NBS SPECIAL PUBLICATION 260-76

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

Standard Reference Materials:

A Standard Reference Material Containing Nominally Five Percent Austenite (SRM 485a)

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Standard Reference Materials:

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A Standard Reference Material Containing Nominally Five Percent Austenite (SRM 485a)

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Abstract

This Standard Reference Material, SRM 485a, is a renewal of SRM 485 , and 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 5 percent by weight AISI type 310 stainless steel powder (austenitic) with AISI type 430 stainless steel powder (ferritic). From this blend, 216 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 216. The curve relates the weight percent nickel from x-ray fluorescence measurements to the volume percentage austenite as determined by quantitative microscopy measurements of 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.

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 is sometimes 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 485a read National Bureau of Standards (NBS) Technical Note 709, [1], 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 confoundments which severely affect the intensities of both the austenite and ferrite lines - preferred granular orientation, large grain size, and residual stress. The previous SRM [2], was found to be reasonably free of these confoundments. Therefore, the present SRM (485a), since it was prepared in a similar manner, should be free of these confoundments, and be very useful in determining 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 [3]. This SRM, 485a, is a renewal of that (1970) SRM. Standard Reference Materials containing 15% and 30% austenite in ferrite have also been prepared.

Following the preparation and production of SRM 485, an automated XRF unit, used to determine the nickel content of each

compact, was purchased. Therefore the characterization procedures used for SRM 485a will not be the same as that used for 485.

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

The purpose of this paper is to present a detailed description of the preparation and characterization of the 216 compacts, and their subsequent certification as SRM 485a.

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 (austentic) 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 16.03 weight percent Cr, and effectively no nickel - .09 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 lies in the fact that the nickel content for each compact is related directly to the austenite content.

Because of the significant differences between the nickel content of the austenitic component (20.41 weight percent) and that of the ferritic component (.09 weight percent), it was possible to use XRF analysis to obtain a very 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 then 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, however, final sizing was done at the National Bureau of Standards.

The austenitic stainless steel powder, passed the 250 mesh screen, but was retained on a 325 mesh screen. This sieving yielded the 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.

Table 1. Chemical Analysis of Stainless Steel Powder.

(Content in Weight Percent)

Elements	310 Stainless Steel	430 Stainless Steel
Chromium	24.99	16.03
Nickel	20.41	0.09
Iron	bal	bal
Carbon	0.05	0.05
Manganese	0.20	0.10
Phosphorus	0.01	0.011
Sulfur	0.007	0.01
Silicon	0.75	0.40

The steps in the preparation of SRM 485a are shown in figure 2. Since this SRM was a renewal of a previously prepared SRM, 485, the powder for SRM 485a was taken from jars of already blended material [3]. A total of 216 units were produced.

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 6μm, and 1μm, diamond paste with mineral spirits as the lubriciant. The temperature of 843°C was chosen as the sintering temperature since it was found [2] that at this temperature diffusion of the nickel into the ferrite material was minimal. A micrograph of a compact, at low and high magnifications, is shown in figure 3.

a

b

Figure 1. Scanning electron micrographs of the powders used to produce SRM 485a. Mag. X160

