

# **Residual Stress Measurement by X-Ray Diffraction — SAE J784a**

Report of Iron and Steel Technical Committee approved  
September 1960 and last revised by Fatigue Design and  
Evaluation Committee August 1971.

SAE Information Report  
First edition published September 1960  
Second printing January 1964  
Second edition published August 1971

Published by:

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## PREFACE

The predecessor of this manual was published in 1960 as SAE TR-182 (SAE J784) and was intended to bring together the most recent developments in x-ray techniques for residual stress measurements. At that time, it was evident that the ability to reproduce results among different laboratories was lacking. In addition, the disadvantages of film techniques, particularly with regard to hard steels, had been given much attention. Consequently, the original manual covered almost exclusively diffractometer methods and recommended a fairly uniform technique based on two exposures and peak position determination by a three-point parabola.

Since the publication of TR-182, the X-Ray Subcommittee of the SAE Iron and Steel Technical Committee, Residual Stresses and Fatigue (now the X-Ray Division of the SAE Fatigue Design and Evaluation Committee), continued to be very active in the application of x-ray diffraction techniques to residual stress measurements. Much of this activity concerned the circulation of stressed and unstressed specimens in a round-robin fashion. The knowledge gained from these specimens illustrated that reproducibility was still not good. An analysis of the data and circulation of annealed tungsten powder as an alignment standard indicated that laboratories were introducing their own individual biases into the measurements, due to a lack of uniformity in the technique used and, in some cases, to the lack of an understanding of the factors which affect absolute stress measurements. Therefore, it was decided that a revision of TR-182 be undertaken in an attempt to correct these practical difficulties. This subsequent publication, HS J784a is the result of that revision.

It is intended that HS J784a will bring together what we as a Committee consider to be the most important aspects of the x-ray techniques. By necessity, it places much emphasis on theoretical aspects but, at the same time, attempts to show how these theoretical considerations are reduced

to a practical technique. A sincere attempt has been made to provide a cohesive manual with the traditional stigmas which seem to plague committee decision being avoided.

As with any publication of this magnitude, many people have participated and it is not possible to list all who have had a part. However, the Committee would like to express their sincere appreciation to the various authors and the reviewers for the part which they played.

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# **Part A**

## **Principles and Methods of Analysis**

## SECTION 1 RESIDUAL STRESSES: TYPES AND SOURCES

### 1.1 INTRODUCTION

There are two kinds of stress to which an object may be subjected:

(1) Applied stress, due to external forces acting on the object.

(2) Residual stress, which remains after all applied forces have been removed. The basic cause of residual stress is non-uniform plastic flow due to some previously performed operation. Some specific causes are welding, casting, quenching, and nonuniform cold working, such as bending, twisting, grinding, and shot peening. Residual stress may be decreased or eliminated by annealing or by certain mechanical treatments. Residual stresses must form a balanced force system within the object, which means that residual compressive stress in one part of the object must be accompanied by residual tensile stress in another part.

Residual stress may be harmful or beneficial. Residual tensile stress at the surface of an object is usually harmful, in that it forms a contributory cause—and sometimes the major cause—of brittle fracture, particularly in fatigue. On the other hand, compressive residual stress at the surface normally increases fatigue strength.

Residual and applied stress add algebraically, as long as their sum does not exceed the elastic limit of the material. Suppose a designer so proportions a part that the maximum applied stress at the surface, due to the service load, is 80,000 psi tension, which he regards as safe. Unknown to him, however, may be the fact that the part already contains 40,000 psi tensile residual stress at the surface, resulting in an actual service stress of 120,000 psi. If the part had been treated to produce 40,000 psi compressive residual stress at the surface, the real service stress would be 80,000 – 40,000 or 40,000 psi tension.

It is clear that engineers must know what residual stress is present, if any, before they can know the actual stress corresponding to a particular service load. The residual stress is most commonly measured by the following two methods:\*

(1) Mechanical Relaxation (Dissection)—A part of the object is removed, by cutting, for example, and the elastic deformation of the remainder is measured. The stress existing before

the cut was made is then calculated from this deformation. This method is inherently destructive and very time consuming.

(2) X-ray Diffraction—This method depends on the fact that the spacing  $d$  of the atomic planes in a crystalline material is altered by stress and that  $d$  can be determined by measuring the angular position of a diffracted x-ray beam. The fractional change in  $d$  is a strain from which the stress can be calculated. It might be thought that a stress-free specimen of the same material would be needed as a standard for comparison. This is not the case. If the strain is measured in two directions in the residually-stressed object, the stress in any chosen direction can be calculated. The x-ray method is nondestructive and fast, if only stress at the surface is to be determined; (because service failures normally begin at the surface of a part, knowledge of the surface residual stress is sufficient for most applications). The x-rays used in diffraction measurements penetrate only a very short distance into a metal. If information about subsurface stress is required, material must be removed to expose the subsurface material to the x-ray beam, and the x-ray method then too becomes destructive.

X-ray diffraction was first applied to the measurement of stress in 1925 (1),\*\* and in those days the diffracted beams were recorded on photographic film. This kind of recording is suitable for materials which give sharp diffraction lines, but it introduces many complications when the material—such as hardened steel—produces very broad lines. A notable advance was made in 1953 (2), when it was shown that residual stress could be quickly measured, even in hardened steel, by means of the new x-ray diffractometer, in which diffracted beams are recorded electronically. Such measurements have since become routine in many laboratories.

The present manual is in two parts. Part A contains a detailed treatment of the x-ray method, including such topics as instrumental factors, specimen preparation, sources of error, tables of numerical factors, etc. Part B is briefer and contains worked out examples of actual stress measurements. Any reader who is completely uninitiated in the x-ray method might do well to go directly to Part B in order to get some idea of the magnitude of the various quantities involved, and even to make some rough measurements immediately. He will then be in a better position to appreciate some of the details of Part A. In short, he might look at the forest before examining the trees.

### 1.2 SOURCES OF RESIDUAL STRESSES

Residual stresses are introduced into metals and other materials by any process—mechanical, thermal, or chemical—that

---

\*A comparison of all the different methods is made in HS-147 "Evaluation of Methods for Measurement of Residual Stress." New York: Society of Automotive Engineers, Inc., 1957.

\*\*Numbers in parentheses designate References at end of this section.

results in a permanent, nonuniform change in shape or volume. The following categories, with specific examples of how residual stresses arise in each category, are described in this section:

- 1.2.1 Mechanical: surface working, forming, assembling.
- 1.2.2 Thermal: welding, casting, heat treatment.
- 1.2.3 Chemical: oxidation, corrosion, electroplating.
- 1.2.4 Combinations of above: abusive grinding, nonthermal-induced phase transformations.

Other examples in these major categories are covered in reviews of residual stresses (3-5).

**1.2.1 MECHANICAL PROCESSES**—Practically all cold working operations develop residual stresses because of non-uniform flow, including:

**1.2.1.1 Surface Working**—In surface working operations such as shot peening, surface rolling, polishing etc., the penetration is shallow, thus deforming the surface more than the interior. The distended surface is held in compression by the interior, while the less worked interior is placed in tension by the heavier worked surface. As is well known, this residual stress pattern is highly desirable in springs, shafts, gears, and bearings where high tensile stresses develop at the surface in fatigue service. The compressive stresses in the worked surface, in effect, reduce the magnitude of the service stress.

The stress pattern may be demonstrated by shot peening a flat test strip on one side only (Fig. 1). In adjusting to the induced stresses, the test strip bows, with the peened surface on

the convex side. This also indicates how an imbalance of residual stress can cause warping of a part.

**1.2.1.2 Drawing**—In rod and wire drawing, the residual stress pattern developed is largely a function of whether or not the penetration is deep. When penetration is shallow (light reductions, small die angle), the residual stresses are compressive at the surface and tensile in the interior, similar to surface working processes. The longitudinal, circumferential (hoop), and radial stress distributions are shown in Fig. 2A for the case of shallow penetration of cylindrical stock. When the penetration is deep, the residual stress patterns for the various directions are now completely reversed from the case of shallow penetration (Fig. 2B). (These patterns for drawing would also apply to swaging and extrusion.)

**1.2.1.3 Rolling**—In flat rolling (rolling with a constant reduction in thickness at each point across the width of the strip), the residual stress pattern is a function of many factors, including roll diameter, reduction per pass, and rolling direction. If strip rolling is carried out in one direction only with shallow penetration, the residual stress will lie in the rolling or longitudinal direction only, with a pattern similar to that for shallow drawing in the longitudinal direction—that is, compression at the surface and tension in the interior.

At the other extreme of deep penetrating conditions (heavy reduction per pass, small roll size), the residual stress pattern cannot be rationalized in simple terms. It is found, though, that the longitudinal residual stress pattern is reversed—the surface is now in tension while the interior is in compression.

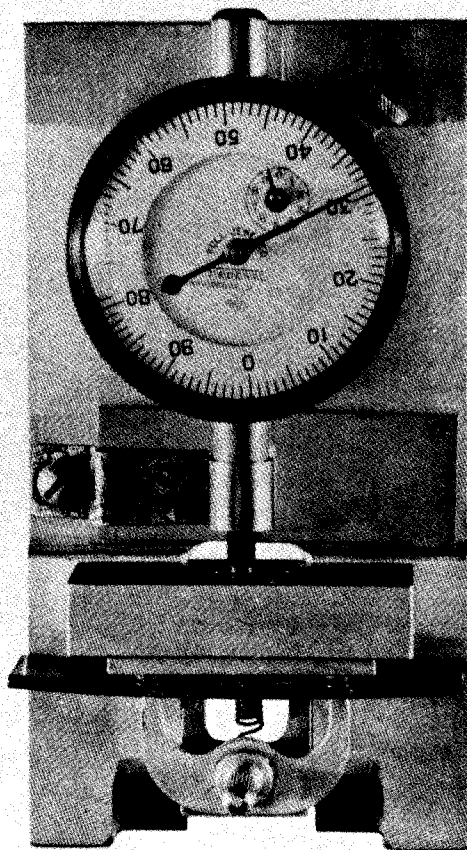


Fig. 1 - Shot peened (on one side only) steel test strip (Ref. 5, used with permission of McGraw-Hill Book Co.)

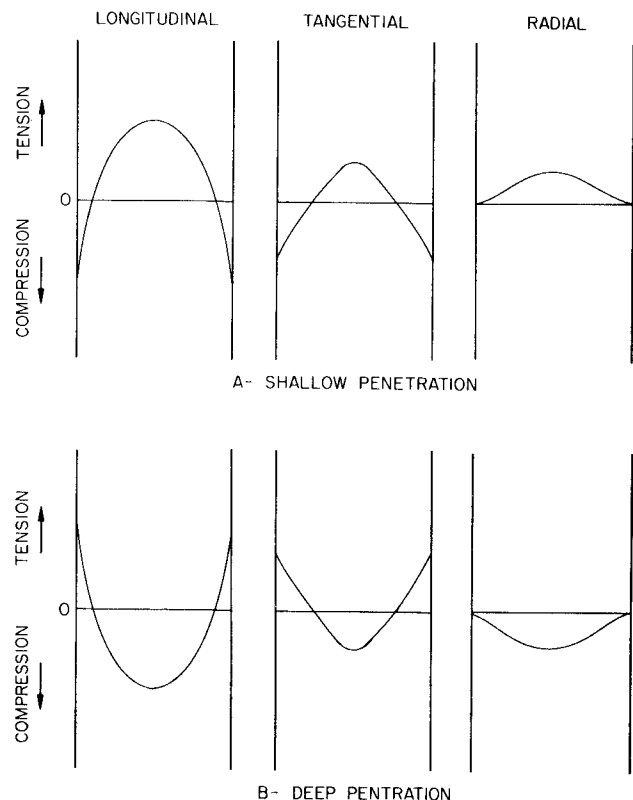


Fig. 2 - Idealized residual stress patterns found in shallow-drawn rods (no phase transformation)

In rolling, and in other cold working processes as well, the amount of deformation in the final pass largely determines the final residual stress pattern.

**1.2.1.4 Grinding and Machining**—With gentle grinding or good machining practice (turning, milling, etc.), metal is not only removed but the surface stress is changed as well because of cold working (6). As in the case of surface working operations, the surface is distended, resulting in the same type of residual stress pattern—surface in compression extending to a depth of a few thousandths of an inch and interior in tension (Fig. 3).

With less than gentle grinding practice (dull wheel, heavier cuts, inadequate coolant), which very often is the normal practice, a thermal effect is also introduced, which places the surface in tension, as shown in Fig. 3. This will be more fully discussed later.

**1.2.1.5 Assembling**—Stresses are often developed in the various components of an assembly during manufacture or con-

struction. (These stresses are often referred to as reaction stresses.) In shrink fitting of a die assembly, for example, the outer ring is shrunk onto the oversize insert. The mismatch between the two components creates tensile hoop (tangential) stresses in the outer ring and compressive hoop stresses in the insert.

An example of highly beneficial residual stresses in assembling a structure is prestressed concrete. Here, steel reinforcing rods are externally loaded in tension while concrete is poured around them. After the concrete has set, the external loading force on the rods is released, and, as the steel and concrete adjust to this new condition, the steel rods are left in tension while the concrete is placed in compression.

An example of possible harmful residual stresses arising from assembly is in large structures such as ships. In this case, it is often necessary to use hydraulic jacks to correct cumulative errors which arise during construction by welding or riveting. After construction, residual stresses are introduced into the assembly when the jacks are removed.

**1.2.2 THERMAL PROCESSES**—Residual stresses arising from thermal processes may be classified as those arising from a thermal gradient alone, as in quenching a hot solid with no phase transformation taking place, and those from a thermal gradient in combination with a phase transformation, as in heat treating steel. Examples from both of these classes are analyzed below.

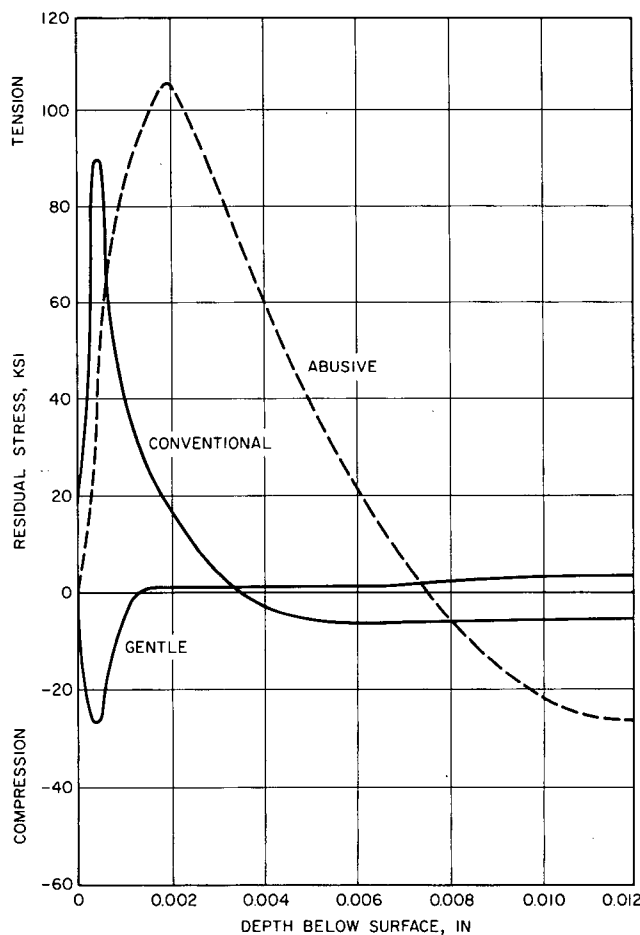
**1.2.2.1 Thermal Gradient**—Examples where residual stresses develop because of a thermal gradient on cooling include quenching, casting, and welding.

**1.2.2.1.1 Quenching**—The general type of residual stress pattern found in quenched cylinders is similar to that shown for shallow drawing (Fig. 2A). The pattern arises as follows: On quenching in the absence of phase transformation, length changes are proportional to temperature changes. Thus, after a short time, the surface during the quench contracts more than the interior, creating a pressure against the core. Since the core does not contract by the same amount, this creates a condition of tension in the rigid outer shell and compression in the core. Irreversible plastic flow then occurs.

This condition is only temporary because, as cooling continues, the core cools through a greater temperature range than the outer shell, and the transient situation of the first part of the cooling is reversed. Due to this temperature lag, the core contracts more than the outer shell. Consequently, the core is placed in tension since the rigid outer shell prevents complete contraction, while the outer shell is in compression. This is purely the result of thermal changes and accompanying plastic deformation with no phase transformation.

Most steels quenched from below about 1200 F would exhibit the residual stress pattern of Fig. 2A. Tempered glass would also show a similar pattern, since it is made by cooling the surface of hot glass sheet by air jets, placing the surface in compression and the interior in tension.

The actual value of the peak stresses reached on quenching increases with an increase in the following factors: quenching temperature, quenching power of the coolant, section size, modulus of elasticity, yield strength, coefficient of thermal ex-



|                     | GRINDING CONDITIONS |                |         |
|---------------------|---------------------|----------------|---------|
|                     | GENTLE              | CONVENTIONAL   | ABUSIVE |
| WHEEL               | A46HV               | A46KV          | A46MV   |
| WHEEL SPEED, FT/MIN | 2000                | 6000           | 6000    |
| DOWN FEED, IN/PASS  | "LS"                | 0.001          | 0.002   |
| GRINDING FLUID      | SULF OIL            | SOL OIL (1:20) | DRY     |

Fig. 3 - Typical residual stress distributions after gentle and normal grinding of hardened steel (stress measured in direction of grinding)

pansion. As peak stresses decrease, the thermal diffusivity of the part is lowered.

1.2.2.1.2 Castings—As in the case of quenched cylinders, castings which undergo no phase transformation would normally have compressive stresses at the surface and tensile

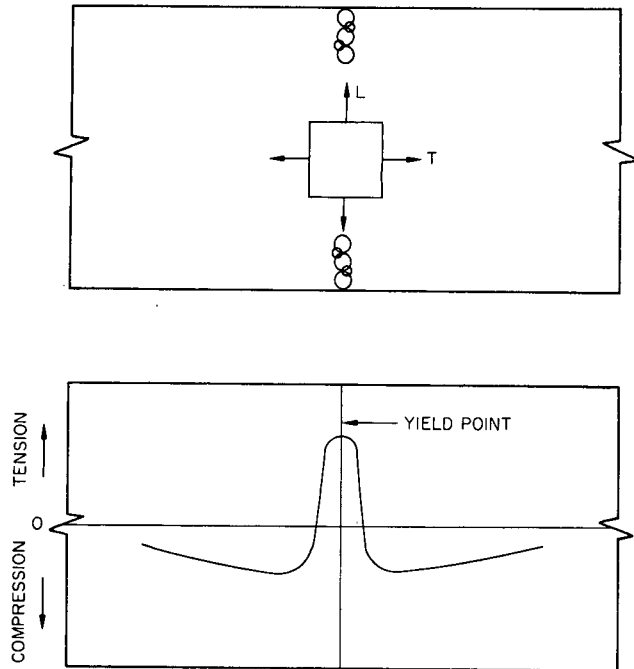


Fig. 4 - Longitudinal residual stresses in butt-welded plate

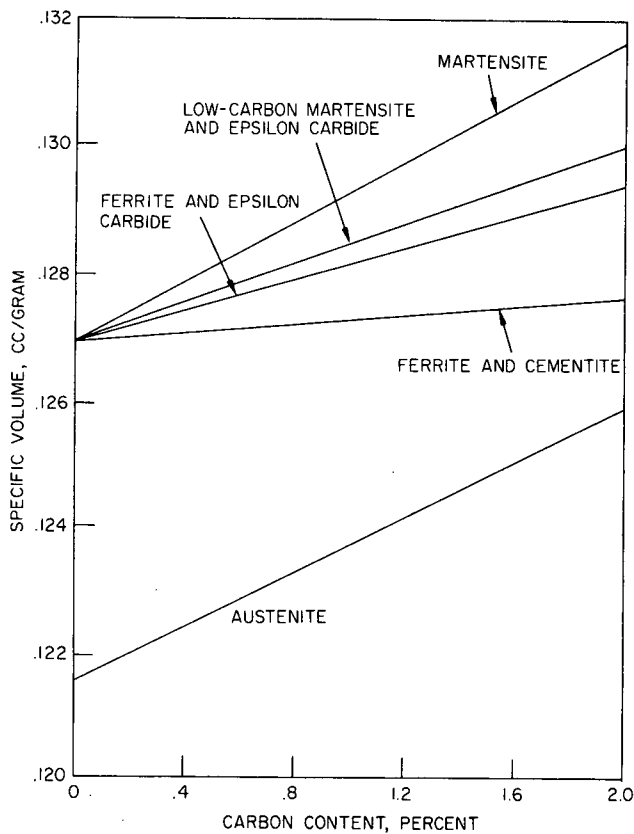


Fig. 5 - Specific volumes of steel phases versus carbon content

stresses in the interior. The situation can be complicated, however, by three major factors:

(1) The mechanical restraint the mold offers to a shrinking casting, as in permanent molding or die casting.

