Reference Materials for Weld Metal Ferrite Content: Gauge Calibration and Material Characterization

The accuracy of FN measuring gauges was established, and reference materials showed a very low standard deviation

BY C. N. McCOWAN, T. A. SIEWERT, D. P. VIGLIOTTI AND C. M. WANG

ABSTRACT. The Ferrite Number (FN) is a measure of the ferrite content in stainless steel welds. Accurate measurement of the FN is important because the relative amounts of ferritic and austenitic phases influence the physical and mechanical properties (as well as general corrosion and stress corrosion cracking resistance) of stainless steel welds. This report documents the procedures used to evaluate several batches of certrifugally cast reference materials and to assign reference values that can be used to calibrate several types of instruments. It also describes some general characteristics of the specimens.

Our initial effort was devoted to finding and reducing sources of uncertainty in the gauges (instruments for measuring ferrite content in units of FN) and in the calibration procedures. After improving our gauges and procedures, we found our calibration lines were nearly linear over the range of 0 to 100 FN and the gauges compared well to gauges used by The Welding Institute (TWI), which had assigned the certified FN values to secondary specimens produced in the past.

The measurements on the reference materials showed the standard deviations in FN for the secondary specimens were typically less than 0.5 FN for the 0 to 30 FN range, and less than 3 FN for the 30 to 100 FN range. The microstructure was found to be a finely dispersed and homogeneous mixture of ferritic and austenitic phases, with a percent ferrite area fraction that was nearly equivalent to the FN (up to about 60 FN) assigned to the reference materials.

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Introduction

Austenitic stainless steel weld metals usually contain a small but controlled amount of ferrite to reduce the tendency of the weld to crack during solidification. Duplex ferritic-austenitic stainless steel welds contain a balance of austenitic and ferritic phases to optimize their mechanical properties and corrosion resistance. Therefore, quantitative measurement of the ferrite content is an important commercial issue, as ranges are commonly specified in contracts and production standards. In the U.S., the amount of ferrite is usually measured magnetically using instruments calibrated according to the American Welding Society standard AWS A4.2, *Standard Procedures for Calibrating Magnetic Instruments to Measure the Delta Ferrite Content of Austenitic and Duplex Austenitic-Ferritic Stainless Steel Weld Metal* (Ref. 1).

KEY WORDS

Ferrite
Ferrite Content
Ferrite Detector
Ferrite Number
Magne-Gauge
Reference Material
Stainless Steel

American Welding Society standard A4.2 specifies procedures for both primary and secondary calibration of the instruments. Primary calibration is based on reference materials of coating thickness, such as the National Institute of Standards and Technology (NIST) Standard Reference Materials (SRMs) 1361 to 1364, while secondary calibration is based on certified specimens of stainless steel. The standard describes the importance of secondary reference materials as the only way of calibrating instruments for which no primary calibration method exists, and the most appropriate standard for in-process checks, being much more durable than the primary reference materials.

At least three different companies or organizations have produced specimens, assigned values and sold sets of secondary Ferrite Number (FN) reference materials during the past 30 years (Ref. 2). One of our goals during development of our internal procedures was to assure ourselves that our calibration scale was comparable to those previously used. Also, we worked closely with Commission II of the International Institute of Welding and with the Welding Research Council's Subcommittee on Welding of Stainless Steel and Nickel-based Alloys to ensure our procedures would maintain, if not raise, the accuracy of the measurements.

Secondary Specimens

The specimens were produced in Russia from centrifugal castings of chromium-nickel-iron alloys. Ferrite content (magnetic phase) was varied by adjusting the composition of the alloy.

The cast specimens approximate the ferrite distribution in a weld deposit and have solidification structures similar to those in welds. Like ferrite in welds, the magnetic response of the ferritic phase varies with the alloy composition (especially with key components such as Cr, Ni or Mo) (Ref. 3). The compositions varied from about 10.3% Ni and 18.2% Cr for specimens near 2 FN, to 5.5% Ni and 28.6% Cr for specimens near 105 FN.

The dimensions of the specimen are approximately 10 x 12 x 20 mm, as shown in Fig. 1. The secondary specimens are sold in sets: 1) a lower-range set with eight specimens distributed over the range of 0 to 30 FN, and 2) a higher-range set of eight specimens distributed over the range of 30 to 120 FN. The specimens are engraved with identification numbers on the 12 x 20 mm face opposite the measurement surface.

The ferrite content is measured in terms of an arbitrary quantity, the FN. The FN of the specimen is determined using a gauge that measures the force required to pull a magnet of known strength off the surface of the specimen. We used a transfer-standard technique to assign numbers to the new reference materials. In other words, we first calibrated gauges (as described in the following sections), then used them to assign FN values to the secondary reference specimens.

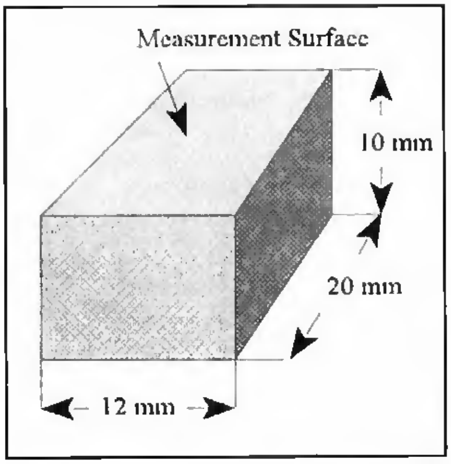

Fig. 1 — FN secondary specimen configuration.

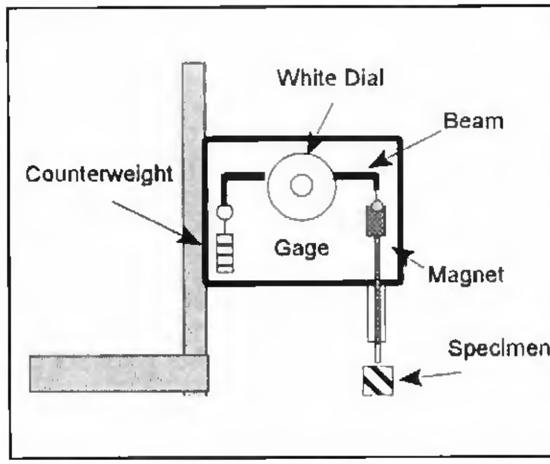

Fig. 2 — Schematic showing the general features of the gauge.

