U. S. Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate

Standard Reference Material 486

15% Austenite in Ferrite

This Standard Reference Material is intended for use in the calibration of x-ray diffraction equipment which is used in determining the amount of retained austenite in ferrous materials over the range of 10 to 20 percent. X-ray diffraction procedures recommended for these determinations require accurate measurements of the integrated intensity for a number of selected peaks (see NBS Technical Note 709). Rotation of the SRM is highly recommended to minimize the effects of inhomogeneities in the distribution of austenite on its face.

For each specimen of SRM 486 (a disk 21 mm in diameter and 2.4 mm thick), the calibrated surface is opposite the side labeled with the certified austenite content given as volume percent. Damage to the calibrated surface renders the certification void.

As described on the back of this certificate, each specimen of this SRM was certified by determining the nickel content by x-ray fluorescence. The calibration of the XRF measurements was based on the austenite content in 12 specially prepared specimens as measured with a quantitative microscope (QM). The certified value is believed accurate to within $\pm$ 0.5 percent austenite.

In special cases SRM 486 may be used as an x-ray fluorescence standard for determining the nickel content in nickel-iron or nickel-chromium-iron alloys.

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

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 R. K. Kirby.

Washington, D.C. 20234
March 6, 1981

George A. Uriano, 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 as a result of blending austenitic stainless steel powder (20.9% Ni) with ferritic stainless steel powder (0.10% Ni). This process permits the use of precise and rapid x-ray fluorescence analysis for the determination of nickel content, and hence the austenite content, of each pressed and sintered specimen.

The powder for SRM 486 was prepared at Federal Mogul Corporation, Detroit, Michigan, and final blending of the powder was done at the Patterson-Kelley Co., Inc., East Stroudsburg, Pennsylvania.

A calibration curve that relates Ni-K$_\alpha$ count rate and austenite content for each specimen of SRM 486 was established using 12 specimens that were prepared for microscopic measurements by etching. Quantitative microscope (QM) methods were used by G. E. Hicho to determine the percentage austenite over an area covering about 90 percent of the surface of each of these etched specimens. The standard deviation for replicate QM determinations of this type was typically 0.07 percent austenite.

Because the etching procedure used to prepare these specimens substantially increases the Ni-K$_\alpha$ count rate, the calibration curve was established for specimens in the unetched condition; the condition in which the SRM is issued. The Ni-K$_\alpha$ count rate is known to be nearly linear over the range (13.4 to 16.9 volume percent) of austenite found in the lot of specimens. Therefore, a linear calibration curve was fitted by S. R. Low using least-square regression methods given in DATAPLOT routines developed by J. J. Filliben, of the Statistical Engineering Division, Center for Applied Mathematics, who served as statistical consultant throughout the development of this SRM.

The certified value for each specimen of SRM 486 is determined from this calibration curve and from the results of a minimum of three measurements of the Ni-K$_\alpha$ count rate obtained by P. A. Pella, M. Darr, and K. F. Heinrich, of the Gas and Particulate Science Division, Center for Analytical Chemistry. Total nickel counts from each of these determinations are in the range 0.87×10^5 to 1.1×10^5. Typically, the standard deviation among the determinations on a given specimen is 400 counts (0.06 percent austenite). Possible sources of systematic error or bias that arose in the establishment of the calibration curve include the QM determinations and the use of etched specimens for the calibration of an unetched SRM specimen. Another possible source of bias in the certified value is related to the porosity level of the calibrated surface. An analysis of these factors indicates that the stated austenite content is probably accurate within $\pm$ 0.5 percent austenite.

Technical details concerning the preparation and evaluation of this SRM are given in NBS Special Publication 260-XX, Standard Reference Materials: A Standard Material Containing Nominally Fifteen Percent Austenite. (In preparation)