(2) The artificial cooling rates (chills) that may be introduced into the casting.

(3) The casting design may be such (for instance, section size may vary) that greatly different cooling rates are produced.

1.2.2.1.3 Butt Welding—Residual stresses are developed in weldments, even in the absence of phase transformation in the solid state. Considering a flat plate butt-welded along a line, the longitudinal stress (stress parallel to the weld line) (Fig. 4) is developed as follows. As the weld metal and heat-affected zone shrink during cooling, they are restricted by the cool surrounding area. Longitudinal tensile stresses are thus introduced in the welded zone, balanced by compressive stresses immediately adjacent.

1.2.2.2 Hardening Heat Treatments—In quench-hardening of steels, the effects of phase transformation are superimposed on those from the thermal gradient. Because of their great industrial importance, both direct-hardening and carburizing steels are analyzed here in some detail. As an aid in following the discussion, the specific volumes of the microstructural phases associated with steel heat treating are given in Fig. 5 so that the volume changes (and thus the stress changes) may be correlated with phase transformation.

1.2.2.2.1 Quenching of Direct-Hardening Steels—In the cool-

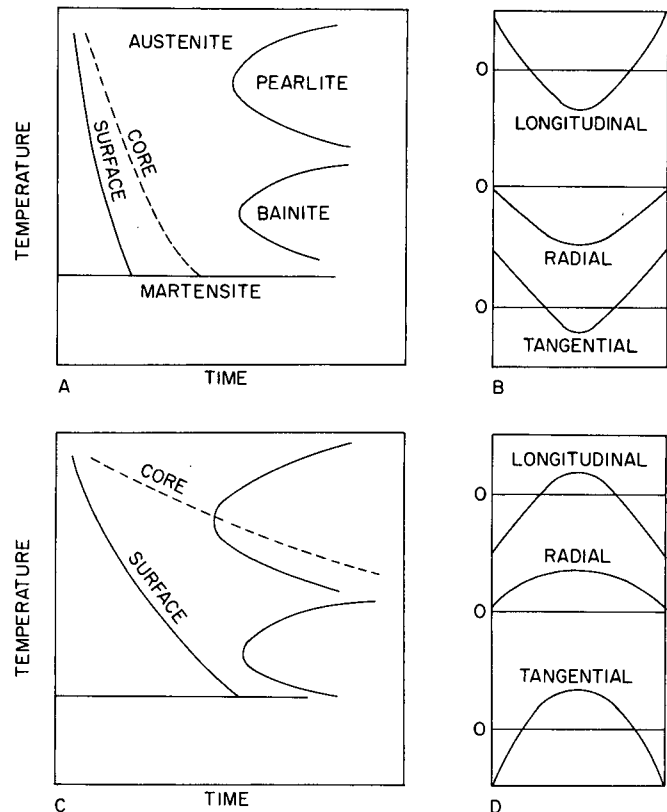


Fig. 6 - Residual stress patterns developed on quench-hardening a steel cylinder. Cooling curves for the surface and core regions of the part are shown relative to the TTT diagram.

ing of direct-hardening steels, the austenite decomposition involves the formation of martensitic, bainitic, or pearlitic structures. In every case there is an expansion, as indicated in Fig. 5.

The residual stress pattern developed depends on many heat treating factors, notably the cooling rate of various zones of the part during quenching. Some patterns for quenched cylinders may be seen in Fig. 6. The cooling curves for the surface and core of the part which develop these patterns are also shown in terms of the TTT diagram.

Consider first the case where the quenching rate was such that the entire bar transformed to martensite (Fig. 6A). The transformation and the attendant volume expansion (about 4%) begins at the surface because it reaches the  $M_s$  temperature first, while the interior is at a higher temperature. The warm ductile austenite in the interior follows the outside expansion plastically. When the inside of the bar transforms to martensite and expands, the cold outside surface is stretched elastically. Thus, the exterior is thrown into tension, the interior into compression. The final residual stress pattern is shown in Fig. 6B. (This assumes that the stresses due to phase transformation are dominant over those developed solely by a thermal gradient.) The danger with this type of residual stress pattern is that the peak tensile stresses generated at the surface may be so high as to cause quench cracking.

A common occurrence in heat treating is that the cooling rate is not sufficiently fast to harden the part completely throughout to martensite, and that some high temperature phase transformation occurs in the interior. This structure is termed a slack-quenched structure. The resulting residual stress pattern for this structure (Fig. 6C) may be completely reversed from the preceding example. Here the sequence of cooling events is as follows. First, the outside expands martensitically, at about the same time that the interior expands pearlitically. The hot austenite-pearlite structures on the inside, being softer than the martensite, deform plastically to re-

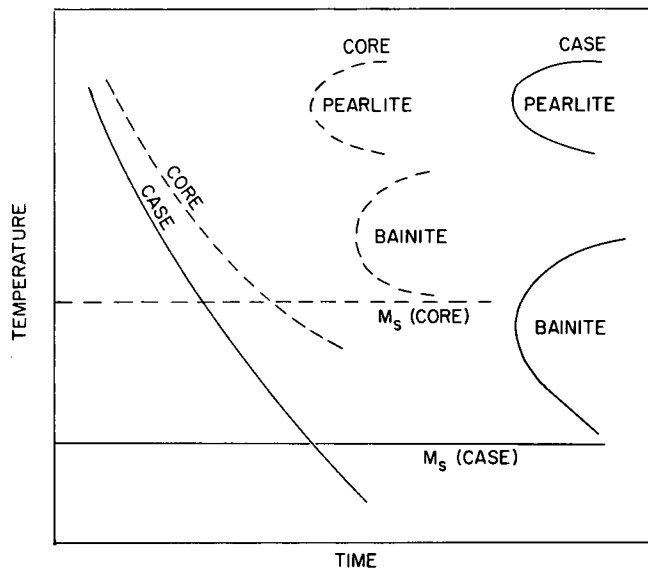


Fig. 7 - Cooling curves of the case and core of carburized steel relative to their TTT diagrams

lieve any stress developed. Later, the inside cools and contracts, producing the same type of residual stress pattern as that due to a thermal gradient alone—compression at the surface and tension in the core (Fig. 6D). This is the kind of pattern to be expected from quenching plain carbon steels in section sizes greater than about 1/4 in., or from quenching relatively large sections of alloy steels with insufficient hardenability.

The residual stress pattern in steel parts hardened by flame or induction heating resembles that in slack-quenched parts, that is, compressive at the surface and tensile below the hardened skin. This pattern results because only the heated skin portion undergoes the austenite-to-martensite transformation in cooling. The volume increase associated with the transformation of the skin portion is resisted by the cold interior, placing the skin in compression and inducing tensile stresses in the underlying metal.

Decarburization of the surface during heating for hardening can lead to a dangerous residual stress state. On the quench, the surface transforms to a pearlitic structure at a relatively high temperature before the underlying material transforms to a martensitic structure. At lower temperatures, when the underlying material transforms to martensite with a volume increase, the rigid outer pearlitic surface is thrown into tension. This explains why many decarburized steels exhibit quench cracking.

1.2.2.2.2 Quenching of Carburizing Steels—In many applications where wear resistance must be combined with high fatigue resistance, carburizing of steel is used to develop compressive surface stresses by controlling the time sequence of

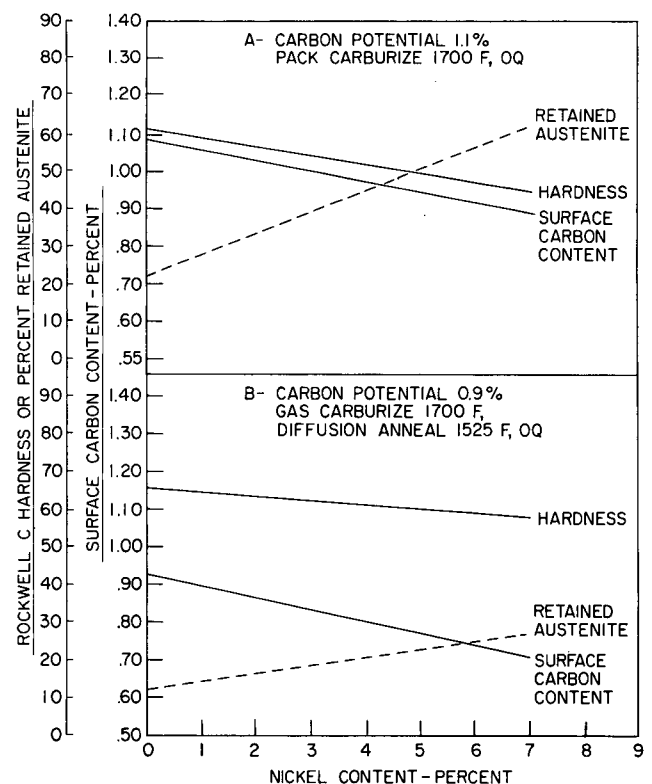


Fig. 8 - Influence of Carbon Potential and nickel content on the case properties of various carburizing steel grades (Ref. 8)

the austenite-to-martensite transformation throughout the part. With a homogenous direct-hardening steel, for example, the surface transforms first during the quenching period; however, by diffusing carbon into the surface before or during the austenitizing step, the process could be reversed and the surface caused to transform last (7). This may be illustrated by considering the cooling curves of the surface and core regions, superimposed on their TTT diagrams (Fig. 7).

As indicated in Fig. 7, the core transforms first to hard martensite due to its higher  $M_s$  temperature. Its volumetric expansion is taken up by yielding of the relatively soft, warmer austenite at the surface. Finally, as the temperature of the surface falls further, the surface material transforms, with its expansion being opposed by the hard martensitic core. This places the surface layers in compression, which is so desirable for fatigue resistance in rotating parts like bearings and gears; the core is in tension.

**1.2.2.2.3 Post-Quenching Treatments**—The as-quenched structure of direct-hardening or carburizing steel usually consists of fresh martensite, retained austenite, and precipitates such as carbides (assuming that no high-temperature products such as bainite or pearlite formed on the quench). The amount of retained austenite present is a function of alloy content (particularly Mn and Ni) and carbon level, as shown in Fig. 8, for various carburized steels (8). Other classes of steels which would show substantial amounts of retained austenite include: tool and high speed steels, PH stainless, maraging, 9% nickel, and martensitic stainless steels. For example, nondeforming tool steels are heat treated to contain as much as 20-30% retained austenite intentionally so that zero dimensional change is experienced on heat treating. The retained austenite in most other steels is present unintentionally.

All quenched structures are metallurgically unstable; that is, the amounts and composition of the phases in these structures can be changed by thermal means, such as tempering. These

microstructural changes can lead to changes in the macro residual stresses as well, since the volumes of the initial phases in the as-hardened structure differ from the final phases, as shown in Fig. 5. Note that martensite has the greatest specific volume, and austenite the least. Also of interest is that the two tempered structures (low-carbon martensite with epsilon carbide and ferrite with epsilon carbide) have smaller specific volumes than the fresh martensite (as-quenched) structure.

**1.2.2.2.4 Cold Treating**—Cold treating, such as subzero processing, is a very effective means of improving the residual stress state in steels with a retained austenite gradient, as in the carburized and hardened steel class or in grades of the direct-hardening class which have not hardened throughout on the quench. In either of these steel classes, the higher retained austenite at the surface results in greater transformation to martensite on cold treating. This produces an expanded surface, and thus a compressive stress state with respect to the core.

The improvement in residual stress by cold treating is convincingly shown by Bond (9). SAE 9310 steel was carburized at 1700 F to a depth of about 0.040 in. The surface carbon content was 0.80%, with an estimated retained austenite content of 30% after oil quenching from 1500 F. The results in Fig. 9 show that the compressive residual stress at the surface of the as-quenched structure was 40,000 psi, which was raised to 60,000 psi compression after cold treating at -120 F. (It is estimated that the retained austenite at the surface is still about 15% even after cold treating. This demonstrates that retained austenite can be quite stable, especially the high alloy austenites, and that cold treating will not necessarily reduce the austenite content down to low levels.)

**1.2.2.2.5 Tempering**—There are at least five opportunities for residual stress and other properties to change upon tempering of quench-hardened steel.

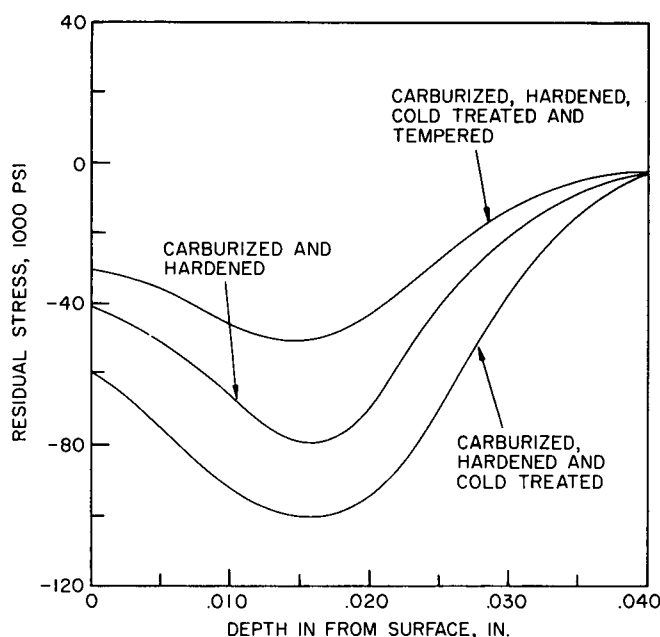


Fig. 9 - Residual stress as a function of depth for carburized SAE 9310 steel (Ref. 9)

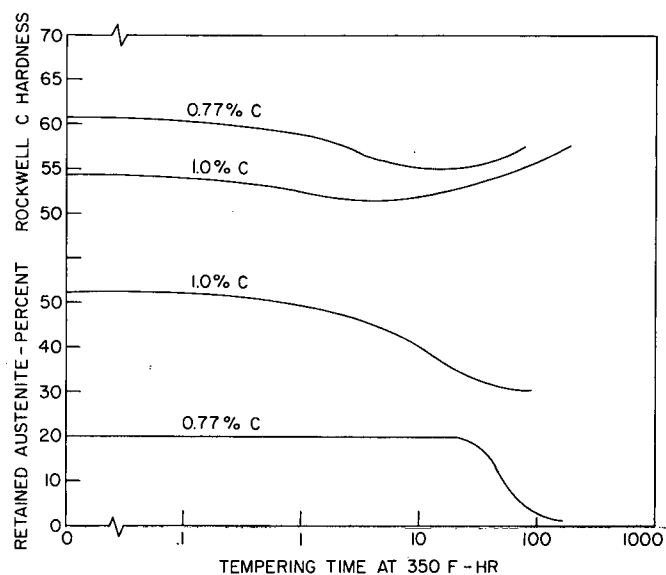


Fig. 10 - Hardness and retained austenite changes on tempering of carburized 4918 steel after oil quenching from 1525 F (Ref. 8)

(1) Considering the thermal effect alone, with no phase transformation, residual stresses can be relieved by plastic yielding on heating for tempering.

(2) The actual tempering reaction, such as the decomposition of the martensite, leads to contraction (Fig. 5).

(3) The tempering of any retained austenite in the structure leads to expansion upon precipitation of alloy carbides from this phase.

(4) Cooling after tempering, particularly if quenching is used, may develop compressive residual stresses, since the surface layers cool faster than the core.

(5) On cooling after tempering, retained austenite, which has been conditioned by the precipitation of alloy carbides during tempering and is thus less stable, may transform to martensite.

These combined tempering effects upon the hardness and retained austenite changes at the surface of carburized 4918 steel (5% Ni, 0.25 Mo) are demonstrated in Fig. 10. Note that the hardness first drops with tempering time as the martensite is tempered, but that eventually the hardness reaches a minimum and then rises with further time as the retained austenite starts to transform to fresh martensite or bainite.

Bond (9) has shown that tempering (300 F) of SAE 9310 after carburizing, hardening, and cold treating has a substantial effect on the residual stress level (Fig. 9). The residual stress was lowered to 30,000 psi compression after tempering, as compared with 60,000 psi compression for the cold treated condition.

Liss, et al. (10), have reported quantitative results on the influence of tempering on the residual stress in a 2-1/8 in. diameter 1045 steel, hardened by water quenching from 1480 F. The surface stress of an untempered bar dropped from a value

of 158,000 psi compression to a value of 86,000 psi compression after tempering at 400 F.

Although the two examples cited above pertain to the decrease in compressive stresses on tempering, it is well known that tempering is beneficial in reducing residual tensile stresses by the decomposition of martensite during the tempering reaction; the higher the tempering temperature, the lower the stress.

1.2.2.2.6 Aging—Precipitation hardening (aging) is a common strengthening mechanism for three major classes of high alloy steels: precipitation hardening (PH) stainless steels, secondary hardening tool steels, and maraging steels. With the secondary hardening steels, such as the high-speed steels, the as-quenched hardened structure is further hardened by precipitation of alloy carbides in the martensitic matrix during the tempering operation. In contrast, the maraging and PH stainless steels, which are essentially carbon-free, can produce a ductile martensite in the quenched condition. Upon reheating (upon aging), intermetallic compounds such as  $\text{Ni}_3\text{Ti}$ , instead of alloy carbides, precipitate out from the supersaturated martensitic matrix.

In all three classes of these alloy steels, stresses may develop in both the precipitate and the enveloping matrix, due to the larger volume of the precipitate compared to the matrix from which it was formed. Tangential tensile stresses (among other stresses) may be expected in the enveloping matrix, as in the case of the shrink-fitted insert previously discussed.

Many steels which are high in nickel and/or manganese are susceptible to an interesting phenomenon, known as austenite reversion, upon aging or heating after hardening. The heating operation may be aging or service exposure at elevated tem-

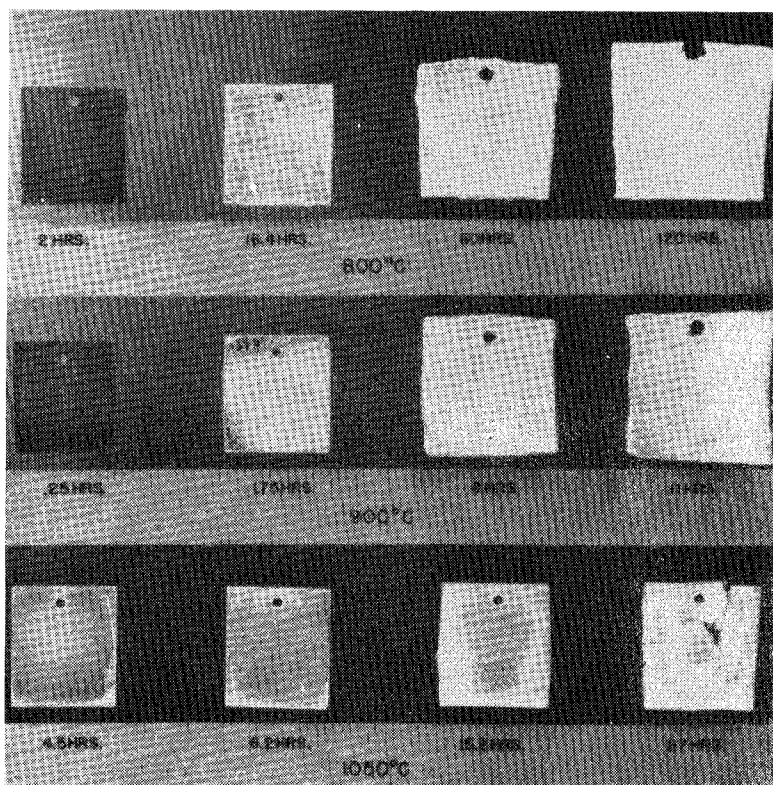


Fig. 11 - Growth of zirconium during air oxidation. Tensile stresses induced in zirconium during oxidation have dragged the metal out to the very edge of the oxidized specimens

peratures. For example, Peters (11) showed the reversion to austenite in maraging steels. An 11% nickel steel had about 20% austenite after aging at 900 F for 3000 hr. With a 30% nickel steel, as much as 75% austenite (from an initial value of 6%) was found on aging at 900 F for 200 hr.

The austenite reversion is believed due to the gradual breakdown of the martensitic phase, approaching the equilibrium three-phase structure of ferrite, austenite, and carbides (and other precipitates such as  $\text{Ni}_3\text{Ti}$ ). The Fe-Mn-C ternary diagram, at the 10% Mn level, shows the presence of austenite as low as 700 F. The Fe-Ni-C ternary diagram would show similar features.

In addition to the maraging steels, other high alloy steels such as PH stainless and 9% nickel steels, because of their relatively high nickel content, may be expected to show the phenomenon of austenite reversion, probably as low as 700 F for long exposure times.