a. as sized 310 stainless steel powder
b. as sized 430 stainless steel powder

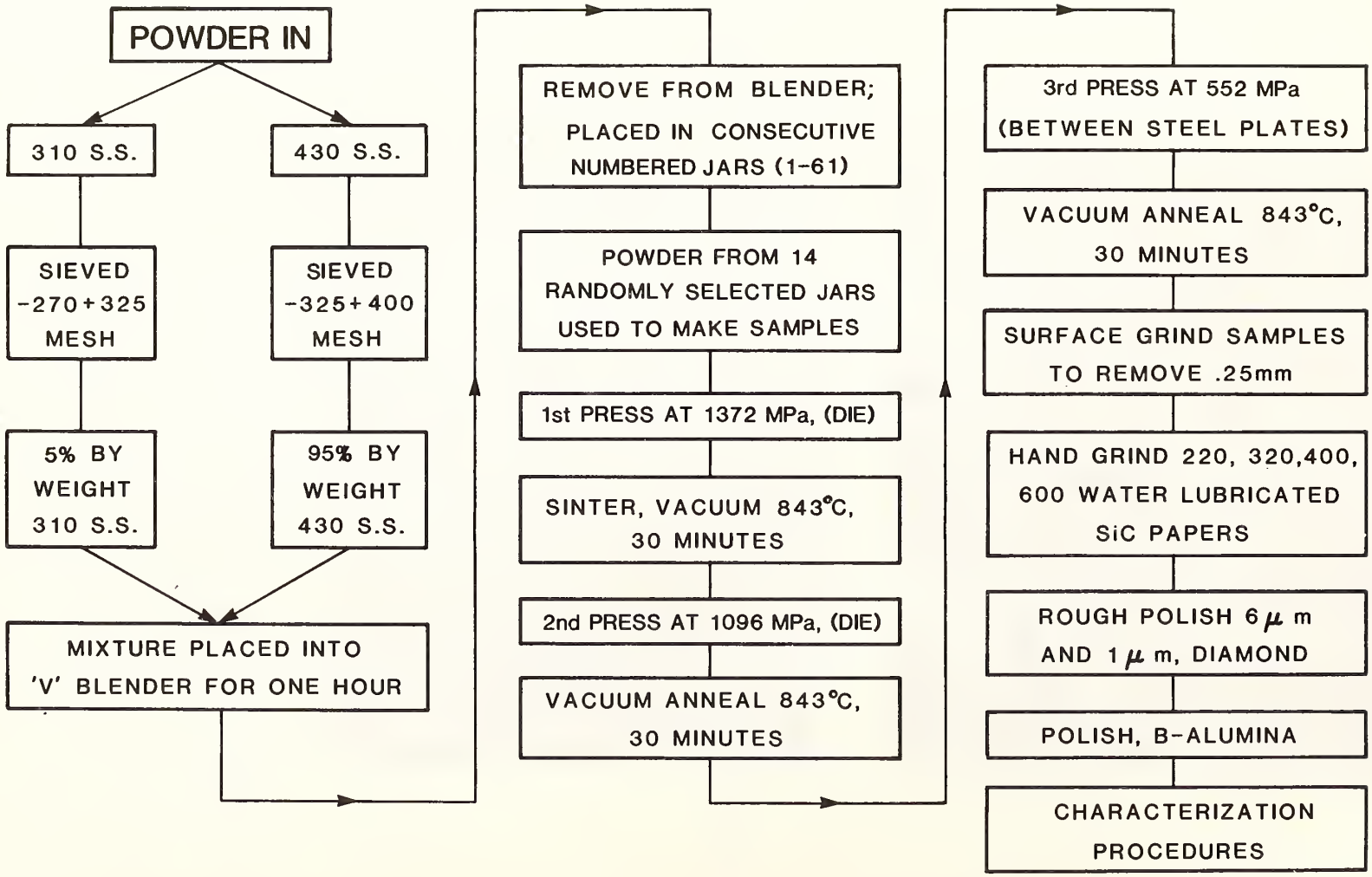
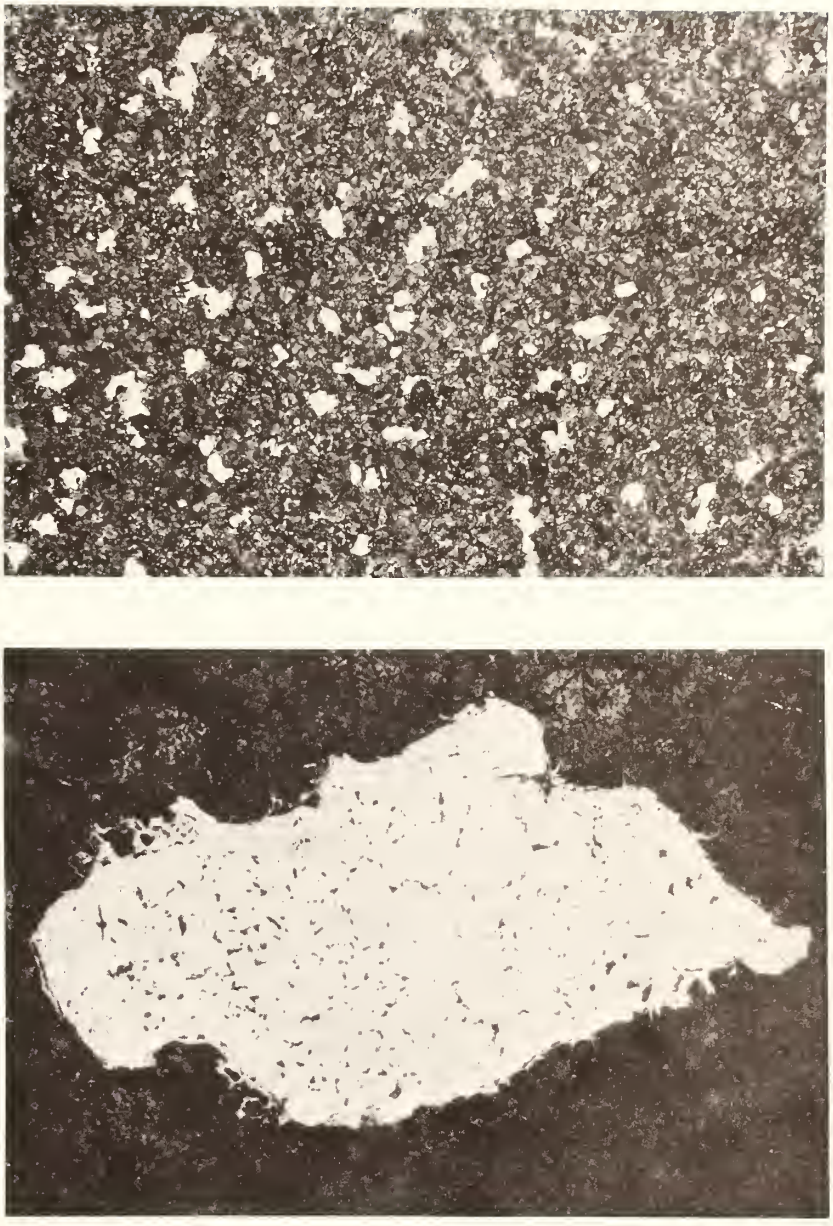


