A HISTORICAL REVIEW OF RETAINED AUSTENITE AND ITS MEASUREMENT BY X-RAY DIFFRACTION

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ABSTRACT

This paper traces the historical development of the methods, techniques, and applications related to the measurement of retained austenite by x-ray diffraction from the early 1930s to the present.

INTRODUCTION

Quantitative determination of retained austenite content in heat-treated steels by x-ray diffraction has provided a reliable means of controlling properties and ensuring quality. Depending on the composition, austenitizing temperature, quenching rate, final quenching temperature, and stress state, a heat-treated steel could contain a significant volume fraction of retained austenite. The role of retained austenite in these microstructures is complex, as it can have both positive and adverse effects on the properties and performance of these steels. Too much retained austenite can result in lower elastic limits, reduced hardness, lower high cycle fatigue life, and dimensional instability.[1] Too little retained austenite, however, can result in poor fracture toughness and reduced low cycle fatigue and rolling contact fatigue life.[1] In the 1930s and 40s a number of methods were developed to determine the amount of retained austenite in a heat-treated steel. The main methods were based on metallographic, magnetic and x-ray diffraction experimental techniques. The metallographic point[2] and linear[3] counting methods were tedious and subject to large errors when the retained austenite content was less than ten per cent. Since austenite is non-magnetic and the structural magnetization of ferrite and martensite are similar, it is possible to determine the amount of retained austenite by magnetic techniques.[4][5] However, reliable measurements by magnetic methods are only possible in the complete absence of cementite. Thermal dilatation has also been employed to determine the amount of retained austenite.[2] When x-ray diffraction methods were developed to replace the difficult metallography and unreliable magnetic methods, it became and remains the most satisfactory technique to determine the amount of retained austenite. The interpretation of the x-ray pattern is straightforward and less than 0.5 percent retained austenite can be detected and quantified employing a standard x-ray diffractometer.

In this manuscript, some of the important early (1930-50) investigations employing x-ray diffraction methods to determine the amount of retained austenite in steels are reviewed. Then some later (1950-70) works are cited which examine the serious experimental problems associated with determination of integrated intensities. Finally, results of recent (1970-00) x-ray diffraction investigations on the relationships among the amounts...
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of retained austenite and the substructure of the austenite and the martensite phases will be presented.

1930 to 1950

The first quantitative determination of retained austenite in heat-treated steel most likely was reported by Tamaru and Sekito in 1931. They also determined the amounts of cementite and martensite in quenched and annealed steels. Deybe-Scherrer patterns were taken by grazing an x-ray beam off the surface of a rotating five-millimeter diameter specimen placed eccentrically in the camera. A thin gold strip, 0.04 mm thick and 4.9 mm wide covered a portion of the specimen surface. The maximum intensities of the gold lines were taken as standards to which the intensities of the austenite lines were compared. Darkening of the film was made quantitative with a deflection type microdensitometer. The conversion factor required to make the determination absolute was obtained from one hundred percent austenitic steel with a composition of 0.78% C and 11.6% Mn that was quenched from 1150°C. The results from the x-ray photographs and microphotometer patterns for these measurements are shown in Fig 1.

A similar technique employing a Phragmén focusing camera and Cr Kα radiation with a thin aluminum foil providing the standard reference lines was reported by Gardner, Cohen and Antia. A drawing of their camera arrangement is shown in Fig. 2. Typical microphotometer traces are shown in Fig. 3. The maximum intensity of the 200 reflection from the aluminum was compared to the maximum intensity of the 200 reflection from the austenite. This method was calibrated with a series of steels whose austenite content was known

Figure 1 - Microphotometer traces from Ref. 5.

Figure 2 - Diagram of Phragmén focusing camera employed by Gardner, Cohen and Antia for determination of retained austenite, from Ref. 8.
from point counting. The calibration became uncertain at austenite contents below ten percent. Specimens of $\frac{1}{2} \times \frac{1}{2} \times \frac{3}{8}$ inch were ground to the curvature of the camera prior to heat treatment. After treatment at least 0.040 inches were removed with emery paper and 0.003 inches were removed by an electrolytic etch. Steels with grain sizes smaller than ASTM 6 (44µm) produced smooth diffraction lines while sizes as large as ASTM 3 (125µm) required movement of the specimen during exposure. By etching away successive layers the retained austenite gradient was defined in five per cent nickel steels that were oil quenched from 925°C. The x-ray results were compared to austenite contents determined by lineal counting techniques and the agreement was within one percent.

![Figure 3](https://example.com/figure3.png)

**Figure 3** - Typical microphotometer traces illustrating various measurements upon which density calculations are based. Determination of the total density (including background) of the aluminum standard line is shown separately at the left, and is generally included to ensure the maximum density does not exceed the linear range of the film, from Ref. 8.

