National Institute of Standards & Technology

Report of Investigation

Reference Material 8481

Secondary Ferrite Number Standard - High Range

Serial No: SAMPLE

This Reference Material (RM) is intended for the calibration of instruments used to measure weld metal ferrite content in accordance with American National Standards Institute (ANSI), American Welding Society (AWS) and International Standards Organization (ISO) normative standards ANSI/AWS A4.2 and ISO 8249 [1,2]. The ferrite content is measured in terms of a method defined quantity, the Ferrite Number (FN). NIST measurements were made in accordance with ANSI/AWS A4.2 [1].

Description and Source of Material: Each RM unit is a set of eight individually measured specimens. Each specimen is approximately 10 mm × 12 mm × 20 mm and has an identification number scribed on one of the 12 mm × 20 mm faces. The measurement surface is the face opposite the surface that contains the identification number. RM 8481 comprises eight specimens having FN values ranging from 30 FN to 120 FN. The related RM 8480 Secondary Ferrite Number Standard - Low Range, comprises eight specimens having FN values ranging from 0 FN to 30 FN.

The RM was made from centrifugal castings of chromium-nickel steel alloys. The ferrite content was varied by adjusting the composition of the alloy. The cast specimens approximate the ferrite distribution in a weld deposit and have a solidification structure similar to that of welds [3]. Like the ferrite in welds, the magnetic response of the ferritic (magnetic) phase varies with alloy composition. The homogeneity of the cast material was demonstrated to be sufficient for use as secondary FN standards [4].

Metrological Traceability: The measurand is ferrite number and is intended for the calibration of instruments used to measure weld metal ferrite content in accordance with ANSI, AWS, and ISO normative standards ANSI/AWS A4.2 and ISO 8249[1,2].

Expiration of Value Assignment: RM 8481 is valid indefinitely, within the measurement uncertainty specified, provided the RM is handled and stored in accordance with instructions given in this Report of Investigation (see "Instructions for Handling, Storage and Use"). Periodic validation of this RM is not required. This report is nullified if the RM is damaged, contaminated, or otherwise modified.

Material Maintenance: NIST will monitor this RM over the period of its validity. If substantive technical changes occur that affect the value assignment before the expiration of this report, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Overall direction and coordination of the technical measurements leading to certification were performed by F. DelRio of the NIST Applied Chemicals and Materials Division. FN measurements and analysis were managed by C.N. McCowan of the NIST Applied Chemicals and Materials Division. Statistical support was provided by J. Wang of the NIST Statistical Engineering Division.

Support aspects involved in issuance of this RM were coordinated through the NIST Office of Reference Materials.

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Report Issue Date: 07 November 2018
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Centrifugal castings were manufactured by Merinov Enterprises (formerly the Mladis Co., Moscow, Russia).

NIST Measurement Procedure: The FN measurements were made with a Magne-Gage-type instrument[(1)] using a #3 magnet, as specified by ANSI/AWS 4.2 [1]. Measurements were made on each specimen in the RM unit in five positions by two operators using two gages. As shown in Figure 1, the five positions are clustered about the center of the specimen face. At each position, five measurements were made by each operator using each gage. The lowest repeatable measurement of the five was retained in accordance with ANSI/AWS 4.2 (to screen out measurements for which the magnet detaches prematurely). In total, 100 measurements were made on each specimen, but only 20 are used in the calculation of the FN value.

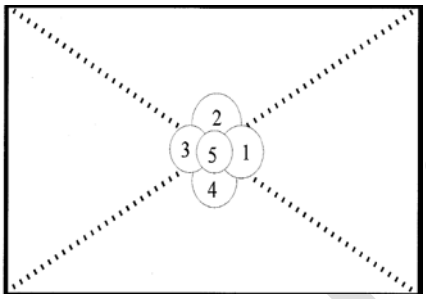

Figure 1. Measurements were made in the five positions shown, on the face of the specimen opposite to the identification number.

Uncertainty Analysis: The RM consists of eight individually measured specimens. Table 1 provides three columns of data on each specimen: the mean and standard uncertainty for position 5 of the specimen, pooled statistics from all five positions on the specimen, and grand means and standard uncertainties for all five positions. The data for position 5 is given as the reference value in accordance with normative standards [1,2]. The pooled statistics and grand averages are provided in Table 1 for information only to more fully describe the variation in FN measurement within a specimen. The pooled statistics combine information from each gage-operator combination. The data used to generate the grand mean considered every measurement as an individual observation regardless of gage, operator, or position.