**1.2.3 CHEMICAL PROCESSES**—Not as well known is the fact that chemical processes—oxidation, corrosion, electroplating—are sources of residual stress, just as are mechanical and thermal processes.

**1.2.3.1 Oxidation and Corrosion**—Large stresses may be generated by the formation of an oxide scale or other reaction product on the surface of a metal during oxidation or corrosion. In oxidation, for example, stresses arise because the oxide usually has a larger volume than that of the metal from which it was formed. If the oxide maintains crystallographic coherency with the underlying metal, then the oxide is in compression, while the metal is placed in tension. These stresses often assume a major role in the oxidation process by influencing spalling, warping, and even the dimensional stability of the metal. For example, zirconium sheet (Fig. 11) shows a huge increase in lateral surface area (as much as 200%) by the tensile stresses introduced during oxidation (12).

**1.2.3.2 Electroplating**—As in oxidation, residual stress may

also be produced in electroplated coatings if the electroplate maintains coherency with the underlying substrate. The crystalline structure of the plate may be distorted or even modified as the two structures (plate and substrate) attempt to adjust to one another.

Another source of stress in the electroplate is believed due to the nonuniform distribution of hydrogen and/or incorporation of impurities from the plating bath.

Residual stresses in the electroplate may be compressive or tensile, depending on the material and plating conditions (–10,000 to +65,000 psi in nickel from chloride-sulfate baths).

**1.2.4 COMBINATION PROCESSES**—The residual stress state developed in a material is not readily interpretable when mechanical, thermal, and chemical effects are combined during processing. A few examples that are amenable to analysis are cited here.

**1.2.4.1 Abrasive Grinding**—When nonideal grinding practice is followed—dull wheel, inadequate coolant, deep cuts—a complex situation exists in that both a mechanical effect (from surface working) and a thermal effect (from excessive heat generation) are operative. If the shrinkage of the surface due to the heating is of greater significance than the mechanical deformation, then the stresses are tensile at the surface. All too familiar is the development of grinding cracks when the tensile stress generated at the surface exceeds the fracture stress.

**1.2.4.2 Nonthermal-Induced Transformation**—In carburized steels and many high alloy steels, austenite may be present in substantial amounts as the minor phase, or as the matrix phase, in stainless steels. The austenite is not metallurgically stable, in that both external stress and radiation may transform the austenite into martensite; thermal means are not necessary. The resulting volume change, of course, can alter the existing residual stress pattern. Some examples are:

(1) Running-in or preservice operations of carburized bearings and gears causing cold working of the surface and trans-

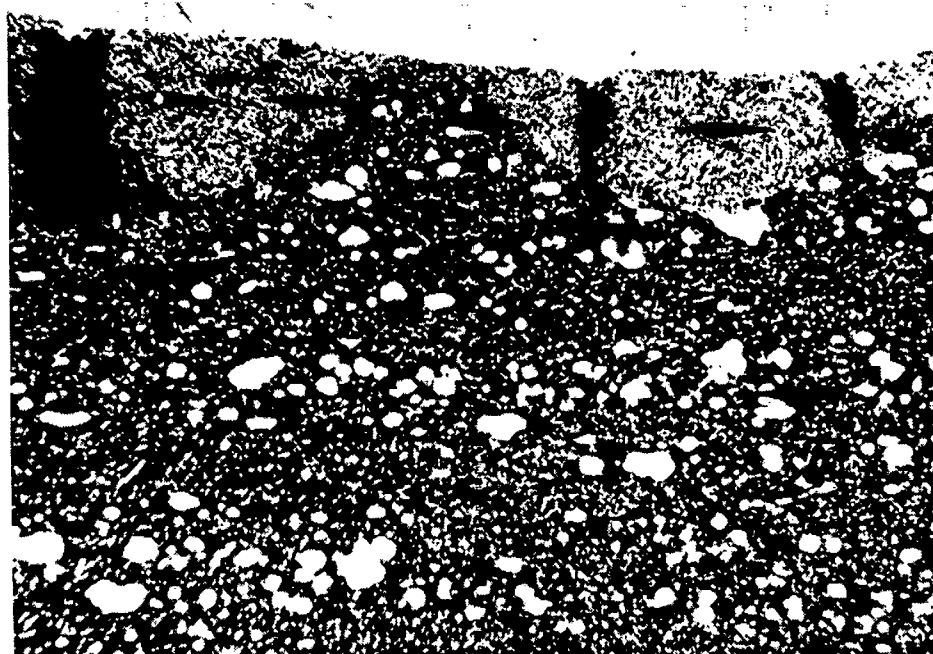


Fig. 12 - Electrical discharge machined surface of hardened high-speed steel (100X) reduced 46% for reproduction

formation of retained austenite.

(2) Shot peening of hardened steel surfaces, again resulting in cold working and transformation of retained austenite.

(3) Mechanical working of austenitic steels, resulting in cold working of the austenite and its partial transformation to martensite (13).

(4) Neutron radiation of hardened steels, causing austenite-to-martensite transformation and change in residual stress in both the martensite and remaining austenite (14).

These examples demonstrate that the amounts of austenite and martensite, and thus the residual stresses, may be altered by nonthermal means. In the past, only the residual stress in the martensitic phase has been deemed of importance in the correlation with fatigue, dimensional stability, and stress corrosion cracking. It would seem that in multi-phase structures which are metallurgically unstable, the austenite content should be examined as well, since failures may originate in this phase, or in the martensite, or at the interface between the two phases where the stress gradient may be high.

1.2.4.3 *Others*—Other common examples where mechanical and thermal processes work in combination to produce residual stress include: assemblies using weldments and castings, flame cutting, rapid local heating of cold drawn material, and newer processes such as electrical discharge machining of hardened steel as demonstrated by cracking at the surface (Fig. 12).

### 1.3 REFERENCES

1. H. H. Lester and R. H. Aborn, Army Ordnance, Vol. 6, 1925-1926, pp. 120,200,283, and 364.
2. A. L. Christenson and E. S. Rowland, ASM Transactions, Vol. 45, 1953, p. 638.
3. W. M. Baldwin, Jr., "Residual Stresses." ASTM Proceedings, Vol. 49, 1949, p. 539.
4. W. R. Osgood (ed.), "Residual Stresses in Metals and Metal Construction." New York: Reinhold Publishing Co., 1954.
5. J. O. Almen and P. H. Black, "Residual Stresses and Fatigue in Metals." New York: McGraw-Hill, 1963.
6. H. R. Letner, "Influence of Grinding Fluids Upon Residual Stresses in Hardened Steel." ASME Transactions, Vol. 79, 1957, p. 149.
7. D. P. Koistinen, "The Distribution of Residual Stresses in Carburized Cases and Their Origin." ASM Transactions, Vol. 50, 1958, p. 228.
8. L. R. Kupfer, "Core and Carburizing Properties of a New Nickel-Base Carburizing Steel." M. S. Thesis, Case Institute of Technology, 1961.
9. W. B. Bond, "X-Ray Diffraction Studies of the Residual Stress Level in an AMS 6260 (SAE 9310) Carburized Case." Norelco Reporter, Vol. 13, 1966, p. 18.
10. R. B. Liss, C. G. Massieon, and A. S. McKloskey, "The Development of Heat Treat Stresses and Their Effect on Fatigue Strength of Hardened Steels." SAE Transactions, Vol. 74, 1966, paper 650517.
11. D. T. Peters, "A Study of Austenite Reversion During Aging of Maraging Steels." ASM Transactions Quarterly, Vol. 61, March 1968, p. 62.
12. C. A. Phalnikar and W. M. Baldwin, Jr., "Scaling of Zirconium in Air." ASTM Proceedings, Vol. 51, 1951, p. 1038.
13. A. J. Sedriks and J. V. Craig, "Formation and Detection of Metastable Austenite in Abraded Surfaces of 18% Ni-Co-Mo Steel." Journal of Iron and Steel Institute, Vol. 203, March 1965, p. 268.
14. G. L. Eggert, "The Influence of Neutron Irradiation on the Dimensional Stability of Steel." Materials Science and Technology for Advanced Applications, Vol. 11, p. 62.

## SECTION 2 PRINCIPLES OF RESIDUAL STRESS MEASUREMENTS

### 2.1 ELEMENTAL STRESS RELATIONSHIPS

The theoretical developments necessary for the treatment of lattice strains and their relation to stresses follow from the classical theory of elasticity. Consider the case of an infinitesimally small cube inscribed in a body subjected to a stress system. Taking the edges of the cube as x, y, and z axes of coordinates, there will be three components of stress acting on each face, as illustrated in Fig. 13.

Consider the cube face normal to the x axis, across which the normal stress  $\sigma_x$  acts in the x direction and two shearing stresses,  $\tau_{zx}$  and  $\tau_{yx}$ , occur, the first subscript indicating the axial direction in which the shear stress acts. Since under equilibrium conditions there will be no translation of the elemental cube,  $\sigma_x = -\sigma_x$ ,  $\sigma_y = -\sigma_y$ , and  $\sigma_z = -\sigma_z$ .

Likewise, the absence of rotation under equilibrium conditions implies equality of the moments of the shearing forces around the various axes, and from this it follows that  $\tau_{yz} = \tau_{zy}$ ,  $\tau_{xy} = \tau_{yx}$ , and  $\tau_{xz} = \tau_{zx}$ . Thus, the state of stress on any element in a homogeneous isotropic solid is completely described by six components: three normal stresses ( $\sigma_x$ ,  $\sigma_y$ ,  $\sigma_z$ ) and three shear stresses ( $\tau_{xy}$ ,  $\tau_{yz}$ ,  $\tau_{zx}$ ).

In a three-dimensional stress system, such as the one from which the infinitesimal cube was taken, the general state of stress on any oblique plane may be resolved into one normal and one shearing stress. The normal stress acting on this plane is given by

$$\sigma_\phi = \sigma_1 \alpha_1^2 + \sigma_2 \alpha_2^2 + \sigma_3 \alpha_3^2 \quad (1)$$

and the shearing stress on the same plane by

$$\tau_\phi = [( \sigma_1 - \sigma_2 )^2 \alpha_1^2 \alpha_2^2 + ( \sigma_1 - \sigma_3 )^2 \alpha_1^2 \alpha_3^2 + ( \sigma_2 - \sigma_3 )^2 \alpha_2^2 \alpha_3^2 ]^{1/2} \quad (2)$$

where:

$\alpha_1$ ,  $\alpha_2$ , and  $\alpha_3$  = Direction cosines between  $\sigma_\phi$ ,  $\tau_\phi$ , and the principal stress axes

For plane stress, where  $\sigma_z = 0$ , the stress system will consist of two normal stresses,  $\sigma_x$  and  $\sigma_y$ , and a shear stress  $\tau_{xy}$ , unless the directions of the normal stresses are principal stress directions, resulting in  $\tau_{xy}$  being equal to zero.

On any oblique plane which again is not a principal plane, the normal stress is given by

$$\sigma_\phi = \sigma_x \cos^2 \phi + \sigma_y \sin^2 \phi + 2 \tau_{xy} \sin \phi \cos \phi \quad (3)$$

while the shearing stress is given by

$$\tau_\phi = \tau_{xy} (\cos^2 \phi - \sin^2 \phi) + (\sigma_y - \sigma_x) \sin \phi \cos \phi \quad (4)$$

From the preceding equations it should be obvious that, whether a two- or three-dimensional stress state exists in a specimen such as the infinitesimal cube, a shearing stress and strain will always exist on a plane which is not aligned along a principal direction. However, to simplify computations of the stress or strain existing in a system, it is always possible to choose an orientation along the principal axes, where the shear stress is zero.

### 2.2 STRESS-STRAIN RELATIONS

From the theory of elasticity, Hooke's law states that stress ( $\sigma_x$ ) is related to strain ( $\epsilon_x$ ) by the modulus of elasticity (E).

$$\sigma_x = E \epsilon_x \quad (5)$$

The tensile force producing the stress in the x-direction produces not only a linear strain in that direction, but also a strain in the transverse directions. The ratio of the transverse strain to the longitudinal strain is called Poisson's ratio ( $\nu$ ). Thus:

$$\epsilon_y = \epsilon_z = -\nu \epsilon_x = -\frac{\nu \sigma_x}{E} \quad (6)$$

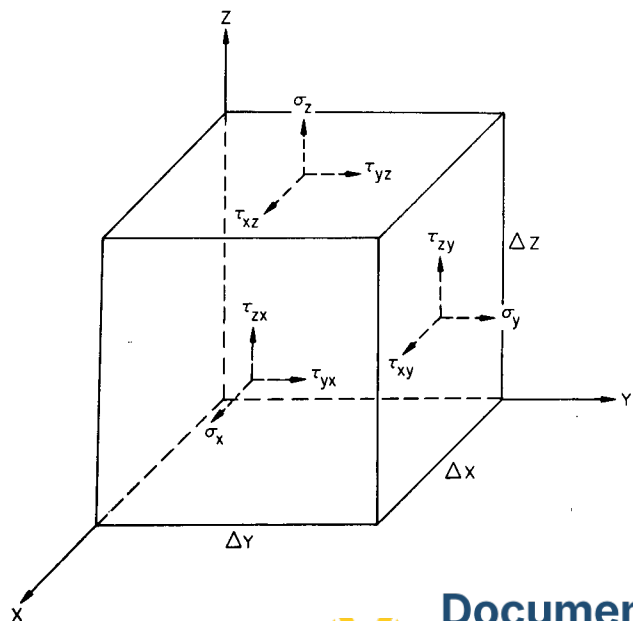


Fig. 13 - Normal stresses,  $\sigma$ , and shear stresses,  $\tau$

For a triaxial stress system existing in a body, Hooke's law states that the strain along any principal direction is due to the stress acting along that axis, plus the superimposed strains resulting from the Poisson effect of principal stresses acting along the other two axes. Taking into account the transverse strains in addition to the longitudinal strains in the directions of the principal axes, the following stress-strain relationships are obtained for a triaxial stress system:

$$\begin{aligned}\epsilon_1 &= \frac{1}{E} [\sigma_1 - \nu(\sigma_2 + \sigma_3)] \\ \epsilon_2 &= \frac{1}{E} [\sigma_2 - \nu(\sigma_3 + \sigma_1)] \\ \epsilon_3 &= \frac{1}{E} [\sigma_3 - \nu(\sigma_1 + \sigma_2)]\end{aligned}\quad (7)$$

For a biaxial state of stress, or plane stress, where  $\sigma_3 = 0$ , then Eq. 7 reduces to

$$\begin{aligned}\epsilon_1 &= \frac{1}{E} (\sigma_1 - \nu\sigma_2) \\ \epsilon_2 &= \frac{1}{E} (\sigma_2 - \nu\sigma_1) \\ \epsilon_3 &= -\frac{\nu}{E} (\sigma_1 + \sigma_2)\end{aligned}\quad (8)$$

It may be noted from the preceding equation that even when the stress along the third axis is zero, the strain along that axis is not zero unless  $\sigma_1 = -\sigma_2$ .

In the case of plane strain,  $\epsilon_3 = 0$  and the preceding stress-strain relationships become

$$\begin{aligned}\epsilon_1 &= \frac{1+\nu}{E} [(1-\nu)\sigma_1 - \nu\sigma_2] \\ \epsilon_2 &= \frac{1+\nu}{E} [(1-\nu)\sigma_2 - \nu\sigma_1]\end{aligned}\quad (9)$$

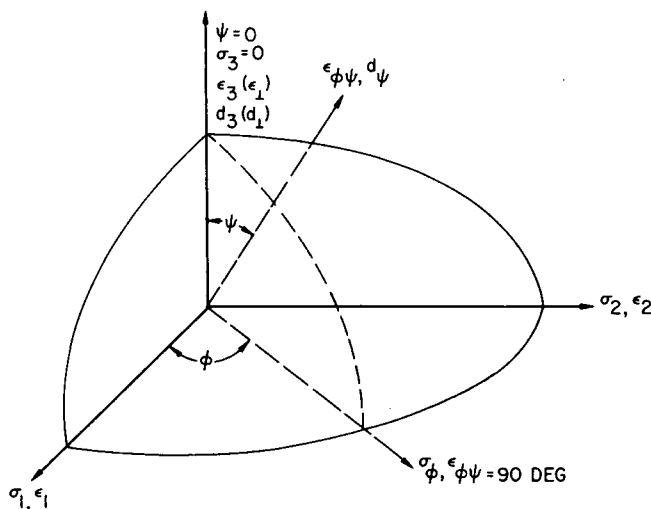


Fig. 14 - Ellipsoids of stress and strain using units employed in x-ray diffraction stress analysis

A more elegant treatment of stress in an isotropic medium is to use a spherical rather than a cubic element of volume. In general, under the action of stresses, a spherical element of volume will be distorted into an ellipsoid.

If the principal stresses or strains are taken parallel to a set of orthogonal axes  $x$ ,  $y$ , and  $z$ , the equation for the strain ellipsoid may be written as

$$\frac{x^2}{\epsilon_1} + \frac{y^2}{\epsilon_2} + \frac{z^2}{\epsilon_3} = 1 \quad (10)$$

Any point  $x_\eta, y_\eta, z_\eta$  on the ellipsoid surface represents the component of normal strain, whose direction and magnitude are approximately but not precisely those of the radius vector to this point.  $\epsilon_{\phi\psi}$  shown in Fig. 14 is a normal strain vector of this sort, and a general relation for its determination, as expressed in terms of the principal strains  $\epsilon_1, \epsilon_2$ , and  $\epsilon_3$ , is

$$\epsilon_{\phi\psi} = \epsilon_1 \alpha_1^2 + \epsilon_2 \alpha_2^2 + \epsilon_3 \alpha_3^2 \quad (11)$$

where:

$$\begin{aligned}\alpha_1 &= \cos\phi \sin\psi \\ \alpha_2 &= \sin\phi \sin\psi \\ \alpha_3 &= \cos\psi = (1 - \sin^2\psi)^{1/2}\end{aligned}$$

These are the direction cosines of the strain vector  $\epsilon_{\phi\psi}$ , with respect to the directions of the principal strains  $\epsilon_1, \epsilon_2, \epsilon_3$ , which define the strain ellipsoid. In a similar manner, we can define a stress ellipsoid for which the normal stress is given by the relation

$$\sigma_{\phi\psi} = \sigma_1 \alpha_1^2 + \sigma_2 \alpha_2^2 + \sigma_3 \alpha_3^2 \quad (12)$$

where:

$$\alpha_1, \alpha_2, \text{ and } \alpha_3 = \text{Direction cosines of stress } \sigma_{\phi\psi}, \text{ with respect to the principal axes}$$

The strain ellipsoid (Eq. 11) is much more useful in calculating stresses, since stress cannot be measured directly but must be computed from strain measurements. Putting values for the principal strains given in Eq. 7 into Eq. 11 for the strain ellipsoid, the general equation relating strains in any direction  $\phi, \psi$  within a body to the acting principal stresses is obtained.

$$\epsilon_{\phi\psi} = \frac{1+\nu}{E} (\sigma_1 \alpha_1^2 + \sigma_2 \alpha_2^2 + \sigma_3 \alpha_3^2) - \frac{\nu}{E} (\sigma_1 + \sigma_2 + \sigma_3) \quad (13)$$

### 2.3 RELATION OF STRAIN EQUATION TO X-RAY STRESS ANALYSIS

Under the surface of a stressed specimen, each element of volume will be subjected to or acted upon by three principal stresses:  $\sigma_1, \sigma_2$ , and  $\sigma_3$ . At the surface, to which x-ray diffraction measurements are confined, plane stress conditions are assumed to exist, and only two principal stresses,  $\sigma_1$  and

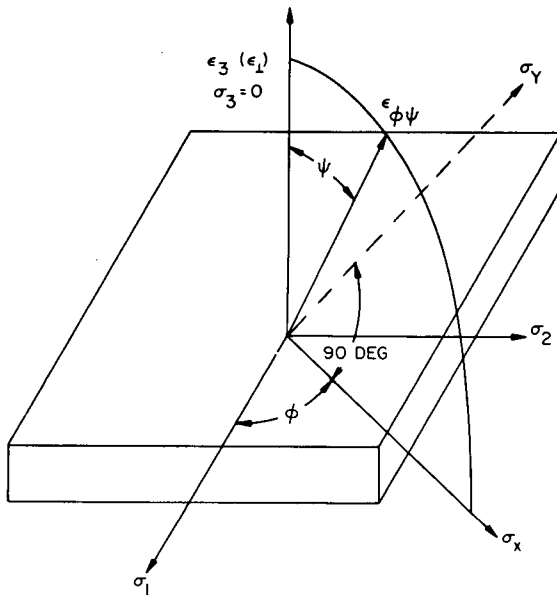


Fig. 15 - Stress in a biaxial system

$\sigma_2$ , lying within the plane of the surface, are possible since  $\sigma_3$ , normal to the surface, is considered to be zero. Nevertheless, as noted in Eq. 8, there are lateral contractions due to Poisson's ratio and there will always be a component of strain  $\epsilon_3$  in the direction of the surface normal.

