ASTM INTERNATIONAL

Designation: E975 – 13

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

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 practice 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 practice.

1. Scope

1.1 This practice 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 practice 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 and health practices and determine the applicability of regulatory limitations prior to use.*

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 (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.

TABLE 1 Calculated Theoretical Intensities Using Chromium K$_\alpha$ Radiation^A

hkl	Sinθ/λ	θ	f	Δf'	Δf''	/F/²	LP	P	T^B	N²	R
(α iron, body-centered cubic, unit-cell dimension a$_o$ = 2.8664Å):											
110	0.24669	34.41	18.474	−1.6	0.9	1142.2	4.290	12	0.9577	0.001803^B	101.5^C
200	0.34887	53.06	15.218	−1.6	0.9	745.0	2.805	6	0.9172	0.001803^B	20.73^C
211	0.42728	78.20	13.133	−1.6	0.8	534.6	9.388	24	0.8784	0.001803^B	190.8^C
(γ iron, face-centered cubic, unit-cell dimension a$_o$ = 3.60Å):											
111	0.24056	33.44	18.687	−1.6	0.9	4684.4	4.554	8	0.9597	0.0004594^B	75.24^C
200	0.27778	39.52	17.422	−1.6	0.9	4018.3	3.317	6	0.9467	0.0004594^B	34.78^C
220	0.39284	64.15	14.004	−1.6	0.8	2472.0	3.920	12	0.8962	0.0004594^B	47.88^C

^{A} Data from "International Tables for X-Ray Crystallography," *Physical and Chemical Tables*, Vol III, Kynoch Press, Birmingham, England, 1962, pp. 60, 61, 210, 213; Weighted K$_{\alpha 1}$ and K$_{\alpha 2}$ value used (λ = 2.29092Å).
^{B} Temperature factor ($T = e^{-2M}$ where $M = B(\sin^2 \theta)/\lambda^2$ and $2B = 0.71$. Also N is the reciprocal of the unit-cell volume.
^{C} Calculated intensity includes the variables listed that change with X-ray diffraction peak position.

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 sample 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 sample, 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:

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

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,

^{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.

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 which is equal to $(1 + \cos^2 2\theta)/\sin^2 \theta \cos \theta$ for normal diffractometric analysis but becomes $(1 + \cos\theta^2 2\alpha \cos^2 2\theta)/(\sin^2 \theta \cos \theta)(1 + \cos^2 2\alpha)$ when a monochromator is used in which diffraction by monochromator and sample 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 sample diffraction, then LP $= (\cos^2 2\alpha + \cos^2 2\theta)/\sin^2 \theta \cos (1 + \cos^2 2\alpha)$,
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 μ_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-plane.

K is a constant which is dependent upon the selection of instrumentation geometry and radiation but independent of the nature of the sample. 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:

$$V_\gamma = (I_\gamma / R_\gamma)/[(I_\alpha/R_\alpha) + (I_\gamma/R_\gamma)] \qquad (1)$$

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

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

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:

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

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

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

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

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 *Sample Preparation:*

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

4.1.2 Rough grinding using a milling tool or high-pressure 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 should reduce the depth of deformation.

4.1.3 Standard metallographic wet-grinding and polishing methods shall be used to prepare samples 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. A final surface polish of 2.36×10^{-4}in. (6-μm) diamond or an equivalent abrasive polish is required. Sample etching, observation for heat effects, and repolishing is a recommended safeguard.

4.1.4 Since deformation caused by dull papers or over-polishing can transform some of the retained austenite, electrolytic polishing or chemical polishing of initial samples of each grade and condition should be used to verify proper metallographic sample preparation. Standard chromic-acetic acid for electropolishing 0.005-in. (0.127 mm) from samples 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 can be used to verify the metallographic polish. Hot-acid etching is not recommended because of selective etching of one phase or along a preferred crystallographic direction.

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

4.1.6 Sample size shall be large enough to contain the Xray beam at all angles of 2θ required for the X-ray diffraction analysis to prevent errors in the analysis. In most cases, an area of 1 in.^{2} (645.16 mm^2) is sufficient, but sample size depends upon the dimensions of the incident X-ray diffraction. 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 must 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.

