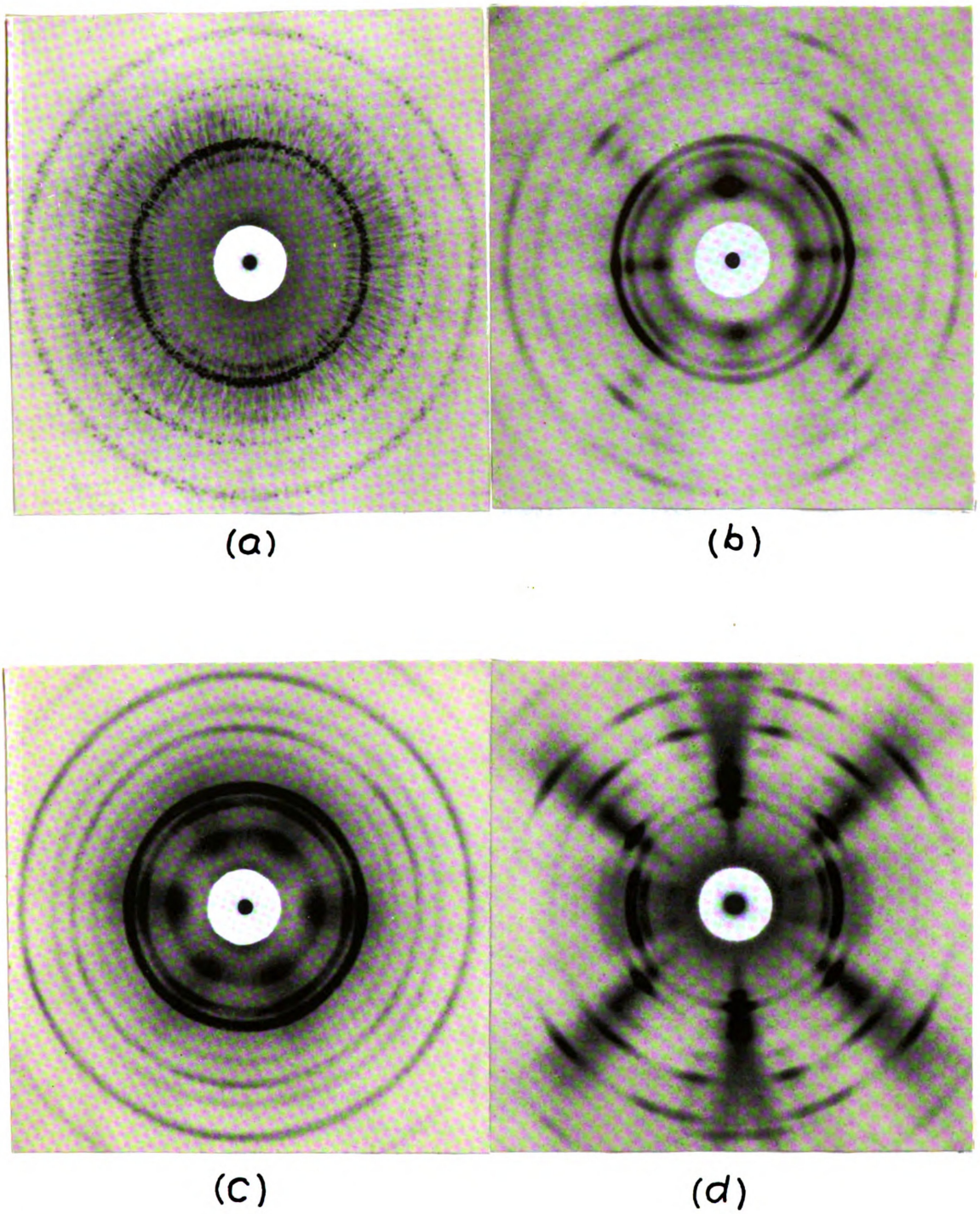


Fig. 24. Changes in structure with steps in rolling of low-carbon sheet steel. (a) One pass, 7 percent reduction; (b) 14 passes, 71 percent reduction; (c) 21 passes, 90 percent reduction; (d) 30 passes, 97 percent reduction.