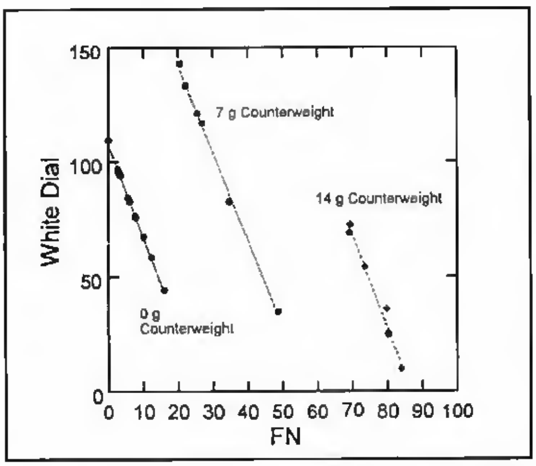

Fig. 3 — Calibration data for gauge 2. The three sets of data show the typical appearance for calibration lines are determined separately for the 0-, 7- and 14-g counterweights, respectively.

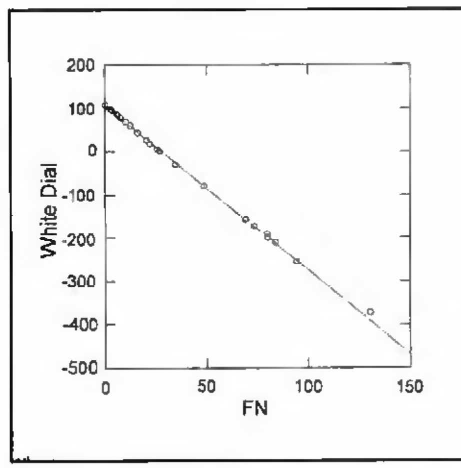

Fig. 4 — Calibration data for gauge 1 using offset white-dial values to allow a continuous linear fit of data over the 0 to 100 FN range.

The FN is expected to be approximately equal to the percent ferrite in stainless steel welds up to about 15, but at FN higher than 15, the FN number is expected to be numerically greater than the ferrite volume percentage. (These concepts are described in more detail in AWS A4.2.)

Gauge Calibration

The gauge used to assign FNs to the secondary specimens is shown schematically in Fig. 2. It is essentially a spring balance, referred to as a Magne-Gauge[1]-type instrument (MGI) in AWS A4.2. The white dial winds a spring as it is turned and this applies a gradually increasing force to lift the magnet off the surface of the specimen. Counterweights can be hung on one side of the balance to increase the applied force. To calibrate this type of gauge, a series of primary coating thickness standards are used to determine a relationship between FN (calculated for each coating thickness) and white-dial values for the gauge. We used the average FN of two gauges to assign certified values to the reference materials in our program. Complete calibration data are given in a research report (Ref. 4).

Frequency

Metrological laboratories often require instruments to be recalibrated at intervals of 6 to 12 months. However, when making our measurements to assign values to secondary reference materials, we checked the gauge calibration at the beginning of each day. The check was made by measuring three primary reference materials in the range of interest and comparing these results to those recorded for the same primary reference materials during the initial calibration. If this check had indicated a recalibration of the gauge (or maintenance) was needed, the full calibration procedure would have been implemented.

Determination of Magnet Strength

The Annexes of AWS A4.2-91 and AWS A4.2M-97 recommend the magnet used for FN measurement have a detachment (tearing-off) force of 510 ±51 FN per newton (5 ±0.5 FN per gram-force). Using a magnet that is too weak will result in falsely high FN values, and using a magnet that is too strong will result in falsely low FN values (with respect to the standardized method).

1. *Tradenames are included for clarity only; no endorsement is implied or intended.*

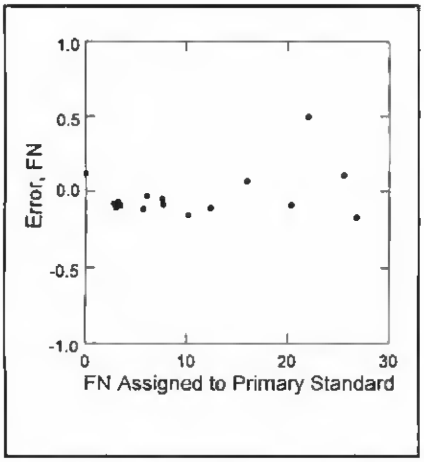

Fig. 5 — The difference in the FN assigned to the primary reference materials and FN calculated from Equation 2 for the range 0 to 30 FN (gauge 1).

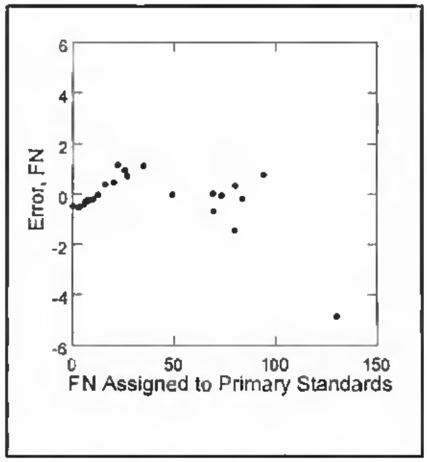

Fig. 6 — The difference in the FN assigned to the reference material and FN calculated from Equation 3 for the range 0 to 100 FN (gauge 1). Only data of 100 FN or less were used for fitting.

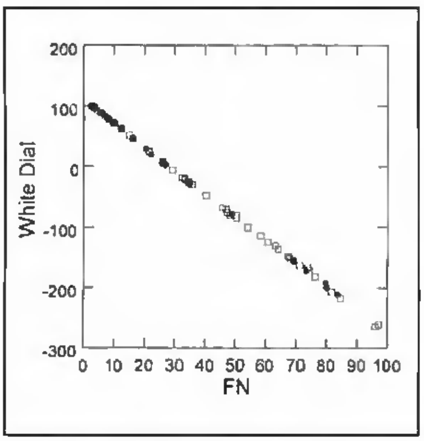

Fig. 7— The primary reference materials used for the calibration of gauge 1 (shown as solid squares here) are compared to 51 other primary reference materials.