Consider Fig. 15, which illustrates part of a flat metal surface in which  $\sigma_1$  and  $\sigma_2$  are the principal stresses;  $\epsilon_1$ ,  $\epsilon_2$ , and  $\epsilon_3$  are the principal strains; and  $\sigma_3$  is equal to zero. The general expression for the strain in a direction making angles  $\phi$  and  $\psi$  with  $\sigma_1$  and  $\sigma_3$ , respectively, is given by Eq. 13.

$$\epsilon_{\phi\psi} = \frac{1+\nu}{E} (\sigma_1 \alpha_1^2 + \sigma_2 \alpha_2^2) - \frac{\nu}{E} (\sigma_1 + \sigma_2) \quad (14)$$

Putting in the appropriate values for direction cosines,  $\alpha_1 = \cos\phi \sin\psi$  and  $\alpha_2 = \sin\phi \sin\psi$ , the working equation for the strain vector  $\epsilon_{\phi\psi}$  may be written as

$$\epsilon_{\phi\psi} = \frac{1+\nu}{E} (\sigma_1 \cos^2\phi + \sigma_2 \sin^2\phi) \sin^2\psi - \frac{\nu}{E} (\sigma_1 + \sigma_2) \quad (15)$$

Noting again Eq. 1 and Fig. 15, it is observed that when  $\psi = \pi/2$  or 90 deg, the surface stress component  $\sigma_\phi$  is given by

$$\sigma_\phi = \sigma_1 \cos^2\phi + \sigma_2 \sin^2\phi. \quad (16)$$

Therefore, substituting Eq. 16 into Eq. 15,

$$\epsilon_{\phi\psi} = \left( \frac{1+\nu}{E} \right) \sigma_\phi \sin^2\psi - \frac{\nu}{E} (\sigma_1 + \sigma_2) \quad (17)$$

This is the basic equation relating lattice strains to stresses in the x-ray diffraction method of stress measurement, and it can be utilized to determine the surface stress  $\sigma_\phi$  in any direction on the surface of the specimen.

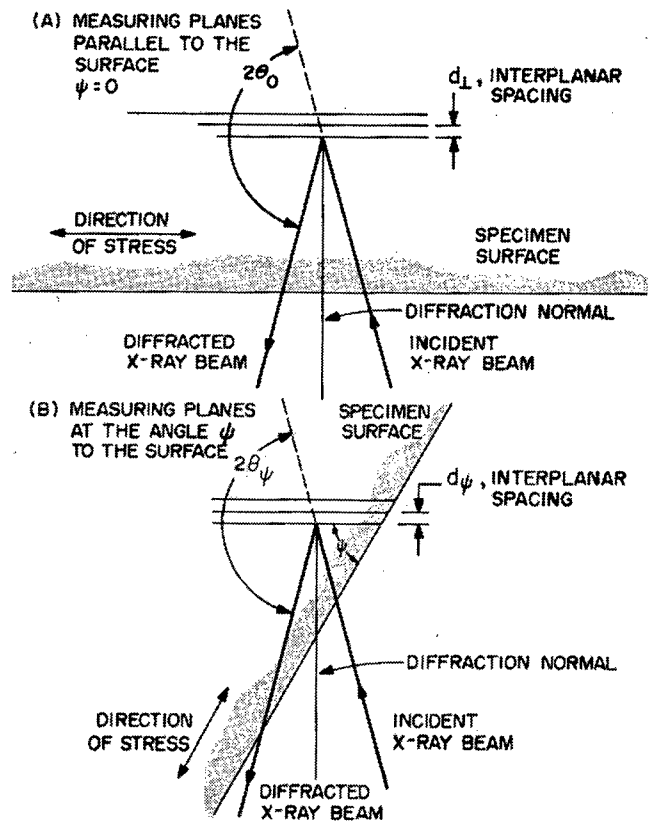


Fig. 16 - Schematic showing orientation of measured lattice planes with respect to specimen surface: (A) specimen at  $\psi = 0$  exposure, (B) specimen rotated  $\psi$  deg

The direction of the surface stress  $\sigma_\phi$  can be made to coincide with the longitudinal, transverse, circumferential, or any other arbitrarily designated direction. Therefore, if the stress in the longitudinal direction of a tube is desired, the tube is positioned on the x-ray stress stage or specimen holder so that the longitudinal direction  $\phi$  is parallel to the plane of the goniometer table rotation and perpendicular to the goniometer axis of rotation. Then two measurements of lattice plane spacings for a particular set of  $hkl$  planes are required—one of strain perpendicular to the surface ( $\psi = 0$  deg) and the second at a rotated angle  $\psi$ , preferably 45 deg or greater, to the surface normal. This is illustrated in Fig. 16. Note that the planes being measured are perpendicular to the incident and diffracted beam bisector, while the measured strains are always along the beam bisector. The stress thus determined is in the direction  $\phi$  on the surface.

In the back reflection region, with the incident beam perpendicular to the surface of the specimen, the planes which reflect lie almost parallel to the surface. The measured spacing  $d_L$  is therefore in the approximate direction of the surface normal. If  $d_o$  is the interplanar spacing corresponding to the unstressed condition, the effective strain normal to the specimen surface will be

$$\epsilon_L = \epsilon_{\phi o} = \frac{d_L - d_o}{d_o} = \frac{\Delta d}{d_o}$$

Since the angle  $\psi$  is equal to zero for  $\epsilon_{\perp}$ , the direction cosines  $\alpha_1$  and  $\alpha_2$  in Eq. 14 are each zero, thus:

$$\epsilon_{\perp} = -\frac{\nu}{E} (\sigma_1 + \sigma_2) \quad (19)$$

Of course, in special cases such as that of a rod in tension or a bar in flexure, where only a single principal stress is operative whose direction is obvious, Eq. 19 reduces to

$$\epsilon_{\perp} = -\frac{\nu}{E} \sigma_1 \quad (20)$$

Consider again the fact that two x-ray exposures are made, one with the incident beam falling almost perpendicular to the surface of the specimen, as in Fig. 16, and the other with the beam inclined at an angle  $\psi$  to the surface normal; and that Eq. 17 covers the general case for calculation of  $\sigma_{\phi}$  from strains measured in these separate directions. By substituting Eq. 19 into Eq. 17 the following simplified form results:

$$\epsilon_{\phi\psi} = \left( \frac{1+\nu}{E} \right) \sigma_{\phi} \sin^2 \psi + \epsilon_{\perp} \quad (21)$$

or

$$\sigma_{\phi} = (\epsilon_{\phi\psi} - \epsilon_{\perp}) \frac{E}{1+\nu} \left( \frac{1}{\sin^2 \psi} \right) \quad (22)$$

The strain  $\epsilon_{\phi\psi}$  is equivalent to the difference in interplanar spacing of the atomic planes which lie perpendicular to  $\psi$  in the stressed and unstressed conditions. If  $d_o$  is considered as the unstressed lattice spacing, it follows that

$$\epsilon_{\phi\psi} = \frac{d_{\psi} - d_o}{d_o} \quad (23)$$

Since  $\epsilon_{\perp} = \frac{d_{\perp} - d_o}{d_o}$ , the quantity  $(\epsilon_{\phi\psi} - \epsilon_{\perp})$  becomes

$$(\epsilon_{\phi\psi} - \epsilon_{\perp}) = \frac{d_{\psi} - d_o}{d_o} - \frac{d_{\perp} - d_o}{d_o} = \frac{d_{\psi} - d_{\perp}}{d_o} \quad (24)$$

There is negligible error by substituting  $d_{\perp}$  for  $d_o$  in the denominator of this expression, thus:

$$(\epsilon_{\phi\psi} - \epsilon_{\perp}) = \frac{d_{\psi} - d_{\perp}}{d_{\perp}} \quad (25)$$

Substituting this into Eq. 22, a more convenient form of that equation results for obtaining the stress in any surface direction  $x$ , as a function of changes in interplanar spacings:

$$\sigma_{\phi} = \sigma_x = \frac{d_{\psi_x} - d_{\perp}}{d_{\perp}} \left( \frac{E}{1+\nu} \right) \frac{1}{\sin^2 \psi} = K^* \left( \frac{d_{\psi_x} - d_{\perp}}{d_{\perp}} \right) \quad (26)$$

Sometimes it is also desired to determine the stress  $\sigma_y$  in the direction perpendicular to  $\sigma_{\phi} = \sigma_x$  (note Fig. 15). By analogy with Eq. 26,

$$\sigma_y = \sigma_x + 90 \text{ deg} = K^* \left( \frac{d_{\psi_y} - d_{\perp}}{d_{\perp}} \right) \quad (27)$$

This indicates that if the specimen is turned 90 deg in the specimen holder, and  $d_{\perp}$  and  $d_{\psi}$  are measured at  $\psi = 0$  and  $\psi = \psi$  as previously described, the normal stress,  $\sigma_y$ , at right angles to  $x$  is determined.

## 2.4 STRESS EQUATION AS FUNCTION OF DIFFRACTION ANGLE

The bulk of stress determinations by x-ray diffraction techniques has been conducted on x-ray diffractometers, wherein the position of the diffracted beam is measured in terms of the angular position  $2\theta$  directly. Therefore, it is more convenient to express the stress equation (Eq. 26) in terms of the angular position  $2\theta$  rather than of the interplanar spacings.

X-ray diffraction from a crystal occurs when Bragg's law is fulfilled:

$$\eta\lambda = 2d \sin \theta \quad (28)$$

where:

- $\eta$  = Integer
- $\lambda$  = Wavelength of x-ray beam
- $d$  = Spacing between reflecting planes
- $\theta$  = Angle of incidence of the beam with these atomic planes

Differentiating Bragg's law we obtain

$$\frac{\Delta d}{d_{(hkl)}} = -\cot \theta_{(hkl)} \Delta \theta \quad (29)$$

which is equivalent to

$$\frac{\Delta d}{d} = \frac{\Delta a}{a} = -\cot \theta \left( \frac{\Delta 2\theta}{2} \right) \quad (30)$$

Combining Eqs. 30 and 26 yields

$$\sigma_{\phi} = (2\theta_{\perp} - 2\theta_{\psi}) \cdot \frac{\cot \theta}{2} \cdot \left( \frac{E}{1+\nu} \right) \cdot \frac{1}{\sin^2 \psi} \left( \frac{\pi}{180} \right) \quad (31)$$

when  $2\theta$  is expressed in degrees.

For stress measurements on a given material, the terms not involving the angle  $\theta$  are constant and Eq. 31 can be expressed as

$$\sigma_{\phi} = K' \cot \theta_{\perp} (2\theta_{\perp} - 2\theta_{\psi}) \quad (32)$$

In practice, the  $\cot \theta$  term has in general been considered

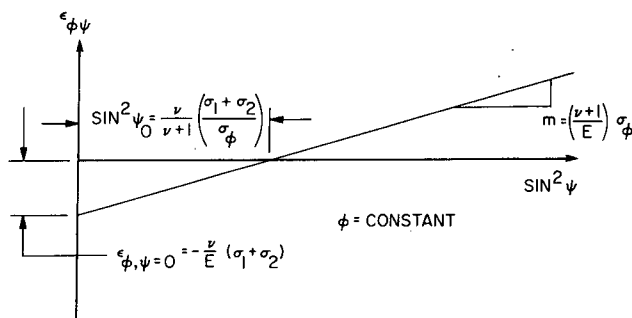


Fig. 17 - Illustration of the  $\sin^2\psi$  method for x-ray stress measurement

constant (see paragraph 6.4), and the final equation for stress in terms of  $2\theta$  becomes

$$\sigma_\phi = K(2\theta_\perp - 2\theta_\psi) = K(\Delta 2\theta) \quad (33)$$

## 2.5 STRESS EQUATION AS FUNCTION OF $\sin^2\psi$

The multiple exposure or  $\sin^2\psi$  technique can be used as an alternate method for strain determination. This method utilizes a diffractometer or a back reflection film camera to measure lattice strains by recording high-angle diffraction lines produced by a collimated x-ray beam. The technique involves the measurement of several values of lattice strains in multiple  $\psi$  directions, where  $\psi$  is chosen so that for successive values of  $\psi$ , the intervals between the corresponding values of  $\sin^2\psi$  are equal. For example, for the  $\psi$  angles of 0, 20, 29, and 36.3 deg, the corresponding values of  $\sin^2\psi$  are 0, 0.1170, 0.2340, and 0.3505 respectively, and the intervals between successive values of  $\sin^2\psi$  are all approximately 0.117. The  $\sin^2\psi$  interval should be selected to yield a usable plot of  $\sin^2\psi$  versus  $\epsilon_{\phi\psi}$ , as described in the following development.

Referring to the basic equation relating lattice strains to stress, Eq. 17 gives the relation

$$\epsilon_{\phi\psi} = \frac{1+\nu}{E} (\sigma_\phi \sin^2\psi) - \frac{\nu}{E} (\sigma_1 + \sigma_2) \quad (34)$$

Representing this equation graphically with  $\epsilon_{\phi\psi}$  as a function of  $\sin^2\psi$  for the distribution of strains in the plane containing the surface normal N, and having the angle  $\phi$  with respect to  $\sigma_1$  (Fig. 14), illustrates the following relations for a biaxial stress state.

Referring to Fig. 17, it is observed that for any angle  $\phi$  in which the lattice strain is measured, the strains  $\epsilon_{\phi\psi}$  are linearly dependent on  $\sin^2\psi$ .

The slope of the plot in Fig. 17 can be found by differentiating Eq. 34 with respect to  $\sin^2\psi$ .

$$m = \frac{\partial \epsilon_{\phi\psi}}{\partial \sin^2\psi} = \left( \frac{1+\nu}{E} \right) \sigma_\phi \quad (35)$$

From the least-mean-square line obtained from the plot of lattice strains as functions of  $\sin^2\psi$ , the calculation of the slope of the line leads directly to the stress component  $\sigma_\phi$  by the relation

$$\sigma_\phi = \frac{m E}{1 + \nu}$$

where:

$m$  = Slope

The intercept of the plot with the  $\epsilon_{\phi\psi}$  axis (at  $\psi = 0$  deg) is given by

$$\epsilon_{\phi\psi} = - \frac{\nu}{E} (\sigma_1 + \sigma_2) \quad (36)$$

By calculating the intercept of the line with the  $\epsilon_{\phi\psi}$  axis, the sum of the principal stresses is determined from the equation

$$(\sigma_1 + \sigma_2) = - \frac{E \epsilon_{\phi\psi}}{\nu}$$

Setting Eq. 34 equal to zero, noting that  $\sigma_\phi = \sigma_1 \cos^2\phi + \sigma_2 \sin^2\phi$ , and solving for  $\sin^2\psi$  yields

$$\sin^2\psi_0 = \frac{\nu}{\nu+1} \frac{(\sigma_1 + \sigma_2)}{\sigma_1 \cos^2\phi_0 + \sigma_2 \sin^2\phi_0} \quad (37)$$

From this equation, the lattice directions  $\phi_0$  and  $\psi_0$ , in which the lattice strains are equal to zero, can be found.

For a complete analysis of a surface stress state, it is necessary to determine the lattice-strain distribution in several planes with different azimuth angles. By this means, the magnitude and direction of the principal stresses can be determined\*.

At the very surface of a specimen subjected to a state of multiaxial residual stresses, the normal stress component is inherently zero. The assumption usually made is that the stress component perpendicular to the surface does not influence the x-ray stress determination because of the small depth of effective penetration of the x-rays into metallic specimens. That is indeed the case with many problems. In other cases, however, where large stress gradients exist near the surface of specimens, the third stress component has to be taken into account. Although the corrections are small, standard methods are available to evaluate this influence.

\*Reference: C. S. Barrett and T. B. Massalski, "Structure of Metals." New York: McGraw Hill, 1966, p. 475.

## SECTION 3 EQUIPMENT AND METHODS OF ANALYSIS

### 3.1 INTRODUCTION

From a reading of the previous section, it is clear that residual stress measurements by x-ray diffraction are in reality precise measurements of lattice strains between specially oriented planes  $hkl$ . With the application of appropriate elastic constants, these lattice strains, which may be expressed as  $\Delta d/d$ ,  $\Delta a/a$ ,  $(\epsilon_\psi - \epsilon_\perp)$ , or  $\Delta 2\theta$  units, are then converted to stress values. The proper elastic constants to be employed for any given metal system and instrumental test condition will be discussed in later sections.

To measure lattice strains, we make use of an x-ray beam of a suitable monochromatic wavelength  $\lambda$ , and record the  $2\theta$  shift in position of a particular high angle diffraction line  $hkl$  as a function of the specimen orientation angle  $\psi$  (Fig. 16). Satisfactory stress determination can be made with independent observations of lattice strains at just two  $\psi$  orientations, such as 0 deg, and 45 or 60 deg. However, multiexposure techniques involving observations of  $2\theta_{hkl}$ , or  $d$  at more than two  $\psi$  orientations, are often used to minimize the random errors associated with individual measurements of strain.

Diffractionmeter and/or film diffraction techniques using back reflection cameras can be used to make the necessary studies of lattice spacing  $d$  or peak position  $2\theta$  at the various  $\psi$  specimen orientations. The principal advantage of the diffractionmeter method is that it produces a purely objective means of determining the  $2\theta$  position of the diffraction peaks and can give useful results on specimens whose diffraction lines are too broad to be handled by film techniques. It is also capable of full automation in the case of problems involving repetitive measurements. Its principal disadvantages are the limitations of size of specimens which can be examined (although the portable diffractionmeter to be described below largely removes this handicap) and the considerably higher initial cost of the equipment compared to corresponding film equipment. The primary disadvantage of the film technique is that experimental errors are larger and hence the method is less accurate. A description of both methods is given below.

### 3.2 DESCRIPTION OF DIFFRACTOMETER EQUIPMENT AND METHODS

Description of the physical proportions and general operating instructions for x-ray diffractometers may be obtained in any textbook on x-ray diffraction or in specific manufacturer catalogues. Therefore, none of this information will be given here. Fig. 18 shows pictures of several common types. However, the functions of the diffractometer and its various com-

ponents which are specifically important to residual stress measurements will be introduced in this section and covered in much greater detail in following sections.

Most x-ray diffractometers are provided with a scanning goniometer which allows scanning from negative  $2\theta$  angles, which in some cases may be as large as  $-110$  deg, to positive angles up to  $165$  deg. The center of the goniometer contains a specimen platform which must be adjustable in three directions so that the desired point on the specimen surface can be made to coincide very precisely with the true axis of rotation of the diffractometer. Means must also be provided, usually a locating tool or dial gage, to permit rapid placement of variably shaped specimen surfaces on the goniometer axis in repetitive or production runs. The specimen holder must be able to rotate about the diffractometer axis independently of the detector motion so that strain ( $\Delta 2\theta$  or  $\Delta d/d$ ) can be measured at various  $\psi$  angles. In instruments in which  $\theta$  and  $2\theta$  drives can be operated independently (omega motion), this facility is already available, but in instruments where these drives are coupled, the specimen platform must have a separate rotary motion for setting  $\psi$  angles.

As will be noted in paragraph 6.3, stress determinations can be made with one of two focusing techniques:

- (1) The stationary method, where the detector remains on the diffractometer circle.
- (2) The parafocus technique, where the receiving slit and/or detector are moved along a radius toward the specimen in order to fulfill the changing beam focusing conditions (Fig. 41) when the various  $\psi$  or specimen orientation angles are used.

There are two variations of the stationary technique: the standard method and the parallel beam method. These differ only in the way that beam divergence and  $\psi$  rotation are handled. The standard stationary technique contains the following components in the beam optics to control horizontal and vertical beam divergence: an incident beam defining slit, incident beam Soller baffles with plates mounted horizontally, diffracted beam Soller baffles mounted horizontally, and a fine receiving slit to limit the specimen irradiated area horizontally from which diffracted radiation is received (Fig. 30). The stationary detector parallel beam method, which is used largely in Japan, makes use of two extra-long Soller baffle slits of low horizontal divergence to collimate or parallel the beam. The parallel beam is described in more detail in paragraph 3.6.

The parafocus technique is used to obtain high diffracted intensities and thus to reduce the time required for a measurement. A robust slide track upon which the detector

and/or receiver slit are mounted, and which will allow precise parafofocusing, can be purchased from the manufacturer or be built by the user. This slide track should be adjustable, so that precise radial alignment can be made and held indefinitely. The correct adjustment is obtained when the observed value of the diffraction angle  $2\theta$  is constant over the full range of  $\psi$  angles, allowing for specimen shape and beam divergence errors (paragraph 6.3), when a stress free specimen such as a fine metal powder is used (see Section 8).