Measurement Repeatability (u_R): The Type A uncertainty in FN measurements taken at a single location (position 5) is due to differences in operators, gages, and magnets used on the gages. u_R is given in Table 1.

Calibration Error (u_C): The Type B uncertainties in FN measurements due to sources of bias include: (1) uncertainty due to variation in the thickness of the coating thickness standards (SRMs 1321, 1363a and 1364a) used to calibrate the gage, (2) uncertainty of the dial readings on the gage, and (3) uncertainty related to the fit of the calibration curve.

The calibration error shown in Figure 2 was determined by simulation. A triangular distribution (± 0.5) was used to model the error of the dial readings from the gage. The root mean square error of the calibration result, based on 10 000 Monte Carlo samples, was used as the standard uncertainty for the calibration error.

Combined Uncertainty (u): The two standard uncertainties (u_R and u_C) can be combined by quadrature addition to obtain the overall standard uncertainty (combined uncertainty). To determine the overall uncertainty for a given specimen, the standard uncertainty for the specimen in Table 1 and the calibration error estimated for the FN level of the specimen in Figure 2 are combined using Equation l.

$$u = \sqrt{u_R^2 + u_c^2} \tag{1}$$

Pooled Uncertainty (u_p): The lowest repeatability estimate is expected for measurements performed by the same operator using the same gage. This within-specimen uncertainty is a good indicator of material homogeneity. These values are deemed particularly characteristic of the specimen when the uncertainties for the four conditions (two gages and two operators) show similar variation.

[(1)] Certain commercial instruments and materials are identified to adequately specify the experimental procedure. Such identification does not imply a recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the instruments or materials are the best available for the purpose.

The mean and standard uncertainties for measurements made at a single location (Position 5, reported in Table 1) include uncertainty due to the differences between the gages and operators. These values are the best indicator of how closely a user measuring the standard might expect to match the NIST measurement at a given location.

The pooled estimate of the variation in the specimen, u_p reported in Table 1, was calculated using Equation 2,

$$u_p = \sqrt{(u_1^2 + u_2^2 + u_3^2 + u_4^2)/4} \quad (2)$$

where u_1 to u_4 are the standard uncertainties calculated for the five measurements at each of the four operator-gage combinations. This statistic best characterizes the overall variation in the FN due to local variations in the microstructure of each specimen.

The grand mean and standard uncertainty for the 20 measurements made on each specimen reported in Table 1 provide NIST's best overall estimate of the FN, and variation in FN that includes all contributions of measurement uncertainty and specimen homogeneity.

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Handling and Storage: Do not expose the RM to magnetic fields, temperatures outside the range of –40 °C to +40 °C, or corrosive atmospheres or solvents. Avoid mechanical damage or material removal (e.g., grinding or polishing) of the measurement face of the RM. Store the RM in the case provided when not in use.

Use: To calibrate an instrument in the range covered by RM 8481, measure the ferrite number at the center of the measurement face (Position 5, see Figure 1) of each specimen supplied. The measurement face is opposite the face on which the specimen identification number is scribed. Consult the instrument user's manual and current normative standards for procedural specifics and data analysis [1,2]. For the purposes of many users, the Position 5 mean FN value (Table 1) will be the value used to calibrate or verify their instrument.

Specimens may be wiped clean before measurement using a soft cloth to free the surface of dust. A mild detergent solution or solvent such as isopropyl alcohol may also be used to clean the surface of any contaminants.

REFERENCES

[1] ANSI/AWS 4.2-97, Standard Procedures for Calibrating Magnetic Instruments to Measure the Delta Ferrite Content of Austenitic and Duplex Austenitic-Ferrite Stainless Steel Weld Metal, American Welding Society, Miami, Fl.

[2] ISO 8249:1995, Determination of Ferrite Number in Austenite Weld Metal Deposited by Covered Cr-Ni Steel Electrodes, ISO, Geneva, Switzerland (1995).

[3] Merinov, P.; *Preparation of Centrifugally Cast Ferrite Standards of the Russian Delegation IIW* Doc. II-1223-93 (II-C-933-93).