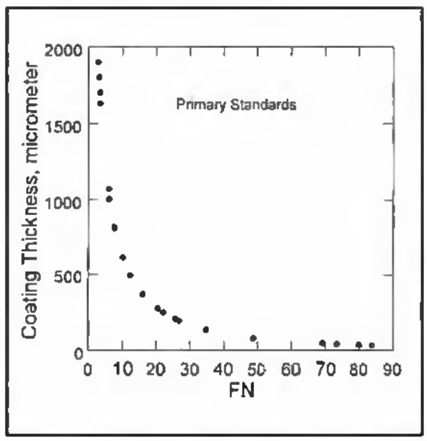

Fig. 8 — The relationship of the coating thickness of the primary reference materials to the FN calculated by Equation 1.

Traditionally, the magnet's strength is measured on MGIs by suspending a 5-g iron weight from the magnet. The spring force (obtained from the dial reading) at the time the weight is lifted just past the balance point of the gauge is divided by the weight to yield the detachment force per gram. Using this procedure, detachment forces of 490 and 541 FN per newton (4.8 and 5.3 FN per gram-force) were determined for gauges 1 and 2.

The magnet's strength can be determined directly from the slope of the calibration line if the Magne-Gauge instrument (MGI) is calibrated in terms of force. We also calibrated our gauges using a digital scale (calibrated by the manufacturer) to determine the force associated with the white-dial readings. To do this, a steel mass was positioned on the scale, below the magnet. As the white dial on the gauge was turned to load the gauge's spring, the force on the mass was recorded. The steel mass was sufficiently large that it would not be lifted off the scale over the range of measurement. Once the white-dial value was correlated to force (linear regression), the force was substituted for the white-dial data and correlated to FN. The slopes of the FN/force regression lines for gauges 1 and 2 were found to be 541 and 561 FN per newton (5.3 and 5.5 FN per g-force), respectively.

The procedures yielded different results, but all values were within the 510 ±51 FN per newton (5 ±0.5 FN per g-force) recommended in the Annex of AWS A4.2.

Table 1 — Calibration Tolerances for Primary Standards (A4.2)

Error (FN)	Measurement Range, (FN)
±0.40	0 to 5
±0.50	5 to 10
±0.70	10 to 15
±0.90	15 to 20
±1.00	20 to 30

Primary FN Calibration

Primary reference materials for coating thickness were used to calibrate the gauges. The FN of the reference materials were calculated using an equation from AWS A4.2:

$$\ln(FN) = 1.8059 - 1.1189\cdot\ln(T) - 0.1774\cdot[\ln(T)]^2 - 0.0350\cdot[\ln(T)]^3 - 0.0037\cdot[\ln(T)]^4 \quad (1)$$

where T is the thickness of the coating in millimeters.

The coating thicknesses used to calibrate the gauges were distributed through the range of 0.01 to 1.9 mm, which corresponds to a range of 0 to 130 FN (Equation 1). To calibrate a gauge, each reference material was measured a minimum of five times and the lowest repeatable white-dial values from these measurements were taken as the calibration value. (The lowest value is taken, as recommended in AWS A4.2, to screen out values associated with premature detachments of the magnet.) Two operators independently calibrated each gauge, and the average of the two white-dial values served as the final calibration value for each standard.

An example of a typical calibration plot is shown in Fig. 3 for the gauge 2 data. The 0 to 30 FN data and two sets of extended FN data are plotted as separate straight lines (0-, 7- and 14-g counterweights). Typically, three or more calibration lines would be needed to cover the range of 0 to 100 FN. According to AWS A4.2, two lines can be fitted to the data in the 0 to 30 FN range, while above 30 FN, separate lines are fitted to data for each counterweight.

AWS A4.2 requires the slopes of the various calibration lines be similar, and specifies the maximum variations allowed between the fitted lines and the FN assigned to the primary reference materials. The allowed variations are given in Table 1.

The requirement that the slopes of the various calibration lines be similar helps ensure the estimates of FN calculated from the calibration lines are more or less continuous over the 0 to 100 FN range. If the slopes are very similar and the intercepts are correct, this approach serves as an accurate calibration procedure. It is awkward, however, to justify why sets of discontinuous data are used to develop independent equations for the calculation of FN, particularly when Hooke's Law says the slopes should be the same. For this reason, we took a slightly different approach and developed offsets to produce a white-dial calibration that was continuous over the range of 0 to 100 FN.

The offsets can be determined in several ways. One way is to measure a primary standard using two counterweights. For example, measuring SRM 1313 on gauge 1 produced a white-dial value of 45.75 with no counterweight, and a white-dial value of 150.75 with a 7-g counterweight. The offset, 105, for gauge 1 is the difference between the two values. The offset for gauge 2 was determined to be 118.

Another way to find the offset is to use a digital scale to determine the white-dial reading that corresponds to equal forces for various counterweight configurations. This approach was used to construct the continuous data sets for gauges 1 and 2. Figure 4 shows the calibration line for gauge 1; the calibration line for gauge 2 was similar and can be found in Ref. 4.

Although this offset procedure is not mentioned in AWS A4.2, it clearly follows the intent of the standard to have similar slopes. From a statistical standpoint, this procedure is expected to improve the calibration, because 1) more data are included, and 2) the data are continuous over the region of interest, 0 to 100 FN. However, the deviation allowed by AWS A4.2 between the FN assigned to the primary reference materials and the calibration line is small, so fitting a single calibration line over the whole FN range may not always be possible.

For this study, we decided each gauge would be calibrated for two FN ranges: 0 to 30 and 30 to 100 FN. To minimize the error at very low FN, only calibration data within the 0 to 30 range were used for the linear regression fits in this range. To retain some of the advantages discussed above, all the calibration data (0 to 100 FN) were used for calibration between 30 and 100 FN.

The equations that best fit the calibration data for gauges 1 and 2 are given in Equations 2 to 5.

