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U.S. DEPARTMENT OF COMMERCE/National Bureau of Standards

Standard Reference Materials:

A Standard Reference Material Containing 2.5 Percent Austenite (SRM 488)

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Standard Reference Materials:

A Standard Reference Material Containing 2.5 Percent Austenite (SRM 488)

G.E. Hicho and E.E. Eaton

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PREFACE

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

A Standard Reference Material Containing Two And One-Half Percent Austenite (SRM 488)

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Abstract

This Standard Reference Material, SRM 488, is intended for the calibration of x-ray diffraction equipment used in determining the amount of retained austenite in hardened steels. The SRM was produced using powder metallurgical techniques and involved blending 2 1/2 percent by weight AISI type 310 stainless steel powder (austenitic) with AISI type 430 stainless steel powder (ferritic). From this blend, 389 compacts were produced and subsequently examined for nickel content by x-ray fluorescence spectrometry. A calibration curve was established using 13 compacts randomly selected from the population of 389. The curve relates the weight percent nickel obtained from x-ray fluorescence measurements to the volume percentage austenite as determined by quantitative microscopy measurements of the area percent. The curve was then used to assign the certified values to the remaining compacts. This SRM may be used as an x-ray diffraction standard for retained austenite or in very special cases as an x-ray fluorescence standard for nickel content.

Key Words: austenite in ferrite; powder metallurgy; quantitative microscopy; retained austenite standard; Standard Reference Material; x-ray fluorescence.

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Introduction

This Standard Reference Material was produced in order to calibrate x-ray diffraction (XRD) equipment used to determine the amount of retained austenite in hardened steels.

The National Bureau of Standards is in the process of producing a series of SRM's containing various percentages of austenite in ferrite. The first of these was issued in 1970 and contained nominally four percent austenite [1]. A second issue of that SRM is available and in addition, 15% and 30% austenite in ferrite SRM's are also available.

In various heat treating processes, steel is heated to a temperature where a face-centered-cubic solid phase called austenite is formed. After a sufficient stabilization time, the steel is quenched into a medium where the austenite may be entirely transformed to a metastable, body-centered-tetragonal solid phase called martensite. However, sometimes the austenite is not completely transformed to martensite. The untransformed (retained) austenite could be detrimental to the properties of the finished product. Often there are requirements as to the maximum amount of retained austenite permitted in hardened steels. Therefore, it is necessary to quantitatively determine the amount of the retained austenite in the finished product.

The amount of retained austenite in steel is usually determined by XRD procedures using a direct comparison of the integrated intensities of a series of both austenite and ferrite diffraction lines. It is highly recommended that the user of SRM 488 read National Bureau of Standards (NBS) Technical Note 709 [2], describing the x-ray method used at the National Bureau of Standards (NBS) to determine the amount of austenite in an austenite-ferrite mixture, prior to using this SRM.

An XRD reference material for retained austenite should be reasonably free of certain metallurgical problems which severely affect the intensities of both the austenite and ferrite lines - preferred granular orientation, large grain size, and residual stress. The previously issued NBS austenite standard (SRM 486 [3]) was found to be reasonably free of these problems. The present SRM (488) should also be free of these problems since it was prepared in a similar manner, and should be very useful in determining the amount of retained austenite in hardened steels.

A separate characterization of the austenite content for each compact is required because a homogenous blend of austenite in ferrite cannot be guaranteed to the degree required. Therefore, each compact is individually certified.

The purpose of this paper is to present a detailed description of the preparation and characterization of the 389 compacts, and their subsequent certification as SRM 488.

General Description of the SRM

This SRM is composed of two constituents whose structures are metallurgically different. The components are AISI type 310 stainless steel (austenitic) and AISI type 430 stainless steel (ferritic) powders. The 310 is a highly stable austenitic stainless steel (24.99 weight percent Cr and 20.41 weight percent Ni) requiring a substantial change in composition to produce a transformation to a structure other than austenite. The 430 contains 16.82 weight percent Cr, and effectively no nickel - .08 weight percent.