Figure 2. Fabrication procedure for SRM 485a.



a

b

Figure 3. General microstructure of a compact.
a. Optical micrograph showing the white austenite particles in the ferrite matrix. Magnification x40.
b. Optical micrograph on an enlarged austenite particle. Magnification x800.

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 485a

Certain basic XRF concepts were used in the determination of the weight percent nickel in SRM 485a. These concepts will now be discussed.

Each compact consists primarily of high purity Fe, Ni, and Cr powders and in the XRF analysis were treated as ternary unknowns. Because of this, it was possible to analyse 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 spectometer, employing a Cr target with an Al filter, operating at 50 kv and 10 ma was used. To reduce the effect of surface inhomogeneity of the austenite, each compact was rotated with its axis tilted approximately 60° to the beam, and examined for approximately 30 seconds.

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 upon this compact and its area percent austenite value was known with high accuracy. Specimens B and C (corresponding to the 5 and 30 area percent austenite compacts) were made from 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 four determinations of nickel in C, was 6.478 with a standard deviation of 0.015 weight percent nickel.

Specimens A, B, and C were then measured for nickel content by XRF along with each set of compacts whose weight percent nickel was to be determined. These same samples were then used to monitor the

output of the unknown samples and were checks on the x-ray spectrometer in that any deviation from the predetermined values of A, B, and C indicated an abnormality in the analyses of the unknowns.

A summary of the XRF precision data on samples A, B, and C is as follows. The mean weight percent nickel for 20 XRF determinations of specimen A was 1.439 with a standard deviation of 0.012 and relative standard deviation of 0.8 percent. The mean weight percent nickel for 14 XRF determinations of specimen B was 1.518 with a standard deviation of 0.014 and relative standard deviation of 0.92 percent. The mean for 14 determinations of specimen C was 6.481 weight percent nickel with a standard deviation of 0.028 and a relative standard deviation of 0.43 percent.

