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Standard Reference Materials:

Homogeneity Characterization of Fe-3Si Alloy

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HOMOGENEITY CHARACTERIZATION OF Fe-3Si ALLOY

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An alloy of iron-3.22 wt. pct. silicon (Fe-3Si) was characterized with regard to chemical homogeneity of iron and silicon at the micrometer level of spatial resolution. This alloy is satisfactory for use as a homogeneous standard for electron probe microanalysis. The samples were cut from coarse-grained sheet stock to a final size of about 3 mm x 3 mm x 0.28 mm thick. Homogeneity was checked by means of quantitative raster scanning in which a square matrix (1.1 mm x 1.1 mm) of individual points is analyzed by the microprobe. Each matrix represents 400 separate analyses. Usually, the same matrix was rerun so that each point was sampled twice. The coefficient of variation for both the iron and silicon is less than one percent. Quantitative microprobe analysis was also carried out on this alloy giving a silicon content of 3.14% and an iron content of 96.9%.

Key words: Fe-3Si alloy; homogeneity testing; metallography; microprobe analysis; spectrometric analysis;
standard reference materials.

INTRODUCTION

This study forms part of the continuing program at the National Bureau of Standards for homogeneity characterization of new and existing standard reference materials, [1-4]. The purpose of this program is to provide standards suitable for the new microanalytical methods, especially electron probe microanalysis. The work described in this paper concerns an iron-3.22 wt. pct. silicon alloy (Fe-3Si). This alloy is coarse-grained sheet stock having the so-called Goss orientation in which a (110) grain is parallel to the surface and [001] is the rolling direction. This material is widely used commercially in power transformers.

This report describes the methods and results of homogeneity characterization of this alloy. The-methods and results of quantitative electron probe microanalysis of the sheet stock are also presented. The Fe-3Si alloy is interesting from this point of view because of the very strong absorption of silicon x-rays by the iron. An appreciable atomic number effect is also present, caused by the difference in atomic number of the pure silicon standard ($Z=14$) and the average atomic number of the Fe-3Si alloy ($Z=25.6$).

In addition to the sheet stock, one specimen of an existing NBS standard, designated SRM-1134 Silicon Steel, was also tested for homogeneity. This material contains 2.89% silicon.

GENERAL DESCRIPTION OF THE PROPOSED NEW STANDARD

The sheet stock was cut into pieces approximately 3 mm x 3 mm. The thickness of the sheet is about 0.28 mm. The material is coarse-grained as revealed by a direct HF etch (figure la). Each new standard is supplied as a single piece, HF etched and ready for metallographic mounting and polishing.

The standard was easily prepared for microprobe examination. The mounted specimens were ground on water-lubricated SiC papers in the usual progression of 80, 220, 400 and 600 grit size. Rough polishing was accomplished on a wheel covered with 6 um diamond impregnated nylon cloth rotating at 125 RPM. Fine polishing was done on a microcloth impregnated with 1/4 um diamond rotating at 125 RPM. The-specimen was finished on an 0.05 um Al_2O_3 impregnated microcloth rotating at 150 RPM. Total polishing time was about 30 min. The specimens were lightly etched in freshly prepared 3% nital. Etching revealed the grain boundaries and produced some pits (figure lb). The specimens were then ready for microprobe examination.

HOMOGENEITY CHARACTERIZATION OF THE Fe-3Si ALLOYS

The original sheet stock was a rectangular pie-ce about 55 mm by 250 mm. Samples for homogeneity testing were taken from positions in the sheet as shown in figure 2. All six of these samples were tested for homogeneity by means of the microprobe.

Figure 1. Structure of the Fe-3Si sheet stock.

a. After HF etch showing typical grain structure x25.

b. After metallographic preparation (see text) x40.

Figure 2. Choice of samples for the homogeneity study.

The basic method used for homogeneity testing was that of quantitative area scanning [5, 6]. In this method, the electron beam is made to move discretely some fixed increment of distance, to dwell for a predetermined period while data are taken and then to move to the next point. A square matrix of points (20 x 20) was examined; the spacing between points was about 54 um. Hence, 400 separate points were examined in an area of slightly over one square millimeter. Usually, this matrix was repeated so that each point was investigated twice.

