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Ferrite measurement in austenitic and duplex stainless steel castings

William J. Ruprecht

To the Graduate Council:

I am submitting herewith a thesis written by William J. Ruprecht entitled "Ferrite measurement in austenitic and duplex stainless steel castings." I have examined the final electronic copy of this thesis for form and content and recommend that it be accepted in partial fulfillment of the requirements for the degree of Master of Science, with a major in Metallurgical Engineering.

Carl D. Lundin, Major Professor

We have read this thesis and recommend its acceptance:

Charlie R. Brooks, R. A. Buchanan

Accepted for the Council:
Carolyn R. Hodges

Vice Provost and Dean of the Graduate School

(Original signatures are on file with official student records.)

To the Graduate Council:

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Ferrite Measurement in Austenitic and Duplex Stainless Steel Castings

A Thesis
Presented for the
Master of Science
Degree
The University of Tennessee, Knoxville

William J. Ruprecht III
December 1999

DEDICATION

This thesis is dedicated, with sincere thanks, to my parents

William J. Ruprecht, Jr

and

Rebecca A. Ruprecht

who have always made my academic and

professional success their first priority.

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ABSTRACT

Practical and accurate non-destructive means for the measurement of the ferrite content of duplex stainless steel castings is a necessity from the specification and service performance consideration standpoints. The ability to determine ferrite rapidly, accurately and directly on a finished casting, in the solution annealed condition, can enhance the acceptance, save on manufacturing costs and ultimately improve service performance of duplex stainless steel cast products. If the suitability of a non-destructive ferrite determination methodology can be demonstrated for standard industrial measurement instruments, the production of cast secondary standards for calibration of these instruments is a necessity. With these concepts in mind, a series of experiments were carried out to demonstrate, in a non-destructive manner, the proper methodology for determining ferrite content. The literature was reviewed, with regard to measurement techniques and vagaries, an industrial ferrite measurement round-robin was conducted, the effects of casting surface finish, preparation of the casting surface for accurate measurement and the evaluation of suitable means for the production of cast secondary standards for calibration were systematically investigated.

It was found that surface finish effects can induce significant differences in measured ferrite content. Several finishes were identified, which when applied (Feritscope® method), resulted in a significant decrease in measured ferrite content on a nominally 74 FN sample (>10 FN and well outside the 2σ variation of $\pm$ 0.5) defined for a polished surface.

An interlaboratory round-robin test series revealed that cast secondary calibration standards can be produced from castings. It was found that for both Magne Gage and Feritscope®, the repeatability ferrite measurement of centrifugal castings surpassed that of statically cast materials. Reproducibility was also unaffected by ferrite measurement technique.

Additional characterization of ferrite content, as a function of depth below a cast surface, revealed that the ferrite content immediately below a cast surface is not indicative of the bulk casting. At least 0.125" of material must be removed to ensure that the measured ferrite content is representative of the bulk casting. Analysis of operator and instrument error, for the Feritscope® showed that error induced by the operator exceeds that of the instrument alone.

Additional tests characterized the Feritscope® by establishing its probe interaction volume (0.050"). Considering instrument repeatability and reproducibility, the Feritscope® was clearly identified as the superior instrument for ferrite measurement. The data obtained from this research program provides recommendations to insure accurate, repeatable and reproducible ferrite measurement and qualifies the Feritscope® for field use on production castings.

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PROGRAM INTRODUCTION

Ferrite measurement techniques evolved after the realization that austenitic stainless steel weld metals, containing a moderate amount of ferrite, were free of hot cracking related weld defects. Ferrite measurement was immediately identified as a method by which engineers could quantify the amount of weld metal ferrite and ensure that their fabrications would be free from hot cracking. The advent of duplex stainless steels further re-emphasized the need for adequate ferrite measurement techniques as a suitable ferrite/austenite phase balance provides adequate mechanical properties and improved corrosion performance. In order to qualify their cast products, reliable means to measure ferrite were developed to assure compliance with industrial practices and customer requirements.

