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Standard Test Method for
X-Ray Determination of Retained Austenite in Steel with Near Random Crystallographic Orientation[1]

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INTRODUCTION

The volume percent of retained austenite (face-centered cubic phase) in steel is determined by comparing the integrated chromium or molybdenum X-ray diffraction intensity of ferrite (body-centered cubic phase) and austenite phases with theoretical intensities. This method should be applied to steels with near random crystallographic orientations of ferrite and austenite phases because preferred crystallographic orientations can drastically change these measured intensities from theoretical values. Chromium radiation was chosen to obtain the best resolution of X-ray diffraction peaks for other crystalline phases in steel such as carbides. No distinction has been made between ferrite and martensite phases because the theoretical X-ray diffraction intensities are nearly the same. Hereafter, the term ferrite can also apply to martensite. This test method has been designed for unmodified commercial X-ray diffractometers or diffraction lines on film read with a densitometer.

Other types of X-radiations such as cobalt or copper can be used, but most laboratories examining ferrous materials use chromium radiation for improved X-ray diffraction peak resolution or molybdenum radiation to produce numerous X-ray diffraction peaks. Because of special problems associated with the use of cobalt or copper radiation, these radiations are not considered in this test method.

1. Scope

1.1 This test method covers the determination of retained austenite phase in steel using integrated intensities (area under peak above background) of X-ray diffraction peaks using chromium K_α or molybdenum K_α X-radiation.

1.2 The method applies to carbon and alloy steels with near random crystallographic orientations of both ferrite and austenite phases.

1.3 This test method is valid for retained austenite contents from 1 % by volume and above.

1.4 If possible, X-ray diffraction peak interference from other crystalline phases such as carbides should be eliminated from the ferrite and austenite peak intensities.

1.5 Substantial alloy contents in steel cause some change in peak intensities which have not been considered in this method. Application of this method to steels with total alloy contents exceeding 15 weight % should be done with care. If necessary, the users can calculate the theoretical correction factors to account for changes in volume of the unit cells for austenite and ferrite resulting from variations in chemical composition.

1.6 *Units*—The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

1.7 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.8 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Significance and Use

2.1 *Significance*—Retained austenite with a near random crystallographic orientation is found in the microstructure of heat-treated low-alloy, high-strength steels that have medium

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[1] This test method is under the jurisdiction of ASTM Committee E04 on Metallography and is the direct responsibility of Subcommittee E04.11 on X-Ray and Electron Metallography.

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(0.40 weight %) or higher carbon contents. Although the presence of retained austenite may not be evident in the microstructure, and may not affect the bulk mechanical properties such as hardness of the steel, the transformation of retained austenite to martensite during service can affect the performance of the steel.

2.2 *Use*—The measurement of retained austenite can be included in low-alloy steel development programs to determine its effect on mechanical properties. Retained austenite can be measured on a companion specimen or test section that is included in a heat-treated lot of steel as part of a quality control practice. The measurement of retained austenite in steels from service can be included in studies of material performance.

3. Principles for Retained Austenite Measurement by X-Ray Diffraction

3.1 A detailed description of a retained austenite measurement using X-ray diffraction is presented by the Society of Automotive Engineers.[2] Since steel contains crystalline phases such as ferrite or martensite and austenite, a unique X-ray diffraction pattern for each crystalline phase is produced when the steel sample is irradiated with X-irradiation. Carbide phases in the steel will also produce X-ray diffraction patterns.

3.2 For a randomly oriented specimen, quantitative measurements of the relative volume fraction of ferrite and austenite can be made from X-ray diffraction patterns because the total integrated intensity of all diffraction peaks for each phase is proportional to the volume fraction of that phase. If the crystalline phase or grains of each phase are randomly oriented, the integrated intensity from any single diffraction peak (*hkl*) crystalline plane is also proportional to the volume fraction of that phase:

$$I_{\alpha}^{\ hkl} = KR_{\alpha}^{\ hkl} V_{\alpha} / 2\mu$$

where:

$$K = \left(\frac{I_o e^4}{m^2 c^4}\right) \times \left(\frac{\lambda^3 A}{32\pi r}\right)$$

and

$$R_{\alpha}^{\ hkl} = \frac{1\left(/F/^{\,2}\, pLPe^{-2M}\right)}{v^{\,2}}$$

where:

$I_{\alpha}^{\ hkl}$ = integrated intensity per angular diffraction peak (*hkl*) in the α-phase,
I_o = intensity of the incident beam,
μ = linear absorption coefficient for the steel,
e,m = charge and mass of the electron,
r = radius of the diffractometer,
c = velocity of light,
λ = wavelength of incident radiation,
A = cross sectional area of the incident beam,
v = volume of the unit cell,
$/F/^2$ = structure factor times its complex conjugate,
p = multiplicity factor of the (*hkl*) reflection,
θ = Bragg angle,
LP = Lorentz Polarization factor for a Bragg-Brentano powder diffractometer is equal to $(1 + \cos^2 2\theta)/\sin^2 \theta \cos \theta$ for normal diffractometric analysis but becomes $(1 + \cos^2 2\alpha \cos^2 2\theta)/(\sin^2 \theta \cos \theta)(1 + \cos^2 2\alpha)$ when a monochromator is used in which diffraction by monochromator and specimen take place in the same plane; 2α is the diffraction angle of the monochromator crystal. If diffraction by the monochromator occurs in a plane perpendicular to the plane of specimen diffraction, then $\mathrm{LP} = (\cos^2 2\alpha + \cos^2 2\theta)/(\sin^2\theta \cos\theta)(1 + \cos^2 2\alpha)$,
e^{-2M} = Debye-Waller or temperature factor which is a function of θ where $M = B(\sin^2 \theta)/\lambda^2$, $B = 8\pi^2 (\mu_s)^2$, where $\mu_s^{\ 2}$ is the mean square displacement of the atoms from their mean position, in a direction perpendicular to the diffracting plane, and
V_{α} = volume fraction of the α-phase.

K is a constant which is dependent upon the selection of instrumentation geometry and radiation but independent of the nature of the specimen. The parameter, *R*, is proportional to the theoretical integrated intensity. The parameter, *R*, depends upon interplanar spacing (*hkl*), the Bragg angle, θ, crystal structure, and composition of the phase being measured. *R* can be calculated from basic principles.

3.3 For steel containing only ferrite (α) and austenite (γ) and no carbides, the integrated intensity from the (*hkl*) planes of the ferrite phase is expressed as:

$$I_{\alpha}^{\ hkl} = KR_{\alpha}^{\ hkl} V_{\alpha} / 2\mu$$

3.3.1 A similar equation applies to austenite. We can then write for any pair of austenite and ferrite *hkl* peaks:

$$I_{\alpha}^{\ hkl} / I_{\gamma}^{\ hkl} = \left[\left(R_{\alpha}^{\ hkl} / R_{\gamma}^{\ hkl}\right)\left(V_{\alpha} / V_{\gamma}\right)\right]$$

3.3.2 The above ratio holds if ferrite or martensite and austenite are the only two phases present in a steel and both phases are randomly oriented. Then:

$$V_{\alpha} + V_{\gamma} = 1$$

3.3.3 The volume fraction of austenite (V_{γ}) for the ratio of measured integrated intensities of ferrite and austenite peak to *R*-value is:

$$V_{\gamma} = \left(I_{\gamma} / R_{\gamma}\right) / \left[\left(I_{\alpha} / R_{\alpha}\right) + \left(I_{\gamma} / R_{\gamma}\right)\right] \tag{1}$$

3.3.4 For numerous ferrite and austenite peaks each ratio of measured integrated intensity to *R*-value can be summed:

$$V_{\gamma} = \left(\frac{1}{q} \sum_{j=1}^{q} I_{\gamma j} / R_{\gamma j}\right) / \left[\left(\frac{1}{P} \sum_{i=1}^{P} I_{\alpha i} / R_{\alpha i}\right) + \left(\frac{1}{q} \sum_{j=1}^{q} I_{\gamma j} / R_{\gamma j}\right)\right] \tag{2}$$

where:

q = total number of austenite peaks, and
P = total number of ferrite peaks.

3.3.5 If carbides are present:

$$V_{\alpha} + V_{\gamma} + V_{c} = 1$$

3.3.6 Then the volume fraction of austenite (V_{α}) for the ratio of measured ferrite and austenite integrated intensity to *R*-value is:

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[2] *Retained Austenite and Its Measurement by X-ray Diffraction* , SAE Special Publication 453, Society of Automotive Engineers (SAE), 400 Commonwealth Dr., Warrendale, PA 15096-0001, http://www.sae.org.

$$V_\gamma = (1 - V_c)(I_\gamma / R_\gamma) / [(I_\alpha / R_\alpha) + (I_\gamma / R_\gamma)] \quad (3)$$

3.3.7 For numerous ferrite and austenite peaks the ratio of measured integrated intensity to *R*-values can be summed:

$$V_\gamma = (1 - V_c) \quad (4)$$

$$\left[\left(\frac{1}{q} \sum_{j=1}^{q} (I\gamma j / R\gamma j) \right) \right] / \left[\frac{1}{P} \sum_{i=1}^{p} (I_a i / R_a i) + \frac{1}{q} \sum_{j=1}^{q} (I_r i / R_r i) \right]$$

3.4 The volume fraction of carbide, V_c, should be determined by chemical extraction or metallographic methods. Adequate X-ray diffraction peak resolution for the identification of carbide peaks is required to avoid including carbide peaks in the retained austenite measurement.

4. Procedure

4.1 *Specimen Preparation:*

4.1.1 Specimens for the X-ray diffractometer shall be cut with a minimum amount of heat effect. Saw cutting rather than abrasive wheel cutting should be for specimen removal whenever it is practical.