4.2.2 A chromium X-ray source with a vanadium metal or compound filter to reduce the K_β radiation is recommended. Chromium radiation produces a minimum of Xray 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 can be used, but none of these provide the resolution of chromium radiation. 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 is used to produce a large number of X-ray 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 can be scanned, and the total peak plus background recorded. Background counts are obtained by counting on each side of the peak for one-half of the total peak counting time. Total background is subtracted from peak plus background to obtain the integrated intensity. Alternatively, software supplied with the diffractometer can be used. In general, a diffractometer scanning rate of 0.5°2θ/min or less is recommended 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 can be

used to obtain integrated intensity. Alternatively, software supplied with the diffractometer can be used. Carbide interference with austenite and ferrite peaks of the more common carbides is shown in Fig. 1.

4.3.3 Another method of determining integrated intensity involves 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 to (200) austenite peak shall range from 1.1 to 1.7 to satisfy the requirement of this practice for a near-random orientation of austenite. Equivalent molybdenum peak ratios range from 0.7 to 0.5.

4.3.5 Assuming a 10 % variation in each peak intensity, chromium peak ratios of integrated intensities for the (211) ferrite peak relative to the (200) ferrite peak range from 8 to 11 to satisfy the requirement of this practice for a near-random orientation of ferrite. Equivalent molybdenum peak ratios range from 1.5 to 2.2.

4.3.6 When either the austenite peak ratio or the ferrite peak ratio is above or below the specified range, this method cannot be used.

4.3.7 Three austenite peaks (111), (200), and (220) and three ferrite peaks (110), (200), and (211) can be obtained with chromium radiation on most X-ray diffractometers. Chromium X-ray diffraction limitations may prevent obtaining the entire peak profile for the (211) peak. In this case, the half-peak

profile can be doubled with some error in background. A densitometer reading of film from a Debye Scherrer camera may also be used. In many cases, the (111) austenite and (110) ferrite peaks interfere with each other and cannot be resolved. Four peak ratios of the resolved ferrite to austenite peaks are adequate to determine the retained austenite content of near randomly oriented samples.

4.3.8 Calculated theoretical intensities, R, for ferrite and austenite peaks are listed in Table 1 using chromium K_α radiation and in Table 2 using molybdenum K_α radiation.

4.3.9 The retained austenite content can be estimated from a number of ferrite and austenite intensity to R-value ratios using Eq 2 assuming no carbides are present.

4.3.10 If the volume fraction of carbide has been determined, the volume fraction of austenite can be determined from Eq 3 for a single set of peaks or from Eq 4 for more than one set of peaks using the theoretical intensities listed in Table 1 for chromium radiation or in Table 2 for molybdenum radiation.

5. Example

5.1 Using chromium radiation, the integrated intensity (area of peak above background) for ferrite peaks (200) and (211) and for retained austenite peaks (200) and (220) were determined. Values of R for each peak were obtained from Table 1.

5.1.1 The measured integrated intensities and values of R are illustrated in Table 3.

5.1.2 From Eq 1 for the α (200) and γ (200) peaks:
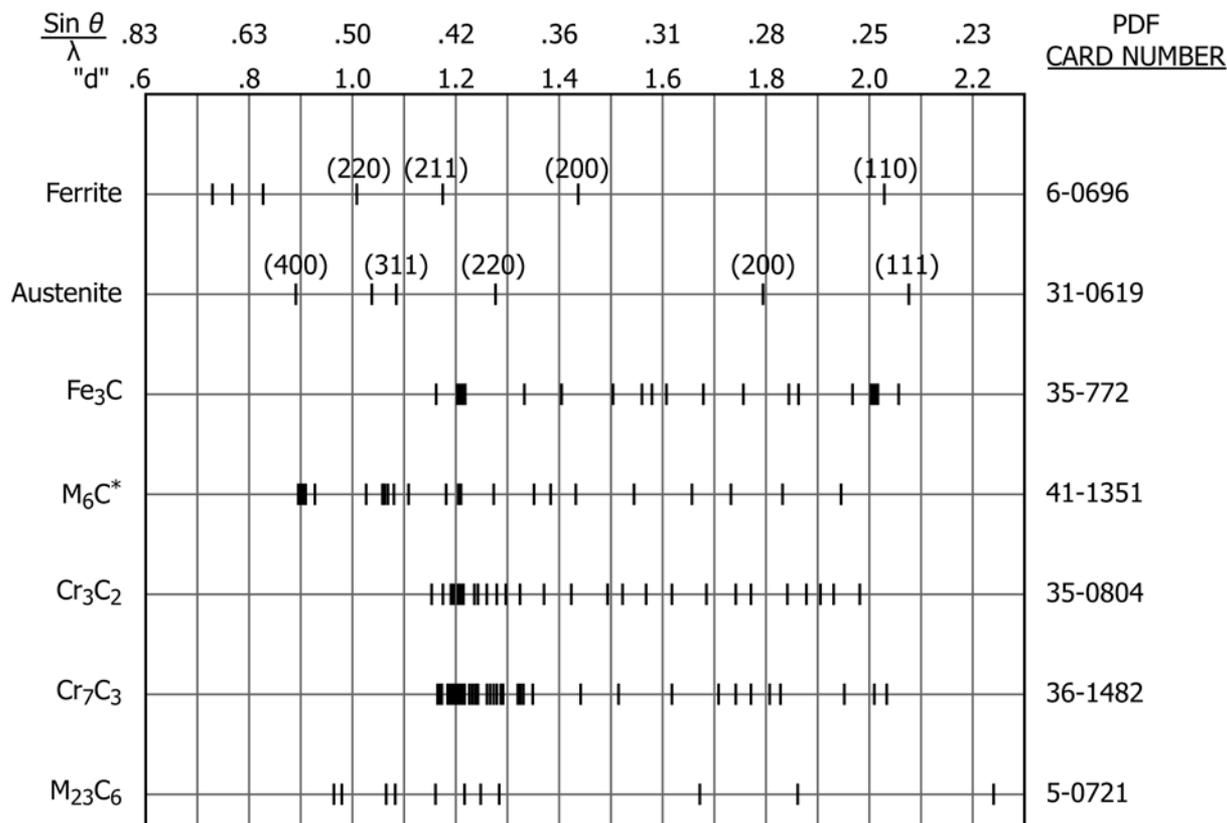
NOTE 1—"M" represents more than one type of metal.