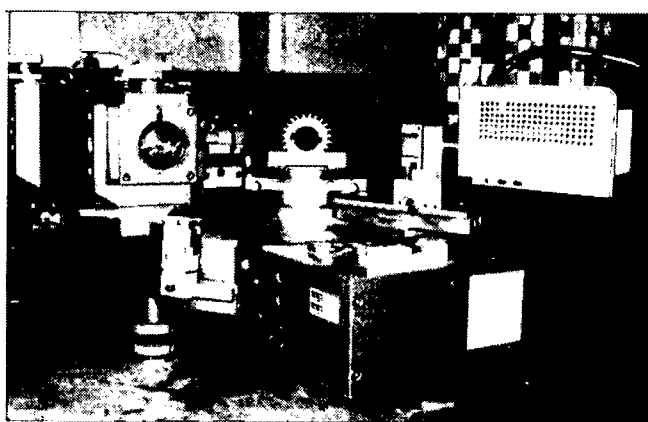
Modern diffractometers also contain electronic components which condition, accumulate, and record the intensity photons picked up by the detector system. Usually both rate meter and scaler circuits are supplied. The output of the rate meter is normally converted to a potential which is directly related to the x-ray diffracted intensity. Thus, intensity can be monitored as a function of the diffraction angles on recorder charts as the desired range of  $2\theta$  is scanned. The recorder trace resulting from this chart-scan technique can then be analyzed graphically to determine the best estimate of the desired diffraction peak.

The scaler circuit permits a measure and electronic accumulation of counts in two modes: scanning and stationary. The scanning mode integrates or totalizes the counts accumulated while scanning over a desired  $2\theta$  range. The stationary mode accumulates counts at a fixed  $2\theta$  position.

The stationary mode is used primarily for residual stress measurements and can have two variations: fixed time scaling, where intensities are measured directly for a fixed time interval, or fixed count scaling, where the times to achieve a certain number of counts are recorded. This latter technique is sometimes called the inverse intensity method.

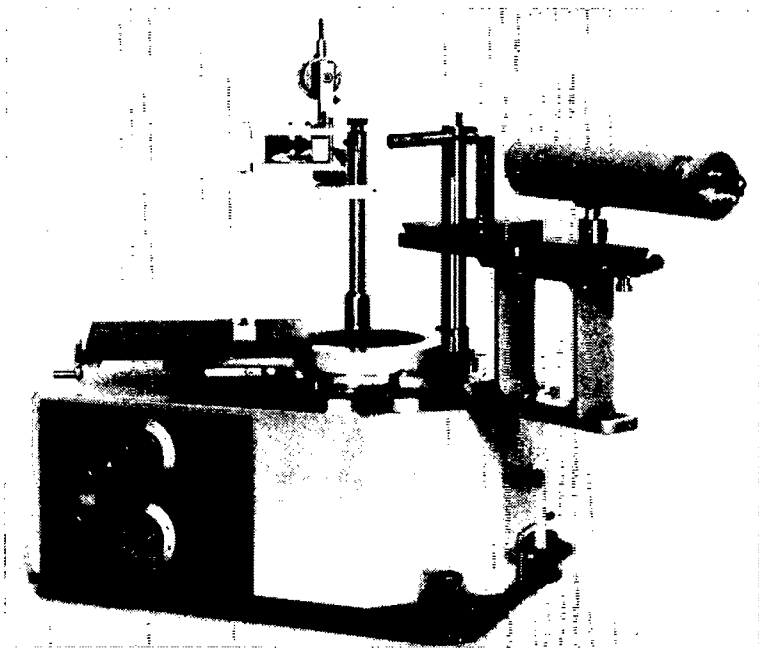
In the measurement of broad and diffuse lines, the output of the rate meter is neither sufficiently accurate nor sufficiently sensitive, and the chart-scan technique is not recommended for specimens yielding such diffraction lines. For such specimens, the scaling methods should be used and the fixed count scaling is the better technique, since it enables the choice and use of a constant probable error.

Usually, the scaling technique for determining the precise location of diffraction peaks is used with the parabola tech-



(A-left)

(B)



(C)

Fig. 18 - Common types of X-ray diffractometers: (A) General Electric Co., (B) Siemens, (C) Philips

nique described in detail in paragraph 7.1. Briefly, the technique involves the precise measurements of diffracted intensity (by scaling methods) at several known angular positions in the vicinity of the peak. From these data, a parabola is calculated which fits the points, and the vertex of this parabola is taken as the peak position. A minimum of three points is necessary but more points may be taken to improve the precision (see paragraph 7.1).

Although many additional factors which influence the diffracted beam and the accuracy of residual stress measurements will be described in detail in following sections, the selection of radiation, slit systems, and optimum detector systems needs to be discussed briefly, since these are equipment items.

Although the peak position is measured in units of  $2\theta$ , the calculation of interplanar spacing  $d$ , through Bragg's Law  $\lambda = 2d\sin\theta$ , involves  $\sin\theta$  rather than  $\theta$ . Thus, with the use of high Bragg angles  $\theta$ , one can obtain a fairly precise value of  $\sin\theta$ , even though the measurement of  $\theta$  is not precise. This can be seen from an inspection of Fig. 19. Thus, one would prefer using the highest practical value of  $\theta$ . From diffractometer design considerations, the maximum practical value of  $\theta$  obtainable is about 83 deg. Stress determinations are usually carried out as close to 165 deg as possible and seldom below 140 deg ( $2\theta$ ). The value of  $\lambda$  is fixed by the available target materials for commercial x-ray tubes. Also, for any given substance, the  $d$  spacing of each family of planes  $hkl$  has a specific value depending on crystal structure. It is fortunate

that workable combinations of wavelength and  $hkl$  planes can be found for most metals and alloys (Section 4).

Two types of detectors are in present use for measurement of diffracted intensity: gas-filled (Geiger and proportional counters) and solid-state detectors such as the scintillation and Si (Li). The proper selection for a given radiation and high  $2\theta$  diffraction angle  $hkl$  depends on the optimum interaction of peak-to-background response and detector efficiency. For example, the proper selection for measurement of stress in steel with Cr radiation is the Xenon filled proportional counter which displays a lower detection efficiency for Cr radiation than the scintillation detector but yields a superior peak in terms of peak/background ratio.

The selection of the optimum x-ray apertures or slit combinations depends chiefly on three factors: specimen size, divergence of incident beam aperture slit, and resolution of  $K_{\alpha 1}$  and  $K_{\alpha 2}$  doublet of the diffracted  $hkl$  beam. The aim is always to produce the maximum diffracted intensity with resolution of peak doublets (if such exist) to reduce the time required for stress measurement. The methods for proper selection will be described in detail in paragraph 6.2. However, typical selections for steel specimens with Cr radiation which obey the above three requirements are listed in Table 1, along with the expected count rates.

Smaller and vertically restricted slit combinations must be used when the above slit combination proves too large in area. Obviously, the counting rate will be greatly reduced, and total time for completion of a stress measurement will thus be greatly lengthened, if the slits are too small.

Certain corrections for  $\theta$  and  $\psi$  dependent factors normally have to be made to the raw diffractometer data before accurate peak determinations can be made. Paragraph 6.3 will consider these in detail.

### 3.3 DIFFRACTOMETER RESIDUAL STRESS TECHNIQUES

**3.3.1 TWO EXPOSURE TECHNIQUE**—The most widely used diffractometer residual stress technique is the two exposure method, where the desired stress parallel to the surface (and goniometer scanning plane) is determined from peak  $hkl$  observations on the selected area at two different  $\psi$  orientations of the specimen surface. Usually one of the  $\psi$  angles is 0 deg—the surface is normal to the beam bisector (Figs. 16 and 22)—and the second is larger—for instance,  $\psi = 45$  or 60 deg; however, any two  $\psi$  angles may be used. One of the stress equations developed in Section 2 can then be used to calculate the surface stress, depending on whether the strain is recorded as  $\Delta 2\theta$  or  $\Delta d/d_1$ :

$$\sigma_{\phi} = K(2\theta_{\perp} - 2\theta_{\psi}) = K(\Delta 2\theta)$$

$$\sigma_{\phi} = K^* \frac{d_{\psi} - d_{\perp}}{d_{\perp}} = K^* \frac{\Delta d}{d_{\perp}}$$

$$\sigma_{\phi} = \frac{E}{1 + \nu} \left( \frac{d_{\psi 2} - d_{\psi 1}}{d_{\psi 1}} \right) \left[ \frac{1}{\sin^2 \psi_2 - \sin^2 \psi_1} \right]$$

Table 1—Typical Specimens with Cr Radiation

| Specimen, Line | Focusing Method | Div/Rec, deg | Typical Count Rate, $\psi = 0$ , cps |
|----------------|-----------------|--------------|--------------------------------------|
| Broad          | Parafocus       | 3/0.2        | 1000/6000                            |
| Fine           | Parafocus       | 1/0.2        | 1000/6000                            |
| Broad          | Stationary      | 1/0.2        | 500/2000                             |

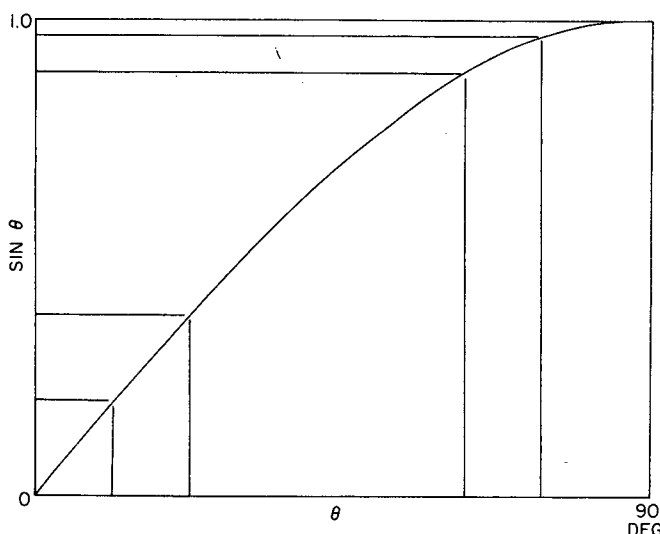


Fig. 19 - Effect of diffraction angle on precision of  $\sin\theta$  determination

The last equation corresponds to the case in which both  $\psi$  angles differ from zero. The two exposure technique may use either parafocus or stationary detector positioning.

**3.3.2 MULTI-EXPOSURE OR  $\sin^2 \psi$  TECHNIQUE**—A more precise determination of  $\sigma_\phi$  can be obtained by making runs at more than two  $\psi$  orientations and plotting the measured strain values for each  $\psi$  orientation as a function of  $\sin^2 \psi$ . The strain may be plotted as  $\epsilon_{\phi\psi}$ ,  $\Delta d/d$ ,  $\Delta a/a$ , or  $\Delta 2\theta$ . An illustration of this procedure is given in Fig. 20. This figure grows out of Eq. 17 of Section 2, which is repeated below

$$\epsilon_{\phi, \psi} = \frac{1+\nu}{E} (\sigma_\phi) \sin^2 \psi - \frac{\nu}{E} (\sigma_1 + \sigma_2)$$

Several important observations can be made with regard to Fig. 20:

- (1) The intercept at  $\psi = 0$  is equivalent to  $\epsilon_{\phi 0} = \epsilon_\perp = -\frac{\nu}{E} (\sigma_1 + \sigma_2)$ .
- (2) The strain values vary linearly with  $\sin^2 \psi$ .
- (3) The slope of the line is equivalent to  $((1+\nu)/E) \sigma_\phi$ , and thus  $\sigma_\phi$  can be determined if the proper elastic constants are used.
- (4) A zero strain will be indicated for a  $\psi$  angle where  $\sin^2 \psi = \left( \frac{\nu}{1+\nu} \right) \left( \frac{\sigma_1 + \sigma_2}{\sigma_\phi} \right)$ .
- (5) The strain values being measured must be reported in terms  $\Delta d/d_0$ ; that is, referred to the unstressed state where  $\Delta d = d_\psi - d_0$ .

Item 5 limits the utility of this particular  $\epsilon$  versus  $\sin^2 \psi$  plot because, as noted before,  $d_0$  often is not known or easily determined, particularly in hardened specimens where very broad diffraction lines result and carbon solution or precipitation influences the lattice spacings.

A similar plot of strain versus  $\sin^2 \psi$  which does not require knowing or determining  $d_0$  is given in Fig. 20. Here the quantity  $(\epsilon_\psi - \epsilon_\perp) = (d_\psi - d_\perp)/d_\perp$  rather than  $\epsilon_\psi$  is plotted against  $\sin^2 \psi$ . Actually,  $\epsilon_\psi - \epsilon_\perp = (d_\psi - d_\perp)/d_0$ , but  $d_0$  in the denominator can be replaced by  $d_\perp$  with very little error. The important idea to note is that  $d_0$  has been eliminated from the numerator. The slope again determines  $\sigma_\phi$ , but in this case the line must go through the origin.

The equations used to determine stress by this method are the following:

$$\sigma_\phi = m \left( \frac{1}{2} s_2 \right)^{-1} \quad (1)$$

where:

$$\left( \frac{1}{2} s_2 \right)^{-1} = \left( \frac{E}{1+\nu} \right)$$

$$m = \text{Slope} = \left( \frac{\delta \epsilon}{\delta \sin^2 \psi} \right)$$

A typical stress determination for steel using the  $\sin^2 \psi$  procedure would be as follows:

(1) Determine the  $2\theta$  position for a given  $hkl$  line using several  $\psi$  specimen orientations, such as  $\psi = 0, 15, 30, 45$ , and  $60$  deg.

(2) Convert these  $2\theta$  values to  $d$  values.

(3) Compute strains as  $\Delta d/d_\perp = (d_\psi - d_\perp)/d_\perp$ .

(4) Plot versus  $\sin^2 \psi$ .

(5) Determine slope of best fit to line. Note that the  $\Delta d/d_\perp$  value at  $\sin^2 \psi = 1$  (extrapolated to  $\psi = 90$  deg) yields the slope directly without further computation.

(6) Using Eq. 35 of Section 2, determine  $\sigma_\phi$  as  $\sigma_\phi = (E/(1+\nu)) \text{slope} = 23,256 (\text{slope})$  in ksi for steel with Cr radiation.

### 3.4 FILM TECHNIQUES

In the case of film techniques\*, the principles of the method are the same as for the diffractometer, but the diffraction angles from the specimen are measured by recording the diffraction maxima on photosensitive film mounted in either a flat or a cylindrical back reflection camera. The usual practice is to attach the camera directly to the head of the x-ray tube (Fig. 24). Two modifications are in general use. The first, known as the two exposure technique, is the exact counterpart of the diffractometer procedure in that two separate exposures are made at the two chosen  $\psi$  angles. This is shown schematically in Fig. 21 for a cylindrical camera.

\*Information supplied by J. F. Norton, Advanced Metals Research Corp., Burlington, Mass.

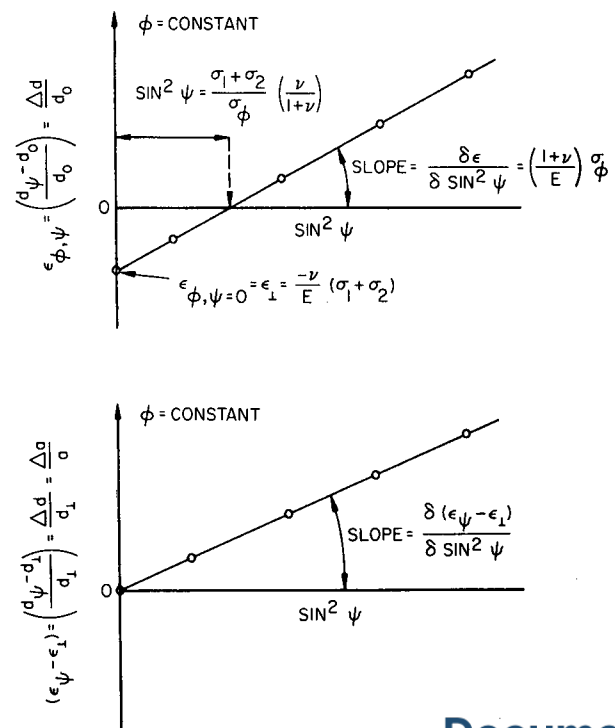


Fig. 20 -  $\sin^2 \psi$  technique for stress determination

The measured quantity in this case is a distance  $S$ , measured along the film from the axis of the incident beam to the center of the diffraction maximum. If it is assumed that the specimen surface lies on the axis of the cylindrical camera whose radius is  $R_0$  then

$$\frac{S_1}{R_0} = \pi - 2\theta_1 = 2\eta_1 \text{ and } \frac{S_2}{R_0} = \pi - 2\theta_2 = 2\eta_2 \quad (2)$$

where  $\theta$  is in radians.

Thus, it can be seen that

$$(2\theta_1 - 2\theta_2) = \frac{S_2 - S_1}{R_0} \quad (3)$$

Substituting Eq. 3 into Eq. 26 of Section 2, and noting that

$$\frac{d_{\psi_2} - d_{\psi_1}}{d_{\psi_1}} = \frac{\cot \theta_0 (S_2 - S_1)}{2 R_0} \quad (4)$$

one obtains the working equation for the calculation of stress

where  $\psi_1 = 0$

$$\sigma_\phi = \left( \frac{E}{1 + \nu} \right) \frac{\cot \theta_0}{2 R_0 \sin^2 \psi} (S_2 - S_1) \quad (5)$$

where  $R_0$  and  $S$  must be measured in the same units.

This may be written as:

$$\sigma_\phi = K_F (S_2 - S_1) \quad (6)$$

where  $K_F$  is the stress constant for this particular technique.

In actual practice, the distance  $S$  is not measured from the axis of the incident x-ray beam but rather from a fiducial mark of some sort that is recorded on each exposed film at a fixed distance from the beam axis (Fig. 22). For the usual operation, the actual position of the fiducial mark need not be known as long as it is constant, because it drops out in the difference  $(S_2 - S_1)$ . However, if the actual diffraction angle or the corresponding  $d$  value is to be calculated from the film measurement, then the true position of the fiducial mark must be determined by exposing a stress free (powder) specimen of known lattice constant. It is desirable to record fiducial marks near both ends of the useful angular range on the film, in order to permit a correction of the measured  $S$  values for film shrinkage during processing.

The second modification is called the single exposure technique and is shown schematically in Fig. 23. This is not to be confused with the one-exposure method referred to in Sec-

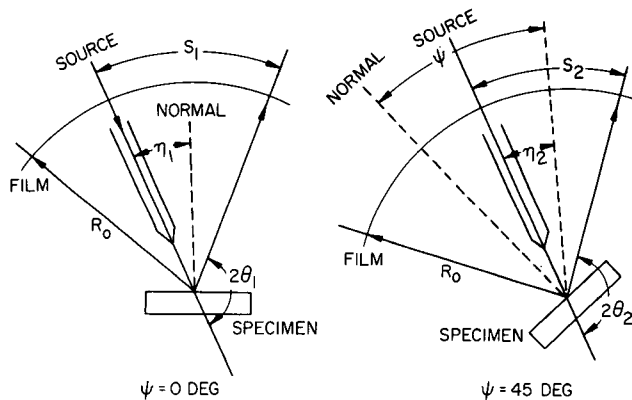


Fig. 21 - Geometry for cylindrical film camera

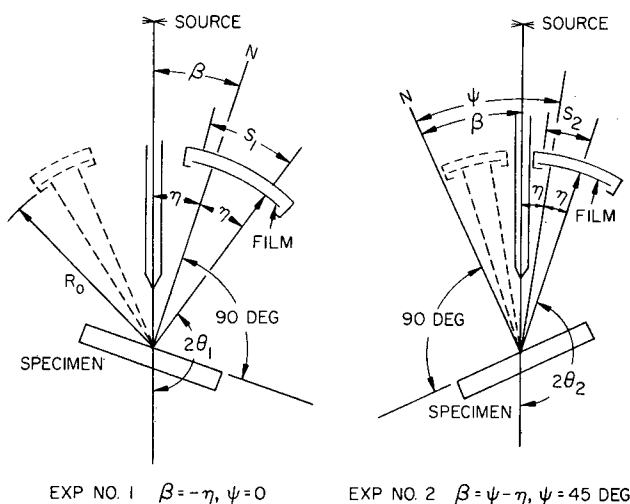


Fig. 22 - Schematic of two exposure film technique

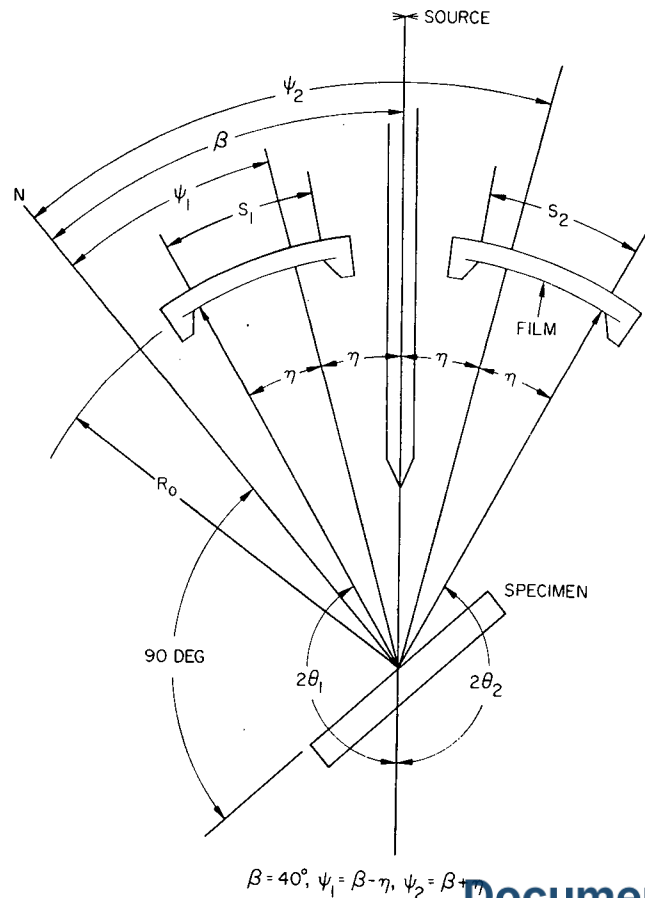


Fig. 23 - Schematic diagram of single exposure film technique

tion 7.5. In this case, the incident beam is inclined at a selected angle  $\beta$  to the surface normal, and the diffracted beams are recorded simultaneously on both sides of the incident beam either on two separate films or on the two ends of a single film. Under these conditions, one side corresponds to a  $\psi$  angle of  $\beta + \eta$  and the other to  $\beta - \eta$ .

