U.S. Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards 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 measurements of the integrated intensity for a number of selected peaks. See Technical Note 709. SRM 488 should serve the calibration needs when measuring at low levels of retained austenite.

The certified austenite content (volume percent) is given on the side opposite the measured surface. 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.

Each specimen of SRM 488 was certified by determining its nickel content by x-ray fluorescence. The calibration of the XRF 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 percent austenite.

The preparation of specimens and the technical measurements leading to certification were directed and coordinated by G. E. Hicho of the Fracture and Deformation Division, Center for Materials Science.

The 310 stainless steel powder used in making SRM 488 was prepared at Federal Mogul Corporation, Detroit, Michigan, while the 430 stainless steel was prepared at the Hoeganaes Corporation, Riverton, New Jersey. Final blending of the powder was done at the Patterson-Kelley Co., Inc., East Stroudsburg, Pennsylvania. Compacting and sintering operations were conducted at the National Bureau of Standards.

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. K. Kirby.

Washington, D.C. 20234
October 6, 1983

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

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Supplementary Information

The austenite content of this SRM is directly related to the nickel content (i.e., weight percent nickel) 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 pressed and 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 measurements for each of these specimens were converted to weight percent nickel. The specimens were then etched so that for a corresponding area (about 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 NBS by J. J. Filliben). The residual standard deviation for the calibration curve was 0.18 area-percent austenite. Filliben and S. Leigh, both of the Statistical Engineering Division, served as statistical consultants throughout the development of the SRM.

The x-ray fluorescence measurements were performed by P. Pella, Gas and Particulate Science Division. Each specimen was observed for a total of thirty seconds 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 inter-element effects. Typically, the standard deviation for the uncertainty due to random error only among the XRF determinations for a single check sample, was 0.02 weight percent nickel. The relative standard deviation for the same sample was approximately 0.9%. While they are believed to be small, systematic errors for both the XRF and QM are included in the uncertainty of the certified values. A comparison of the measurements for the same sample using XRF and atomic absorption spectrometry indicated that no systematic error could be detected within the precision of the XRF 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 ± 0.3 percent.