Diffraction Notes

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RETAINED AUSTENITE MEAUREMENT

Retained austenite strongly influences the properties of steel. The fatigue life, ductility, toughness, hardness, yield strength, and machinability can all depend upon austenite content. Accurate measurement of the volume percent-retained austenite is of critical importance in the optimization of heat treatment procedures.

Austenite is an FCC phase, stable above 735 deg. C, which either transforms to ferrite, carbides, and martensite, or remains as unstable retained austenite when cooled to room temperature. The products of the transformation depend upon the alloy chemistry, cooling rate, lowest temperature exposure, plastic deformation, and other factors that affect the diffusion of carbon from the FCC austenite crystal structure. If carbon diffuses out of the unit cell, carbides and BCC ferrite are formed. If the carbon is retained by rapid quenching, BCT martensite is formed.

Of the various methods of measuring austenite content, x-ray diffraction is generally considered the most reliable. X-ray diffraction patterns depend upon both the crystal structures and amounts of the phases present in the sample. If the crystals are randomly oriented, the intensity of the diffraction peaks produced by each phase is proportional to the amount of the phase present.

The intensities of the austenite and ferrite diffraction peaks in a steel are given by,

$$I_{ai} = \frac{KR_{ai}}{2U} C_a$$

And

$$I_{fj} = \frac{KR_{fj}}{2U} C_f$$

where $_{ai}$ and $_{fj}$ designate the ith austenite and jth ferrite peaks, I is the integrated intensity, R is a constant for the peak selected, K is an instrumental constant, U is the sample absorption coefficient, and C is the volume fraction present.

Dividing the equations and assuming $C_a + C_f = 1$ yields the relation upon which the direct comparison method for x-ray diffraction austenite analysis is based:

$$Ca = \left[\frac{R_{ai} \;\; I_{fj}}{R_{fj} \;\; I_{ai}} + 1 \right]^{-1}$$

The austenite fraction is calculated from the ratio of the austenite and ferrite diffraction peak intensities. The constants, R, can either be determined empirically or calculated.

At Lambda Research, the method of Averbach and Cohen (1) is used in accordance with ASTM specification E975-84. The integrated intensities of the austenite (200) and (220), and the ferrite (200) and (211) diffraction peaks are measured on automated diffractometers, providing four austenite/ferrite peak intensity ratios. A sample data reduction is shown in Table I for NBS standard reference material No. 487 containing 31.0% austenite, which is routinely checked for quality control. For forged steel samples, standard deviations of ± 2% are typical, with a detection limit on the order of 0.2%.

The use of multiple diffraction peaks minimizes the effects of preferred orientation and allows interference from carbides to be detected. The horizontal diffractometers used allow for a wide variety of sample shapes and sizes weighing up to 50 lbs. A device which simultaneously rotates and oscillates small samples rapidly in the x-ray beam can be used to integrate the diffracted intensity over many orientations, allowing reliable measurement on highly textured or coarse-grained samples.

TABLE I
X-RAY DIFFRACTION
DIRECT COMPARISON METHOD
VOLUME PERCENT RETAINED AUSTENITE ANALYSIS
000075.001 NBS SRM 48731.08% Austenite 1/.5nspr 1s 8710D21DC

PHASE	(HKL)	BG1	I	BG2	IO
FERRITE	200	12659	31663	12903	6101
FERRITE	211	26123	110878	29861	54894
AUSTENITE	200	6503	16684	6397	3784
AUSTENITE	220	12874	32721	13585	6262

PERCENT RETAINED AUSTENITE BY (HKL) PAIRS

AUSTENITE	FERRITE	VOL. PERCENT
200	200	29.0
220	200	30.5
200	211	29.6
220	211	31.1

AVERAGE PERCENT AUSTENITE = 30.1 STAND. DEV. = 0.9
95 PERCENT CONFIDENCE INTERVAL IS 28.4 TO 31.7

The austenite distribution as a function of depth can be determined with resolution on the order of 0.005 in. by electropolishing to remove layers without plastically deforming the material and transforming the unstable austenite. Austenite measurements should be made on electropolished surfaces to remove interfering oxides and eliminate any plastically deformed surface layer. The austenite distribution from the surface to a depth of nominally 0.030 in. through the hardened case of a pinion tooth is shown in Figure 1. The austenite content is low in the decarburized surface to a depth of nominally 0.002 in. Beneath the decarburized layer, the austenite content diminishes nearly linearly through the hardened case. The retained austenite and residual stress distributions can be measured simultaneously.

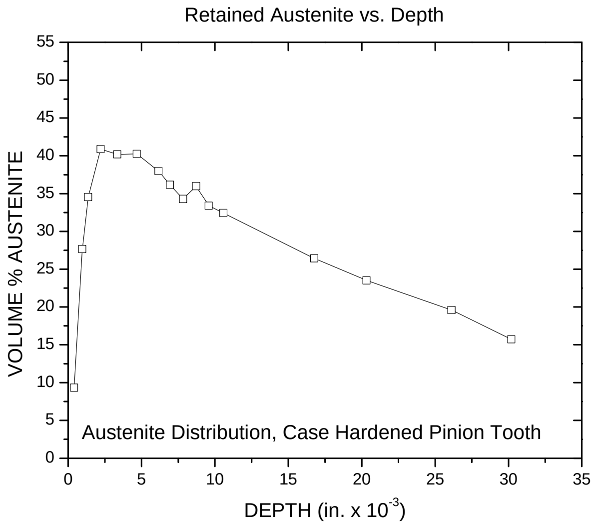

Figure 1

(1) B.L. Averbach and M. Cohen, AIME, Vol. 176, 401, (1948)

EQUIPMENT ACQUISITION

An automated Huber diffractometer adapted for large steel sample analysis is now on line. The instrument has a maximum sample capacity in excess of 500 lbs. and a beam clearance of approximately 10 in. The new system is running with software developed at Lambda Research to provide fully automatic multiple psi tilt residual stress measurement with automated diffraction peak acceptance, counting time, allowed error adjustment and complete correction for subsurface residual stress measurement.

Lambda Research has acquired two additional GE/Diano horizontal diffractometers. The instruments are being automated and modified for residual stress and retained austenite measurements on massive samples, with an x-ray beam clearance of over 8 in. When the new equipment is fully operational, Lambda Research will have seven automated diffractometers running under the control of the central laboratory computer system.

NEW PERSONNEL

Jerome Gnoose has joined our staff as a laboratory technician. Jerry has an M.S. Degree from the University of Southern Mississippi in Geology. Natalie Mainprize, who joined our staff as an administrative secretary, has completed several years towards a degree in business management.

AIME CONFERENCE PAPER

Paul Prevèy of Lambda Research will be presenting a paper at the TMS annual meeting in Phoenix, AZ on January 25. The paper, entitled, “Limitations of Non-Destructive Surface X-Ray Stress Measurement,” will outline the potential problems in using x-ray diffraction for surface residual stress measurement. The influence of secondary surface treatments on the shallow layer sampled by the x-ray beam, difficulties in interpreting results, and errors due to penetration of the x-ray beam into the subsurface stress gradient, will be covered.