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. The selection was done in the following manner.

Arbitrarily, selection was started at the fourth lowest weight percent nickel value. That compact and every 22nd compact was selected until the total population was exhausted. From the population of 216 compacts, 13 compacts were chosen for the calibration curve.

The calibration curve is a plot of the volume percent, 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). The development of the calibration curve follows.

In order to obtain a more accurate value of the area percent austenite when using the QM method, it was necessary to examine each calibration compact for surface porosity prior to the austenite determination. In the QM determination for porosity, the surface was considered as being divided into individual quadrants, each 0.625 mm square. There are 34 of these quadrants in a row, and 34 rows. The area percent porosity for each quadrant was obtained and entered into the computer's memory. An area equal to about 90% of the sample's total area was subsequently used to establish a mathematical relationship which yielded a mean porosity, and a

standard deviation for each compact's surface. If a compact had excessive porosity it was discarded. Once the porosity was determined for each compact, the compacts were stained.

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. One reagent, Murakami's satisfied our needs. This reagent, 10 grams sodium hydroxide (NaOH), 10 grams potassium ferricyanide ($K_3 Fe(CN)_6$), and 100 mL distilled water, when heated to about 100°C, stained the compact to the extent that under the microscope 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 components 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 compacts surface, a normalization of the data using the standard deviation was performed.

The equation used in the normalization procedure was (A-B)/S, where A is the area percent austenite value for that quadrant, and B and S were the mean and standard deviation obtained for the compact's surface. The value obtained for each quadrant 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 excesive inhomogeneity of the austenite, the compact was discarded. None of the calibration compacts showed any severe inhomogeneity to the extent that it would be discarded.

Since a total of three hours was needed for each set of observations only two orientations of the compact were used. The characterization steps used for this SRM are shown in figure 4.

The calibration curve was fitted using least-square regression methods. The variance representing the fit of the calibration curve's experimental data points was 0.04 area percent austenite.