Following the blending of the powders, the powders were pressed into compacts and subsequently sintered, repressed, and vacuum annealed. The finished compacts were approximately 21 mm (.83 in) in diameter and 2.4 mm (.094 in) thick. Only one surface of the SRM was polished, and it is that surface which is certified as to the austenite content. No surface preparation of the SRM is necessary - in fact damage to the surface renders the certification void.

The main concept of the SRM is that the austenite content for each compact is related directly to the nickel content. Because of the significant differences between the nickel content of the austenitic component (20.41 weight percent) and that of the ferritic component (.08 weight percent), it was possible to use x-ray fluorescence analysis to obtain a precise measurement of total nickel counts on the surface of the compact. The total nickel counts were then corrected to weight percent nickel.

Using a calibration curve (described later) and the values obtained by XRF analyses, it was possible to assign a volume percent austenite to each compact.

Preparation of the Compacts

The austenitic and ferritic powders used to make this SRM were produced by water atomization techniques. The preliminary sizing of the powders was done by the manufacturer. Final sizing however, was done at the National Bureau of Standards.

The austenitic stainless steel powder passed a 250 mesh screen, but was retained on a 325 mesh screen. This sieving yielded 310 stainless steel powder in the particle size range of 53 to 44 micrometers. The ferritic stainless steel powder passed a 325 mesh screen, but was retained on a 400 mesh screen. This yielded 430 stainless steel particles in the size range of 44 to 37 micrometers. Micrographs of the powders are shown in figure 1. Each powder was then sampled and chemically analyzed. The results of the chemical analyses are shown in Table 1.

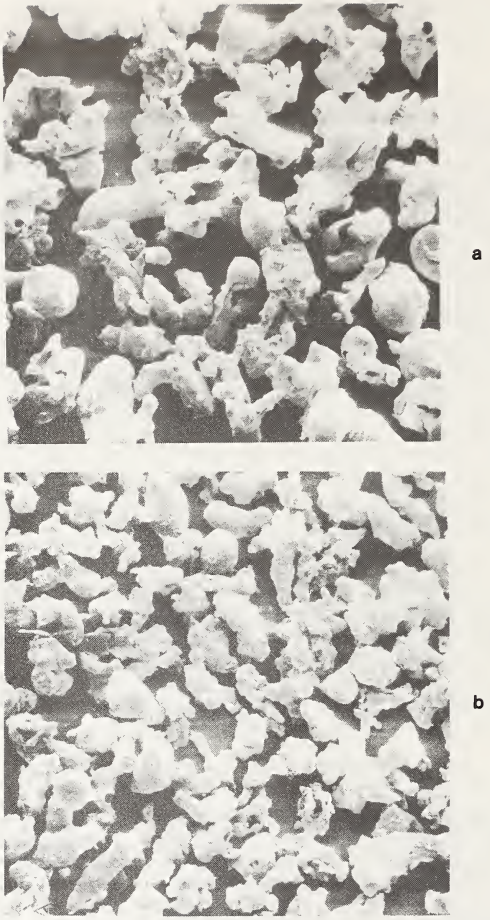

Figure 1. Scanning electron micrographs of the powders used to produce SRM 488. Mag. X160
a. as sized 310 stainless steel powder
b. as sized 430 stainless steel powder

Table I. Chemical Analysis of Stainless Steel Powder.
(Content in Weight Percent)

Elements	310 Stainless Steel	430 Stainless Steel
Chromium	24.99	16.82
Nickel	20.41	0.08
Carbon	0.05	0.02
Manganese	0.20	0.15
Phosphorus	0.01	0.005
Sulfur	0.007	0.008
Silicon	0.75	0.83
Iron	bal	bal

The steps followed in the preparation of SRM 488 are shown in figure 2.

After three cycles of pressing and heat treatment (843°C, 30 minutes) in a vacuum, each compact was hand ground on silicon carbide papers, and then polished on felt cloths using 6μ and 1μ diamond paste. Mineral spirits was used as a lubriciant during polishing. The temperature of 843°C was chosen as the sintering and annealing temperature since it was found [3] that at this temperature diffusion of the nickel into the ferrite was minimal. Micrographs of a compact, at low and high magnifications, are shown in figure 3.

As the final step in the procedure, each compact was polished on microcloth using β-alumina as the abrasive and distilled water as the lubricant. Upon completion of this step, the weight percent nickel of each compact's surface was determined using x-ray fluorescence spectrometry.

