National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 488

2 1/2% Austenite in Ferrite

This Standard Reference Material (SRM) is intended for use in the calibration of X-ray diffraction equipment that is used in determining the amount of retained austenite in hardened steels. X-ray diffraction procedures require accurate measurement of the integrated intensity for a number of selected peaks.[1] SRM 488 should serve calibration needs when measuring at low levels of retained austenite.

Each SRM specimen was certified by determining its nickel content by X-ray fluorescence. The calibration of the X-ray fluorescence measurement was based on the austenite content of 13 specimens that were measured using a quantitative microscope (QM). The certified value is accurate to within ± 0.3% austenite.

The certified austenite content (vol. %) is given on the side opposite the measured surface.[2] Rotation of the SRM is recommended to minimize the effects of surface inhomogeneity that may exist on the surface of the SRM. Damage to the certified surface renders the certification void.

The 310 stainless steel powder used in making this SRM was prepared at Federal Mogul Corp., Detroit, MI; the 430 stainless steel was prepared at the Hoeganaes Corp., Riverton, NJ. Final blending of the powder was performed at the Patterson-Kelley Co., Inc., East Stroudsburg, PA. Compacting and sintering operations were conducted at the National Institute of Standards and Technology (NIST).

The preparation of specimens and the technical measurements leading to certification were directed and coordinated by G.E. Hicho of the NIST Metallurgy Division.

The X-ray fluorescence measurements were performed by P.A. Pella of the NIST Inorganic Analytical Research Division.

Statistical support and consultation throughout the development of this SRM were provided by J.J. Filliben and S.D. Leigh of the NIST Statistical Engineering Division.

The technical and support aspects involved in the original preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by R.K. Kirby. Revision of this certificate was coordinated through the Standard Reference Materials Program by N.M. Trahey.

This Certificate of Analysis has undergone editorial revision to reflect program and organizational changes at NIST and at the Department of Commerce. No attempt was made to reevaluate the certificate values or any technical data presented on this certificate.

Gaithersburg, MD 20889
April 12, 1993
(Revision of certificate dated 10-6-83)

Thomas E. Gills, Acting Chief
Standard Reference Materials Program

(over)

SUPPLEMENTAL INFORMATION

The austenite content of this SRM is directly related to the nickel content (i.e., wt %* Ni) as a result of blending austenitic stainless steel powder (20.41% Ni) with ferritic stainless steel powder (0.09% Ni). This process permits the use of precise and rapid X-ray fluorescence analysis for the determination of nickel content, hence the austenite content, of each sintered specimen.

A calibration curve was established using 13 specimens selected randomly from the entire population of specimens produced. The Ni-K_α counts from X-ray fluorescence measurement for each of these specimens were converted to weight percent nickel. The specimens were then etched so that for a corresponding area ($\sim$ 90%), the area percent austenite could be obtained using a quantitative microscope (QM). The standard deviation for repeated QM determinations was typically 0.28 area percent austenite. The area percent austenite for each etched calibration sample was then plotted versus its previously determined X-ray fluorescence value and a linear calibration curve was obtained using DATAPLOT (a least-square regression routine developed at NIST). The residual standard deviation for the calibration curve was 0.18 area percent austenite.

Each specimen was observed for a total of 30 s for Ni, Cr, and Fe. The concentration of these elements was determined, using combined Rasberry-Heinrich procedures and the Naval Research Laboratory (NRLXRF) programs for correction of interelement effects. Typically, the standard deviation for the uncertainty due to random error only among the X-ray fluorescence determinations for a single check sample, was 0.02 wt % Ni. The relative standard deviation for the same sample was approximately 0.9%. While they are believed to be small, systematic errors for both the X-ray fluorescence and QM are included in the uncertainty of the certified values. A comparison of the measurements for the same sample using X-ray fluorescence and atomic absorption spectrometry indicated that no systematic error could be detected within the precision of the x-ray fluorescence measurements at the nickel levels observed. A possible source of systematic error in the QM determination is the porosity of the specimen. An analysis of these errors indicates that the stated austenite content is accurate within $\pm$ 0.3%.

*wt % = mg/kg x 10^{-4}

REFERENCES

[1] Bechtold, C.J., An X-Ray Diffraction Method for Determining the Amount of Austenite in an Austenite-Ferrite Mixture, Natl. Bur. Stand., (U.S.) Tech. Note 109, February, 1972.

[2] Hicho, G.E., and Eaton, E.E., Standard Reference Materials: A Standard Reference Material Containing 2.5 Percent Austenite, (SRM 488), NBS Spec. Publ. 260-86 (December, 1983).