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Standard Reference Materials:

PREPARATION AND HOMOGENEITY CHARACTERIZATION OF AN AUSTENITIC IRON–CHROMIUM–NICKEL ALLOY

U.S. DEPARTMENT OF COMMERCE
National Bureau of Standards

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Standard Reference Materials:

Preparation and Homogeneity Characterization of an Austenitic Iron–Chromium–Nickel Alloy

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CONTENTS

LIST OF TABLES

LIST OF FIGURES

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PREPARATION AND HOMOGENEITY CHARACTERIZATION OF AN AUSTENITIC IRON-CHROMIUM-NICKEL ALLOY

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An alloy of weight fraction 0.710 iron, 0.183 chromium and 0.107 nickel was characterized at the micrometer level of spatial resolution by means of electron probe microanalysis. This alloy, designated SRM 479, is of suitable homogeneity for use as a standard in microanalytical techniques. The coefficient of variation for each of the three elements is not more than 1.5% based on about 800 analyses involving five different specimens. There is no statistically significant variation in composition within specimens or from specimen to specimen. Electron probe microanalysis was carried out using different instrumental conditions and operators. SRM 479 is supplied as a disk about 4.6mm diameter by 1mm thick.

Key words: Austenitic stainless steel; electron probe microanalysis; Fe-Cr-Ni alloy; homogeneity testing; metallography; stacking fault energy; Standard Reference Material.

I. INTRODUCTION

The National Bureau of Standards maintains a continuing program to provide standards suitable for quantitative microanalytical methods such as electron probe microanalysis, spark source mass spectrometry, and laser probe analysis. Several standard materials have already been certified under this program [1-6].[1] This report describes the methods and results of homogeneity characterization of the first ternary alloy to be so certified - an austenitic Fe-Cr-Ni alloy designated SRM 479.

This alloy was produced at NBS as a research material for a project concerned with the measurement of stacking fault energies [7]. Those results indicated a high degree of solute homogeneity on a microscopic level. Additional thermomechanical treatment led to material of suitable homogeneity for use in microanalytical methods of analysis. The material preparation and the results of quantitative electron probe microanalysis of the constituent three elements will be presented in detail.

II. PREPARATION OF THE ALLOY

The alloy was melted by the arc fusion method using appropriate amounts of the raw material in the form of chips and small pieces. The raw material purities furnished by the suppliers were: Ni-99.98%, Cr-99.95%, and Fe--99.95%. An inert gas atmosphere of argon, gettered by titanium, was established within the furnace at a pressure of about 0.4 atmospheres. A non-consumable thoriated tungsten electrode was used to melt the charge of approximately 55g. The ingot was melted four times, twice on each side, and allowed to solidify in a round depression in the cooled, copper base plate. A final remelting was conducted in a well of rectangular cross-section in the base plate to produce an ingot about 15cm x 1cm x 1cm. The expected composition,

[1]Figures in brackets indicate the references at the end of this paper.

assuming no material loss, was 10.6% Ni, 18.8% Cr, 70.6% Fe.

A portion of the ingot was reserved for rolling thin foil, a small amount for chemical analysis, and the remainder fabricated for the present standard. Initially, the balance of the ingot was annealed for 1 hr. at 1120°C within a sealed quartz tube containing helium at a pressure of 0.3 atmospheres. The ingot was then swaged to about 60% reduction in diameter, sealed again in quartz and homogenized for 5 days at 1120°C. Two subsequent swaging and annealing steps produced the final round rod of 4.6mm diameter x 220mm long. On each occasion the ends were cropped and the surface etched deeply in a 20% HNO_3 $\pm$ 3% HF solution. No lubrication was used during the mechanical processing. Each heating treatment was concluded by quenching the sealed ingot into water. Five disks, 1mm thick, were cut from the final rod at each end and at the 1/4, 1/2, and 3/4 positions. These disks were used for electron microprobe characterization of the material. Chemical analysis performed on a representative portion of the ingot indicated a composition of 10.7% Ni, 18.3% Cr, 0.012% C, the balance Fe.