[4] Sugaya, Y.; Aihara, T.; *IIW SC-II-C 6th Round Robin on Ferrite Secondary Standards*, (August 1993) IIW Doc. II-1228-93 (II-C-935-93).

Report Revision History: 07 November 2018 (Editorial changes); 06 March 2000 (editorial change); 23 March 1999 (original report date).

Users of this RM should ensure that the Report of Investigation in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-2200, fax (301) 948-3730, e-mail srminfo@nist.gov, or via the Internet https://www.nist.gov/srm.

Table 1: Reference and Information Values for RM 8481, Serial Number H-Sample

Relative FN Level	Specimen ID	Reference Value (FN)		Information Values (FN)			
		Mean and Standard Uncertainty, u_R (for position 5)		Pooled Mean and Standard Uncertainty, u_P		Grand Mean and Standard Uncertainty, u_C	
9	10-68	--2.5	--68	--	--	--	--
10	29-1631	--4.5	-0.26	--	--	--	--
11	30-2133	--9.1	--77	--	--	--	--
12	11-289	--7.6	--65	--	--	--	--
13	24-1573	--73.6	-- 12	--	--	--	--
14	14-460	--5.0	--83	--	--	--	--
15	21-401	--7.2	--.00	--	--	--	--
16	23-800	--2.0	--40	--	--	--	--

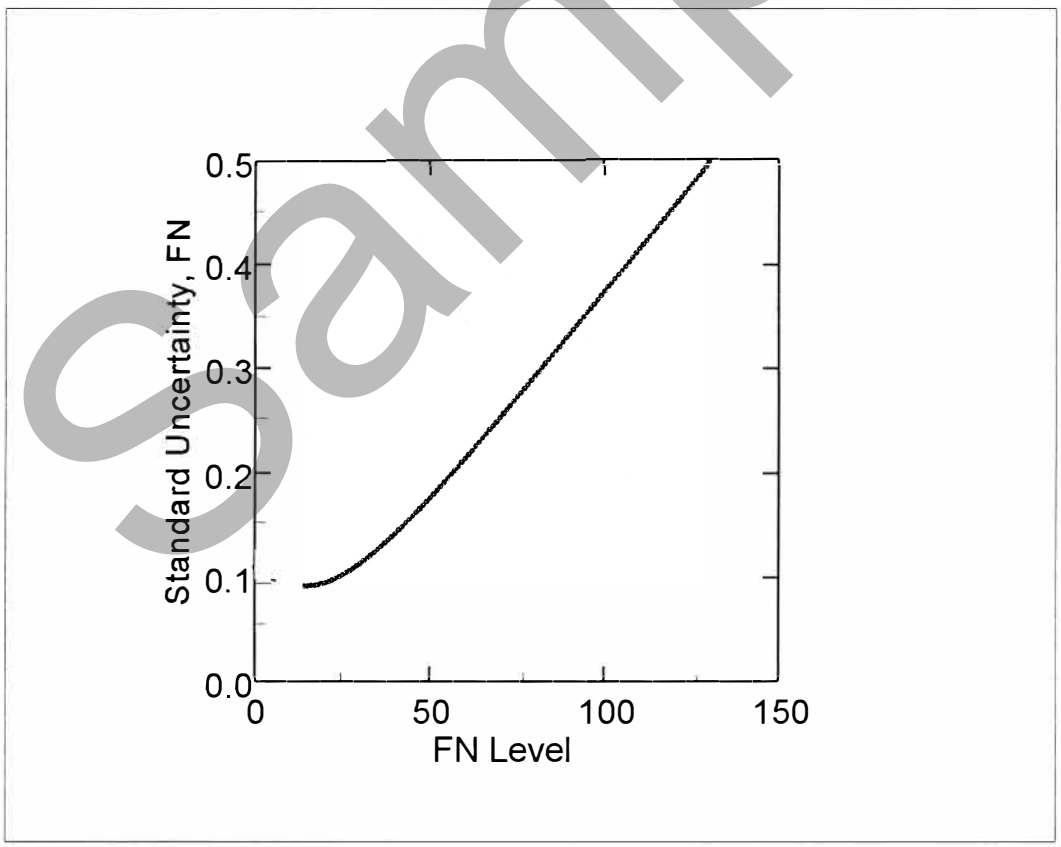

Figure 2: Calibration error (S_R) for the secondary standards.