The equation for the stress is then:

$$\sigma_\phi = \left( \frac{E}{1 + \nu} \right) \frac{\cot \theta_0}{2R_0 [\sin^2 (\beta + \eta) - \sin^2 (\beta - \eta)]} (S_2 - S_1) \quad (7)$$

This can be simplified to:

$$\sigma_\phi = \left( \frac{E}{1 + \nu} \right) \frac{1}{4R_0 \sin^2 \theta \sin 2\beta} (S_2 - S_1) \quad (8)$$

$$= K'_F (S_2 - S_1) \quad (9)$$

and if  $\beta$  is chosen as 45 deg, the  $\sin 2\beta$  term drops out.

In the single exposure technique, employing both sides of the film holder, it is necessary to establish the precise relative positions of the fiducial marks by a standardization procedure with a stress-free specimen of known lattice constants.

Eqs. 5 and 8 have been developed for a cylindrical diffraction camera of radius  $R_0$  with the specimen at the center. If a flat film camera is employed with the film perpendicular to the incident x-ray beam and at a distance  $R_0$  from the specimen, the quantity  $\sec^2 2\theta$  must be inserted in the denominator of the second term in Eqs. 5 and 8.

A word should be said about the relative merits of the single exposure and the two exposure techniques when using the film camera. At diffraction angles above about 75 deg, the two exposure technique has the smaller stress constant and in principle should be more sensitive. However, it is difficult to achieve this advantage in actual practice because the conditions for alignment of the specimen with respect to the center of the camera are considerably more critical than is the case with the single exposure technique. Experience has shown that under normal experimental conditions the reproducibility of the two methods is about the same. The shorter exposure time and the relaxed alignment requirements make the single exposure technique the favored choice in many practical applications.

The x-ray camera (Fig. 24) must be provided with means for adjusting the area of interest on the surface of the specimen precisely to the center of the cylindrical camera or to the fixed distance of the flat camera. One method is to use a mechanical pointer attached to the collimator tube, the tip of which is exactly on the axis of the x-ray beam and at the proper distance from the film. Contact is established either by means of a thin feeler gage inserted between the pointer tip and the specimen surface or by some sort of electrical contact indicating device. Precise alignment requires a delicate adjustment screw mechanism either on the x-ray tube mounting or the specimen support.

Another method makes use of a reference powder of known

lattice constants placed on the specimen surface. A substance is selected that produces a sharp diffraction line on the film adjacent to the line from the specimen and, since this line corresponds to a known and constant diffraction angle, each film is self-calibrating and corrections can be made for variations in the specimen to film distance. The use of a reference powder practically doubles the exposure time and its proper application is a rather delicate operation. From the point of view of precision, the reference powder and a well-designed mechanical pointer give essentially equal results.

Several methods have been used to measure the diffraction line positions on the films. The most common and probably the most practical method employs a standard film measuring box such as those used for conventional x-ray diffraction films. Readings are made with a scale and vernier, repeated observation being made and averaged. For sharp lines and for the sharp fiducial marks, a viewing magnification of perhaps 2x is desirable but, as the lines become broad, magnification is detrimental. There is a certain subjective character to the line reading process, but it is surprising how consistent an experienced observer can be, even when the diffraction lines are broad. Reproducible results require practice and young eyes. It must be remembered, however, that the film techniques are difficult to apply to hard steels, due to the problems associated with determining the peak position of a broadened line. For hard steels, diffractometers, which produce a profile of the diffraction line, are definitely superior.

Numerous attempts have been made to measure the diffraction line positions by means of microphotometer traces of the films but, in general, the results have been disappointing. The traces are comparable to the continuously recorded chart records obtained with the diffractometer but measurements made on these traces are inferior to those obtained by precise point counting techniques, for which the diffractometer is well suited.

The film cameras normally used are not designed to fulfill the focusing conditions discussed in connection with the dif-

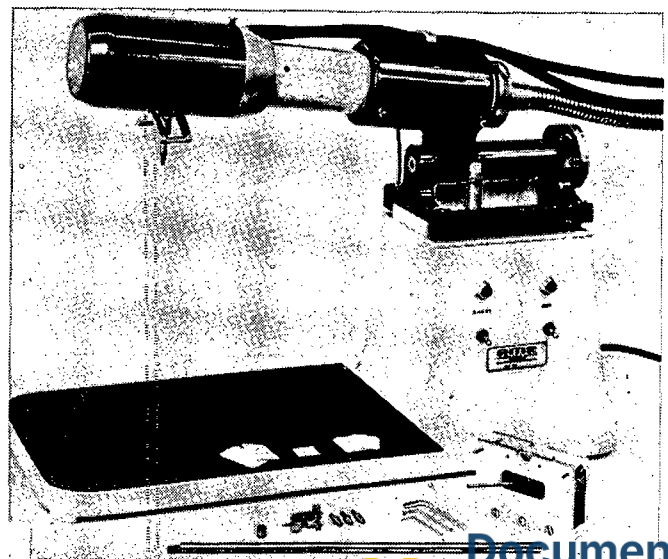


Fig. 24 - Typical film unit for stress measurement

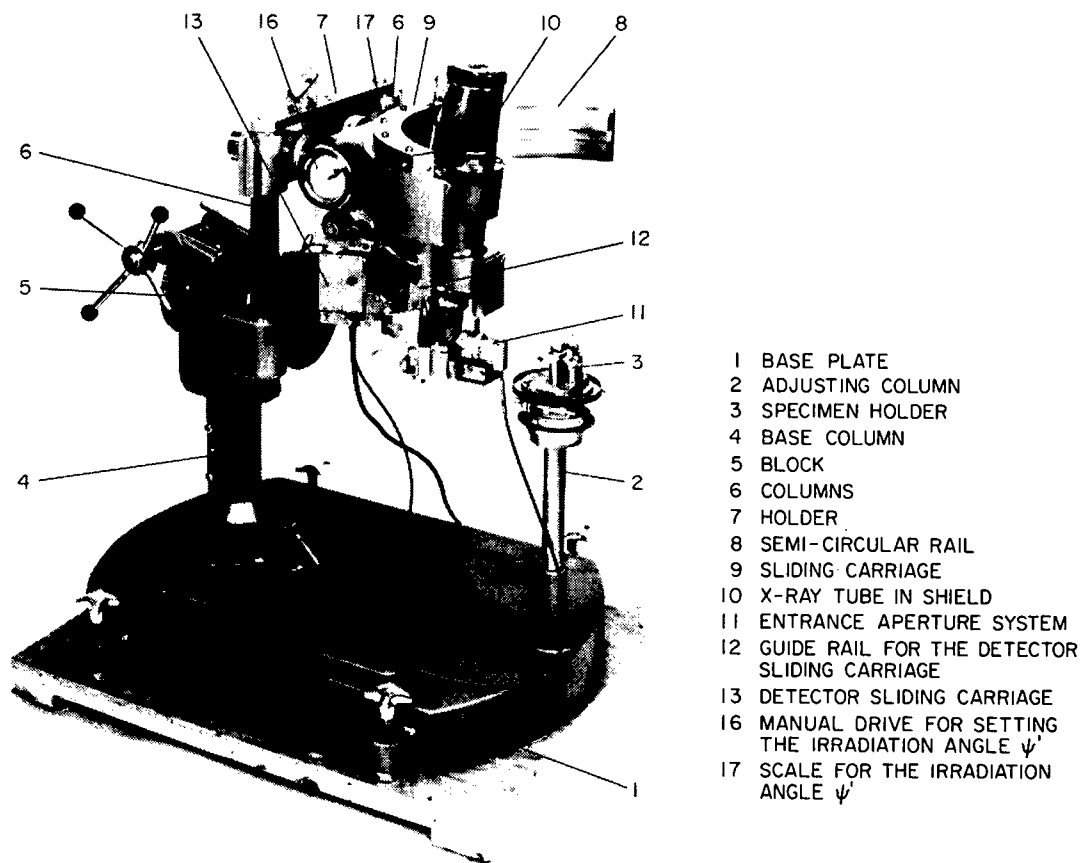


Fig. 25 - Goniometer for stress measurements

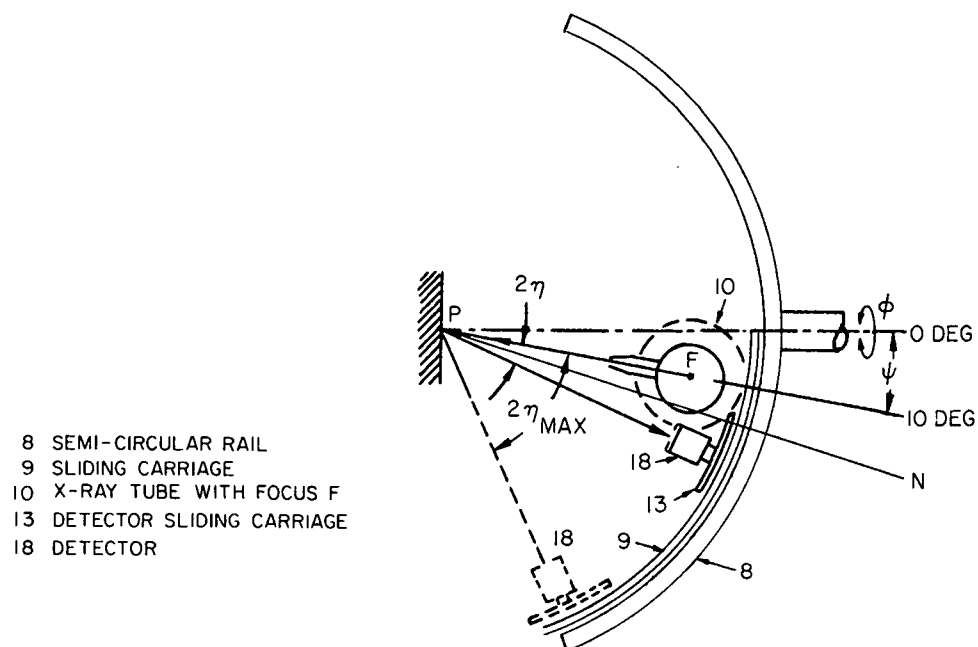


Fig. 26 - Schematic diagram of the portable diffractometer in place for stress measurement



Table 2—Measuring Apparatus, Method, and Conditions of X-ray Diffraction Technique

| Measuring Apparatus           | Parallel Beam Type X-ray Stress Analyzer |
|-------------------------------|------------------------------------------|
| Characteristic x-ray          | Co- $K_{\alpha}$ , Cr- $K_{\alpha}$      |
| Filter                        | Fe, V                                    |
| Diffraction plane             | (310), (211)                             |
| Slit (restricted)             | 5 × 10 mm                                |
| Tube voltage                  | 30 kV                                    |
| Tube current                  | 5-10 mA                                  |
| High voltage (counter tube)   | 1,150 V                                  |
| Time constant                 | 16 sec                                   |
| Goniometer scan speed         | 1 deg/min                                |
| Chart full scale              | 250 cps                                  |
| Chart speed                   | 20 mm/min                                |
| (Specimen—Plain Carbon Steel) |                                          |

fractometer. The combination of an x-ray tube with a fine focal spot and a collimator with a small aperture results in an incident beam of such small angular divergence that focusing is not a problem, and the lines of the normal and oblique exposure are of essentially equal sharpness.

### 3.5 PORTABLE DIFFRACTOMETER TECHNIQUE

A hybrid product of the diffractometer and the portable film unit is the portable diffractometer\*. This unit is designed to be moved to the part and placed against it. The portable diffractometer measures  $hkl$  peak positions in the back reflection region (113 to 163-2 $\theta$ ). Alignment of the unit is checked both optically and with reference material. The detector used is a proportional counter with a preamplifier. Fig. 25 shows the goniometer and Fig. 26 is a diagram of the unit in place for stress measurement.

Stress can be determined by either the two or multi-exposure  $\sin^2 \psi$  method. In order to cover various diameters of the stress ellipsoid, the goniometer can be rotated around the  $\phi$  axis. The irradiation orientation angle  $\psi'$  is read from the scale (17 in Fig. 25) in tenths of a degree from the manual drive itself.

The detector sliding carriage (13 in Fig. 25) is passed through the diffractometer angle range either manually or mechanically by means of a synchronous motor. The diffraction angle  $2\theta$  is read with an accuracy of 0.01 deg.

The measuring direction is along the bisecting line of the angle  $2\eta$  formed by incident and diffracted beams. The normal to the surface, the incident, and the diffracted beams all lie in the same plane. The trace of this plane on the specimen surface is the direction of the stress component  $\phi$ , which is determined during the measurement. Because the radiation source, rather than the specimen, is rotated, and because the detector is between the surface normal and the x-ray source

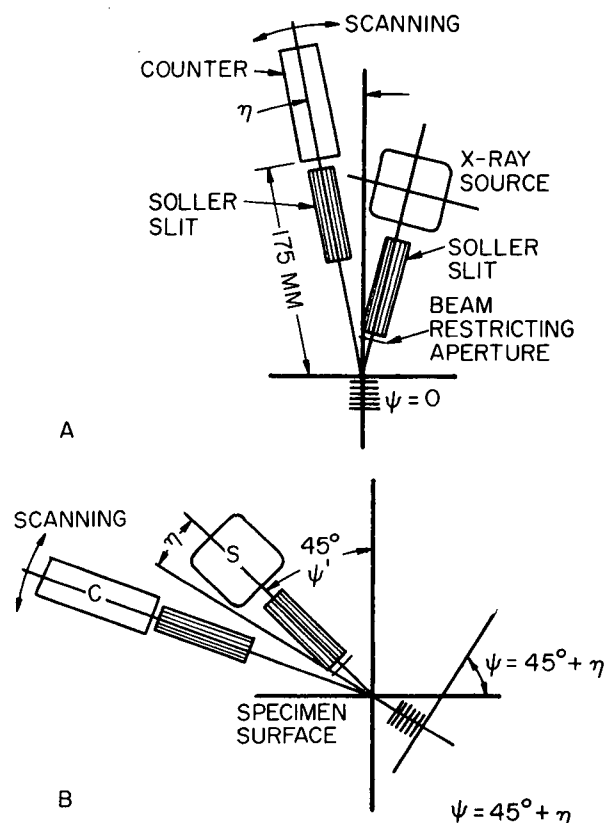


Fig. 27 - Parallel beam type diffractometer for stress measurement

at the higher  $\psi$  angles, the specimen orientation angle  $\psi$  is negative with this technique.

This unit is too new to give data on the precision of peak shift measurements. A logical assumption would be that the accuracy falls somewhere between that of the regular diffractometer and the film camera. The advantage gained from this type of unit is that it enables one to apply diffractometer principles and known correction factors to large parts.

### 3.6 PARALLEL BEAM DIFFRACTOMETER TECHNIQUE

A modified diffractometer technique which appears to be a cross product of the portable diffractometer and the two-exposure film geometry is used in Japan to measure stress\*. This stationary detector technique is called the parallel beam method because long Soller slits (104 mm) with closely spaced vertical baffles are used to limit horizontal divergence or to parallel the incident and diffracted beams. Typical horizontal divergence angles for the Soller slits are between 0.17-0.35 deg. A separate beam-restricting aperture of rectangular shape is employed behind the incident beam Soller slit to limit both the irradiated area on the specimen and vertical divergence as well. Fig. 27 shows the geometry of the parallel beam diffractometer. Note that, as with the portable method, the specimen is always fixed (not rotated). Instead, the x-ray

\*Siemens America, Inc., 1180 Avenue of the Americas, New York, New York 10036.

\*Information supplied by S. Taira, Faculty of Engineering, Kyoto University, Kyoto, Japan.

source, and in fact the whole diffractometer, is rotated relative to the specimen surface to make lattice strain observations at the various required  $\psi$  specimen orientations. With this diffractometer geometry, the  $\psi$  angle for plotting  $\sin^2 \psi$  diagrams or for direct calculation of stress with the two exposure technique is equal to the sum of  $\psi'$ , the incident radiation angle, and  $\eta$ , the complementary diffraction angle.

Typical instrumental conditions for this technique are given in Table 2. Obviously, the greatest disadvantages of the parallel beam method—compared to the focusing methods—are lower beam intensities and resolutions; hence, comparative times per stress determination would be longer. Also, because there is

no relative  $\theta$  rotation of the specimen, a systematic error in  $2\theta$  peak location must be recognized, especially with broad line specimens. This is also true in the portable diffractometer technique. Because of the variation in  $\psi$  angle, a different absorption factor must be used.

These shortcomings are, however, far outweighed by the fact that specimen positioning is very simple, since there is no necessity for paying attention to focusing conditions, even in the case of oblique  $\psi$  beam conditions. Also, sample positioning errors, which can be very large with the stationary and parafocus techniques, appear to be very small (Fig. 43).

## SECTION 4 SELECTION OF RADIATION, REFLECTIONS, AND FILTERS

### 4.1 RADIATION AND PEAK COMBINATIONS

The main consideration in selecting a radiation and a diffraction peak for a given material is to obtain diffraction at a high  $2\theta$  angle with high intensity and peak-to-background (P/B) ratio. A high  $2\theta$  angle is desired because the stress is proportional to  $[\cot \theta (\theta_{\psi=0} - \theta_{\psi=\psi})]$ . A low value of  $\cot \theta$  and, therefore, a large measured  $(\theta_{\psi=0} - \theta_{\psi=\psi})$  occur at high  $2\theta$  angles for a given stress, corresponding to the greatest precision. The  $2\theta$  angle should be greater than 130 deg, although the 220 peak with Cr  $K_{\alpha}$  radiation at 128 deg is used for  $\gamma$ -Fe. Diffraction angles with various radiations are given for  $\alpha$ -Fe in Table 3, and for several other materials of interest in Table 4. Diffraction peaks in the angular range of interest are indicated. Some peaks normally at too high an angle may be accessible if the sample is an alloy.

Relative intensities of peaks in the desired range with a given radiation can be estimated from the data for the material in the ASTM Powder Diffraction File, from a quick diffractometer trace, or from calculations. Calculated intensities for  $\alpha$ -Fe with the diffractometer arrangement are given in Table 3. These are peak maximum (rather than peak area) intensities using the appropriate Lorentz polarization factor of  $(1 + \cos^2 2\theta)/\sin^2 \theta$  (for a point, not for integrated intensity). This should be a better measure than integrated intensity of the ability to determine peak position. Calculations were based on the  $K_{\alpha_1}$  peak only; however, the change in resolution of the  $K_{\alpha}$  doublet with diffraction angle will affect maximum intensity. It is seen that the multiplicity factor over the range of angles of interest is a good indication of intensity. Relative intensities using different radiations can probably be best determined experimentally.

The P/B ratio depends on how nearly monochromatic the radiation is. This is affected by filters and instrumentation. The technique is determined by the material being analyzed, as discussed in paragraph 4.2 on filtering.

There are also other considerations. Doublet separation may be a problem with a partially hardened or cold worked material. The peak may not be sharp enough to use only the  $K_{\alpha_1}$  component, and may not be broad enough to use the  $K_{\alpha}$  doublet. Possible solutions to this problem which involve the choice of radiation are to use a  $K_{\beta}$  peak, which is monochromatic, or to use Mo  $K_{\alpha}$  radiation, which has a large doublet separation (1).<sup>\*</sup> The 732,651 peak of Fe is probably the best choice using Mo radiation.