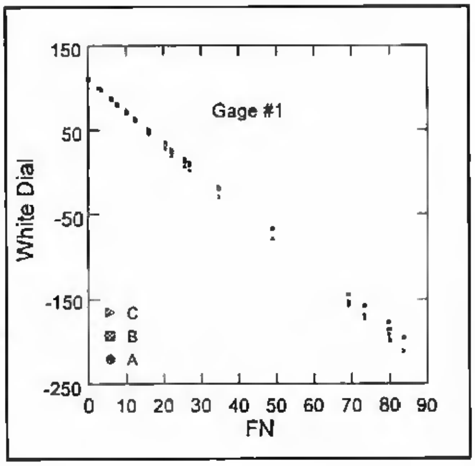

Fig. 9 — The white-dial reading vs. FN for the A, B and C magnets for Magne-Gauge no. 1. The A, B and C magnets are all no. 3 magnets.

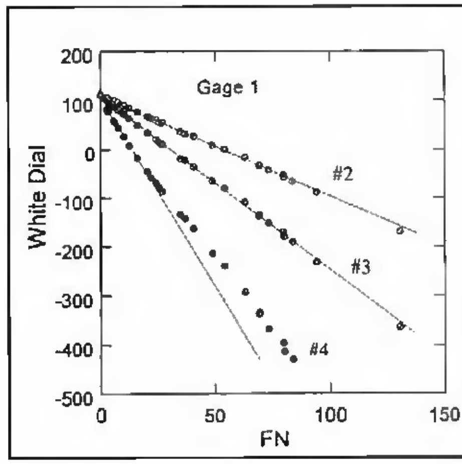

Fig. 10 — The effect of using nos. 2, 3 and 4 magnets on gauge calibration data (magnetic strength increases with magnet number).

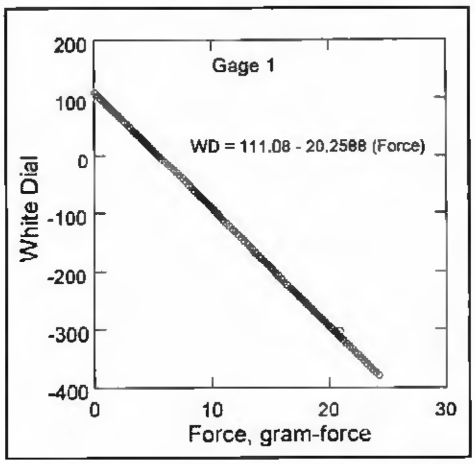

Fig. 11— The relationship of the white-dial reading to the force measured using a digital scale for gauge 1.

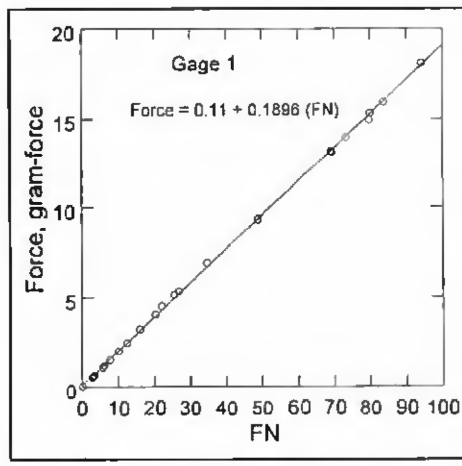

Fig. 12 — The relationship of force to FN for gauge 1.

Gauge 1

$$FN = 27.35 - 0.247\ (WD) \quad \text{for 0 to 30 FN} \tag{2}$$

$$FN = 28.30 - 0.261\ (WD) \quad \text{for 0 to 100 FN} \tag{3}$$

Gauge 2

$$FN = 26.33 - 0.244\ (WD) \quad \text{for 0 to 30 FN} \tag{4}$$

$$FN = 27.04 - 0.252\ (WD) \quad \text{for 0 to 100 FN} \tag{5}$$

where WD is the white-dial reading of the gauge.

Overall, the fits are good, as indicated by the relatively small differences between the predicted and assigned FN values for primary reference materials (Figs. 5 and 6). The standard errors for the slopes of Equations 2 through 5 were typically 0.001 (Ref. 4). All the R-squared values for the equations exceeded 0.999.

Equations 3 and 5 come very close to satisfying the tolerances required by AWS A4.2 over the range of 0 to 100 FN. However, neither equation quite meets the allowed tolerance of ±0.40 FN between 0 and 5 FN. The largest error for Equation 3 in the 0 to 5 FN range is 0.51 FN. For Equation 5, all calibration points meet the required tolerances of AWS A4.2, except the 0 FN point, which has an error of 0.58 FN. The errors are reasonable, but the lack of full compliance for the extended FN equations (Equations 3 and 5) makes it necessary to use Equations 2 and 4 for FN calculations within the 30 FN range. These equations are both well within the required tolerance of AWS A4.2.

The two equations that best fit the 0 to 30 FN data (Equations 2 and 4) have slopes slightly lower than those for the equations developed for the higher FN

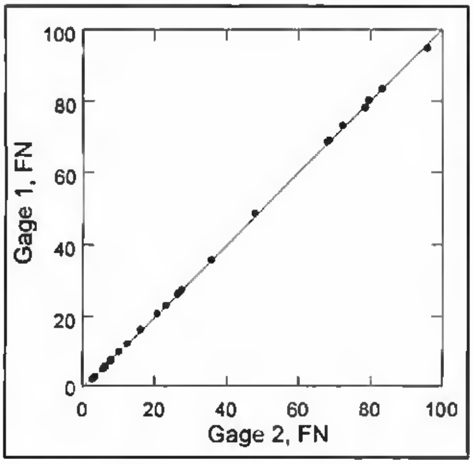

Fig. 13 — The FN calculated using Equations 2 and 3 for gauge 1 and Equations 4 and 5 for gauge 2.

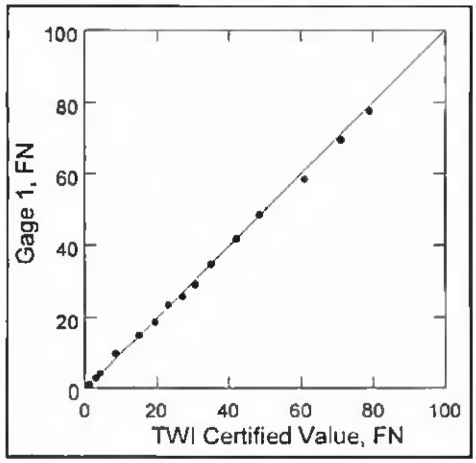

Fig. 14 — The FN measured by gauge 1 plotted vs. the FN certified by TWI.