The Ferrite Measurement program was conceived with the ideology that an increased database, with regard to current ferrite measurement techniques, will benefit producers and users of stainless steel castings. Utilizing available instrumentation, a series of "round-robin" tests have been implemented to study lab-to-lab variation in traditional magnetic and modern electronic ferrite measurement techniques. Since the implementation of this program (February 1998), the Materials Joining Research Group (University of Tennessee – Knoxville) conducted a survey of literature and initiated studies into the characterization of castings. Studies involving ferrite content measurement as a function of surface roughness were designed. Efforts to characterize ferrite content as a function of depth from the surface of a casting were implemented.

Additionally, this research effort has moved toward the development of a practice to manufacture cast secondary standards, which are required for the calibration of electronic ferrite measurement equipment.

This increased knowledge base has a direct impact upon industrial corporations that manufacture duplex stainless steel castings. Analysis of ferrite typically requires a more time consuming and possibly destructive analysis in which castings are sectioned for metallographic analysis or resized to complement an instrument. With the validation of improved techniques, the amount of expended labor and energy usage can decrease while productivity can improve. It is the desire of this research effort that a marked reduction in energy usage and associated material and labor costs shall result from an increased understanding of new ferrite determination techniques and their applicability to industry.

CHAPTER II

PROJECT GOALS

The following project goals have been defined for this program:

- Comparison of metallographic, magnetic and electronic permeability methods of ferrite measurement and assessment of statistical repeatability for each method.
- Examination of variations in ferrite content by performing surface-to-core depth profile measurements on castings.
- Examination of the effect of surface finish on measurement capability.
- Development of standard ferrite measurement procedures.
- Development of a methodology for the production of Cast Secondary Standards.
- Publication of research and guidance in ferrite measurement.

CHAPTER III

LITERATURE REVIEW

Introduction

A critical review of published literature has been conducted to define methods of ferrite measurement and means of round-robin testing for measurement validation. Special attention has been paid to relevant technical specifications (AWS A4.2) as well as research articles. This review is primarily concentrated on applicable ferrite measurement techniques and their inherent capability and accuracy. The following section, "Importance to Industry", describes the desire of industrial producers and users of stainless steel castings to obtain repeatable and cost effective methods of ferrite determinations for their finished products.

Importance to Industry

Producers and users of stainless steel castings have recognized the need to accurately quantify the microstructure of their finished product. With increasing demand being placed upon quality and reliability by institutions like the International Standardization Organization (ISO 9000 / ISO 9001), engineers have recently become concerned with their ability to accurately quantify the ferrite content in a casting, and thus to verify the capability of their manufacturing processes. Additionally, efforts to

eliminate destructive evaluation, as a method to qualify castings, have yielded to new developments in ferrite measurement techniques.

With the advent of new technology for non-destructive evaluations of ferrite content, new options have been introduced to foundries, consumers and engineers. Prior to examining current techniques, a review of "Advances in Ferrite Measurement" was compiled from a series of Adam's Lectures presented at the American Welding Society's annual meetings and then subsequently published in the Welding Journal.

Advances in Ferrite Measurement

In his 1974 Adams Lecture, W.T. DeLong summarized the subject of ferrite measurement for the 55th annual American Welding Society (AWS) Meeting. During his lecture, DeLong recounted the characteristics of ferrite and its importance in the field of welding. Dating his lecture material prior to World War II, DeLong was able to characterize early observations of the effect of ferrite on cracking, fissuring, mechanical properties and corrosion performance of weldments.[1]

As a part of his lecture, DeLong recounted methods of ferrite measurement including calculation of ferrite from chemistry, metallography, magnetic measurement, x-ray diffraction and magnetic permeability. His critique of each available method, as applied to weld metal substrates consisting of austenitic stainless steels, revealed the following observations:[2]

- Ferrite determination from chemistry had been evaluated and was considered a statistically viable option for ferrite prediction through the application of

appropriate constitution diagrams. The Schaeffler and DeLong diagrams were the only applicable diagrams which incorporated alloy chemistry into ferrite content prediction.

