U. S. Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate

Standard Reference Material 493

Spheroidized Iron Carbide (Fe_3C) in Ferrite

This Standard Reference Material (SRM) consists of spheroidized iron carbide (Fe_3C) in ferrite and is issued primarily for the calibration of x-ray diffraction and Mössbauer equipment. SRM 493 is a wafer approximately 29 mm (1 1/8 in) square and 2.4 mm (0.094 in) thick. The determination of iron carbide is often important in itself, and the accurate determination of retained austenite may depend in part upon the capability to determine iron carbide. This standard, when used in conjunction with austenite standards, will allow calibration of equipment used to analyze the relative amounts of two of the most important microstructural constituents in steel (ferrite and austenite).

The certified value for the amount of iron carbide as a percent by volume is:

$$14.23 \pm 0.30.$$

This value is the average of the results of a quantitative optical method involving 1620 measurements. The probability is about 95 percent that the average iron carbide concentration in any wafer is within the indicated interval.

The overall direction and coordination of the material preparation and the technical measurements leading to certification of the iron carbide percent by volume were performed under the chairmanship of T.R. Shives in association with M.R. Meyerson and W.F. Gerhold, NBS Metallurgy Division.

The statistical analysis was performed by J. Mandel.

A Quantitative Television Microscope (QTM) was used by T.R. Shives and L.C. Smith for the quantitative determination of the volume percent iron carbide. Thirty fields (approximately 0.008 mm^2 per field) from each of three cross sections from each of eighteen of the approximately 390 wafers produced were analyzed. These eighteen wafers were chosen by a statistically based random selection. The standard error of the average of the 1620 measurements was 0.035 percent Fe_3C. As a check on the accuracy of the QTM method, 36 measurements were made by means of the NBS Electromechanical Drum Scanner (EDS) by L.C. Smith and G.A. Moore. Six fields from each of six of the cross sections analyzed by the QTM method were analyzed by the EDS method. The average result obtained was 13.98 percent Fe_3C with a standard error of 0.24 percent Fe_3C.

Essentially, all the carbon present in this material should be in the form of iron carbide. (There is a very small amount of carbon soluble in ferrite at equilibrium at room temperature.) Based on this, the carbon analysis indicates that the iron carbide should range between 14.0 and 14.5 volume percent. This provides an additional check on the accuracy of the certified value.

A relatively high-purity iron-carbon alloy was chosen for this standard to avoid the formation of carbides other than iron carbide. The material was prepared by alloying carbon with molten electrolytic iron under vacuum. The resulting single ingot was hot forged to a bar (about 31 mm square). The melting and forging operations were done by the Naval Research Laboratory in Washington, DC.

The forged ingot was given a homogenization treatment and sliced into wafers which were heat treated to produce spheroidized iron carbide in a ferrite matrix. After heat treatment, both faces of the wafers were surface ground.

The wafers are issued with surface ground faces. For x-ray work, the face to be used must be properly prepared in accordance with good metallographic practice. Picral is recommended as an etchant. When all effects of grinding and flowed metal have been removed, the wafer is ready for use. It may be used for x-ray work in either the etched or unetched condition.

(over)

Mössbauer Data

The Mössbauer spectrum from SRM 493 consists of two overlapping six-line magnetic hyperfine spectra, one from the ferrite matrix and one from the spheroidized Fe_3C. This SRM may be used to calibrate Mössbauer effect spectrometers operating in the backscatter mode with detectors set to detect either the 14 keV gamma-ray or the 6.3 keV x-ray of ^{57}Fe.

Certified Values:

Ferrite Spectrum

Isomer shift (referred to pure iron as zero)	0.000 ± 0.005 mm S^{-1}
Magnetic field splitting	33.0 ± 0.1 T
Quadrupole splitting	0.00 ± 0.01 mm S^{-1}

Iron Carbide Spectrum

Isomer shift (referred to pure iron as zero, positive velocity refers to source and absorber approaching each other)	0.17 ± 0.01 mm S^{-1}
Magnetic field splitting	20.9 ± 0.3 T
Quadrupole splitting	0.00 ± 0.01 mm S^{-1}
Ratio of the Fe_3C spectral area to the total spectral area	0.16 ± 0.02

These results are based on a series of 30 measurements on seven randomly selected samples.

Recommendations for use:

The following parameters will give best results when using the Mössbauer effect to determine the amount of Fe_3C present in SRM 493 or an unknown sample:

(1) use the constant acceleration mode with a velocity range of ± 6 mm S^{-1} or greater;

(2) use a source containing at least 15 mCi of ^{57}Co in a solid matrix such as Pd or Cr;

(3) use a solid angle of gamma-ray irradiation from the source less than 0.03 steradians;

(4) use a computer program to least squares fit the spectrum as the sum of two magnetic hyperfine field spectra, one from the ferrite and one from the Fe_3C;

(5) there is a small (about one percent) contribution to the spectrum from retained austenite on the surface of the SRM which should be taken into account in the computer fit to the spectrum in order to improve the accuracy.