Characterization of SRM 488

Certain basic XRF concepts were used in the determination of the weight percent nickel in SRM 488. These concepts will now be discussed.

Although an alloy steel, each compact may be considered to consist of high purity Fe, Ni, and Cr powders. In the XRF analysis, these elements were treated as unknowns. Because of this, it was possible to analyze for all three elements in each compact using the combined procedure of Rasberry-Heinrich [4] and the Naval Research Laboratory [5] programs for the correction of interelement effects.

An AXS, automated wavelength x-ray spectrometer, employing a Cr target with an Al filter, operating at either 50KV, 10MA, or 50KV, 15MA was used. To reduce the effect of surface inhomogeneity of the austenite, each compact was rotated at about 10 RPM with its axis tilted approximately 60° to the beam, and examined for approximately 30 seconds.

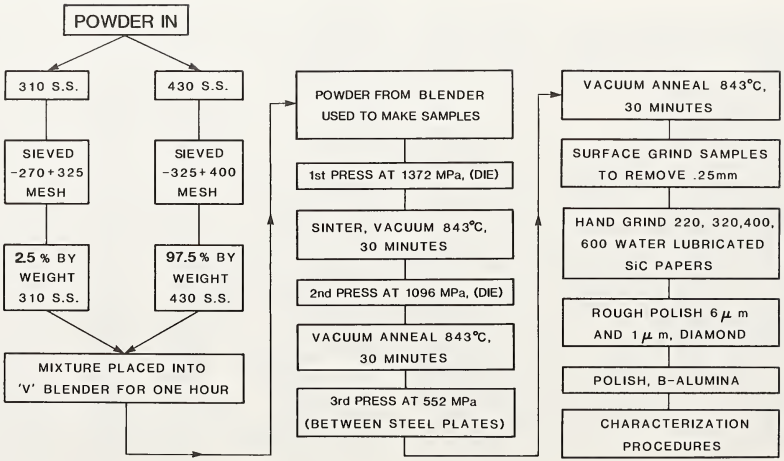

Figure 2. Fabrication procedure for SRM 488.

Figure 3. General microstructure of a compact.
a. Optical micrograph showing the white austenite particles in the ferrite mixture. Mag. X50.
b. Optical micrograph of an enlarged austenite particle. Mag. X800.

For check purposes during XRF examination, three specimens, each containing a different weight percent nickel, were prepared. Specimen A, containing 1.439 weight percent nickel, was prepared in the same manner as described previously; i.e., press, sinter, press, etc. An extensive number of measurements using a quantitative microscope (QM) were performed on this compact and its area percent austenite value was known with high accuracy. Specimens B and F (corresponding to the 5 and 2 1/2 area percent austenite compacts) were made using pure chromium, nickel, and iron powders. But, unlike A, they were vacuum melted into buttons and subsequently machined to fit a holder. Filings from these compacts were individually examined for nickel content using atomic absorption spectrometry. The mean for six determinations of weight percent nickel in B was 1.511 with a standard deviation of 0.009. Likewise, the mean for two determinations of nickel in F, was 0.8163. Specimens A, B, and F were then used as calibration checks on the x-ray spectrometer during the analysis of each compact.

A summary of the XRF precision data on samples A, B, and F is as follows. The mean weight percent nickel for 13 XRF determinations of specimen A was 1.437 with a standard deviation of .011 and relative standard deviation of 0.8%. The mean weight percent nickel for 13 XRF determinations of specimen B was 1.509 with a standard deviation of 0.010 and a relative standard deviation of 0.70%. The mean for 4 determinations of specimen F was 0.809 with a standard deviation of 0.019.

Following the XRF determination of the weight percent nickel for each compact, the total population was ranked from lowest to highest weight percent nickel in order to select compacts to be used in the establishment of the calibration curve. It should be noted that the nickel content ranged from 0.605 to .825 weight percent nickel. This was benefical in that a limited number of calibration samples would then be required to establish the calibration curve.