- The statistical accuracy of metallographic measurements (point counting) was highly influenced by the ferrite colony size, and the introduction of automated techniques had done little to improve upon operator variances. It was also observed that changes in ferrite content within the same substrate made quantification representative of the entire sample difficult.
- Magnetic measurements, using commercially available instruments, were defined to be a suitable method of quantifying ferrite content. Such devices are discussed further in this review.
- The use of x-ray diffraction as a ferrite measurement technique was applicable. However, diffraction patterns were diffuse in nature and subject to interpretation. It was concluded that sufficient accuracy was unattainable using this technique.
- Magnetic permeability measurements had not yet been accurately researched. Although proposals had been submitted on this subject, insufficient research had been conducted to validate such a technique.*

*Note: Future developments would later validate this method of ferrite measurement.

Incorporating these techniques into a world-wide round-robin test series, the International Institute of Welding (IIW), Subcommission IIC and the Advisory

Subcommittee of the High Alloys Committee of the Welding Research Council (WRC), initiated two doctrines in 1974. They are presented as follows:

1) Based upon the round-robin test series, the WRC Advisory Subcommittee proposed that the term "Ferrite Number" (FN) replace conventional "percent ferrite" as a method to quantify ferrite content. The lack of appropriate universal calibration procedures and reference standards had produced significant lack of agreement between laboratories. At that time, FN was meant to directly replace "percent ferrite" on a 1:1 basis.[3]

Note: Future research would reveal that the 1:1 correlation of FN to "volume percent ferrite" is only acceptable for low ferrite contents (0-10 FN), such as that present in the majority of austenitic stainless steel weld metals. The application of ferrite measurement techniques to duplex stainless steels would require further testing to define appropriate correlations.

2) The lack of standardized testing methods produced significant variability in the data acquired from IIW round-robin testing. Furthermore, measurements between laboratories suggested that further work was required to institute a universal system of ferrite measurement.[4]

Data from IIW round-robin testing enabled the WRC to establish a standard practice for quantifying ferrite content using available techniques. The publication of AWS A4.2, "Standard Procedures for Calibrating Magnetic Instruments to Measure the Delta Ferrite Content of Austenitic Stainless Steel Weld Metal", was the among the first steps to provide a universal calibration procedure for magnetic instrumentation.

Developments in ferrite measurement techniques continued for the next 20 years before another review of applicable techniques was performed. In that time, the FN

system was explored through a series of round robin test series and AWS A4.2 undertook a series of revisions to incorporate newly developed techniques.

Dr. D. J. Kotecki revisited the topic of ferrite measurement in his 1997 article entitled, "Ferrite Determination in Stainless Steel Welds – Advances since 1974" (Reference 23). Describing the revisions to AWS A4.2 and recounting research efforts encompassing the previous 20 years, the following items were highlighted:

- Extension of the Ferrite Number (FN) System:

  With the advent of new stainless steel alloys (duplex), the need to characterize materials, whose ferrite content exceeded 28 FN, was established.[5] The FN system was studied with various modifications, including extrapolation, calibration with new coating thickness standards and the development of cast secondary standards.*

  **Note: Coating thickness standards (primary standards) and cast secondary standards will be examined in following sections of this review.

- Ferrite Number vs. Ferrite Percent:

  The relationship between ferrite number and ferrite percent was explored utilizing weld metal samples. However, it was determined that the morphology and distribution of weld metal ferrite promoted unwanted effects during metallographic characterization. Such effects included a lack of agreement between laboratories, utilizing metallographic techniques, due to the fineness and irregular morphology of weld metal ferrite.

  However, such adverse morphologies were not present in cast materials. In general, the ferrite size was significantly coarser and more regularly shaped than weld

metal ferrite. A comparison of point counting and magnetic measurements revealed that the ratio of ferrite number to ferrite percent was not uniform over the entire FN scale. It was established that the correlation was roughly 1:1 for FN values of 0-28. However, above 28 FN the correlation deviated. Examinations, during experimental trials, suggested that this correlation could be approximated using a ferrite number to ferrite percent ratio of 1.4:1. However, a lack of agreement between laboratories left this issue in dispute among researchers.[6]

- Future Work:

  Dr. Kotecki suggested that the issue of "ferrite number vs. ferrite percent" needed further study. However, his suggestions indicated that the lack of agreement between laboratories, to establish a firm correlation, did not preclude the successful use of the FN system.