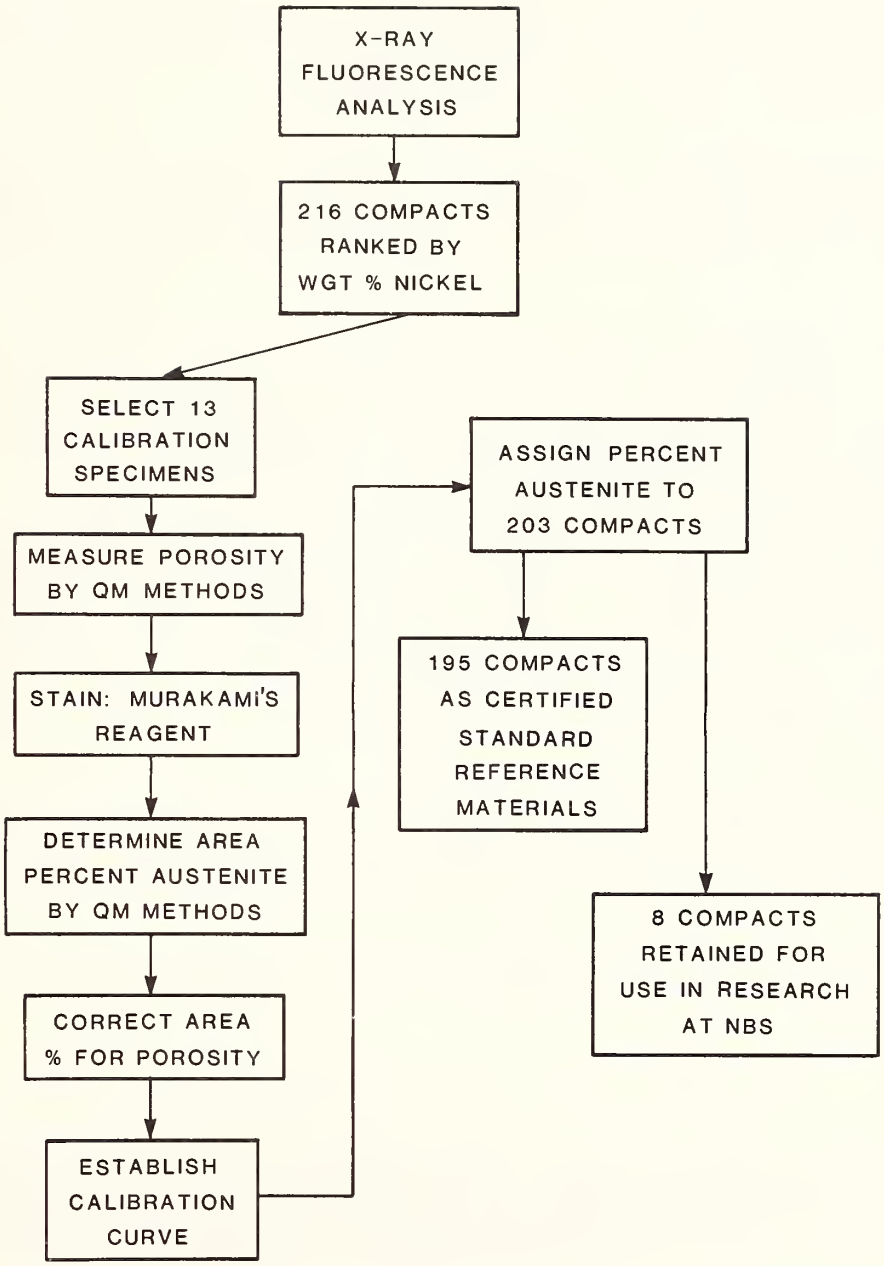


Figure 4. Characterization steps for SRM 485a.

The correlation coefficient, a measure of the fit of the curve to the data points was found to be 0.97. The equation for the curve using the 13 calibration points is as follows:

Volume percent austenite = 4.758W - 1.121

where W represents the weight percent nickel as determined by XRF spectometry. The calibration curve, along with the data points used to develop this curve, is shown in figure 5.

A question arose as to what uncertainity (i.e. standard deviation) should be assigned to each SRM compact. It was noted that two prime factors in this analysis were the QM determination for area percent austenite itself and the associated porosity correction.

A statistical analysis of the data revealed that the variance of fit of the porosity correction contributed less than 2% of the total variance of a single austenite determination. In addition, an analysis of the austenite values for replicate determination of the calibration samples revealed that the variance associated with short term replication errors was found to be less than 10% of the variance of fit for the calibration curve. These results raised the question whether the lack of fit of this calibration curve was due to sample problems, long term errors (i.e., measurement variability), or sample idiosyncrasies. Analysis of the data proved the latter true, therefore a smaller uncertainty should be assigned to the certified value since we are only dealing with the imprecision of the calibration curve.

This conclusion was corroborated in an QM experiment performed on a single sample over a period of 14 days. An analysis of the QM measurements for a single 5% austenite sample showed a variance of 0.06 which is comparable to the variance of fit listed previously.

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

$$SD = (0.20)\ [1/13 + 3.0812\ (\ X-1.2171)^2]^{1/2} \qquad [7]$$

Considering that this uncertainty encompasses the errors associated with both the QM determinations of the porosity and the austenite, and the errors associated with the XRF measurements, we believe the certified value assigned to each compact is within $\pm$