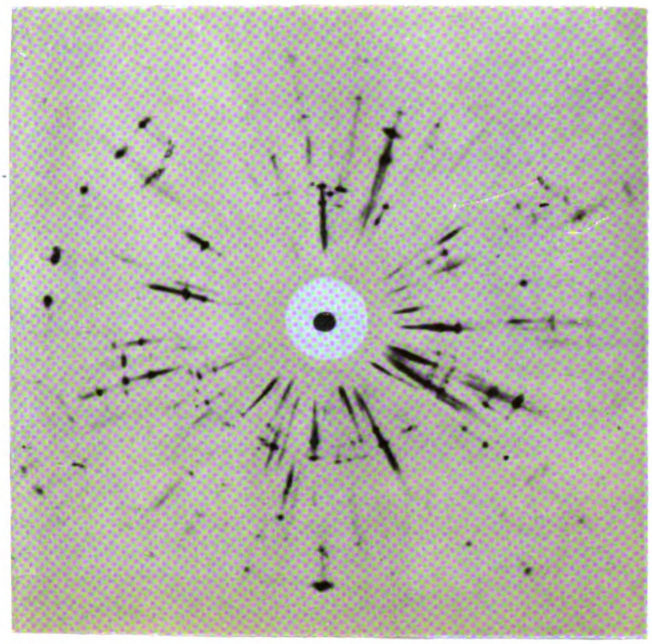
From a whole series of steel samples the following average results were obtained:

Appearance of	percent reduction
Continuous rings (fragmentation)	27
Sharp rings	38
Six-point fiber pattern	54
Four-point fiber pattern	76

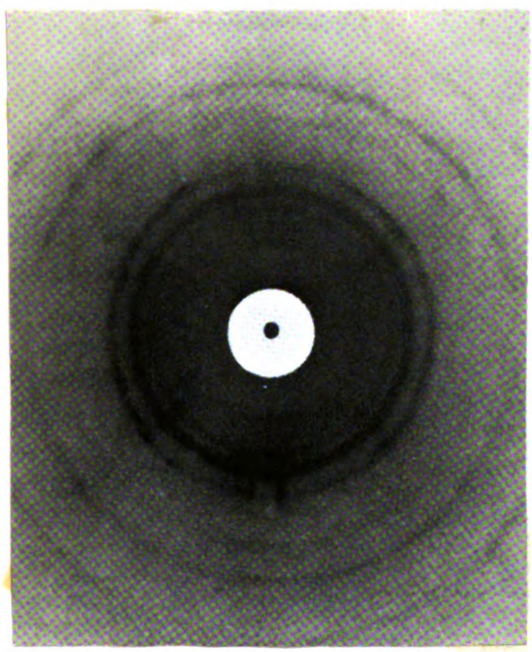
Such values are greatly dependent upon the type of mills, chemical composition, thickness, grain size, and orientation in the original material.

The x-ray diffraction patterns of Fig. 24 were obtained with the x-ray beam perpendicular to both the plane of the surface of the metal and the rolling direction. If, however, similar patterns were taken perpendicular to the rolling direction, but parallel to the rolling plane the preferential orientation of the grains would be noticeable after 15 to 30 percent reduction, depending again upon the above-mentioned variables. The diffraction pattern continues to be a six-pointed figure upon further reduction instead of changing to a four-point at approximately 76 percent.

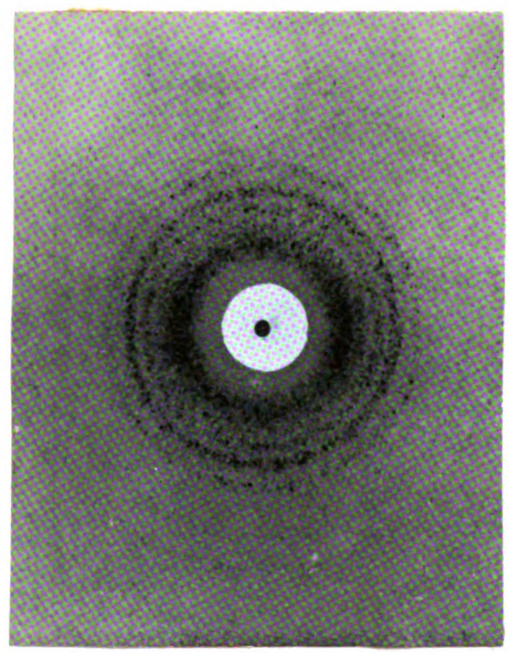
An interesting study is that of the effect of annealing upon the grain structure of deformed or strained materials. A variation in annealing temperatures can produce grains that are recrystallized with either a preferred orientation or a random orientation. Fig. 25 shows the change in grain orientation obtained with two different annealing processes. The material is cast steel.