Because of this retained austenite, do not use conversion electron detection to obtain the Mössbauer spectrum.

Useful References:

L.H. Bennett, I. Weisman, and L.J. Swartzendruber, "Nuclear Magnetic Resonance and Mössbauer Effect", Techniques of Metals Research Vol. VI, part 2, E. Pasaglia, Editor, Interscience, N.Y. 1972.

L.J. Swartzendruber and L.H. Bennett, "Retained Austenite Developed During Surface Grinding of a Carbon Steel", Scripta Met. 6, 737 (1972).

L.J. Swartzendruber, L.H. Bennett, E.A. Schoeffer, W.T. DeLong, and H.C. Campbell, "Mossbauer-Effect Examination of Ferrite in Stainless Steel Welds and Castings", Welding Journal, January 1974.

W. Wilson and L.J. Swartzendruber, "A Flexible Least Squares Mössbauer Effect Fitting Program", Computer Physics Commun. 7, 151 (1974).

The technical measurements and analyses leading to the certification of the Mössbauer data were performed by L.J. Swartzendruber of the NBS Metallurgy Division.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R.E. Michaelis and L.J. Kieffer.

March 7, 1985
Gaithersburg, MD 20899
(Revision of Certificate
dated 8-28-70)

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

Mössbauer Data

The Mössbauer spectrum from SRM 493 consists of two overlapping six-line magnetic hyperfine spectra, one from the ferrite matrix and one from the spheroidized Fe_3C. This SRM may be used to calibrate Mössbauer effect spectrometers operating in the backscatter mode with detectors set to detect either the 14 keV gamma-ray or the 6.3 keV x-ray of ^{57}Fe.

Certified Values:

Ferrite Spectrum

Isomer shift (referred to pure iron as zero)	0.000 ± 0.005 mm S^{-1}
Magnetic field splitting	33.0 ± 0.1 T
Quadrupole splitting	0.00 ± 0.01 mm S^{-1}

Iron Carbide Spectrum

Isomer shift (referred to pure iron as zero, positive velocity refers to source and absorber approaching each other)	0.17 ± 0.01 mm S^{-1}
Magnetic field splitting	20.9 ± 0.3 T
Quadrupole splitting	0.00 ± 0.01 mm S^{-1}
Ratio of the Fe_3C spectral area to the total spectral area	0.16 ± 0.02

These results are based on a series of 30 measurements on seven randomly selected samples.

Recommendations for use:

The following parameters will give best results when using the Mössbauer effect to determine the amount of Fe_3C present in SRM 493 or an unknown sample:

(1) use the constant acceleration mode with a velocity range of ±6 mm S^{-1} or greater;

(2) use a source containing at least 15 mCi of ^{57}Co in a solid matrix such as Pd or Cr;

(3) use a solid angle of gamma-ray irradiation from the source less than 0.03 steradians;

(4) use a computer program to least squares fit the spectrum as the sum of two magnetic hyperfine field spectra, one from the ferrite and one from the Fe_3C;

(5) there is a small (about one percent) contribution to the spectrum from retained austenite on the surface of the SRM which should be taken into account in the computer fit to the spectrum in order to improve the accuracy.

Because of this retained austenite, do not use conversion electron detection to obtain the Mössbauer spectrum.

Useful References:

L.H. Bennett, I. Weisman, and L.J. Swartzendruber, "Nuclear Magnetic Resonance and Mössbauer Effect", Techniques of Metals Research Vol. VI, part 2, E. Pasaglia, Editor, Interscience, N.Y. 1972.

L.J. Swartzendruber and L.H. Bennett, "Retained Austenite Developed During Surface Grinding of a Carbon Steel", Scripta Met. 6, 737 (1972).

L.J. Swartzendruber, L.H. Bennett, E.A. Schoeffer, W.T. DeLong, and H.C. Campbell, "Mössbauer-Effect Examination of Ferrite in Stainless Steel Welds and Castings", Welding Journal, January 1974.

W. Wilson and L.J. Swartzendruber, "A Flexible Least Squares Mössbauer Effect Fitting Program", Computer Physics Commun. 7, 151 (1974).

The technical measurements and analyses leading to the certification of the Mössbauer data were performed by L.J. Swartzendruber of the NBS Metallurgy Division.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R.E. Michaelis and L.J. Kieffer.

March 7, 1985
Gaithersburg, MD 20899
(Revision of Certificate
dated 8-28-70)

Stanley D. Rasberry, Chief
Office of Standard Reference Materials