It may be desirable to use a more or less penetrating radiation. More penetration would be desirable if grains are large because there is a chance for more material to diffract. The condition of a very thin surface layer will not have as much effect if significant diffraction also occurs from below it. Less penetrating radiation is desired if there is a steep stress gradient, such as that produced by grinding or shot peening. The correction to the measured stress depends on the depth of penetration in relation to the stress gradient.

For a given material, the use of a particular crystallographic direction may be desirable. If there is preferred orientation, an  $hkl$  may have to be selected which gives adequate intensity from planes both normal to and at an angle  $\psi$  to the surface. If measured mechanical values of the polycrystalline elastic constants are used in the stress equations rather than experimentally determined or calculated x-ray values, a particular  $hkl$  may be most appropriate. When the equations in paragraph 6.4 on the stress factor are applied to the calculation of the elastic modulus appropriate for mechanical measurement of polycrystalline material, a value of 1/5 is used for  $\tau = (h^2 k^2 + k^2 \ell^2 + \ell^2 h^2)/(h^2 + k^2 + \ell^2)^2$ . Therefore, assuming fair agreement between calculated and measured values, an  $hkl$  corresponding to a value of  $\tau$  near 1/5 should result in most accuracy for anisotropic materials.

### 4.2 FILTER CRITERIA

Sources of undesired radiation are the  $\beta$  component, the white (continuous) radiation which peaks at about 0.5 Å, and fluorescent x-rays generated in the sample. Decreasing the x-ray tube voltage will lower the intensity of white radiation to some extent in the short wavelength (high energy) region, but the characteristic intensity will also be decreased. Filter materials and the method of calculating required thicknesses used to suppress the  $K_{\beta}$  peak of various radiations are tabulated in x-ray texts (1). A  $\beta$  filter will also decrease the diffracted white radiation at the short wavelength side near the  $K_{\alpha}$  peak. The best position for the filter is determined by the sample material and radiation (2). In the case of Cr radiation and an Fe sample, the Fe fluorescent radiation caused by the continuum will also be decreased by a V filter in the diffracted beam. Co radiation is the best choice for specimens of copper and nickel for the same reason. If Co radiation is used with an Fe sample, the Fe filter should be placed in the incident beam to remove the  $K_{\beta}$  radiation, which will cause Fe to fluoresce. The long wavelength fluorescent radiation from an Al sample is not a problem because it is highly absorbed in the air path between sample and detector. When a weak peak is being analyzed, the  $\beta$  filter, which also decreases the  $K_{\alpha}$  intensity, may be eliminated if no  $\beta$  peak interferes with the peak used.

<sup>\*</sup>Numbers in parentheses designate References at end of Section.

Table 3—Diffraction Angles and Intensities for  $\alpha$ -Fe

| $h^2 + k^2 + l^2$ | hkl           | d, Å<br>$a_0 = 2.8664 \text{ Å}$ | Multiplicity | 2 $\theta$ , deg           |                          |                          |                          | Relative Maximum Intensity, % |       |       |       |
|-------------------|---------------|----------------------------------|--------------|----------------------------|--------------------------|--------------------------|--------------------------|-------------------------------|-------|-------|-------|
|                   |               |                                  |              | CrK $\alpha_1$<br>2.2896 Å | CoK $\alpha_1$<br>1.7889 | CuK $\alpha_1$<br>1.5405 | MoK $\alpha_1$<br>0.7093 | Cr                            | Co    | Cu    | Mo    |
| 2                 | 110           | 2.0269                           | 12           | 68.78                      | 52.37                    | 44.67                    | 20.15                    | 100.0                         | 100.0 | 100.0 | 100.0 |
| 4                 | 200           | 1.4332                           | 6            | 106.03                     | 77.23                    | 65.02                    | 28.65                    | 14.6                          | 10.8  | 12.4  | 15.2  |
| 6                 | 211           | 1.1702                           | 24           | 156.09                     | 99.70                    | 82.33                    | 35.28                    | 45.6                          | 18.1  | 20.4  | 27.4  |
| 8                 | 220           | 1.0134                           | 12           |                            | 123.92                   | 98.94                    | 40.97                    |                               | 6.0   | 5.9   | 7.5   |
| 10                | 310           | 0.9064                           | 24           |                            | 161.35                   | 116.37                   | 46.06                    |                               | 9.9   | 8.7   | 9.0   |
| 12                | 222           | 0.8275                           | 8            |                            |                          | 137.14                   | 50.75                    |                               |       | 2.6   | 2.0   |
| 54                | 721, 633, 552 | 0.3901                           | 96           |                            |                          |                          | 130.78                   |                               |       |       | 0.78  |
| 56                | 642           | 0.3830                           | 48           |                            |                          |                          | 135.59                   |                               |       |       | 0.37  |
| 58                | 730           | 0.3764                           | 24           |                            |                          |                          | 140.86                   |                               |       |       | 0.18  |
| 62                | 732, 651      | 0.3640                           | 96           |                            |                          |                          | 153.90                   |                               |       |       | 0.65  |
| 64                | 800           | 0.3583                           | 6            |                            |                          |                          | 163.59                   |                               |       |       | 0.04  |

Table 4—Diffraction Angles for Several FCC Materials

| $\gamma$ -Fe      |          |              |                        |                   |                   |                   | Al                       |                   |                   |                   |
|-------------------|----------|--------------|------------------------|-------------------|-------------------|-------------------|--------------------------|-------------------|-------------------|-------------------|
| $h^2 + k^2 + l^2$ | hkl      | Multiplicity | d, Å                   | 2 $\theta$ , deg  |                   |                   | d, Å                     | 2 $\theta$ , deg  |                   |                   |
|                   |          |              | $a_0 = 3.60\text{\AA}$ | CrK $_{\alpha 1}$ | CoK $_{\alpha 1}$ | CuK $_{\alpha 1}$ | $a_0 = 4.0494\text{\AA}$ | CrK $_{\alpha 1}$ | CoK $_{\alpha 1}$ | CuK $_{\alpha 1}$ |
| 3                 | 111      | 8            | 2.08                   | 66.84             | 50.98             | 43.50             | 2.3379                   | 58.64             | 44.99             | 38.47             |
| 4                 | 200      | 6            | 1.80                   | 78.99             | 59.59             | 50.67             | 2.0247                   | 68.86             | 52.43             | 44.72             |
| 8                 | 220      | 12           | 1.273                  | 128.17            | 89.30             | 74.48             | 1.4317                   | 106.19            | 77.33             | 65.10             |
| 11                | 311      | 24           | 1.085                  |                   | 110.98            | 90.41             | 1.2209                   | 139.32            | 94.21             | 78.23             |
| 12                | 222      | 8            | 1.039                  |                   | 118.79            | 95.67             | 1.1690                   | 156.67            | 99.84             | 82.44             |
| 16                | 400      | 6            | 0.900                  |                   | (167.28)          | 117.71            | 1.0124                   |                   | 124.15            | 99.08             |
| 19                | 331      | 24           | 0.826                  |                   |                   | 137.69            | 0.9290                   |                   | 148.66            | 112.02            |
| 20                | 420      | 24           | 0.805                  |                   |                   | 146.22            | 0.9055                   |                   | 162.11            | 116.57            |
| 24                | 422      | 24           |                        |                   |                   |                   | 0.8266                   |                   |                   | 137.45            |
| 27                | 511, 333 | 32           |                        |                   |                   |                   | 0.7793                   |                   |                   | 162.51            |

| $h^2 + k^2 + l^2$ | hkl | Multiplicity | Cu                     |                |                |                | Ni                     |                |                |                |
|-------------------|-----|--------------|------------------------|----------------|----------------|----------------|------------------------|----------------|----------------|----------------|
|                   |     |              | d, Å                   |                | 2θ, deg        |                | d, Å                   |                | 2θ, deg        |                |
|                   |     |              | $a_0 = 3.6150\text{Å}$ | CrK $\alpha_1$ | CoK $\alpha_1$ | CuK $\alpha_1$ | $a_0 = 3.5238\text{Å}$ | CrK $\alpha_1$ | CoK $\alpha_1$ | CuK $\alpha_1$ |
| 3                 | 111 | 8            | 2.0871                 | 66.53          | 50.75          | 43.32          | 2.0345                 | 68.49          | 52.16          | 44.49          |
| 4                 | 200 | 6            | 1.8075                 | 78.60          | 59.32          | 50.45          | 1.7619                 | 81.05          | 61.02          | 51.85          |
| 8                 | 220 | 12           | 1.2781                 | 127.20         | 88.83          | 74.12          | 1.2459                 | 133.53         | 91.77          | 76.38          |
| 11                | 311 | 24           | 1.0900                 |                | 110.30         | 89.93          | 1.0625                 |                | 114.68         | 92.93          |
| 12                | 222 | 8            | 1.0436                 |                | 117.99         | 95.14          | 1.0172                 |                | 123.12         | 98.44          |
| 16                | 400 | 6            | 0.9038                 |                | (163.55)       | 116.92         | 0.8810                 |                |                | 121.94         |
| 19                | 331 | 24           | 0.8293                 |                |                | 136.48         | 0.8084                 |                |                | 144.65         |
| 20                | 420 | 24           | 0.8083                 |                |                | 144.68         | 0.7879                 |                |                | 155.67         |

Table 5—Comparison of Detectors for Diffraction from Nonfluorescing Powders  
(from Dowling, et al., Ref. 5)

| Counter                    | MoK $\alpha$            |     |                  | CuK $\alpha$            |     |                 | CrK $\alpha$            |     |                 |
|----------------------------|-------------------------|-----|------------------|-------------------------|-----|-----------------|-------------------------|-----|-----------------|
|                            | Detection Efficiency, % | P/B | Figure of Merit* | Detection Efficiency, % | P/B | Figure of Merit | Detection Efficiency, % | P/B | Figure of Merit |
| Geiger (A)**               | 14                      | 27  | 10               | 51                      | 46  | 20              | 59                      | 18  | 13              |
| Proportional (Xe) - no PHD | 13                      | 16  | 5                | 63                      | 57  | 30              | 60                      | 16  | 11              |
| - PHD                      | 12                      | 44  | 13               | 56                      | 146 | 69              | 58                      | 92  | 64              |
| Scintillation - no PHD     | 100                     | 10  | 25               | 100                     | 12  | 10              | 100                     | 5   | 6               |
| - PHD                      | 90                      | 44  | 100              | 89                      | 134 | 100             | 90                      | 93  | 100             |

\*Relative value of (detection efficiency) x (peak-to-background ratio).

\*\*Corrected for dead time.

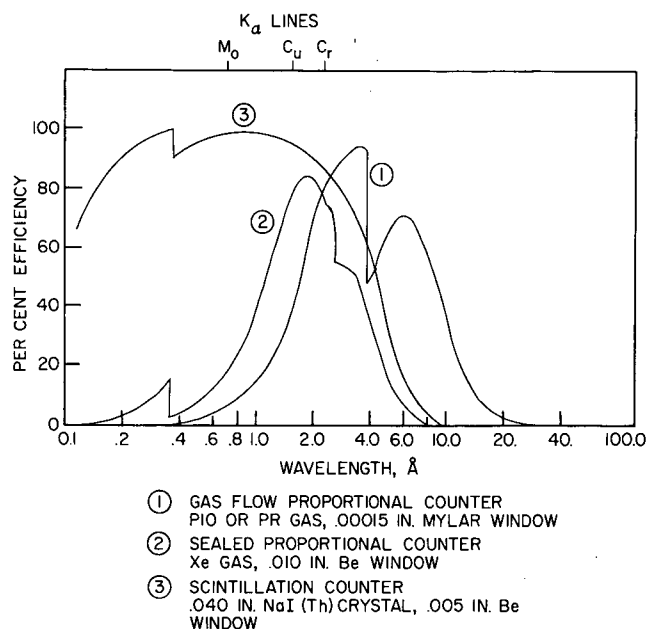


Fig. 28 - Comparison of counting efficiency for several commonly used detectors

The counter tube may also discriminate against white radiation, as shown in Fig. 28. An Xe filled proportional counter is a good choice for the longer wavelengths of Cu through Cr because the efficiency is high in this region and low for the white radiation of shorter wavelengths (3, 4). Although the efficiency of the scintillation counter is slightly higher in this region, it is almost 100% efficient over the entire diffraction wavelength region from Cr K on down. Therefore, background is high. This counter is the best choice for Mo, however (Fig. 28).

The scintillation counter is normally used in conjunction with pulse height discrimination (PHD). When used with Cr through Cu radiations, this allows the white radiation including the  $\lambda/2$  peak to be discriminated against. With Mo, however, PHD is less effective because the characteristic wavelength is near that of the white radiation peak. Because of the low resolution of the counters, especially scintillation, a  $\beta$  filter is still needed to remove white radiation just on the short wavelength side of K $\alpha$ . When used with the proportional counter and Cr through Cu, PHD decreases white radiation be-

tween that removed by the  $\beta$  filter and that discriminated against due to counter characteristics. Sample fluorescent radiation can be decreased by the baseline setting if its wavelength is sufficiently longer than that of the characteristic radiation used. When this difference is small, as in the case of Cu radiation and an Fe sample, an improvement in P/B ratio can be obtained with the right PHD settings (4, 5). As shown in Table 5, when considering a "figure of merit" defined as counting efficiency  $\times$  P/B ratio, the scintillation counter with PHD is by far the best choice for Mo radiation and is also slightly better than the Xe filled proportional counter with PHD for Cu (4, 5). When the proportional counter is used with PHD, it is superior to the Geiger counter.

Another method used to discriminate against fluorescent radiation when using Cu with an iron sample is a crystal monochromator in the diffracted beam. A monochromator in the incident beam can be used to remove the white radiation which causes fluorescence of a Cr sample, which cannot be discriminated against when Cr radiation is used. A monochromator may be used to improve P/B ratio, but one must consider the usual decrease in diffracted intensity associated with monochromators. In addition one must apply the correct LPA factors corresponding to monochromated conditions (6, 7).

#### 4.3 REFERENCES

1. A. Taylor, "X-Ray Metallography." New York: John Wiley and Sons, Inc., 1961, p. 750.
2. B. W. Roberts, "Filter and Crystal Monochromator Techniques." International Tables for X-Ray Crystallography. Birmingham: Kynoch Press, Vol. III, 1962, p. 73.
3. J. Taylor and W. Parrish, "Absorption and Counting—Efficiency Data for X-Ray Detectors." Rev. Sci. Instr., Vol. 26, 1955, p. 367.
4. E. W. Nuffield, "X-Ray Diffraction Methods." New York: John Wiley and Sons, Inc., 1966, p. 177.
5. P. H. Dowling, et al., "Counters for X-Ray Analysis." Philips Technical Review, Vol. 18, 1956/1957, p. 262.
6. L. I. Mirkin, "X-Ray Analysis of Polycrystalline Materials." New York: Consultants Bureau, 1964.
7. R. W. James, "The Optical Principles of the Diffraction of X-Rays." G. Bell and Sons Ltd., 1948.

## SECTION 5 SPECIMEN PREPARATION

### 5.1 EFFECT OF SURFACE CONDITION

The surface resulting from heat treatment will affect measurement of residual stress. Scale will decrease diffracted intensity and should be removed by some method (such as acid cleaning or pickling) which will not introduce stress. Stress can be measured in the material below a thin oxide layer without its removal, however. Decarburization also affects stress, as shown in Fig. 29 (1).<sup>\*</sup> A 0.001 in. layer of ferrite at the surface, which shows up white in the photomicrograph, is followed by a layer to a depth of 0.010 in. in which a carbon gradient exists. The decrease in carbon toward the surface is indicated by the decrease in hardness. Residual compressive stress also decreases toward the surface, as does the x-ray peak breadth. A measure of the stress in this layer may be desired. However, if the stress in the case produced by a given heat treatment is to be investigated, any affected surface layer must be removed by electropolishing.

Surface roughness has also been found to affect measured stress. Grooves produced by machining are found to affect the

stress measurements in the direction across the grooves as the roughness increases, but not the measurements along the grooves (2). When a rough surface is produced by machining, shot peening, etc., diffraction at large  $\psi$  angles will occur mainly from the tops of asperities. This material may be in a different stress state than that at the base of the asperities. The intensity will also be decreased because of absorption.

A ground or machined surface also contains residual stresses which depend on the operating conditions of the removal technique used. Grinding produces stresses to a depth of about 0.005 in. which vary with wheel hardness, depth of cut, coolant, etc. (3, 4). Machining in general is thought to produce stresses to depths of several thousandths of an inch (5). Again, if the stress before machining is desired (such as the stress from heat treatment in a finish machined part), the surface layer of about 0.005 in. at the position of measurement must be removed by a stress-free method such as electropolishing.

### 5.2 MATERIAL REMOVAL

In the removal of material for stress measurements in depth, machining can be used only for rough removal due to the stresses produced. Metallographic polishing must be followed by etching. Electrical discharge machining has also been found to affect stress at depths up to a few thousandths of an inch (5, 6), and can be used only for fast initial material removal.

The only stress-free methods appear to be electrolytic and chemical polishing. In electropolishing, the electrolyte and operating conditions used depend on the material or alloy. Techniques used for various materials are discussed by Tegart (7); a list is also given in Ref. 8. Also, a survey of the X-Ray Division of the SAE Fatigue Design and Evaluation Committee was made to determine what electropolishing techniques are in use. A condensation of results of the survey, including data obtained from members of the Society of Materials Science, Japan, is given for ferrous alloys in Table 6. Entries are grouped according to the type of solution, with the materials listed for which they are used.

Solutions in most extensive use are based on phosphoric or perchloric acids. The former are used mostly in this country; information on the latter in Table 6 is principally from Japan. Perchloric solutions produce a better surface finish than phosphoric (9), although those from the latter are entirely adequate for stress measurement. The temperature of a perchloric solution must be maintained below a certain level due to the explosive hazard. For precautions to be used when mixing and using these solutions, see Refs. 7 and 8.

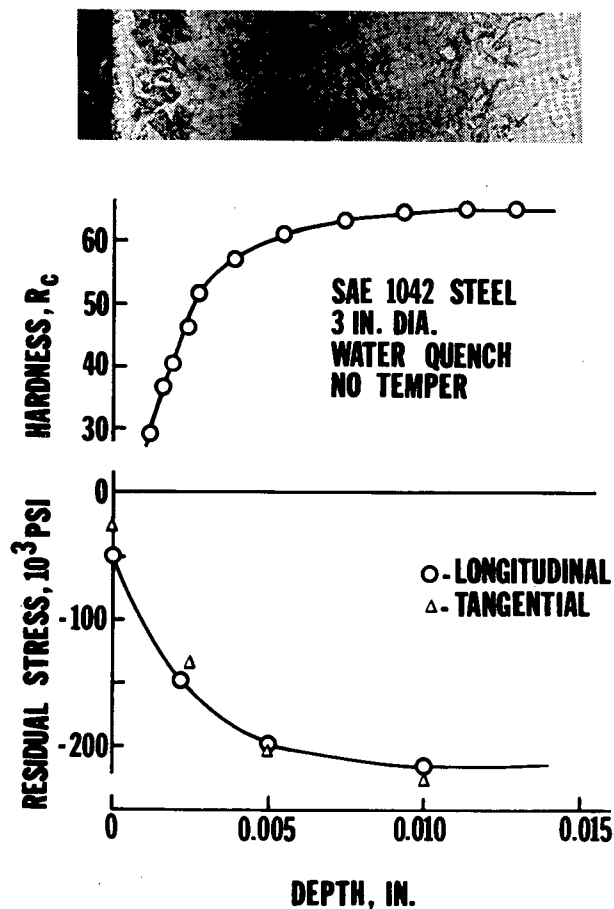


Fig. 29 - Effect of decarburization on residual stress (Ref. 1)

<sup>\*</sup>Numbers in parentheses designate References at end of Section.