(a)

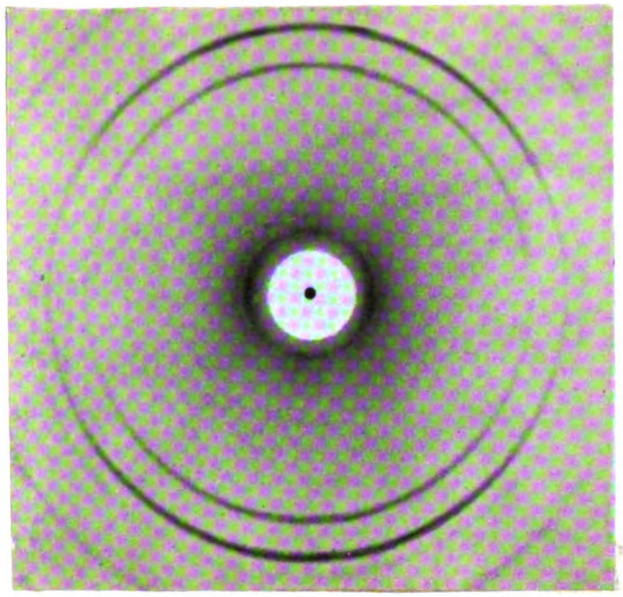


(b)

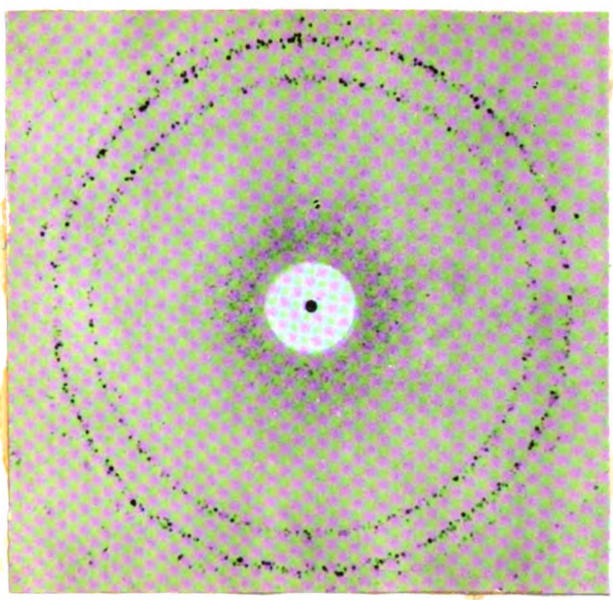


(c)

Fig. 25. Diffraction studies of cast steel annealing.
(a) Original structure, showing internal strain;
(b) Commercial anneal, showing some remaining detrimental structure;
(c) Ideal annealed structure of same steel.



(a)



(b)

Fig. 26. Effects of annealing an 80 percent reduced sample of low-carbon steel at different temperatures. (a) 1200° F.; (b) 1500° F.

Fig. 26 shows a similar effect upon a strained low-carbon steel when annealed at different temperatures, the same process being used for both samples.

Although comprehensive work in combining x-ray research with fatigue tests has not yet been completed, progress has been made, and more should be expected. The chief difficulty has been in providing specimens sufficiently thin for x-ray analysis. An example of such research is the study of duraluminum airplane propellers. After detailed study of all portions of the blade it was found that the surface near the hub was the focus of all component stresses, and diffraction patterns were made at different times. The results of these studies are shown with diffraction patterns in Fig. 27. Through this study, improvements in propeller design, and in the composition and properties of the alloy have been made in the past few years.

(a) (b) (c)

Fig. 27. Patterns for aluminum-alloy airplane propellers. (a) Unused propeller, (b) after 900 flying hours, (c) same propeller after 1,400 flying hours. Grain groth and distortion with increasing use are indicated.

The three examples of x-ray diffraction studies were chosen for they were representative of such studies as reviewed in most published literature. More examples might have been given but I felt that they would be extraneous. However, it can be observed from these examples and the text of the thesis that x-ray diffraction can and will play an even more important role in the study of fabrication processes than it does at the present time. Such studies will assure a finished product that will be the finest that can be manufactured and, at the same time, be produced most economically.