FIG. 1 Example of Carbide Interference

TABLE 2 Calculated Theoretical Intensities Using Molybdenum K_α Radiation[A]

hkl	Sinθ/λ	θ	f	Δf'	Δf''	/F/^{2}	LP	P	T[B]	N^2 [B]	R[C]
(α iron, body-centered cubic, unit-cell dimension a_o = 2.8664Å):											
110	0.24669	10.10	18.474	0.4	1.0	1428.9	62.15	12	0.9577	0.001803	1840
200	0.34887	14.36	15.218	0.4	0.9	978.9	29.71	6	0.9172	0.001803	288.6
211	0.42728	17.68	13.133	0.4	0.9	735.8	18.95	24	0.8784	0.001803	530.0
220	0.49338	20.53	11.652	0.3	0.9	574.6	13.62	12	0.8413	0.001803	142.5
310	0.55161	23.08	10.542	0.3	0.9	473.4	10.47	24	0.8057	0.001803	172.8
222	0.60426	25.43	9.685	0.3	0.9	402.0	8.396	8	0.7716	0.001803	37.56
321	0.65268	27.64	9.012	0.3	0.9	350.1	6.949	48	0.7390	0.001803	155.6
400	0.69774	29.73	8.480	0.3	0.9	311.6	5.892	6	0.7078	0.001803	14.06
330	0.74006	31.73	8.054	0.3	0.9	282.4	5.099	12	0.6778	0.001803	21.12
411								24			42.23
420	0.78010	33.67	7.713	0.3	0.9	260.1	4.489	24	0.6492	0.001803	32.80
332	0.81817	35.55	7.437	0.3	0.9	242.7	4.017	24	0.6217	0.001803	26.23
422	0.85455	37.40	7.211	0.3	0.9	228.9	3.647	24	0.5954	0.001803	21.51
431	0.88945	39.21	7.022	0.3	0.9	217.7	3.360	48	0.5702	0.001803	36.10
510								24			18.05
521	0.95542	42.77	6.719	0.3	0.9	200.3	2.972	48	0.5230	0.001803	26.94
440	0.98675	44.53	6.591	0.3	0.9	193.2	2.853	12	0.5009	0.001803	5.97
433	1.01712	46.29	6.472	0.3	0.9	186.7	2.775	24	0.4797	0.001803	10.75
530								24			10.75
442	1.04661	48.06	6.357	0.3	0.9	180.5	2.735	24	0.4594	0.001803	9.81
600								6			2.45
532	1.07529	49.84	6.244	0.3	0.9	174.5	2.730	48	0.4400	0.001803	18.14
611								24			9.07
620	1.10322	51.63	6.133	0.3	0.8	168.1	2.759	24	0.4214	0.001803	8.46
541	1.13047	53.46	6.022	0.3	0.8	162.4	2.822	48	0.4036	0.001803	16.01
622	1.15707	55.32	5.913	0.3	0.8	157.0	2.922	24	0.3865	0.001803	7.67
631	1.18307	57.22	5.805	0.3	0.8	151.6	3.061	48	0.3702	0.001803	14.87
444	1.20852	59.19	5.700	0.3	0.8	146.6	3.245	8	0.3545	0.001803	2.43
543	1.23344	61.23	5.598	0.3	0.8	141.7	3.484	48	0.3395	0.001803	14.51
550								12			3.63
710								24			7.25
640	1.25787	63.37	5.503	0.3	0.8	137.3	3.792	24	0.3252	0.001803	7.33
552	1.28183	65.64	5.414	0.3	0.8	133.2	4.193	24	0.3114	0.001803	7.53
633								24			7.53
721								48			15.05
642	1.30535	68.08	5.332	0.3	0.8	129.4	4.731	48	0.2983	0.001803	15.80