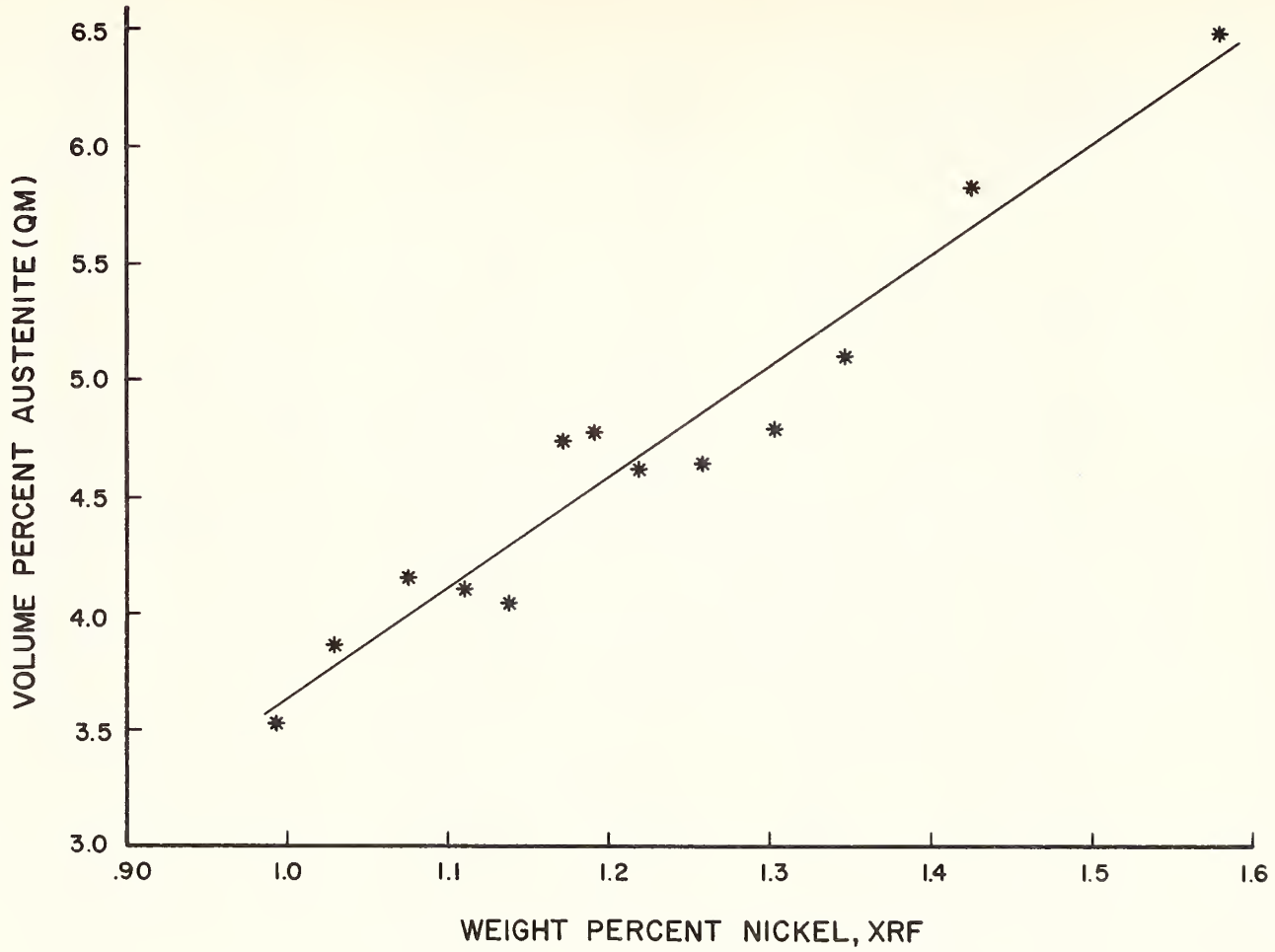


Figure 5. Calibration curve showing the volume percent austenite as a function of weight percent nickel.

0.25 of the assigned volume percent austenite. The distribution of the percent austenite for the 195 compacts is shown in figure 6. The values for the population ranged from 3.58 to 5.55 volume percent austenite.

Conclusions

SRM 485a is satisfactory for issuance as a standard for x-ray determination of retained austenite in hardened steels. The range for the 195 certified compacts was found to be 3.58 to 5.55 volume percent austenite. The mean for this population was approximately 4.55 volume percent austenite. In using SRM 485a, care must be taken not to alter the certified face. During x-ray diffraction examination of the compact, 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.

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And finally to our secretaries P. Salpino and C. Gallupe, both of whom worked so diligently on this manuscript.