APPENDIX

Characteristics and interpretation of x-ray diffraction photographs.

Characteristic appearance of photographs	Interpretation	Remarks
Sharp spots, circular of nearly circular, falling on the circumference of two rings. Large in number but each of small size.	Absence of strain. Grain size about 10^{-3} cm.	When a small aperture (0.5 mm) is used in the x-ray cassette and the spots are very sharp, each of them may be seen to be doubled, which is due to a record being obtained on both sides of a duplitised film.
Sharp spots as above, but large in size and few in number.	Absence of strain. Grain size about 10^{-2} cm.	The spots from large grains are very seldom as sharp as those from small grains.
Large spots on the circumference of a ring surrounded by many others of feeble intensity not situated on a ring but irregularly distributed over the film.	Large spots indicate grains about 10^{-2} cm. or larger. The feeble non-ring spots indicate presence of much larger grains.	The use of small apertures would probably not enable the feeble spots to be observable with the usual short exposure times.
Diffuse spots, broadened chiefly in a radial direction.	Lattice distortion denoting a state of strain.	Increase of the strain causes increase of the diffusion of the spots.
Spots broadened circumferentially or tailed spots.	Fracture of grains into smaller components, each having a slight tilt with respect to its neighbor.	These spots are usually few in number, and spots radially broadened are invariably present.
Sharp continuous doubled ring, background clear.	Absence of strain, grain size between 10^{-3} and 10^{-4} cm.	

Characteristic appearance of photographs	Interpretation	Remarks
Diffuse continuous, background more dense than above.	Considerable lattice distortion or grain size of less than 10^{-4} cm.	Use of a small aperture usually increases the contrast of the diffuse rings to the background, but may reduce this contrast with some complex steels.
Very broad band of feeble intensity whose limits merge into the dark background.	Grain size less than 10^{-4} cm. or a highly strained condition.	Not possible to distinguish between a small particle size and a highly strained condition, but highly strained condition implies a fine particle size. The converse is not necessarily true.
The doubled ring broken into symmetrically dispersed arcs, or a continuous ring with symmetrical locations of increased intensity and increased width.	Preferred orientation. Absence of large strains when arcs are sharp and clearly defined. Large strains indicated by continuous non-uniform rings.	On occasions the preferred orientation effect can be confused with a pattern composed of very large spots circumferentially broadened.
Changes of ring diameter.	Change of lattice dimensions of the unit cell as the result of either small changes of composition or large elastic strains.	

Derivation of Equation

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Bragg's Equation

$$n\lambda = 2d \sin\theta$$

Allowing n to be equal to unity

$$\sin\theta = \frac{\lambda}{2d}$$

Differentiation with θ and d variables

$$\cos\theta\, d\theta = -\frac{\lambda}{2}\frac{1}{d^2}\,dd = -\frac{\sin\theta}{d}\,dd$$

$$d\theta = -\frac{dd}{d}\,\frac{\sin\theta}{\cos\theta}$$

$$d\theta = -\frac{dd}{d}\tan\theta$$

QED

Stress Analysis by X-Ray Diffraction

By
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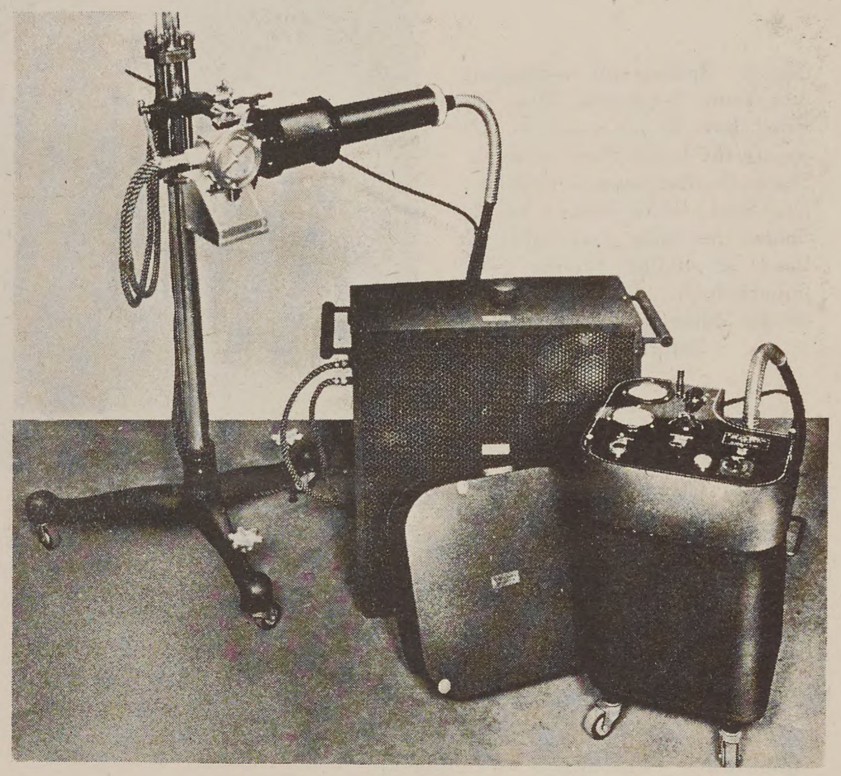