NOTE 1—Since most steels containing retained austenite are relatively hard, abrasive cutoff wheels are frequently used. If adequate cooling is not used, heat effects from abrasive cutoff wheels can be substantial and, in some cases, can transform retained austenite.

NOTE 2—Rough machining using a milling tool or coarse grinding can deform the surface and transform some of the retained austenite to a depth that is greater than the surface depth analyzed. Final milling or rough grinding cuts limited to a depth of 0.010-in (0.254 mm) or less will reduce the depth of deformation.

4.1.2 Standard metallographic wet-grinding and polishing methods shall be used to prepare specimens for X-ray analysis. Grit reductions of 80, 120, 240, 320, 400, and 600 silicon carbide or alumina abrasives may be used, but other valid grit combinations may also be used.

4.1.2.1 The final surface polish shall be 2.36×10^{-4}in. (6-μm) diamond or an equivalent abrasive polish.

4.1.2.2 Specimen etching, observation for heat effects, and repolishing should be conducted as a safeguard.

4.1.3 Since deformation caused by dull papers or overpolishing can transform some of the retained austenite, electrolytic polishing or chemical polishing of initial specimens of each grade and condition should be used to verify proper metallographic specimen preparation. Standard chromic-acetic acid for electropolishing 0.005-in. (0.127 mm) from specimens ground to 600 grit or specific chemical polishing solutions for a particular grade of steel polished to a 2.36×10^{-4}in. (6-μm) finish may be used to verify the metallographic polish. Hot-acid etching should not be used because of selective etching of one phase or along a preferred crystallographic direction.

4.1.4 If measurement of the retained austenite content on the surface of a specimen is desired and the specimen can be mounted in the diffraction system, no preparation is needed.

4.1.5 Specimen size shall be large enough to contain the X-ray beam at all angles of 2θ required for the X-ray diffraction analysis to prevent errors in the analysis.

NOTE 3—In most cases, an area of 1 in.^{2} (645.16 mm^2) is sufficient, but specimen size depends upon the dimensions of the incident X-ray diffraction.

4.1.5.1 When using molybdenum radiation, select peaks in the range from 28° to 40° 2θ for best results.

4.2 *X-Ray Equipment:*

4.2.1 Any diffraction system may be used that consists of an x-ray source, an angular measurement capability, and an x-ray detection system. The system shall be capable of obtaining the entire diffraction peak along with adjacent background levels, capable of detecting at least two separate austenite reflections and a ferrite reflection, and capable of normalizing any equipment-specific intensity biases not accounted for by R-factors. Two separate ferrite reflections should be measured; however, in alloys with known carbide interference, only the unaffected ferrite reflection may be measured.

4.2.2 A chromium X-ray source with a vanadium metal or compound filter to reduce the K_β radiation is should be used.

NOTE 4—Chromium radiation produces a minimum of X-ray fluorescence of iron. Chromium radiation provides for the needed X-ray diffraction peak resolution and allows for the separation of carbide peaks from austenite and ferrite peaks.

4.2.3 Other radiation such as copper, cobalt, or molybdenum may be used, but none of these provide the resolution of chromium radiation.

NOTE 5—Copper radiation is practical only when a diffracted-beam monochromator is employed, because iron X-ray fluorescence will obscure the diffracted peaks.

4.2.4 A molybdenum source with a zirconium filter may be used to produce a large number of X-ray diffraction peaks.

4.3 *X-Ray Method*—X-ray diffraction peaks from other crystalline phases such as carbides shall be separated from austenite and ferrite peaks. The linearity of the chart recorder or photographic film shall be verified prior to utilizing this method for older systems using these recording media.

4.3.1 Entire diffraction peaks minus background under the peaks shall be recorded to obtain integrated peak intensities. Peaks without carbide or second phase interference may be scanned, and the total peak plus background recorded. Obtain background counts by counting on each side of the peak for one-half of the total peak counting time. Subtract the total background from peak plus background to obtain the integrated intensity. Alternatively, software supplied with the diffractometer may be used. In general, the diffractometer scanning rate should be 0.5°2θ/min or less to define the peaks for austenite contents of less than 5 %.

4.3.2 Where carbide or other phase X-ray diffraction peak interference exists, planimeter measurements of area under the austenite and ferrite peaks on X-ray diffraction charts may be used to obtain integrated intensity. Alternatively, software supplied with the diffractometer may be used.

NOTE 6—Details of the correction techniques are outside the scope of this test method. Carbide interference with austenite and ferrite peaks of the more common carbides is shown in Fig. 1.

4.3.3 The integrated intensity may be determined by cutting peak areas from the charts and weighing them with an analytical balance.

4.3.4 Assuming a 10 % variation in each peak intensity, chromium peak ratios of integrated intensities (areas under the peaks minus background) for the (220) austenite peak relative