In order to examine such a large matrix, non-dispersive x-ray analysis was used. The use of curved crystal spectrometers would lead to serious defocusing of the x-rays if the matrix is 50 um x 50 um or larger. The x-ray output from the specimen was analyzed by a lithium-drifted silicon detector. The spectrum contains Si Ka,B and Fe Ka and Fe KB peaks. Single channel analyzers with wide enough windows to enclose the entire Si and Fe peaks were set. A third single channel analyzer was set between as an indicator of background levels.

Certain precautions must be followed when using the procedure outlined above. Total count rate cannot be raised to high levels or the Si(Li) detector will be overloaded. Furthermore, line to background ratios are low in comparison to those obtained with spectrometers. Therefore, long counting times per point may be required.

The experimental conditions chosen were a 15kV operating voltage coupled with a monitor current of about 0.26 pamp. The specimen current was approximately 0.09 pamp. The Si signal-to-noise ratio was 1.2 to 1 and that of the iron was 4 to 1. A forty-second fixed counting time resulted in about 50,000 counts to be accumulated in the Si channel and 170,000 counts in the Fe channel.

Runs were made overnight; the data were punched directly onto paper tape in computer-compatible format. Stability of the instrument was checked by directing the monitor current, which is proportional to the actual beam current [7], through a voltage-to-frequency converter and scaling the output. Monitor current drift then is a direct measure of instrumental drift. For a usual run, drift of monitor current was about 0.3% per hour.

After completion of a run, the paper tape was read onto a magnetic tape. Statistical and spatial analysis of the data were performed with the aid of a program called TOPO [8]. This program does statistical analysis of the raw intensities. The first fifty individual matrix points and each matrix row of points (twenty) are then plotted. A Poisson plot, percent occurrence in terms of sample standard deviations) versus square root of intervals about the mean, is displayed. All points deviating more than four sigma from the mean are labeled. Next, apparent concentrations for each point are printed out. The program assumes that the mean value is equal to the concentration of the element in question and does a linear comparison to obtain the apparent concentrations. Concentration deviations are also printed.

If data are available from reruns of the same matrix of points, the program then looks for trends and makes statistical drift and overlap corrections. All of the information listed above is printed out for this corrected data except the concentration information. The program prepares relative topographs for each element in terms of standard deviations and absolute topographs in terms of absolute percent increments.

RESULTS OF HOMOGENEITY TESTING

Statistical data for each Fe-3Si sample tested are shown in Table I, together with data from the existing SRM (to be discussed later). From these data a conservative estimate of the coefficient of variation, equivalent to 100s divided by the mean x-ray count, is one percent in the entire lot of standards.

The matrices used in homogeneity testing were set around grain boundaries and triple-points of grains to emphasize any possible concentration variations associated with these structural details. A portion of the TOPO program results for Sample 3 is shown in figures 3 - 5. Figure 3 shows the raw intensity data from two runs over the same matrix. The maximum range of groups of twenty points, corresponding to one row, is about 300 counts for silicon and 800 counts for iron.

Table 1. Statistical Profile of the Samples Characterized.

Sample	Sample Number, Element	Total Points Analyzed	Mean X-ray Count	Square Root of Mean X-ray Count	Sample Standard Deviation, s
Fe-3Si	1-Si	400	50947	226	469
Fe-3Si	2-Si	800	50520	225	401
Fe-3Si	3-Si	800	51279	226	252
Fe-3Si	4-Si	400	51611	227	233
Fe-3Si	5-Si	400	51382	227	237
Fe-3Si	6-Si	800	52554	229	335
Fe-3Si	1-Fe	400	173611	416	1301
Fe-3Si	2-Fe	800	170447	413	1130
Fe-3Si	3-Fe	800	169547	412	481
Fe-3Si	4-Fe	400	169334	412	338
Fe-3Si	5-Fe	400	169708	412	500
Fe-3Si	6-Fe	800	167499	409	529
SRM-1134	Si Run 1	800	50848	225	239
SRM-1134	Si Run 2*	648	99822	316	386
SRM-1134	Fe Run 1	800	171955	415	494
SRM-1134	Fe Run 2*	648	342492	585	1281

*Counting time was doubled for the second test of SRM-1134 and the matrix was 18xl8 points.