Fig. 1. X-ray Equipment for Back-Reflection Method of Stress Analysis

THE X-ray back-reflection method is the only means available of accurately analyzing existing stresses in a structure without measuring the unstressed structure. With it, the actual stress can be calculated from the diffraction negative. Parts subject to strain in service can be checked to determine if a change in the crystalline structure of the material has taken place. Once such an analysis has been made, the information obtained can be used to predict the changes that may occur in similar structures. The successful use of this method depends on the availability of precision-built equipment; on care in etching the specimen and in setting the exposure; and on painstaking evaluation of the results.

The X-ray equipment in use at the St. John X-Ray Laboratory is shown in Fig. 1. One unit (right) contains the high-voltage transformer and controls; the water pump (center), which circulates cooling water to the X-ray tube, is mounted in a cabinet. The self-rectifying X-ray tube, supported on a flexible tube stand, is of the ray-proof and shock-proof type.

The back-reflection instrument is mounted directly against the X-ray tube window. It consists of a camera and a motor to oscillate the camera during the exposure. The X-ray beam is directed through an extremely fine collimating bore on the specimen being investigated, the crystals of which reflect it back onto the X-ray film, forming a circular pattern. Superimposed over this pattern is one of a standard material, usually gold. Since the pattern of this standard always appears the same, it is measured for comparison in the evaluation of the results.

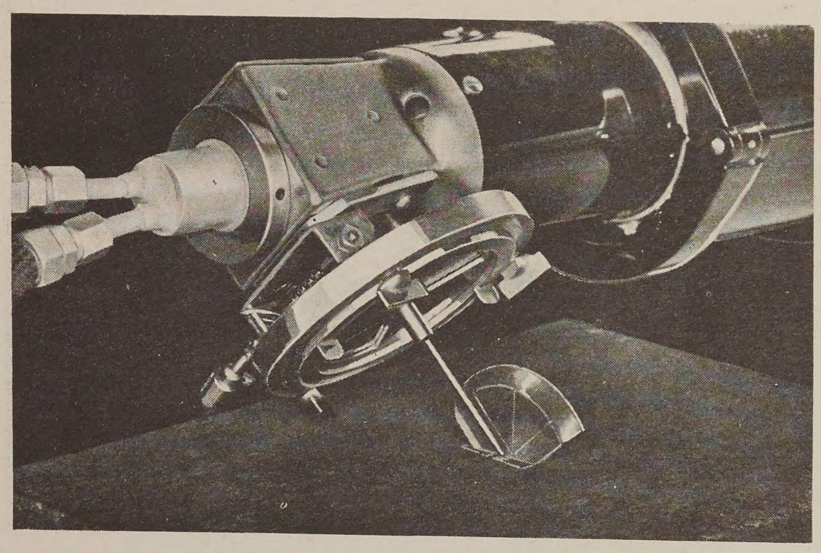

Fig. 2. An Exposure Set-up for Determining the Stress due to Welding in a 1-inch Steel Plate. The Pin in the Middle of the Disk is Used to Obtain the Proper Distance between the Object and Film, and is Removed when Making the Negative. A Protractor Helps to Align the Equipment at the Correct Angle, Usually 45 Degrees