It was apparent that the only universal baseline to evaluate castings and weldments was a direct determination of the amount of ferrite present. This further necessitated the need for a correlation between ferrite number and ferrite volume percent. It was also suggested that current ferrite measurement techniques were not applicable for the characterization of heat-affected zones in comparison to the unaffected base metal or weld metal. Due to the relatively narrow width of the heat-affected zone, no available technique had been able to adequately characterize this region. Although specifications required a destructive metallographic examination to determine the ferrite content of the heat-affected zone, this specification was not accepted due to a lack of reproducibility within the same weldment. It was concluded that a new breed of technology of ferrite

measurement techniques needed to be developed to combat this situation. Finally, the constitution diagrams commonly used to predict the ferrite content based upon alloy chemistry required further development to allow for additional alloying elements and variations in cooling rate due to different joining processes.[7]

Having clearly defined the past, present and future research efforts regarding ferrite measurement techniques, it was evident that this area had undergone a significant amount of change and investigation since its conception in the 1940's. The current review concentrates on defining each appropriate ferrite measurement technique, paying careful attention to evaluate its efficacy.

Review of Measurement Techniques

A variety of techniques have been developed to determine the amount of ferrite present in a substrate. Ferrite measurement has been performed using the following techniques:

- Metallographic Point Counting
- Constitution Diagrams
- Magnetic Attraction
- X-Ray Diffraction
- Mössbauer Effect
- Magnetic Permeability
- Magnetic Saturation

Among the above techniques, x-ray diffraction and the Mössbauer effect have been applied to only laboratory experimentation. The principles governing x-ray diffraction and interpretation of diffraction spectra have long been characterized, however, the Mössbauer effect suggested interesting new principles.

L. J. Schwartzendruber discussed the Mössbauer effect in 1974 in a Welding Journal Research Supplement article entitled "Mössbauer – Effect Examination of Ferrite in Stainless Steel Welds and Castings".[8] When applied to alloy systems, it was found that different phases within a metal yield differing Mössbauer spectra. It was also found that the relative areas contained within the spectra were directly proportional to the amount of each phase present.[9] In comparing this techniques with others, Schwartzendruber commented that the Mössbauer technique was a valid method to conduct ferrite measurement. However, its application was limited to laboratory testing and cannot be readily utilized in the field.

Measurement by magnetic saturation involved saturating a given interaction volume with a magnetic field and measuring the associated magnetic response. K. Bungart (et al.) discovered that such measurements were highly influenced by alloy chemistry and the chemical composition of the ferrite. It was found that the saturation magnetism of the ferrite was governed by its chemistry.[10] Therefore, accurate ferrite measurements could only be obtained if the saturation magnetism of ferrite was established as a function of chemical composition. This technique has not been developed for commercial use. These principles were also examined as a part of Schwartzendruber's examination of the Mössbauer effect.

Having defined the less common ferrite measurement techniques, emphasis is now placed upon the use of metallographic point counting, constitution diagrams and magnetic instrumentation as viable methods of ferrite measurement.

Metallographic Point Counting

ASTM E562 is the "Standard Practice for Determining Volume Fraction by Systematic Manual Point Count." This specification may be applied to any microconstituent or phase which is metallographically identifiable. The principles governing this method are clearly defined in the specification. A two-dimensional metallographic sample is prepared and examined at an appropriate magnification. A grid is then superimposed over the image and the operator counts the number of points which fall within the desired phase or microconstituent. Statistical analysis reveals the fraction of points which fall within the desired phase and the volume fraction is then calculated.[11] When correctly implemented, this technique is an excellent method for determining the volume fraction of a desired phase or microconstituent. However, accuracy is often influenced by many factors, including the following:

- Homogeneity
- Quality of Sample Preparation
- Grid Density
- Magnification of the Substrate
- Operator Interpretation of the Microstructure

Attempts to mechanize this technique, using computer software, often decreased analysis time but still required the use of a trained technician. Although accurate, this technique requires a significant amount of preparation and analysis time. Preparation includes metallographic polishing to a 0.05 micron finish and the application of a suitable etching technique. Etching techniques are tailored to a specific microconstituent. Additionally, this technique is destructive in nature, requiring that a sample be extracted from the component or substrate. It was also limited to the number of fields examined and the location of the removed sample.