Figure 3. Plots of data values taken directly from TOPO program averaged in row groups of twenty for (a) Si and (b) Fe versus position in the matrix. Total time for two runs of the matrix (800 positions) is 10.5 hours.

Table I shows that s Si is 252 counts and s Fe is 481 counts for Sample 3. Relative topographs in terms of these s values are shown in figure 4. These topographs show that four points in 800 are greater than 4s from the mean in iron. No points in 800 fall into this category in silicon. There is some correlation of positive deviations in iron with negative deviations in silicon. However, figure 5 shows that in terms of absolute percent, all deviations are small. All 800 determinations of silicon were in the range 2.94 to 3.34 percent silicon. For iron, all 800 determinations were in the range 95.9 to 97.9 percent. For the iron, 667 points of the 800 total - over 80% were in the range 96.7 to 97.1 percent.

From these and similar data on all specimens tested, we conclude that the Fe-3Si sheet stock is entirely satisfactory for use as a homogeneity standard in electron probe microanalysis. There appears to be some correlation of positive and negative deviations for iron and silicon in the samples studied. But in terms of absolute percent, this correlation may be ignored for the intended application. Structural details such as grain boundaries and triple-points do not play a significant role. These conclusions are based on analysis at more than 3700 individual points in the samples investigated.

Figure 4. Relative topography plotted directly by TOPO program, for (a) Si and (b) Fe in sample 3. The s values for Si and Fe were 252 and 481 counts,, respectively. Deviations from the mean are shown by symbols indicated in the Key.

Figure 5. Topographs plotted directly by the TOPO program showing absolute deviations from the mean concentrations for Si and Fe (Sample 3).

a. Absolute topograph for silicon. Concentration = 3.14%. All 800 points lie in the range 2.94 to 3.34% Si.

b. Absolute topograph for iron. Concentration 96.9%. All points lie in the range 95.9 to 97.9%; 667 points of 800 lie in the range 96.7 to 97.1%.

PRELIMINARY HOMOGENEITY CHARACTERIZATION OF SRM 1134, SILICON STEEL

These tests of SRM 1134 comprising 800 and 648 point determinations indicated that this material appears to be homogeneous at micrometer levels of spatial resolution. However,, we emphasize that no extensive tests of this material were performed. The results were derived from two separate matrices run on a single specimen cut from the SRM 1134 disk which had been selected at random from stock.

QUANTITATIVE ELECTRON PROBE MICROANALYSIS OF THE Fe-3Si

In addition to the homogeneity studies just described, quantititative microanalysis was also carried out on sample #6 from the middle of the original Fe-3Si sheet. For this purpose, data were collected by using curved-crystal spectrometers. For the silicon analysis, an ADP crystal, sealed proportional detector system was used; for iron analysis, a LiF crystal sealed proportional detector system was used. Monitor current was set at 0.1 uamp. An operating voltage of lOkV was chosen. Count rates for the iron and silicon in the specimen were 5300 cts/sec and 600 cts/sec, respectively. One hundred and four separate points were analyzed representing a systematic traverse of several grains and including analyses at triple-points and grain boundaries.

The 10 kV operating voltage was chosen in order to reduce the effect of uncertainties in the microprobe absorption correction for silicon [9]. Ten kilovolts was the lowest operating voltage to provide satisfactory iron intensity. Pure iron and silicon were used as reference standards.

The required corrections for the data were: (1) background subtractions for the sample and standards, (2) a detector dead-time correction for the iron, (3) absorption, and, (4) atomic number.

(1) Ziebold's method for background correction was used [10]. The spectrometers were maintained on the iron and silicon peaks and adjacent elements used to determine the background level. For the Fe, background was determined on Mn and for Si, on Al. A weight-fraction average of the background measured from each element individually can be used to obtain the specimen background level [10].

(2) Dead-time correction was made according to the methods described by Heinrich, et al [7]. The value of the deadtime, t, was 2.3 usec.