The calibration curve is a plot of the volume percent austenite, which is equivalent to the area percent [6] austenite value as determined from QM procedures, along the ordinate and the weight percent nickel value as determined by XRF along the abscissa. It is this calibration curve which permits the assignment of a volume percent austenite from a corresponding weight percent nickel (XRF).

As stated, the QM method is used to determine the area percent austenite on the calibration compact's surface. However prior to this determination, it was necessary to ascertain the area percent porosity of the compact's surface since this value is needed to correct the area percent austenite determined later.

A polished compact is inserted into a holder and placed on the microscope's stage. Mathematically the compact's surface, and part

of the holder is divided into cells, each 0.625 mm square. There are 34 cells in a row and 34 rows. During the scanning of the compact, the area percent porosity is obtained for each cell and recorded so that it can be used later. Once the 1156 cells have been scanned, an equation is used to extract data equivalent to an area 90% of the compact's surface. The data are then statistically analysed, and the appropriate mean porosity, standard deviation, etc. obtained. Following the porosity determination, the area percent austenite was determined.

In order to determine the area percent of austenite on the compact's surface using QM methods, the austenite (the 310 stainless steel component) had to "stand out" from the ferrite background. An extensive amount of research was conducted in order to find a stain that permitted this. Murakami's reagent, (10 grams potassium hydroxide (KOH), 10 grams potassium ferricyanide (K_3 Fe $(CN)_6$), and 100 mL distilled water), when heated to about 100°C, stained the compact so that the austenite (310) was unattacked and the ferrite was stained dark red. Hence it was now possible to use the QM to determine the percentage of white particles (i.e. austenite) on the component's surface.

Following the same QM procedure described previously for the porosity determination, each of the 13 calibration compacts were stained with Murakami's reagent and the area percent austenite and standard deviation determined.

In order to ascertain the extent of inhomogeneity of the austenite over the compact's surface, a normalization of the data using the standard deviation was performed.

The relationship used in the normalization procedure was (A-B)/S, where A is the area percent austenite value for that cell, and B and S are the mean and standard deviation obtained for the compact's surface. The value obtained for each cell was subsequently plotted and a pattern was obtained which revealed the extent of inhomogeneity that existed on the compact's surface. If the normalization revealed a surface with excessive austenite inhomgeneity, the compact was discarded. None of the calibration compacts showed any severe inhomogeneity to the extent that it would be discarded. The characterization steps used for this SRM are shown in figure 4.

The calibration curve was fitted using least-square regression methods. The calibration data are plotted in figure 5. The equation for the curve using the 13 calibration points is as follows:

$$\text{Volume percent austenite} = 4.066X - 0.546$$

where X represents the weight percent nickel as determined by XRF spectrometry.

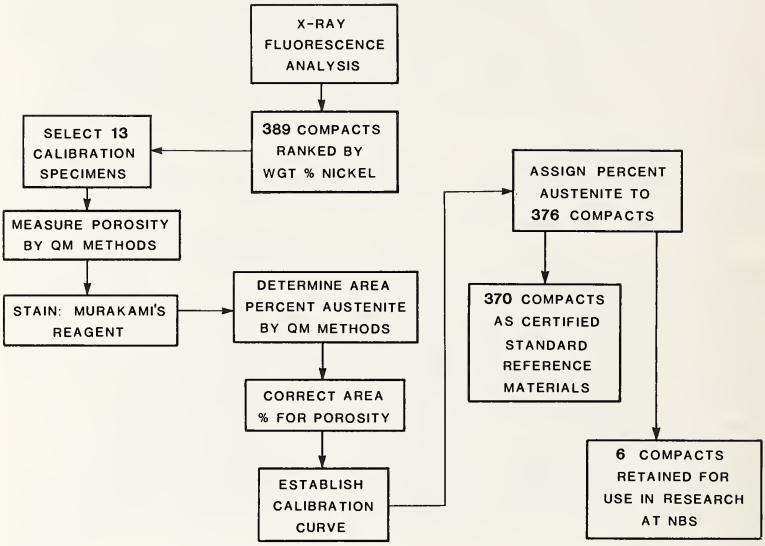

Figure 4. Characterization steps for SRM 488.