To avoid the relatively high backgrounds in diffraction patterns of steels from fluorescence of the iron and atoms of the alloying elements, Averbach and Cohen[9] used monochromatic Co K$_\alpha$ radiation. They obtained the radiation by diffracting the incident beam from the 200 face of a rock salt crystal. These investigators were first to employ the more reliable total diffracted energy, which is proportional to integrated intensity, to determine retained austenite content. Their method made the presence of a standard unnecessary because, in effect, the coexisting martensite is used as an internal standard. Micro-photometer traces of the diffraction patterns recorded on the film along with the equations used to calculate total diffracted energy are shown in Fig. 4.

1950 to 1970

With the development of diffractometer technology, the ease of making quantitative measurements of retained austenite using x-ray diffraction increased. Employing integrated intensities from x-ray diffractometer traces, an empirical equation was developed by Koistinen and Marburger[13] to predict the amount of retained austenite in 0.37 to 1.1% C steels. See Eqn. 1. This equation was based on the composition of the steel and the quenching temperature. Steel composition is important because it affects the martensite start temperature as indicated in Eqn 2.[6] It was also demonstrated by, Durin and Ridal[10] that the amount of retained austenite determined by x-ray diffraction agreed well with both metallographic and magnetic measurements.
%Retained Austenite = \exp(-1.10 \times 10^{-2} (M_s - T_{\text{quench}})) \quad \text{Equation 1}

M_s (^\circ C) = 561 - 474C - 33Mn - 17Cr - 17Ni - 21Mo \quad \text{Equation 2}

With the advent and refinement of diffractometer technology, however, came the difficulty in accurate quantitative measurements of retained austenite in textured samples. This problem was described by Miller[11] who formulated the multiple peak approach to minimize this effect. The increase in precision of retained austenite measurements for a highly textured sample as a result of increasing the number of diffractions peaks analyzed has been presented by Voskamp[14] and is shown graphically in Fig 5. Miller[11] also designed and constructed a rotating and tilting specimen holder to obtain data that was more random in character from textured specimens.

\[ P_\alpha = \text{const} \cdot \frac{1}{V_\alpha} F^2 m (L.P.) e^{-2M} V_\alpha A(\theta) \]

\[ P_\alpha = \text{const} \cdot RV_\alpha A(\theta) \quad \text{Eq 1} \quad \text{Eq 2} \]

where:
- \( P_\alpha \) = power per unit length of diffraction line for a particular diffraction line of substance \( \alpha \), in arbitrary units
- \( V_\alpha \) = volume of unit cell of substance \( \alpha \), in \((\text{kX})^3\) units
- \( F \) = structure factor per unit cell
- \( m \) = multiplicity of diffracting plane
- \( (L.P.) \) = Lorenz and polarization factor
- \( e^{-2M} \) = Debye-Waller temperature factor
- \( V_\alpha \) = volume of substance \( \alpha \) irradiated, in \( \text{cm}^3 \)
- \( A(\theta) \) = sample absorption factor
- \( \theta \) = Bragg angle

**Figure 4** - Microphotometer traces from Ref. 9 along with the equations for total diffracted energy

1970 to 2000

In September of 1979, the x-ray division of SAE Fatigue Design and Evaluation Committee put together an information manual on retained austenite and its measurement by x-ray diffraction identified as SP-453 and titled: Retained Austenite and Its Measurement by X-Ray Diffraction.[15] This manual provided a single resource
summarizing and describing the techniques for retained austenite measurements as well as methods for its control.

Investigations of the relationships among the amounts of retained austenite and the substructure of the austenite and the martensite phases performed by Makinson, et.al. [12], have provided insight as to the transformation of retained austenite under rolling contact stresses. By examining case carburized tapered roller bearing races, x-ray diffraction was used to establish the profile of both the volume fraction of retained austenite and the corresponding particle size through the case region of these bearings. These results are shown in Fig. 6. The maximum diffracting particle size occurs sub-surface at a depth of approximately 250µm. At shallower depths the diffracting particle size decreases due to the higher carbon content. This increasing carbon content increases the tetragonality of the martensite phase that in turn increases the amount of deformation of the austenite phase. This increased amount of deformation produces defects and is indicated by a smaller diffracting particle size. The diffracting particle size also decreases at depths greater than 250µm. Although the carbon content decreases toward the core, which decreases the tetragonality of the martensite, the total amount of martensite increases which leads to greater deformation of the remaining austenite phase and subsequent smaller diffracting particle size.

CONCLUSIONS

A review of the literature concerning the measurement of retained austenite in steel has shown that early investigators, using film methods, developed the basic principles and techniques for the measurement of retained austenite in steel using x-ray diffraction by 1950. Since then, work focused on the use of these methods with more sophisticated and accurate equipment, and the use of this equipment and modification of the basic equations to solve more difficult problems. Today, the measurement of retained austenite in steel has become a routine and often automated procedure used in a variety of production and experimental work to routinely measure the retained austenite content in
steels to less than 0.5 percent.

REFERENCES