Table 6—Electrolytic Polishing of Iron and Steel

| Material <sup>a</sup>            | Solution Composition <sup>b</sup>                                                                                   | Cathode Material         | Cathode-to-Specimen Distance, in. <sup>c</sup> | Voltage | Current Density, amp/in. <sup>2</sup> | Temperature, F <sup>b</sup> | Agitation Necessary? | Time (min.) to Remove 0.001 in. |
|----------------------------------|---------------------------------------------------------------------------------------------------------------------|--------------------------|------------------------------------------------|---------|---------------------------------------|-----------------------------|----------------------|---------------------------------|
| <b>Phosphoric Acid Solutions</b> |                                                                                                                     |                          |                                                |         |                                       |                             |                      |                                 |
| All steels                       | 3 parts orthophosphoric acid (86%)                                                                                  | Stainless steel          | 1-5                                            | 8-15    | 4-10                                  | 75-140                      | No                   | 1-10                            |
| Cast iron                        | 2 parts sulfuric acid (96%)                                                                                         | Lead<br>Copper           |                                                |         |                                       |                             |                      |                                 |
| All steels                       | 5 parts orthophosphoric acid                                                                                        | Stainless steel          | 0.25                                           | 40      | ~20                                   | 100                         | Yes                  | 2                               |
| Mild steel                       | 4 parts sulfuric acid<br>36 ml orthophosphoric acid                                                                 | Stainless steel          | 2.5                                            | 10      | 10                                    | 170                         | Yes                  | 3                               |
| Hardened steel                   | 18 ml sulfuric acid<br>9 ml water<br>2 ml dextrose                                                                  |                          |                                                |         |                                       |                             |                      |                                 |
| Mild steel                       | 100 ml orthophosphoric acid                                                                                         | Stainless steel          | 1.6                                            | 20      | 6-10                                  | 195                         | Yes                  | 2-4                             |
| Hardened steel                   | 50g chromium trioxide                                                                                               |                          |                                                |         |                                       |                             |                      |                                 |
| All steels                       | Rough polishing—<br>1000 ml orthophosphoric acid<br>1500 ml sulfuric acid<br>250 ml water<br>125g chromium trioxide | Stainless steel          | ~0.25                                          | 40      | 40-44                                 | 65-150                      | Yes                  | 1                               |
|                                  | Finishing—<br>9 parts (wt.) orthophosphoric acid<br>1 part (wt.) chromium trioxide                                  | Stainless steel          | ~0.25                                          | 40      | 20-36                                 | 65-150                      | Yes                  | 2.5 (75 F)<br>1 (150 F)         |
| <b>Perchloric Acid Solutions</b> |                                                                                                                     |                          |                                                |         |                                       |                             |                      |                                 |
| Mild steel                       | 1 part perchloric acid                                                                                              | Mild steel               | 1-3                                            | 12-50   | 1-2                                   | Below 86                    | Yes                  | 2-5                             |
| Hardened steel                   | 4 parts acetic anhydride                                                                                            | Stainless steel          |                                                |         |                                       |                             |                      |                                 |
| Hardened steel                   | 1 part perchloric acid<br>9 parts acetic anhydride                                                                  | Carbon                   | 0.2                                            | 30-40   | 3-4                                   | 75                          | No                   | 2.5                             |
| Mild steel                       | 185 ml perchloric acid                                                                                              | Stainless steel          | 0.5-1.2                                        | 40-50   | —                                     | Below 86                    | Yes                  | 1-8                             |
| Hardened steel                   | 765 ml acetic anhydride<br>50 ml water                                                                              | Aluminum                 |                                                |         |                                       |                             |                      |                                 |
| Hardened steel                   | 1 part perchloric acid                                                                                              | Stainless steel          | 0.6                                            | 70      | 5-6                                   | 66-86                       | Yes                  | 1.3-2.5                         |
| Cast iron                        | 10 parts ethanol                                                                                                    |                          |                                                |         |                                       |                             |                      |                                 |
| Hardened steel                   | 50 ml perchloric acid<br>750 ml ethanol<br>140 ml water                                                             | Stainless steel          | 0.1                                            | ~50     | —                                     | 75                          |                      | 2-3                             |
| Mild steel                       | 70 ml perchloric acid                                                                                               | Titanium                 | 0.3                                            | 25      | 12                                    | 75                          | Yes                  | 1                               |
| Hardened steel                   | 750 ml ethanol (increase if steel is soft)<br>100 ml ethylene glycol<br>monobutyl ether<br>90 ml water              |                          |                                                |         |                                       |                             |                      |                                 |
| Hardened steel                   | 60 ml perchloric acid                                                                                               | Stainless steel          | 0.12                                           | 30      | 4-6                                   | 75                          | Yes                  | ~1                              |
| Austenitic steel                 | 700 ml ethanol<br>100 ml butyl cellusolve<br>140 ml water                                                           |                          |                                                |         |                                       |                             |                      |                                 |
| <b>Other Solutions</b>           |                                                                                                                     |                          |                                                |         |                                       |                             |                      |                                 |
| Cast iron                        | 405 ml acetic acid<br>21 ml water<br>15 g chromium trioxide                                                         | Stainless steel<br>Steel | 1-2                                            | 30-50   | —                                     | Below 150                   | Yes                  | 5                               |
| All steels                       | 1 part acetic anhydride                                                                                             | Stainless steel          | 1.5                                            | 15      | 1.2                                   | 32                          | Yes                  | 5-10                            |
| Cast iron                        | 1 part nitric acid                                                                                                  |                          |                                                |         |                                       |                             |                      |                                 |

<sup>a</sup>Mild steel—pure iron, ferritic steel  
Hardened steel—martensitic steel, alloy steel  
Cast iron—all types

<sup>b</sup>Observe the necessary precautions when using perchloric acid and acetic anhydride (7-8).  
<sup>c</sup>Small distances of 0.1-0.25 in. probably refer to spot polishing apparatus.

Different types of electrical circuits are used for electropolishing, depending on the current required, etc. (7). For low currents, a battery power supply may be sufficient. For large capacity, however, a rectifier operating on 110-440 V is required. When layers are to be removed from large areas, the specimen is immersed in a bath. If a small area is to be polished, the surrounding material can be protected with lacquer or tape. For "spot" polishing to prepare a small area of a part of any size for stress measurement, a portable unit may be used in which the electrolyte is circulated to the part.

A thin oxide film is easily removed by electropolishing, but a thick scale should be removed by pickling or by mechanical means. Failure to do so results in an uneven surface because some areas begin to polish sooner than others.

Chemical polishing is applicable to some materials with varying success, but this method is not as well controlled as electropolishing. Solutions and procedures are described by Tegart (7).

### 5.3 FACTORS AFFECTING MINIMUM SPECIMEN SIZE

The size a specimen has to be is of concern, both in making up specimens to investigate the effects of various processes on residual stress, and in removing a representative sample from a part too large to be analyzed as a whole. The geometry of the part influences the minimum specimen size. It is generally acknowledged that cylinders, both thin walled and solid, should be a minimum of two diameters in length to retain the stress distribution in a longer part (5, 10). Stress should also be measured at least a diameter from the end of a heat treated bar. Plates should be cut to a length and width of at least two times the thickness in order to avoid end effects; structural stresses may be relieved, however (10, 11). If there is a question as to how close a cut can be made with other geometries, such as gear teeth, it may be necessary to monitor with strain gages as discussed in paragraph 5.4.

Different stress producers may require different test specimen sizes. The study of processes which affect the entire specimen thickness, such as heat treatment or severe rolling, will require the same specimen section size as the particular part simulated. Machining or shot peening, on the other hand, result in only superficial deformation. Thickness beyond a fairly small value, such as 1/4 in., would not be a factor, and stress could be measured fairly near an edge. Geometry would not have as much effect either.

### 5.4 COMBINATION STRAIN-GAGE DISSECTION AND X-RAY TECHNIQUES

In some cases it is necessary to section a part to make it accessible by x-ray, although cutting will relax residual stress. Some examples are large parts or structures, parts in which stress is to be measured in a bore or hole, and gear teeth, in which stress is to be measured at a location not accessible without cutting away other teeth or cutting the gear.

If stress only at the surface is to be measured, strain-gages may be mounted at the location in the directions of the principal stresses in the surface, and the relaxed strain can be determined from readings before and after cutting (initial-final).

Relaxed stress is obtained assuming biaxial stress. The remaining stress in the sectioned member is then measured by x-ray at an adjacent area and added algebraically to the relaxed stress to obtain the initial stress.

The stress distribution through a member such as a hollow cylinder or plate may also be obtained (10, 11). Gages are mounted on both faces, and the relaxed stresses resulting from cutting out an element are calculated. Disregarding the radial stress, the assumption may be made that the relaxed stress through the section in a given direction parallel to the surface varies linearly between the two surface values. The remaining stresses in depth can then be measured by layer removal and x-ray as an alternative to strain gage methods. After correcting for the effects of stressed layers removed (paragraph 7.4), this stress at a given point is added algebraically to the corresponding relaxed stress, and the initial stress distribution is obtained.

There is the danger, when sectioning or removing layers, that yielding will occur because of the alteration of the stress distribution (5). Relaxed stresses determined from strain-gage readings will then be in error. A technique of dissection should be used in which this will not happen, depending on the stress pattern present.

### 5.5 REFERENCES

1. Unpublished work in the Research Department of Caterpillar Tractor Co.
2. S. Taira and J. Arima, "X-Ray Investigation of Stress Measurement (on the Effect of Roughness of Specimen Surface)." Proceedings of the Seventh Japan Congress on Testing Materials. Kyoto: The Society of Materials Science, 1964, p. 21.
3. H. R. Letner, "Residual Grinding Stresses in Hardened Steel." ASME Transactions, Vol. 77, 1955, p. 1089.
4. H. R. Letner, "Influence of Grinding Fluids Upon Residual Stresses in Hardened Steel." ASME Transactions, Vol. 79, 1957, p. 149.
5. A. A. Denton, "Determination of Residual Stresses." Metallurgical Reviews, Vol. 11, 1966, p. 1.
6. J. G. Roberts, "Residual Stresses and Hardness in Steel by X-Ray Diffraction and Metal Fatigue." Paper 467B presented at SAE Automotive Engineering Congress, Detroit, January 1962.
7. W. J. McG. Tegart, "The Electrolytic and Chemical Polishing of Metals in Research and Industry." New York: Pergamon Press, 1959.
8. American Society for Metals, "Metals Handbook." 1954 Supplement, p. 170.
9. J. Heyes, "Electrolytic Polishing of Steel." Metal Finishing, Vol. 60, December 1962, p. 36.
10. "Methods of Residual Stress Measurement." Handbook Supplement SAE J936, New York: Society of Automotive Engineers, Inc., December 1965.
11. R. P. Frick, G. A. Gurtman, and H. D. Meriwether, "Experimental Determination of Residual Stresses in an Orthotropic Material." Journal of Materials, Vol. 2, No. 4, 1967, p. 719.

## SECTION 6 CONTROL OF INSTRUMENTAL FACTORS

### 6.1 ALIGNMENT

**6.1.1 ALIGNMENT TERMINOLOGY AND GENERAL REQUIREMENTS**—In order to have a common terminology for the purpose of discussing the various motions required for diffractometer alignment, the nomenclature illustrated in Figs. 30 and 31 will be used. It should be noted that the horizontal placement of the goniometer in Fig. 31 is done for the purpose of discussion only, and is not meant to imply that it is necessarily restricted physically to the horizontal plane.

To insure that the geometric arrangement as shown in Fig. 31 is satisfied, the following basic conditions must be met:

(1) The receiving slit, the incident beam slits, and the axis of the focal spot must be made parallel to each other and perpendicular to the plane of the goniometer. The extension of the axis of the tube focal spot must be made to pass through the pivot point of the goniometer.

(2) The line (ROS) connecting the receiving slit and axis of the focal spot must be made to pass over the center of rotation (that is, through the extended axis of rotation). This requires the determination of the position of the center of rotation (O).

(3) In addition to the requirements on the x-ray optics, the measurement of stress requires a radial adjustment to facilitate the receiving slit being relocated at the point where the diffracted beam will be focused for  $\psi$  angles other than 0 deg. As discussed in following sections, the parafofocusing geometry of the diffractometer causes the focus of the diffracted beam to move in toward the center of rotation with an increase in the value of the  $\psi$  angle above 0 deg. The relevance to alignment becomes obvious when it is realized that the receiving slit must

be moved on a *true* radius of the diffractometer circle. If this condition is not met, large  $\Delta 2\theta$  shifts can be introduced.

(4) Optimum diffractometer alignment requires a determination of "true zero," which may not be the same as that indicated on the goniometer vernier wheel. However, since the macrostress measurement involves a  $\Delta 2\theta$  determination, the inclusion of a true zero correction is not necessary because both peaks are shifted equally.

**6.1.2 ALIGNMENT OF OPTICS**—Although the general outline given in the previous section is applicable to all diffractometers, the different physical arrangements produced by various manufacturers result in slightly different alignment techniques for each type of equipment. This is illustrated by the General Electric and Norelco diffractometers shown in Figs. 32 and 33, respectively. At the present time, the main difference in equipment (aside from horizontal and vertical goniometers) is the manner in which the x-ray tube support and the goniometer are coupled. In some cases (such as General Electric), the tube support is not connected directly to the goniometer and the focal plane must be adjusted independently. In the case of General Electric equipment, the x-ray tube socket and the goniometer base plate can both be moved independently in order to bring the focal plane into the proper position with respect to the slits. In other cases (such as Picker), the x-ray tube support and goniometer are coupled directly, so that there is no need for movement of the goniometer baseplate. However, in both cases, the requirements outlined in paragraph 6.1.1 must be satisfied.

Due to the various types of equipment, it is not possible to

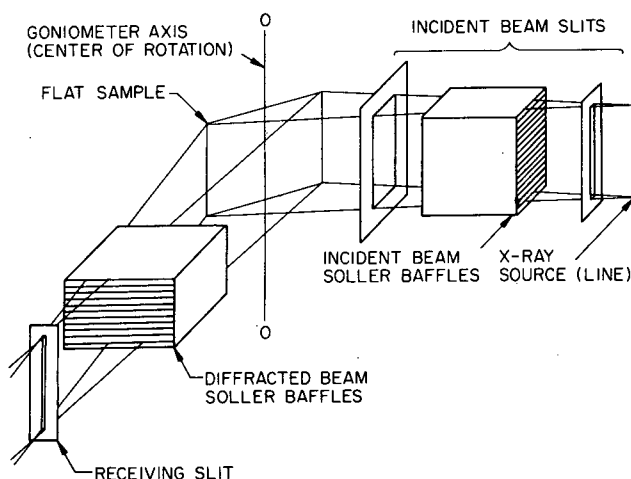


Fig. 30 - X-ray optics for parafofocusing geometry

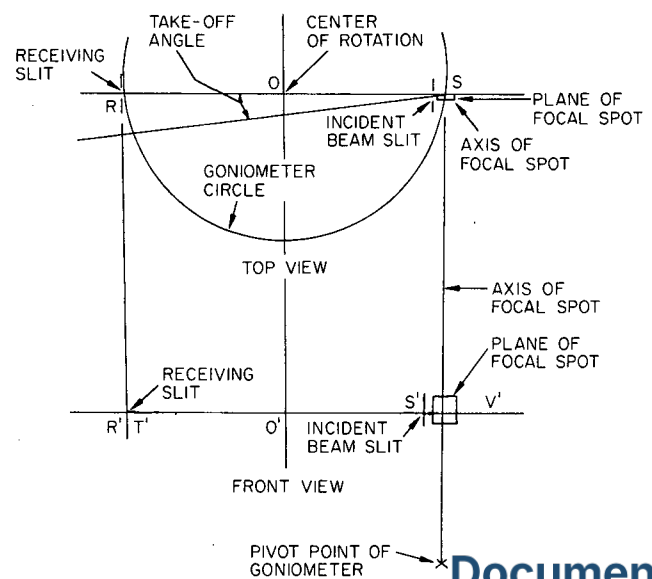


Fig. 31 - Diffractometer geometry

give a detailed account of each; therefore, the following discussion will be limited to the essential requirements and basic techniques. In addition, it should be stressed that the various equipment manuals supplied with the diffractometers should be read and understood before undertaking any precise alignment.

The final alignment is preceded by a preliminary alignment when a tube is changed or major adjustments are made. This is a mechanical alignment employing gages that are furnished with specific type units, which to a close approximation put plane  $R'T'$  in the same plane as  $S'V'$  and parallel to the plane of the goniometer.

The first step in a final alignment procedure consists of selecting a takeoff angle (Fig. 31) that will be a good compromise between intensity and resolution (choose a smaller angle for better resolution and lower total intensity). The tube should then be located with the line focus parallel to the axes of the slits. While referring to the related instruction manual, the proper lineup beam slit, narrow detector slit, and accompanying medium or high resolution detector Soller baffles should be installed. Employing proper filters for the tube used, the proper adjustments should be made so that the maximum beam intensity is obtained with the main protractor set at  $0.00^\circ 2\theta$ . The beam position is checked by comparing intensity readings at equal intervals on both sides of  $0.00^\circ$  (for example,  $\pm 0.03^\circ$ ).

In the case of equipment which has the x-ray tube and goniometer separated, the extension of the plane of the focal spot must be made to pass through the pivot point of the goniometer by adjustment of the goniometer base plate. To facilitate this operation, it is convenient to use a two hole shutter in the filter pocket between the detector slit and counter tube, which allows viewing either the topmost or bottommost (approximately 10%) portion of the x-ray beam. The intensities need not be the same for top and bottom since the reason for this check is to determine by beam position if adjustments are necessary to correct the tilt of the focal spot axis with re-

spect to the goniometer axis. In the case of the direct coupled goniometer and tube support, the same requirement must be satisfied to insure the correct specimen to focal point distance. However, the adjustment must be made at the x-ray tube socket rather than with the goniometer baseplate.

The accuracy of the alignment can be checked at this point in the procedure by choosing a powder sample that gives a strong  $K_\alpha$  doublet line at about  $40^\circ 2\theta$  and recording the apparent resolution. The powdered sample should be small grained (passing 200-320 mesh screen), strain free, and free from ultrafine particles. Annealed tungsten powder has proven to be a very good reference sample for this check, giving a strong line at about  $40^\circ$  with copper radiation for which the separation of the copper  $K_\alpha$  doublet is about  $0.10^\circ$ . If the resolution is satisfactory, the valley between the peaks drops to near  $1/2$  maximum intensity for the weaker  $K_{\alpha_2}$  line. If the resolution is not satisfactory, the alignment around zero should be rechecked by determining whether equal intensities are obtained at equal plus and minus deviations from zero.

One must remember that the apparent width of the  $K_\alpha$  line, as recorded, is wider than the diffracted line by the width of the detector slit used. The resolution of lines near the limiting condition will be partially lost if the time constant of the rate meter is greater than about half the time-width of the slit.

Thus, no matter what diffraction equipment is being aligned, the basic requirements are that the slit axes, the axis of the center of rotation, and the axis of the focal spot be coplanar, parallel within this plane, and perpendicular to the plane of

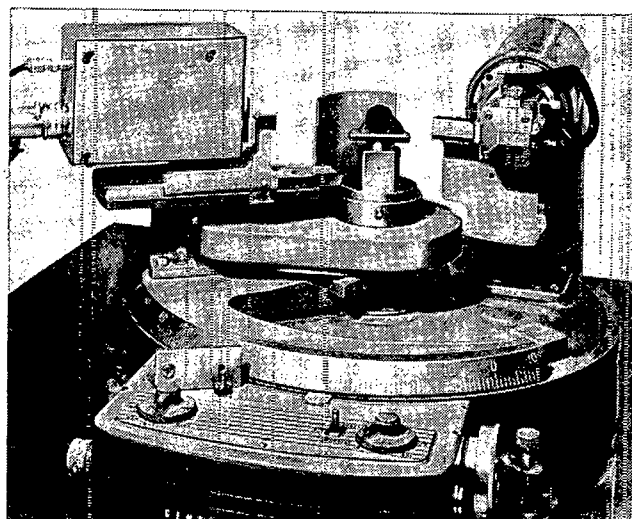


Fig. 32 - General Electric diffractometer (courtesy of General Electric Co., X-ray Department)

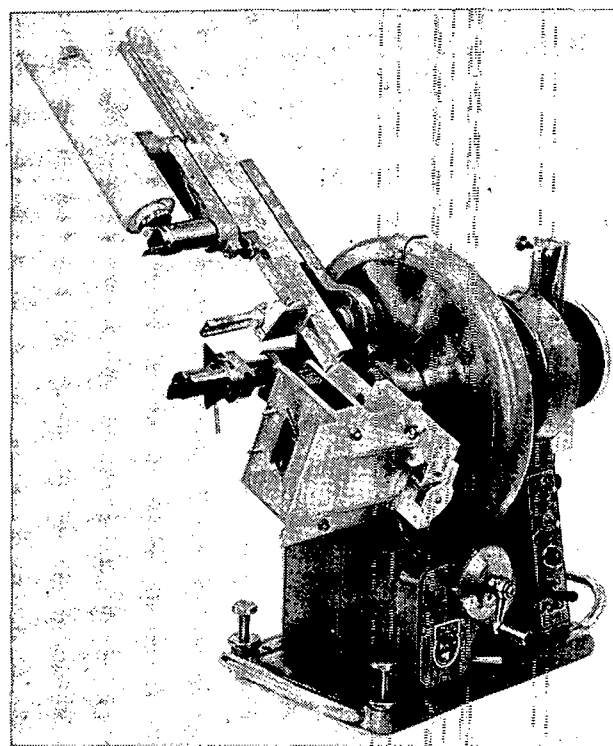


Fig. 33 - Norelco diffractometer (courtesy of North American Philips Co.)