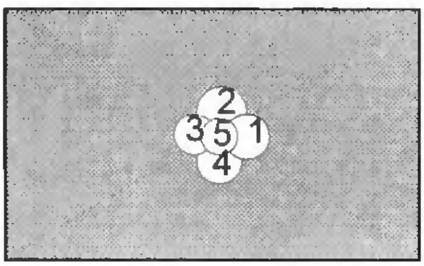

Fig. 15 — Measurements were made in the five positions shown, on the face of the standard opposite the identification number.

range (Equations 3 and 5). This suggests the calibrations are not ideal. (The error plot in Fig. 6 shows a trend at low FN, confirming the data for gauge 1 are fit better by two lines having slightly different slopes, but six calibration points for gauge 2 between 0 and 10 FN have errors very near to 0.)

Calibration Errors

The major contributors to errors in the calibration of the gauges include operator's measurement error (and bias), uncertainty in the thickness of the primary reference materials (±5% of the coating thickness), errors due to the gauge, errors due to magnetic effects and errors associated with the coefficients determined for the line fits.

Some differences in the measurements made by the two operators are apparent in the data. One operator tended to measure the FN slightly lower than the other. Hopefully, the average of the measurements for the two operators is near the average that would be found for a larger population of operators measuring the samples. Since these differences are to be expected for users of the reference materials, we did not change our measurement procedures or attempt to train the operators to produce more similar results. These data, however, show that the magnitude of the difference between operators is significant in relation to the maximum error allowed by AWS A4.2, and some influence of the gauges and samples on the magnitude and sign of the differences is also apparent.

Sources of thickness errors in the primary reference materials used to calibrate the gauges were evaluated by comparing the calibration measurements to the measurements from the 51 other primary reference materials gathered from various sources (Ref. 4). All of the reference materials were measured by the same operator on the same gauge, and then compared to the calibration data for the gauge. As shown in Fig. 7, there are no significant outliers in the data. Equations for lines fit to the calibration data and the independent data (51 other primary reference materials) are virtually identical. From this, we conclude all the primary FN standards used for our calibrations are within the ±5% thickness tolerances expected of them. As confirmation, we sent our primary standards back to the NIST laboratory that is responsible for assigning certified thickness to these specimens, and all were found to be within the certified ranges.

The error at low FN (between the fitted line and actual measurements at 0 FN measured for the gauges and the lowest of the primary FN reference materials) is typically greater than 0.2 FN, which is large, considering that only a ±0.4 FN error is allowed in the 0 to 5 FN range for primary gauge calibrations. Although better agreement in this critical low FN range would be desirable, the lowest FN primary reference materials used for our calibration are well within ±5% uncertainties certified for their thicknesses. At 2.5 FN (2-mm coating thickness), for example, a ±5% error in the thickness of the standard translates to about a 0.4 FN difference. This points out the importance of determining accurate data at 0 FN for these gauges (to help weight the calibration data) and indicates primary reference materials with lower uncertainties in the very low FN range are needed to more accurately calibrate the commercially important 0 to 10 FN range.

Accurate measurements of the thickness of the nonmagnetic coatings become more difficult as the thickness decreases. Fortunately, the relationship between the thickness of the primary reference materials and FN (Fig. 8) is more tolerant of measurement errors as thickness decreases (FN increases). Although small differences in thickness have a more pronounced effect on FN, a larger error in FN can be tolerated in this region (high FN range). This is because the accuracy of the measurement is less critical to the performance and properties of alloys at high FN.

The strengths of the magnets used on the gauges also affect the calibrations. As shown in Fig. 9, using no. 3 magnets of slightly different strengths (all within the tolerance allowed for no. 3 magnets) results in different calibrations for the gauges. As long as the calibration data is linear, this does not present a problem. However, our evaluation of significantly stronger magnets (no. 4 magnets) indicated that magnet strength has an effect on the linearity of the calibration data. In Fig. 10, the calibration data for a no. 4 magnet is shown to diverge from the line drawn to fit the low FN data (FN <20). As already discussed, the data for the no.3 magnets (our calibration data) are also best fit by two lines, indicating a slight nonlinearity, so it appears that magnet/sample interactions affect the linearity of the calibration, which would tend to increase the uncertainty of the calibration.

The changes in slope of the data tend to occur at around 15 to 20 FN for the no. 4 magnets, and at 20 to 30 FN for the no. 3 magnets (on both of our gauges). These FN ranges correspond roughly to the knee of the FN/coating-thickness line in Fig. 8.

Force Calibration

It is difficult to directly compare the gauges used for FN measurement. The comparison can be simplified, however, by replacing the arbitrary white-dial values of the calibration plot with a force. By calibrating the white-dial scale in this manner, only the uncertainty in accuracy of the primary standard and the difference in the strength of the magnet used on the gauge remains.

As shown in Fig. 11, the relationship of the white dial to the force measured on the digital scale is linear, as would be expected by Hooke's Law. This result is comforting and serves as an excellent check on the gauge overall. The overlaps in the data for the various counterweights used (0, 7, 14, 21 and 25 g) are also apparent in Fig. 11. At any point within these overlaps, the offset of the data sets can be determined. We also found that the zero for the gauge was most consistently determined from force data.

Rearranging the terms of the linear equations developed from the white-dial and force data, we find

$$\text{Force} = 5.48 - 0.0494(\text{WD}) \quad \text{Gauge 1} \tag{6}$$

$$\text{Force} = 5.01 - 0.0457(\text{WD}) \quad \text{Gauge 2} \tag{7}$$

Equations 6 and 7 show that for a given white-dial value, gauge 1 applies more force at the sample than gauge 2. Here, there is no effect of magnet strength, because the magnet never detaches from the steel mass.

To relate force to FN, Equations 6 and 7 were substituted for the white-dial data and plotted against the FN data for the primary calibration of gauge 1 in Fig. 12. The result is a plot for which both the X and Y axes are traceable to calibration standards. The arbitrary white-dial scale has been eliminated.