730	1.32846	70.76	5.258	0.3	0.8	126.1	5.489	24	0.2856	0.001803	8.55
651	1.37350	77.46	5.130	0.3	0.8	120.5	8.796	48	0.2620	0.001803	24.03
732								48			24.03
(γ iron, face-centered cubic, unit-cell dimension a_o = 3.60Å):											
111	0.24056	9.84	18.687	0.4	1.0	5845.0	65.51	8	0.9597	0.0004594	1351
200	0.27778	11.39	17.422	0.4	1.0	5098.0	48.43	6	0.9467	0.0004594	644.3
220	0.39284	16.21	14.004	0.4	0.9	3332.6	22.88	12	0.8962	0.0004594	376.7
311	0.46064	19.11	12.355	0.3	0.9	2575.3	15.97	24	0.8601	0.0004594	390.0
222	0.48113	19.99	11.908	0.3	0.9	2397.5	14.44	8	0.8484	0.0004594	107.9
400	0.55556	23.26	10.472	0.3	0.9	1869.5	10.29	6	0.8032	0.0004594	42.59
331	0.60540	25.48	9.668	0.3	0.9	1602.7	8.358	24	0.7709	0.0004594	113.9
420	0.62113	26.20	9.438	0.3	0.9	1530.2	7.849	24	0.7604	0.0004594	100.7
422	0.68041	28.92	8.674	0.3	0.9	1301.5	6.270	24	0.7199	0.0004594	64.77
333	0.72169	30.86	8.231	0.3	0.9	1177.4	5.423	8	0.6909	0.0004594	16.21
511								24			48.64
440	0.78567	33.94	7.670	0.3	0.9	1029.3	4.414	12	0.6452	0.0004594	16.16
531	0.82168	35.73	7.414	0.3	0.9	965.1	3.978	48	0.6192	0.0004594	52.42
442	0.83333	36.32	7.339			946.6	3.854	24	0.6108	0.0004594	24.57
600								6			6.14
620	0.87841	38.63	7.080	0.3	0.9	884.4	3.444	24	0.5782	0.0004594	19.42
533	0.91076	40.34	6.918	0.3	0.9	846.6	3.214	24	0.5549	0.0004594	16.65
622	0.92128	40.90	6.869	0.3	0.9	835.3	3.149	24	0.5474	0.0004594	15.88
444	0.96225	43.15	6.691	0.3	0.9	794.9	2.943	8	0.5182	0.0004594	4.46
551	0.99187	44.82	6.571	0.3	0.9	768.3	2.837	24	0.4973	0.0004594	11.95
711								24			11.95
640	1.00154	45.38	6.533	0.3	0.9	760.0	2.811	24	0.4906	0.0004594	11.56
642	1.03935	47.62	6.385	0.3	0.9	728.0	2.742	48	0.4644	0.0004594	20.44
553	1.06683	49.30	6.278	0.3	0.9	705.3	2.728	24	0.4457	0.0004594	9.46
731								48			18.91
800	1.11111	52.15	6.101	0.3	0.8	665.8	2.773	6	0.4162	0.0004594	2.12
733	1.13685	53.90	5.997	0.3	0.8	644.7	2.842	24	0.3995	0.0004594	8.07
644	1.14531	54.48	5.962	0.3	0.8	637.6	2.873	24	0.3940	0.0004594	7.96
820								24			7.96
660	1.17851	56.88	5.824	0.3	0.8	610.3	3.033	12	0.3730	0.0004594	3.81
822								24			7.61
555	1.20281	58.74	5.723	0.3	0.8	590.7	3.199	8	0.3580	0.0004594	2.49
751								48			14.92
662	1.21081	59.37	5.691	0.3	0.8	584.5	3.264	24	0.3531	0.0004594	7.43