SRM 479 is issued as a disk, approximately 1mm thick x 4.6mm diameter, in the as-cut condition after electrical discharge slicing. A typical microstructure found on a metallographically prepared and etched transverse surface is shown in Fig. 1. The as-deformed grain structure is evident. Small pits and voids that were not completely removed in the processing treatments are to be seen in the section. Inclusions were not detected in the specimens examined.

III. PREPARATION FOR MICROPROBE ANALYSIS

This material was easily prepared for microprobe examination. Mounted specimens were ground on water-lubricated SiC papers in the usual progression 80, 220, 400 and 600 grit size. Rough polishing was done on a canvas cloth impregnated with 6μm diamond particles. Fine polishing was done on a Selvyt cloth impregnated with 1/4μm diamond particles. Both rough and fine polishing were carried out with the wheels rotating at 125 RPM. The specimen was finished using a Gamal cloth impregnated with 0.05μm Al_2O_3. Wheel rotation was 150 RPM. Total time was about 30 minutes to complete specimen preparation. Unetched specimens were characterized and analyzed with the electron probe microanalyzer.

IV. HOMOGENEITY CHARACTERIZATION

Five specimens were taken from the rod; one from each end and one from the 1/4, 1/2 and 3/4 positions. The specimens were tested for homogeneity to determine whether any significant composition gradients were present in both the transverse and longitudinal directions of the rod.

Homogeneity testing was carried out with a microprobe operating voltage of 20kV. The Kα radiation from all three elements was monitored by spectrometers equipped with LiF crystals and sealed proportional counters. The x-ray emergence angle was 52.5°. Each element peak was found by scanning the peak profile by hand. Monitor current was adjusted to give about 25,000 counts per second on pure iron. Under these conditions, a 40 second counting interval on the specimen gave about 900,000 Fe counts, 340,000 Cr counts and 270,000 Ni counts.

The homogeneity test on each transverse specimen face consisted of collecting data along two diameters at right angles to one another and four chords of the specimen; points were spaced 200μm apart. The chord paths were one-fourth of the specimen diameter and parallel to the diameters discussed above. The procedure was repeated twice on different days with different orientation of the specimen. Approximately 160 points total were taken on each specimen.

In evaluating the results, the coefficient of variation in percent, CV, was taken to be the most useful indicator of homogeneity. The value of CV is defined as

$$CV = \frac{100s}{N} \ (\%)$$

where s is the standard deviation, in counts, for a particular data array (s is obtained in the usual fashion) and N is the average total number of counts.

In no case was CV greater than 1.5% for any element present. Therefore, a conservative estimate of CV for the entire lot is 1.5% for all three elements, iron, chromium and nickel.

On any given day of testing, at least two different specimens were tested under the same experimental conditions. These were checked by an "outside count" method [8] to see whether, statistically, the two specimens were significantly different. The specimens always passed the test. Hence, we conclude that all five specimens tested are not statistically different from one another in terms of composition.

The implications of the CV value of 1.5% must now be considered. The width of a 99% confidence interval is

$$W_{99} = \pm C_i \frac{t_{n-1}^{99}}{\sqrt{n}} \frac{(CV)}{100} \qquad (1)$$

where C_i is the true chemical weight fraction of element i in SRM 479; CV is the coefficient of variation, 1.5% in this case; n is the number of trials or analyses; and t_{n-1}^{99} is the Student's t value for 99% confidence for (n-1) degrees of freedom.

If "n" is arbitrarily made equal to sixteen in Eqn. (1), then

$$W_{99} = \pm C_i \frac{2.947}{4} (0.015) = \pm 0.0111 C_i \qquad (2)$$

Hence, if one is willing to run sixteen separate points for each element in SRM 479, the 99% confidence interval widths and mean values would be:

Iron = 0.710 $\pm$ 0.0080

Chromium = 0.183 $\pm$ 0.0020

Nickel = 0.107 $\pm$ 0.0012

The time to collect data for sixteen points is not prohibitive, being about 30 minutes.