The FN-force calibration equations developed from this data are

$$\text{FN} = -0.57 + 5.27\ (\text{Force}) \quad \text{Gauge 1} \tag{8}$$

$$\text{FN} = -0.61 + 5.52\ (\text{Force}) \quad \text{Gauge 2} \tag{9}$$

These slopes, 541 and 561 FN per newton (5.27 and 5.52 FN per g-force), for gauges 1 and 2, respectively, are defined as the detachment force by AWS A4.2 but are actually a calibration factor. A decrease in the calibration factor relates to an increase in magnet strength. At an FN of 80, for example, the calibrations indicate a 1560 N force (15.3 g-force) is needed to detach the magnet on gauge 1, and a 1490 N (14.6 g-force) is needed to detach the magnet on gauge 2. The differences in the applied force needed to detach the magnets decrease with decreasing FN.

Summary of Primary Calibrations

Overall, we conclude the gauge calibrations are within the accuracies required by AWS A4.2, and using these gauges and calibrations, we can certify secondary reference materials that will have accuracies that will meet or exceed those of past producers of these materials. This conclusion is supported by practical verifications we performed to check the performance of our gauges. For example, when the FNs calculated using gauges 1 and 2 are compared (Fig. 13), only slight differences are apparent, and the FN values calculated for these gauges are in good agreement throughout the 0 to 100 FN range. To verify our gauges compared well to gauges previously used for certifying these materials, FN was measured on secondary reference materials from TWI. As shown in Fig. 14, FN measurements made on our gauge 1 agree well with the certified values assigned to the specimens by TWI. The agreement for the gauge 2 data showed a similar trend. This result indicates continuity between the FN values assigned by NIST and those assigned by TWI.

Clearly, the use of commercial gauges and the current calibration practices are not ideal for use in our FN reference material program. At this point, there is not an adequate understanding of the variables contributing to calibration error, particularly those influencing the linearity of the calibration. However, we are satisfied with the performance of our gauges and our calibration procedures (for now).

We found several of the calibration procedures we incorporated into our program useful and suggest they be considered as requirements (or recommendations) in AWS A4.2. Specifically, we found adding the extra procedural step of force calibration was useful and plan to continue to track the performance of the gauges in this manner. It provides 1) detailed information on the linearity of the gauges, 2) a way to separate magnetic coupling variables from mechanical variables, and 3) a means to better compare the gauges to one another. Torsion balances have been evaluated in the past for FN measurement, so this more direct approach is not new (Ref. 5). But, by simply adding a force calibration to the existing procedure for calibrating white-dial gauges, the best of both types of measurement devices can be realized, specifically, the accuracy and design of the MGIs and the traceability of the force calibration to a digital scale.

In addition, the determination of 0 FN using a balance (rather than operator judgment) and the use of continuous data from 0 to 100 FN for the calibration of gauges for measurements at high FN should be considered in AWS A4.2. The use of a balance to determine 0 FN is simple and removes operator's bias for this critical datum. The use of continuous data in the calibration of extended FN ranges is more consistent with the principles on which the linear calibrations are based, and will likely help reduce variations in slopes obtained when fitting smaller groups of calibration data (for various counterweights) independently.

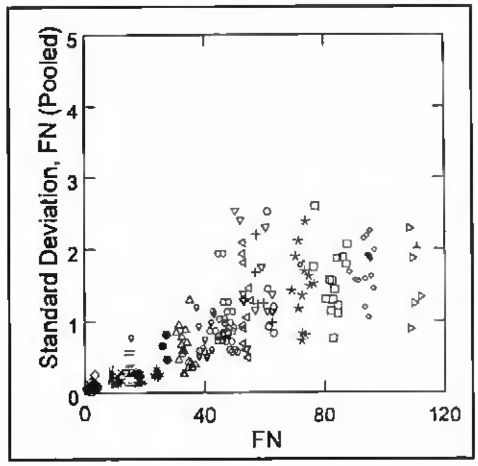

Fig. 16 — The pooled standard deviation for the batch of secondary FN reference materials showing variation similar to, but often lower than, the grand standard deviation for the specimens. Each symbol refers to a different FN range.

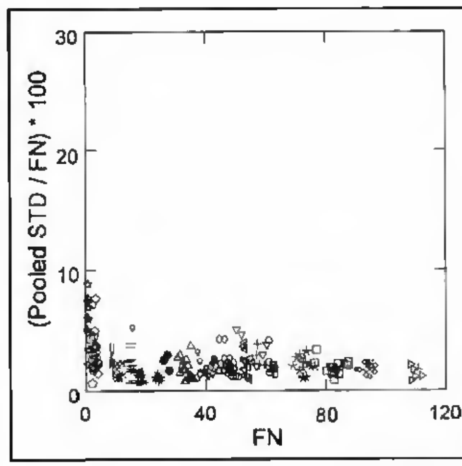

Fig. 17 — The pooled standard deviation normalized by FN. Each symbol refers to a different FN range.

Measurement of Secondary Reference Materials

Measurement Procedure

Measurements were made on the sec-

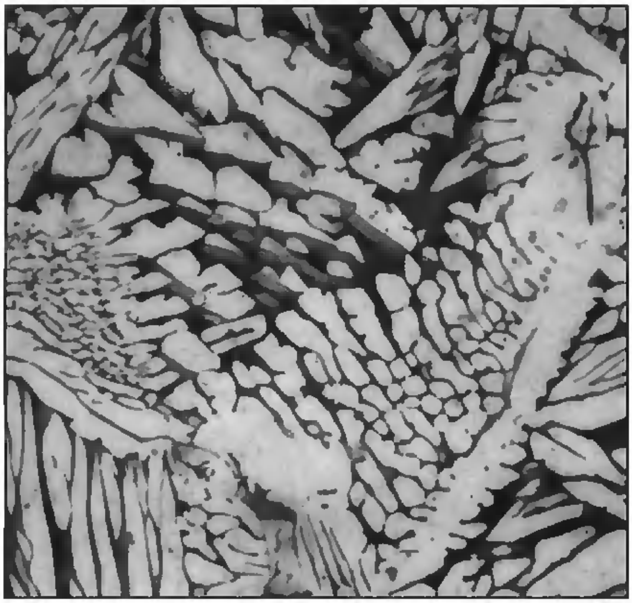

Fig. 18 — Example of the deeply etched microstructure for a specimen with an FN near 42 (160 by 160 μm area).