Because metallographic point counting is a destructive test and requires extensive preparation and analysis time, significant effort is placed upon the development of techniques which were non-destructive and labor efficient. Scientists and engineers next placed their focus on the effect of alloy chemistry on the amount of ferrite present.

Constitution Diagrams

Schaeffler, DeLong and WRC constitution diagrams introduced a non-destructive method to relate alloy composition to the amount of ferrite present in an alloy. The development of such a technique eliminated the need to destructively analyze a component, given that an accurate chemical analysis could be performed.

"Schaeffler Diagram"

The introduction of the Schaeffler diagram (1949) provided the first method to calculate ferrite percent in a non-destructive manner. Schaeffler mathematically correlated chromium and nickel equivalents, which were readily calculated based upon the alloy chemistry, to the amount of ferrite present. Based upon the amount of nickel, carbon, manganese, chromium, molybdenum, silicon and niobium (columbium) present, a brief reference to this diagram quickly estimated the amount of ferrite present (Figure 1).[13]

C. J. Long and W. T. DeLong cited the inherent problem of nitrogen additions during welding in their 1973 article entitled "The Ferrite Content of Austenitic Stainless Steel Weld Metal" (Reference 35). Although DeLong had published his own constitution diagram, accounting for nitrogen levels in weld metal, he identified an inherent problem associated with Schaeffler's diagram. Ferrite content varied with the amount of nitrogen present. As Schaeffler had not addressed this issue, nitrogen levels became a source of experimental error to be addressed in the next generation of constitution diagrams.[14]

"DeLong Diagram"

W. T. DeLong (et. al.)[15] revised the Schaeffler diagram in 1956 by adding the effect of nitrogen to the nickel equivalent. Citing a weighting factor of 30 for the effect of nitrogen, DeLong proposed a significant relationship between nitrogen concentration and ferrite formation (Figure 2).