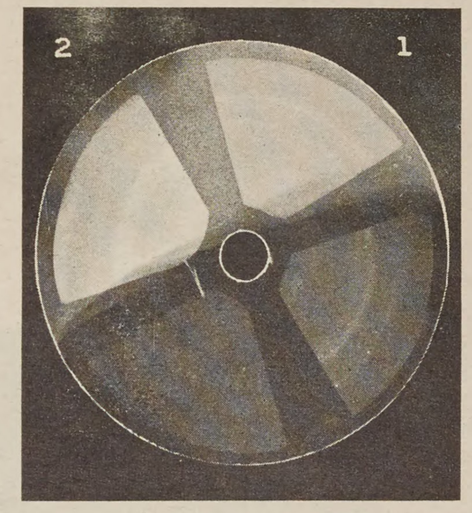

Fig. 3. Radiograph is Divided into Four Segments, Two of which have been Treated to Intensify the Lines. Pattern No. 1 Shows the Exposure of Normalized Steel, while Pattern No. 2 Shows the Same Part under a Load of 40,000 Pounds per Square Inch. The Broadening of the Lines is Due to Plastic Deformation

The actual exposure set-up is depicted in Fig. 2. In order to obtain reliable results, it is of utmost importance that all dimensions be accurately maintained. Thus alignment pins keep the proper object-to-film distance, and a protractor helps in aligning the camera at the desired angle, which usually is set at 45 degrees to the surface of the specimen. The angle of reflection varies, however, depending on the crystallographic plane that reflects the rays.

Fig. 3 shows a typical back-reflection radiograph. The circular film is divided into four segments, opposite segments registering an exposure. In the illustration, one half of the print appears darker. In the lighter portion the X-ray patterns stand out more clearly. This half of the film has been treated with a special intensifying agent in order to strengthen the lines, which must be measured with extreme care. Pattern No. 1 shows the exposure of normalized steel. The outer circle in both segments is the gold reference line. Pattern No. 2 is obtained from the same test bar as pattern No. 1, but under a 40,000 pounds per square inch load. The broadening of the iron lines is due to plastic deformation. In order to determine the stress caused by this load, the distance between the gold reference line and the iron line is measured.

This is accomplished by means of a vernier scale mounted on an optical stage, as seen in Fig. 4. These measurements cannot be made too exactly. The lattice parameter of the iron crystal is, of course, extremely small, and any variations due to stress are of the order of a billionth of an inch. For this reason, the work must be carried out with extreme care, and the results—the X-ray pattern on the film—should be clearly defined for exact measurement to within 0.001 inch.

The applications of this method in the machinery field are too numerous to recount here. It is particularly suited for determining changes in residual stresses. The importance lies in the fact that the examination can be carried out on an object in the field without removing or destroying it. In addition, the X-ray back-reflection method has been used successfully to determine the stress in bridges, ships, rails, car wheels, airplanes, etc.

While the method described is satisfactory and in general use at present, new and improved techniques now being studied may simplify the procedure considerably. It is possible to eliminate the use of a reference material and to employ a special projection apparatus with low magnification to evaluate the results. The film can be projected directly on a stress scale, avoiding mathematics in the final stress analysis.

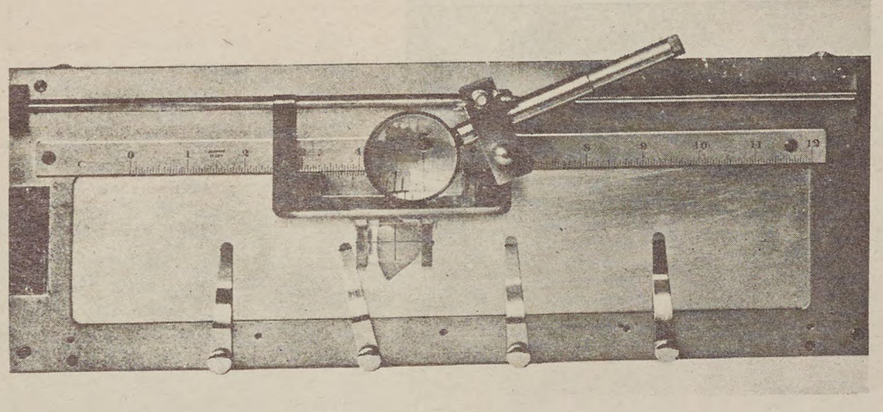

Fig. 4. A Vernier Mounted on an Optical Stage is Used to Measure the Distance between the Line of a Standard Reference Material and the Material being Investigated. From These Measurements, the Stress in Any Particular Spot in a Structure can be Calculated

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