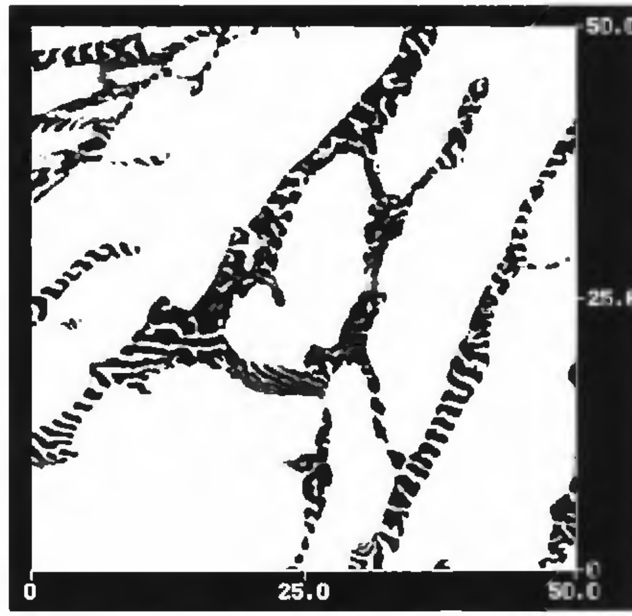

Fig. 19 — MFM image of magnetic domains in the secondary FN standard specimen in Fig. 18. The larger white regions show the morphology of the austenite phase. The black-and-white striped regions show the ferritic phase, indicating the size and orientation of magnetic domains. Units in micrometers.

Table 2 — Example Data for a Secondary FN Standard

Gauge (Operator)	P1	P2	P3	P4	P5	Mean	STD
Gauge1, (1)	51.79	51.53	52.57	52.31	51.53	51.94	0.47
Gauge 1, (2)	52.75	52.75	53.50	53.00	53.00	53.00	0.31
Gauge 2, (1)	51.26	49.96	51.53	53.09	52.57	51.68	1.22
Gauge 2, (2)	53.00	53.25	53.00	53.50	52.75	53.10	0.29
Mean 52.20	51.87	52.65	52.97	52.46	52.43		
STD 0.82	1.47	0.84	0.50	0.65			

Note: Statistics for each of the five positions (P1 through P5) are shown (units are FN).

ondary reference materials in five positions, by two operators using two gauges. As shown in Fig. 15, the five positions are clustered about the center of the specimen face. At each position, five measurements were made (by each operator on each gauge), but only the lowest repeatable measurement of the five was retained. (This portion of the procedure is to screen out measurements for which the magnet detaches prematurely, in accordance with AWS A4.2.) In all, 100 measurements were made on each standard, but only 20 were retained for the permanent data record and the calculation of the certified FN value.

Data Analysis

The data for a secondary standard were evaluated by calculating the mean and standard deviation (STD) for each gauge and operator combination, and each specimen position. In the example data record shown in Table 2, these calculations are shown by row and column, respectively.

Typically, the STD is lowest for measurements performed by the same operator on the same gauge (row). This STD is the best indicator of variation in FN for the sample because it is due primarily to differences in FN measured at the five specimen locations. These values are deemed particularly characteristic of the specimen when the STD for the four conditions (two gauges, two operators) show similar variation. In this example, the measurements made with gauge 2 by operator 1 have much higher variation than the other three conditions. This likely indicates a measurement error, and we used this approach to flag the data files that needed further scrutiny and remeasurement. The mean values are also helpful in detecting measurement errors, assuming the means for a given gauge are similar if the operators have made good measurements. The mean and STD for the measurements made at a single specimen location (column) include variation due to the differences between the gauges and operators. These values are the best indicator of how closely a customer measuring the standard might expect to match our measurement at a given sample location. Because users of the reference materials are instructed to make their measurements at the center of the sample face, the mean for specimen position 5 is defined as the certified value for the secondary standard.

A grand average and STD, and a pooled STD were also calculated. The grand average and STD for the 20 measurements made on the specimen provide our best overall estimate of the FN, and variation in FN, because differences in FN due to sample location are included. The pooled estimate, S, of the variation in the specimen is our best estimate of the variation in FN due solely to the sample. The value S is calculated as shown in Equation 10, where s_1 to s_4 are the standard deviations calculated for the five measurements for each of the four operator-gauge combinations.

$$S = \sqrt{\frac{s_1^2 + s_2^2 + s_3^2 + s_4^2}{4}} \qquad (10)$$

Data Trends

As we developed the certification data on the secondary reference materials, we were able to determine a representative measure of the standard deviation in the measurements for the batch. An overview of the data is presented in Figs. 16 and 17. The various symbols used in these figures represent the rings from which the samples were taken.

The grand standard deviations of the secondary reference materials increased with increasing FN, as might be expected. However, the pooled standard deviations shown in Fig. 16 indicate the FN variations in the specimens do not necessarily continue to increase with increasing FN. The pooled standard deviations for the specimens show that above about 50 FN, the variation might be expected to remain below 3 for good specimens. A group of specimens between 50 and 60 FN were found to have unusually high standard deviations and were diverted for research uses rather than being used as secondary FN reference materials.

In Figure 17, the pooled standard deviation data are normalized with respect to FN. The relative variation in FN is shown to be highest within the 0 to 5 FN range, indicating the influence of different gauges and operators on the FN measurements is most significant at low FN. This 10% variation for the normalized pooled statistics (Fig. 17) best represents the FN variation inherent to the specimens in the 0 to 5 FN range.

Since this is the first batch of secondary specimens for which these statistical data have been compiled, we cannot compare the quality of these specimens with those of specimens produced in the past. The variation in the FN of the specimens does appear to be reasonable, considering the various requirements for measurement accuracy in AWS A4.2. These and future data will be used to develop better documentation on the quality of secondary FN reference materials. The data will also be used to support and update the requirements of AWS A4.2.

Certification of Secondary Reference Materials and Discussion of Errors

Each set of FN reference materials (RM) contains eight individual specimens. A table that accompanies the set provides three types of data on each specimen and a plot reflecting the calibration error. The table includes the average and STD for position 5, the pooled statistics and the grand values for each specimen.

The FN value at the center of the sample (position 5) is defined as the reference value. The pooled statistics and grand averages are provided for information only, to more fully describe the variation in FN measurement within a specimen. More discussion of the uncertainty analysis is included in Ref. 4.