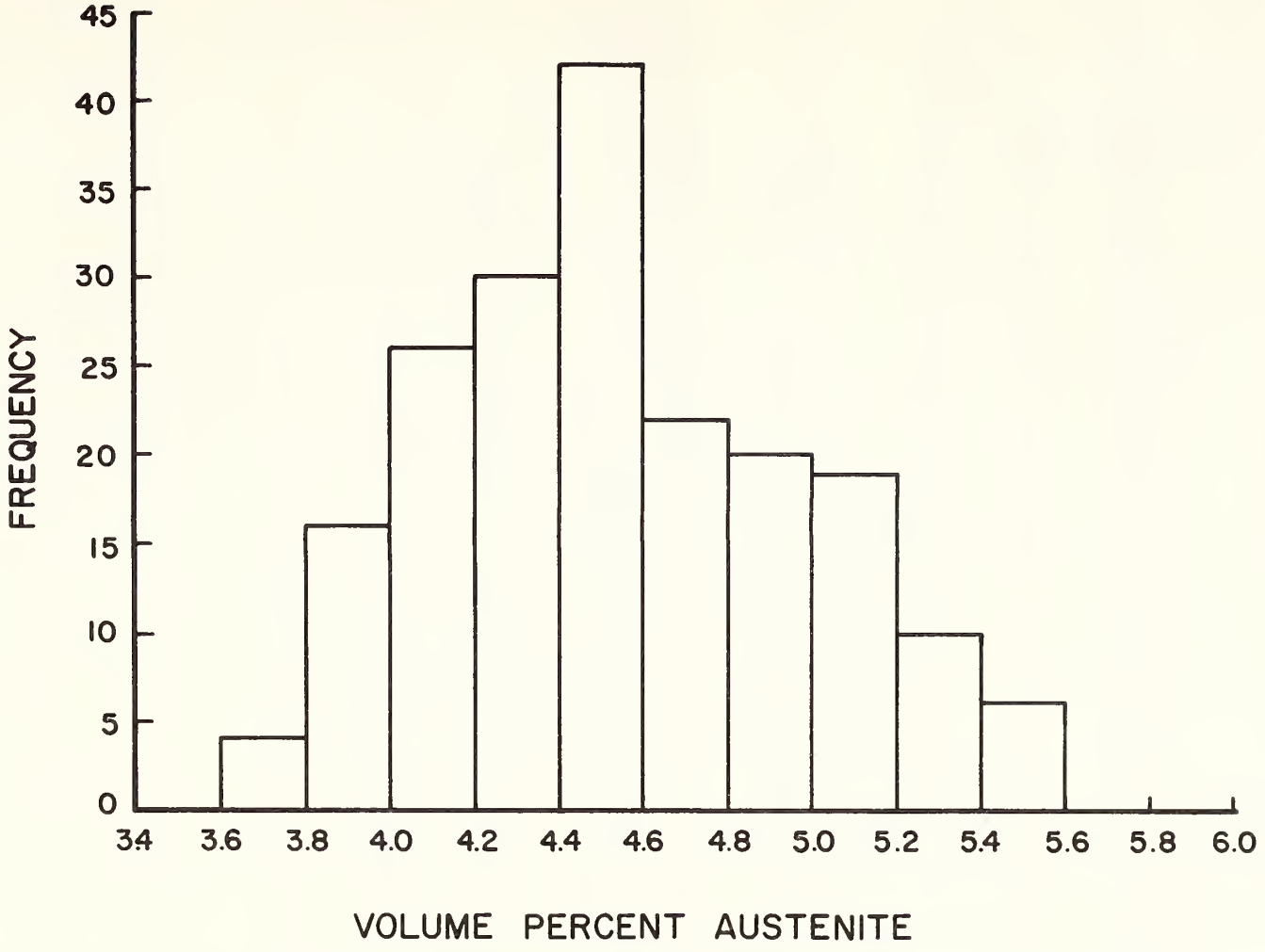

Figure 6. Histogram showing frequency of occurrence versus the volume percent austenite.

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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 485a, is a renewal of SRM 485 , and 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 5 percent by weight AISI type 310 stainless steel powder (austenitic) with AISI type 430 stainless steel powder (ferritic). From this blend, 216 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 216. The curve relates the weight percent nickel from x-ray fluorescence measurements to the volume percentage austenite as determined by quantitative microscopy measurements of 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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