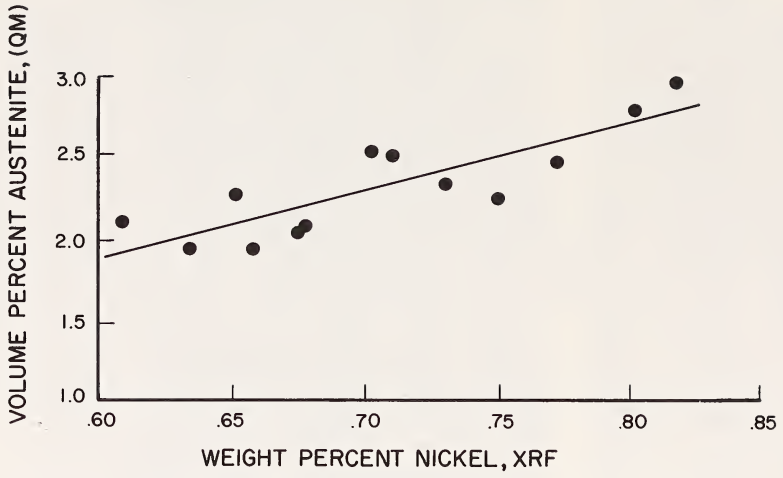

Figure 5. Calibration curve showing the volume percent austenite as a function of weight percent nickel.

The equation to be used for the standard deviation (SD), i.e. uncertainty of a single compact based on errors of the calibration curve is as follows:

$$SD = (.177)\,[1/13 + 19.799\,(X-.7067)^2]^{1/2} \qquad [7]$$

where X represents the weight percent nickel as determined by XRF spectrometry. Using this equation, the calibration curve uncertainty does not exceed ±0.10 volume percent austenite. However, a larger uncertainty is assigned to each certified compact, namely ±0.30 volume percent austenite. This uncertainty encompasses the uncertainty associated with the calibration curve, involving both the QM and XRF measurements, and the potential bias in the measured values. Sources of known bias are primarily the porosity correction applied to each specimen, and the QM threshold setting used for each of the calibration specimens.

Summary

SRM 488 is satisfactory for issuance as a standard for x-ray determination of retained austenite in hardened steels. The range for the 389 certified compacts is within the range of 1.91 to 2.96 volume percent austenite. The mean content for these compacts is 2.23 volume percent austenite. The initial blend was prepared so that it contained 2.5 weight percent austenite and this corresponds to a volume percent of 2.41. Thorough statistical analysis of the data associated with the QM and XRF measurements could not reconcile this difference between these two values. Consideration of the sources of potential bias in all the measurements also could not account for this difference. The most likely explanation presumes that the austenite distribution throughout the volume of each compact is not uniform. Recall that some compacts were rejected because the surface distribution of austenite was not sufficiently uniform, but no studies were conducted on the volume distribution. Since only the prepared, certified surface of the SRM is to be used, this effect if present should have no consequence in a proper measurement program.

In using SRM 488 in an x-ray measurement series, it is highly recommended that the compact be rotated in order to minimize the effect of possible inhomogeneity of the certified surface. In addition, the user should read NBS Technical Note 709 prior to using the SRM.

Acknowledgements

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5. AUTHOR(S)

G. E. Hicho, E. E. Eaton

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11. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)

This Standard Reference Material, SRM 488, is intended for the calibration of x-ray diffraction equipment used in determining the amount of retained austenite in hardened steels. The SRM was produced using powder metallurgical techniques and involved blending 2 1/2 percent by weight AISI type 310 stainless steel powder (austenitic) with AISI type 430 stainless steel powder (ferritic). From this blend, 389 compacts were produced and subsequently examined for nickel content by x-ray fluorescence spectrometry. A calibration curve was established using 13 compacts randomly selected from the population of 389. The curve relates the weight percent nickel obtained from x-ray fluorescence measurements to the volume percentage austenite as determined by quantitative microscopy measurements of the area percent. The curve was then used to assign the certified values to the remaining compacts. This SRM may be used as an x-ray diffraction standard for retained austenite or in very special cases as an x-ray fluorescence standard for nickel content.

12. KEY WORDS (Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)

austenite in ferrite; powder metallurgy; quantitative microscopy; retained austenite standard; Standard Reference Material; x-ray fluorescence.

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