V. ELECTRON PROBE MICROANALYSIS

As a final test, four different microprobe operators analyzed the specimens by means of electron probe microanalysis using pure iron, chromium and nickel as standards. The 1/4, 1/2, 3/4 specimens were used for analyses. The first of these was tested by analyst A, the second by B and the third by C. Analyst D collected data from all three specimens. Sixteen determinations were made for all elements. Results were obtained with instrumental operating voltages of 15, 20, 25, and 30kV respectively. Count rates were set so that pure iron gave about 12,000 counts per second to minimize coincidence loss uncertainties [9]. Instrument stability was checked by observing monitor current variations during the course of the analysis. Drift was less than one percent for all runs. Data from standards were obtained both before and after data from the specimen. No significant variation of the data from standards was observed.

Raw data from standards and specimen were entered in the microprobe data reduction program COR. This program has been described in detail elsewhere [10]. The results obtained are listed in Table I. These results show no significant specimen-to-specimen variations and reaffirm that the CV of 1.5% is reasonable. The analyses for iron and nickel are within about 1% relative for all operating voltages. The chromium analyses tend to be high, the most likely cause being the large characteristic fluorscence correction for chromium - about 20%. Errors in this correction have been discussed in detail by Heinrich and Yakowitz [11]. The fluorescence yield factors for nickel and iron may be the source of error in the case of the analyses in Table I.

VI. CONCLUSIONS

The ternary alloy consisting of 0.710 iron, 0.183 chromium and 0.107 nickel as determined by chemical analysis, is suitable for use as a microanalytical standard. This alloy, SRM 479, has a chemical homogeneity, expressed as coefficient of variation of not more than 1.5% for each element present. The individual region for which this coefficient of variation is valid is approximately a 1.5μm diameter sphere of the material. As an additional characterization, the stacking fault energy of this alloy has been determined from electron transparent thin foils in the temperature range 25°C to 325°C by Latanision and Ruff [7].

SRM 479 should be useful in the study of the fluorescence correction in microprobe analyses. This material will also be useful in any laboratory concerned with the quantitative microanalysis of stainless steels. SRM 479 represents the first ternary alloy to be certified under the homogeneity characterization program of the NBS Office of Standard Reference Materials.

The authors thank Mr. D. P. Fickle for his efforts in the preparation of SRM 479; Dr. K. F. J. Heinrich for use of the electron microprobe; Mrs. M. M. Darr, Mr. C. E. Fiori and Mr. R. L. Myklebust for electron probe microanalysis of SRM 479. Thanks are also due to Mr. Myklebust for aid in preparing the data for the computer. We thank Mr. R. A. Paulson for the chemical analysis of SRM 479. Finally, we acknowledge the initial characterization efforts of Dr. R. M. Latanision (RIAS) as part of his NAS-NRC Postdoctoral Research Associateship.

TABLE I. Electron Probe Microanalysis Results for Fe, Cr and Ni in SRM 479. Standards were pure Fe, Cr and Ni. True Concentrations in weight fractions: Fe = 0.710; Cr = 0.183; Ni = 0.107.

Element	Voltage (kV)	k*	Calculated Concentration	CV (%) (16 points)	Analyst
Fe	15	0.702	0.708	0.87	A
Fe	20	0.688	0.701	0.60	B
Fe	25	0.696	0.717	0.50	C
Fe	30	0.680	0.711	0.58	D
Cr	15	0.210	0.184	0.50	A
Cr	20	0.221	0.192	1.23	B
Cr	25	0.224	0.192	1.32	C
Cr	30	0.226	0.193	0.97	D
Ni	15	0.105	0.109	1.26	A
Ni	20	0.099	0.107	1.37	B
Ni	25	0.095	0.107	0.70	C
Ni	30	0.091	0.107	1.15	D

*The value of k is defined as the background-coincidence loss corrected ratio of intensities between the unknown and standard.

VII. REFERENCES

[1] Michaelis, R. E., Yakowitz, H. and Moore, G. A., J. Res., Nat. Bur. Stand. (U.S.) 68A, 343 (1964).

[2] Yakowitz, H., Vieth, D. L., Heinrich, K. F. J. and Michaelis, R. E., Advances in X-ray Analysis 9, 289 (1966).

[3] Yakowitz, H., Vieth, D. L. and Michaelis, R. E., Nat. Bur. Stand. (U.S.) Spec. Publ. 260-12 (1966).

[4] Yakowitz, H., Michaelis, R. E. and Vieth, D. L., Advances in X-ray Analysis 12, 418 (1969).