(3) For the absorption correction,, Heinrich's modification [11] of Philibert's equation [12] was employed to calculate the absorption factor, f(X). The value of the electron stopping power, a, is given as $4.5\text{x}10^{5}/(E^{1.67} - E_c^{1.67})$ with E and E_c being the operating and critical excitation potentials for the x-ray line being used for analysis.

Mass absorption coefficients were taken from Heinrich's tables [13]. The value of i(X) computed under these conditions and with a 10 kV operating voltage was 0.76 for Si and 0.996 for Fe in the specimen. These values of f(X) meet the criteria suggested for minimizing absorption correction uncertainties[9].

(4) The atomic number effect is determined by both electron backscattering and electron retardation, which depend upon the average atomic number of the target. Therefore, if there is a difference between the average atomic number of the specimen given by ($Z = EC_i Z_i$) and that of the standard, an atomic number correction is required. For Fe-3 Si, the value of Z is 25.64, therefore a somewhat larger effect would be expected for Si (Z=14) analysis than for Fe (Z=26) analysis. For Si analysis in Fe-Si the magnitude of the atomic number correction is in the opposite direction to that of the absorption correction.

The atomic number correction formulation given by Duncumb and Reed [14] was used. Electron backscatter factors, R, given by these authors were chosen since they best approximate the few experimental data available [15]. Bethe's non-relativistic relation for electron stopping power, S, was used with the ionization potential, J, given by Berger and Seltzer [16].

Heinrich and Yakowitz have investigated error propagation in the atomic number correction term [15]. In general, the magnitude of this term decreases as $U = E/E_c$

increases - but very slowly (five percent for a tenfold increase in U). But the uncertainty in the atomic number correction term remains remarkably constant as a function of U. This is because the R and S uncertainties tend to counterbalance one another [15]. Thus, no increase in the error due to the atomic number correction is to be expected at low U values and hence the choice of 10 kV as operating voltage is still valid for obtaining the highest accuracy.

No characteristic fluorescence correction is required for the iron analysis. Secondary characteristic fluorescence of Si Ka by Fe Ka is negligible [17]. No correction for fluorescence effects caused by the x-ray continuum was made. This correction is negligible in the analysis of Fe-3Si alloys [18].

All of the relations for converting measured data to concentration values have been programmed for computer reduction. The measured intensity ratio (k) was computed as C, the concentration, was incremented by 0.005 concentration units in the regions 0 C_{Si} 0.15 and 0.85 < C_{Fe} 1.0. The results are shown in figure 6 and served as calibration curves for the Fe-Si analyses.

The results for iron and silicon may be summarized as follows:

1. Silicon content :3.14% (104 determinations)
2. Observed range of silicon content :2.89% to 3.29%

Figure 6. Calibration curves used for quantitative electron probe microanalysis of (a) silicon and (b) iron. C is the calculated concentration and k is the background and deadtime corrected intensity ratio of silicon or iron in the alloy to pure silicon or iron. Valid at 10 kV operating voltage and x-ray take-off angle of 52.5o.

3. We may be 99% confident that the interval 3.12 and 3.16% would bracket the mean silicon concentration given by the microprobe.

4. Iron content :96.9% (104 determinations)

5. Observed range of iron content :94.4 to 99.4%

6. We may be 99% confident that the interval 96.6 and 97.2% would bracket the mean iron concentration given by the microprobe.

These values may be compared with the values of 3.22 percent silicon and 96.7-96.8 percent iron (by difference) determined by chemical analysis. The value of 3.22% for silicon is estimated accurate to within plus or minus 0.02 percent by the analyst. Agreement between chemical analysis and electron probe microanalysis is considered satisfactory.

CONCLUSIONS

The sheet stock containing 3.22% Si and 96.7-96.8% Fe is entirely suitable for use as a microprobe analysis standard. A conservative estimate of the coefficient of variation for both iron and silicon is ±1%.

The results of quantitative electron probe microanalysis gave 3.14% Si and 96.9% Fe as compared to 3.22% Si and 96.7 to 96.8% Fe (by difference) given by chemical analysis. Correction of microprobe data was carried out using only the set of input parameters described in the text.

The existing standard, SRM 1134, Silicon Steel, was subjected to preliminary homogeneity testing. The results indicate that this material is homogeneous at micrometer levels of spatial resolution.

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