Microstructure

We have examined the microstructures of secondary reference materials, selected over a broad range in FN. The specimens chosen for these destructive evaluations were those with the highest variation in FN. These samples were used to study the microstructures for the various FN levels selected and to identify microstructural features that might explain the high variation in FN measured for these particular specimens. The specimens were prepared for evaloations by both light-microscopy and atomic force microscopy. The primary features of interest for these evaluations were the ferritic phase content and oniformity, the "grain size," magnetic domain size and solidification flaws.

Grain Size

The grain size (cell or dendrite packet size) is difficult to quantify in many of these microstructures. However, the grain size, which is delineated by differences in dendrite orientations, can be estimated qualitatively in many of the samples (particularly for specimens of 30 FN or more). Etching to enhance grain contrast was used occasionally to help delineate the boundaries, but in many of the alloys an almost continuous austenitic phase (white) marks the grain boundaries well enough to roughly estimate the grain size for our purposes. The apparent grain diameters for the specimens evaluated ranged from about 100 to 300 µm. This range in grain diameter appears to be common to the specimens that were evaluated, and it is likely that the average grain size for the specimens is well below 300 µm. Assuming the magnetic measurements made with the FN gauge cover microstructural regions of near 1 mm^2, then the relative scale of the ferrite/austenite morphologies are reasonable, and similar to those expected for stainless steel welds.

Ferrite Content

The ferritic phase content of the specimens was estimated using light microscopy and image analysis. The measurements were made on planes parallel to, and just below the plane on which the FN measurements were made. The specimens were lightly ground (800 grit) and polished to remove surface damage, then etched using a Beraha II solution for the evaluations. Area fraction counts were made at two different magnifications, and for each count, 100 adjacent fields were measured. For the lower magnification, the total area of each count was about 2.5 mm^2 and for the higher magnification, the total field area was about 0.64 mm^2.

Typically, several counts were made at each of the two magnifications. The influence of the etch (light vs. deep etching condition) on the area counts was significant in some cases, as is always the case. However, with appropriate intensity thresholds, similar area fractions were counted for both conditions. The deeply etched samples (Fig. 18) were found to provide excellent contrast for the analysis, particularly for the higher FN levels.

The results of the area-fraction counts were surprising. We found a 1:1 correlation between FN and the ferrite area fraction measured (up to at least 50 FN). It was expected that above 10 FN, the two measurements would start to diverge, and by 50 FN be significantly different[2]. We remeasured several of the samples to check the result and believe them to be accurate (within about 5%).

Three of the samples were evaluated by X-ray diffraction and alternative etching techniques to determine whether they might contain sigma phase, which is nonmagnetic and could be incorrectly identified (and counted) as ferrite in the light-microscopy results. No sigma phase was present in the samples.

Solidification Flaws

Microstructural evaluations showed the centrifugal cast FN specimens have solidification flaws, as might be expected. These flaws can be quite minor and would not be expected to result in significant deviation for FN measurements. Larger flaws were observed, however, that could cause variation for FN measurements. We expect these types of flaws were the main reason for unacceptable FN variation in a few of the reference specimens. These specimens were rejected and used only for the microstructural studies.

Atomic-Force Microscopy

The microstructure (by atomic-force microscopy [AFM]) and the magnetic fields (by magnetic-force microscopy

2. Area fraction counts of the percent ferrite in stainless steel weld have shown the percent ferrite and FN to be approximately equal up to 10% ferrite. At levels of more than 10% ferrite, FN measurements have been shown to overestimate the percent ferrite in welds.

[MFM]) for a low-FN specimen and one with an FN near 42 were also evaluated. These evaluations showed that the phases delineated in light microscopy, AFM and MFM are the same phases: Magnetic domain images show the magnetic ferritic phase is correctly and clearly revealed by light microscopy and AFM.

The characteristic microstructural features (austenite islands, bordered by ferrite) are closely spaced, as shown in Fig. 19. The width of the austenitic phase separating the magnetic ferrite phase is typically less than 25 μm wide in this example. This fine structure shows many dendrites are included in each measurement by the 1-mm-diameter magnet of the gauge. The magnetic-force image shows the magnetic domains in the ferrite phase have characteristic dimensions near 2 or 3 μm, giving more information on the fine distribution of the magnetic fields through these specimens.

Summary

Overall, we conclude the gauge calibrations are within the accuracies required by AWS A4.2, and, using these gauges and calibrations, we can certify secondary reference materials that will have accuracies that will meet or exceed those of past producers of these materials. This conclusion is supported by practical verifications done to check the performance of our gauges and by comparisons to the FN values assigned to secondary reference materials by TWI.

We find several of the calibration procedures that we incorporated into our program useful, and suggest they be considered as requirements (or recommendations) in AWS A4.2. Specifically, we find adding the extra procedural step of force calibration is useful. It provides 1) detailed information on the linearity of the gauges, 2) a way to separate magnetic coupling variables from mechanical variables and 3) a means to better compare the gauges to one another. By simply adding a force calibration to the existing procedure for calibrating white-dial gauges, the best of both types of measurement devices can be realized, specifically, the accuracy and design of the Magne-Gauge instruments and the traceability of the force calibration to a digital scale.

In addition, the determination of 0 FN using a digital scale (rather than operator judgment) and the use of continuous data from 0 to 100 FN for the calibration of gauges for measurements at high FN should be considered in AWS A4.2. The use of a balance to determine 0 FN is simple and removes operator's bias for this critical datum. The use of continuous data in the calibration of extended FN ranges is more consistent with the principles on which the linear calibrations are based, and will likely help reduce variations in slopes obtained when fitting smaller groups of calibration data (for various counterweights) independently.

Estimation of the ferritic phase content of the specimens by light microscopy and image analysis revealed a 1:1 correlation between FN and the ferrite area fraction measured (up to at least 50 FN). It was expected that above 10 FN, the two measurements would start to diverge, and by 50 FN be significantly different. We remeasured several of the samples to check the result, and believe the results to be accurate (within about 5%). X-ray diffraction and alternative etching techniques were unable to detect any sigma phase, one possible explanation for this anomalous result.

Atomic force microscopy revealed that the width of the austenitic phase separating the magnetic ferrite phase is typically less than 25 μm wide. This fine structure indicates many dendrites are included in each measurement by the 1-mm-diameter magnet of the gauge, explaining the low scatter in the measurements. Also, the magnetic-force image shows the magnetic domains in the ferrite phase have characteristic dimensions near 2 or 3 μm.

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