TABLE 2 *Continued*

hkl	Sinθ/λ	θ	f	Δf'	Δf''	/F/²	LP	P	T^B	N² ^B	R^C
840	1.24226	61.99	5.564	0.3	0.8	560.4	3.585	24	0.3343	0.0004594	7.41
753	1.26534	64.06	5.475	0.3	0.8	543.8	3.905	48	0.3209	0.0004594	15.03
911								24			7.51
842	1.27294	64.78	5.446	0.3	0.8	538.5	4.030	48	0.3165	0.0004594	15.15
664	1.30289	67.81	5.339	0.3	0.8	519.0	4.666	24	0.2996	0.0004594	8.00
931	1.32492	70.32	5.268	0.3	0.8	506.3	5.352	48	0.2876	0.0004594	17.18
844	1.36083	75.27	5.164	0.3	0.8	487.9	7.391	24	0.2685	0.0004594	10.68
755	1.38193	79.15	5.107	0.3	0.8	478.0	10.26	24	0.2577	0.0004594	13.93
771								24			13.93
933								24			13.93

^A Data from "International Tables for X-Ray Crystallography," *Physical and Chemical Tables*, Vol. III, Kynoch Press, Birmingham, England, 1962, pp 60, 61, 210, 213; Weight $K_{\alpha 1}$ and $K_{\alpha 2}$ value used ($\lambda = 0.71069\text{Å}$).
^B Temperature factor ($T = e^{-2\,M}$) where $M = B(\sin^2\theta)/\lambda^2$ and $2B = 0.71$. Also N is the reciprocal of the unit-cell volume.
^C Calculated intensity, R, includes the variables listed that change with X-ray diffraction peak position.

TABLE 3 Measured Integrated Intensities and Values of K

Peak –	α (200)	γ (200)	γ(220)	α (211)
I	1.00	1.00	1.41	9.50
R	20.73	34.78	47.88	190.8
I:R	0.04824	0.02875	0.02945	0.0497

$$V_\gamma = \frac{\dfrac{1.00}{34.78}}{\dfrac{1.00}{20.73}+\dfrac{1.00}{34.78}} = 0.373 \text{ or } 37.3\,\% \text{ retained austenite} \quad (5)$$

5.1.3 From Eq 2 for all four peaks:

$$V_\gamma = \frac{\frac{1}{2}(0.02875+0.02945)}{\frac{1}{2}(0.04824+0.04979)+\frac{1}{2}(0.02875+0.02945)} = 0.373 \quad (6)$$

6. Precision and Bias

6.1 *Precision*—On the basis of an interlaboratory test program this method produces an intralaboratory repeatability of 3 % and an interlaboratory reproducibility of 4 % both at the 95 % confidence level.[3] These estimates were derived from measurements of specimens containing about 2.5 %, 5 %, and 15 % by volume austenite in a medium carbon steel. These measures of precision will be degraded with increasing alloy content and also near the minimum detectability limit of 2 %.

[3] Hinton, R. W., "Interlaboratory Evaluation of ASTM Practice for X-ray Determination of Retained Austenite in Steel with Near-random Crystallographic Orientation" (Practice E975), *Journal of Testing and Evaluation*, Vol 15, No. 2 March 1987, pp. 95–100.

6.2 *Bias*—No bias estimate is available because there is no independent test method to determine an accepted reference value from retained austenite. Use of this practice produces comparable values from one facility to another while utilizing a variety of X-ray diffraction instruments.

7. Report

7.1 For this practice, the accompanying report shall contain the following:

7.1.1 Name of the organization and person performing the analysis.

7.1.2 Date the analysis was completed.

7.1.3 Material type.

7.1.4 Specimen description, size, and location.

7.1.5 X ray system used for the analysis.

7.1.6 Radiation used for the analysis.

7.1.7 Beam size or collimator used.

7.1.8 Depth where analysis was performed.

7.1.9 Specimen rotation (Yes / No).

7.1.10 Specimen translation (Yes / No).

7.1.11 The austenite and ferrite peaks used for the analysis.

7.1.12 Approximate carbide volume percent.

7.1.13 Carbide correction (Yes / No).

7.1.14 Volume percent retained austenite.

7.2 Any other information regarding the test procedures deemed necessary shall be based upon purchaser-testing laboratory agreements.

8. Keywords

8.1 austenite; crystallographic orientation; ferrite; martensite; retained austenite; X-ray diffraction