Determination of Volume Percent Retained Austenite by X-Ray Diffraction

INTRODUCTION

Hardening of steels requires heating to an austenitic phase and quenching to room temperature to produce a hard martensitic phase. Austenite is an FCC phase that is stable above a temperature of 735 C. Due to incomplete transformation some austenite is retained at room temperature. Retained austenite can dramatically decrease the mechanical properties of the steel. Properties such as fatigue strength, toughness, hardness, yield strength and machinability can be influenced by retained austenite.

Austenite can transform in service as a result of thermal cycles, plastic deformation, or shock. Shot peening, for example, will transform the austenite on the surface of gear teeth. Exposure to extreme cold renders the austenite increasingly unstable as the temperature diminishes. The transformation of austenite to ferrite involves a nominal 4% volume increase. A linear dimensional increase on the order of the cube root of that would lead to seizure and excessive interference in precision gearing and bearings. Accurate measurement of the retained austenite levels is important in the development and control of a heat treatment process.

X-ray diffraction is considered to be the most accurate method of determining the amount of retained austenite in steels. Lambda Research is the only accredited independent laboratory that uses Bragg-Brentano diffractometers for measurement of retained austenite, as required by ASTM[1] and SAE[2]. Most other labs now use

position sensitive detector (PSD) based systems for speed and portability that cannot be calibrated by direct calculation of the relative phase intensities and must use reference samples that usually are not of the correct structure. As a result, the retained austenite measurements can have a proportional error as much as 20% of the value reported. It was for this reason that the NIST austenite standards were withdrawn, based upon work performed at Lambda Research, which showed that the various austenite standards were not self consistent, leading to inaccuracies in quality assurance testing for retained austenite.

RETAINED AUSTENITE MEASUREMENT TECHNIQUE

For a randomly oriented sample, the integrated intensity from any diffraction peak is proportional to the volume fraction of that phase. Intensities of the austenite (γ) and ferrite (α) diffraction peaks in a steel are:

$$I_\gamma = \frac{KR_\gamma c_\gamma}{2\mu}$$

$$I_\alpha = \frac{KR_\alpha c_\alpha}{2\mu}$$

Where I is the integrated intensity, c is the volume fraction of each respective phase, and μ is the linear absorption coefficient for the steel. K is a constant that is dependent upon the selection of instrumentation geometry and radiation but independent of the sample. R depends upon the interplanar spacing, the Bragg angle, crystal structure and composition of specimen. R is a term that can be calculated from first principals. Lambda calculates R for each steel alloy tested per SAE[2].

Dividing the above equations, and given the sum of the volumes of the austenite and martensite phases equals one, yields the following equation.

$$c_\gamma = \left[\frac{R_\gamma I_\alpha}{R_\alpha I_\gamma} + 1 \right]^{-1}$$

The austenite fraction is determined from the ratio of the austenite and ferrite diffraction peak intensities and the values of R for each phase. This method is more commonly known as the "Direct Comparison" method.

Lambda uses the method of Averbach and Cohen[3] in accordance with ASTM[1]. The integrated intensities of the austenite (200) and (220), and the ferrite (200) and (211) diffraction peaks are measured on the automated diffractometers, providing four austenite/ferrite peak intensity ratios. The use of multiple diffraction peaks minimizes the effects of preferred orientation and allows interference from carbides to be detected.

Carbides can produce diffraction peaks that interfere with the austenite and/or martensite peaks potentially causing significant errors in measurement. Lambda identifies any carbide phases that may be present by obtaining a diffraction pattern of the specimen to be tested. The carbides are identified and the austenite and martensite diffraction peaks are chosen to eliminate interference from the carbide phases.

A device known as a "Miller fixture" is used at Lambda to rotate the specimen around the surface normal and oscillate it through an angular range of $\pm$ 45 deg. The fixture is designed to minimize the effects of preferred orientation during collection of the diffraction peak integrated intensities.

EXAMPLES

Figures 1 and 2 show examples of retained austenite distributions as functions of depth. A depth resolution of less than 0.0005 in. is achievable by electropolishing to remove layers without inducing plastic deformation and transformation of unstable austenite. Austenite measurements should be made on electropolished surfaces to remove interfering oxides and eliminate any plastically deformed surface layer. The austenite distributions shown indicate low austenite content in the decarburized near-surface layer. Beneath the decarburized layer the austenite content diminishes through the hardened case.
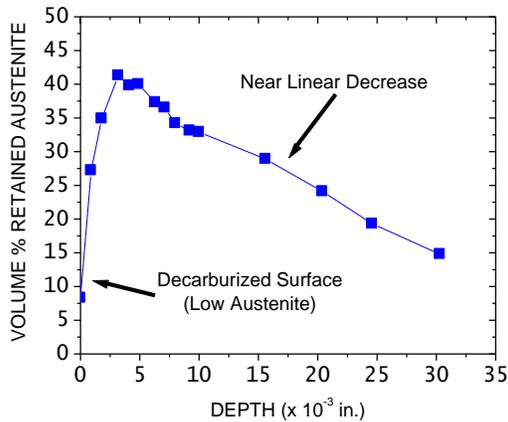
Figure 1 – Percent retained austenite in 8620 steel carburized gear tooth showing peak retained austenite levels at a nominal depth of 0.004 in.
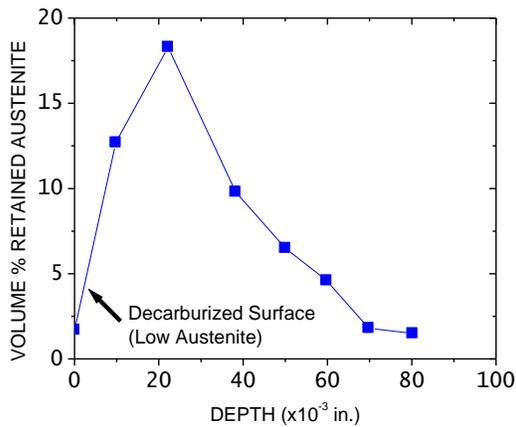

Figure 2 – Percent retained austenite in 8720 steel carburized component showing peak levels of retained austenite at a nominal depth of 0.02 in.

REFERENCES

1. ASTM, "Standard Practice for X-Ray Determination of Retained Austenite in Steel with Near Random Crystallographic Orientation," Standard E975-03, American Society for Testing and Materials, Nov. 1, 2003.

2. "Retained Austenite and Its Measurement by X-Ray Diffraction," SAE Special Publication 453, SAE, Warrendale, PA 15096.

3. B.L. Averbach and M. Cohen, Trans. AIME, Vol. 176, 1948, p. 401.

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