[5] Yakowitz, H., Fiori, C. E. and Michaelis, R. E., Nat. Bur. Stand. (U.S.) Spec. Publ. 260-22 (1971).

[6] Heinrich, K. F. J., Myklebust, R. L. and Rasberry, S. D., Nat. Bur. Stand. (U.S.) Spec. Publ. 260-28 (1971).

[7] Latanision, R. M. and Ruff, A. W., Jr., Met. Trans. 2, 505 (1971).

[8] Tukey, J. W., Technometrics 1, 31 (1959).

[9] Heinrich, K. F. J., Vieth, D. L. and Yakowitz, H., Advances in X-ray Analysis 9, 208 (1966).

[10] Heinrich, K. F. J., Henoc, J. and Myklebust, R. L., NBS Tech Note (1973), (in preparation).

[11] Heinrich, K. F. J. and Yakowitz, H., Mikrochim. Acta 5, 905 (1968).

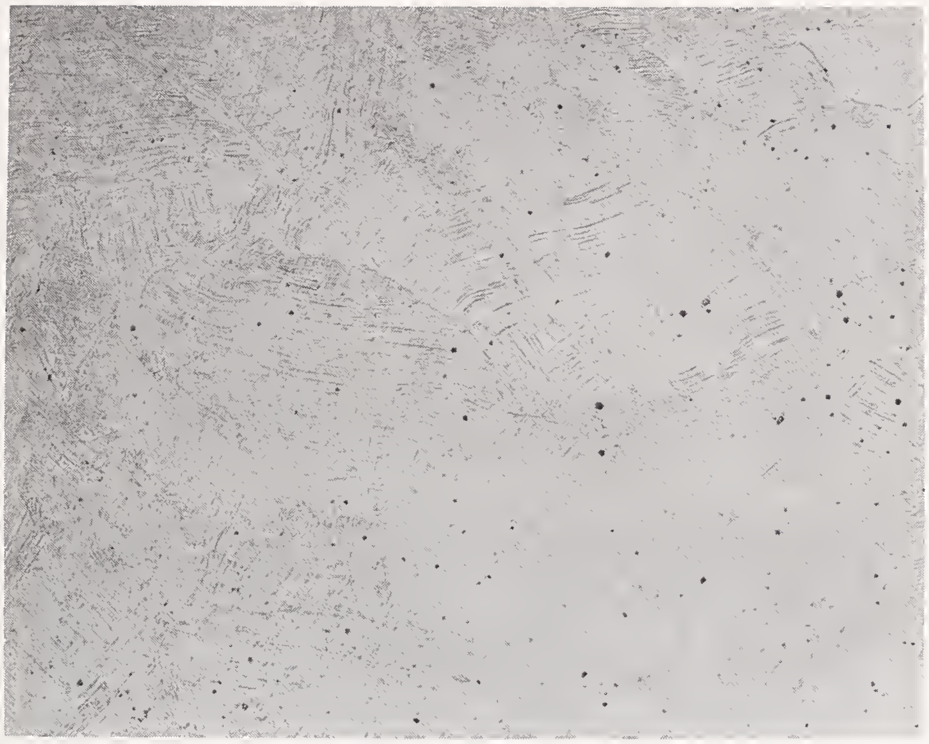

FIGURE I. Microstructure observed on a transverse surface after polishing and etching. Magnification 160X.

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An alloy of weight fraction 0.710 iron, 0.183 chromium and 0.107 nickel was characterized at the micrometer level of spatial resolution by means of electron probe microanalysis. This alloy, designated SRM 479, is of suitable homogeneity for use as a standard in microanalytical techniques. The coefficient of variation for each of the three elements is not more than 1.5% based on about 800 analyses involving five different specimens. There is no statistically significant variation in composition within specimens or from specimen to specimen. Electron probe microanalysis was carried out using different instrumental conditions and operators. SRM 479 is supplied as a disc about 4.6mm diameter by 1mm thick.

17. KEY WORDS (Alphabetical order, separated by semicolons) Austenitic stainless steel; electron probe microanalysis; Fe-Cr-Ni alloy; homogeneity testing; metallography; stacking fault energy; Standard Reference Material.

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