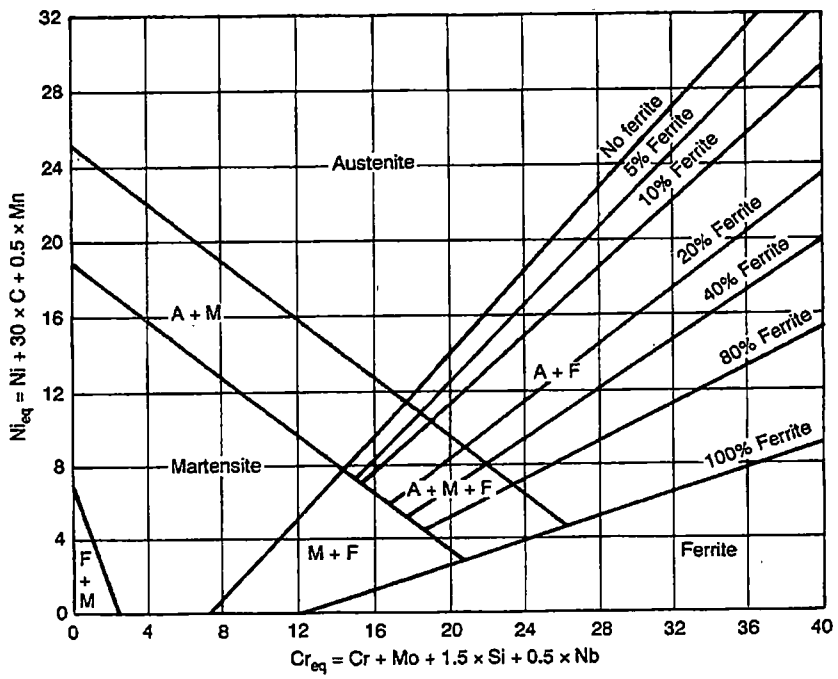

Figure 1. Schaeffler Diagram

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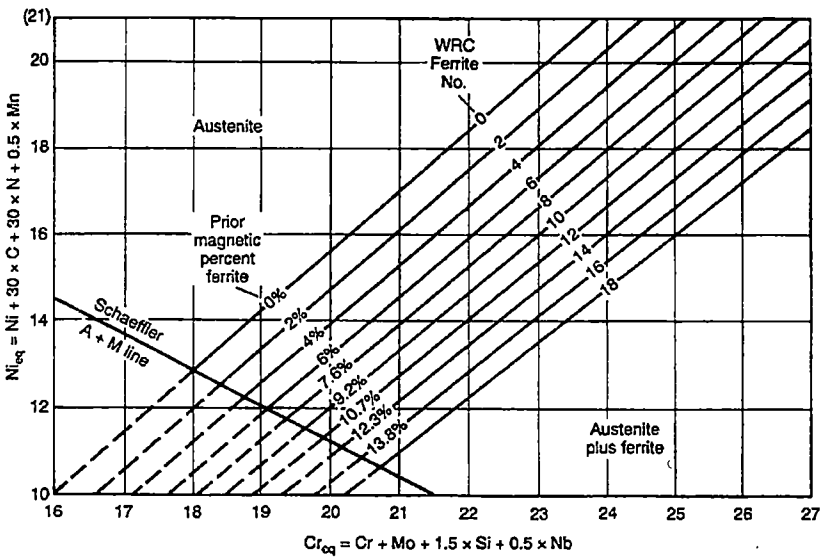

Figure 2. DeLong Diagram

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The major advantage of the DeLong diagram was its introduction of nitrogen as a significant factor in ferrite formation. Nitrogen, an austenitizer, retards the formation of ferrite. DeLong postulated that variations in welding technique and atmospheric conditions could affect the nitrogen content in weld metal, thus affecting the amount of ferrite formed during solidification of the weld pool. His work increased the accuracy of the Schaeffler diagram and revealed that his estimations predicted increased ferrite over that of Schaeffler, for a given chemistry.[16]

"WRC 1988 Diagram"

In 1988, T. A. Siewert, C. N. McCowan and D. L. Olson published the WRC 1988 constitution diagram (Reference 49). This diagram accounted for the following flaws in the Schaeffler and DeLong diagrams:

- The DeLong diagram is essentially a finely tuned subset of the Schaeffer range, designed specifically for the 300-series stainless steel welds containing small amounts of ferrite.[17] The refined nature of the DeLong diagram forced engineers to reference the Schaeffler diagrams for alloys containing more than 15% ferrite. As previously defined, the Schaeffler diagram did not have the improved degree of accuracy or accountability for nitrogen that the DeLong diagram developed.
- The effect of manganese on ferrite formation had been incorrectly established. An improved database revealed that the original 0.5 weighting factor should have been changed to unity (1), based upon work performed by E. R. Szumachowski and D. J. Kotecki.[18]

- A study by R. H. Espy revealed that the effect of nitrogen on ferrite formation resulted in a decreased value of the nitrogen coefficient in the nickel equivalent. Espy suggested that the nitrogen coefficient be lowered from 30 to 20.[19]
- The effect of silicon on weld metal ferrite had been examined by D. J. Kotecki. The results of his study revealed that the 1.5 silicon weighting factor used in both the Schaeffler and DeLong diagrams was inaccurate. Kotecki's work suggested that the weighting factor be reduced to 0.1 [20,21] Kotecki conducted a similar study to investigate the effect of molybdenum and concluded that its coefficient be reduced from 1.0 to 0.7.[22]

Siewert, McCowan and Olson concluded that, based upon the studies of elemental effects on ferrite formation, there was significant need to develop a new constitution diagram for the prediction of weld metal ferrite content. The WRC 1988 diagram (Figure 3) was then developed according to the following goals:[23]

- Development of a database containing recent FN data and new compositions.
- Evaluation of the accuracy of the Schaeffler and DeLong diagrams.
- Determination of which elements were not properly incorporated in these diagrams.
- Development of an improved predictive diagram that was continuous over the range of 0-100 FN.

The development of the WRC 1988 diagram improved the applicable ferrite range, reestablished the appropriate manganese, molybdenum, nitrogen and silicon contents, improved accuracy over the DeLong and Schaeffler diagrams and included solidification boundaries that correspond to changes in FN response.[24]

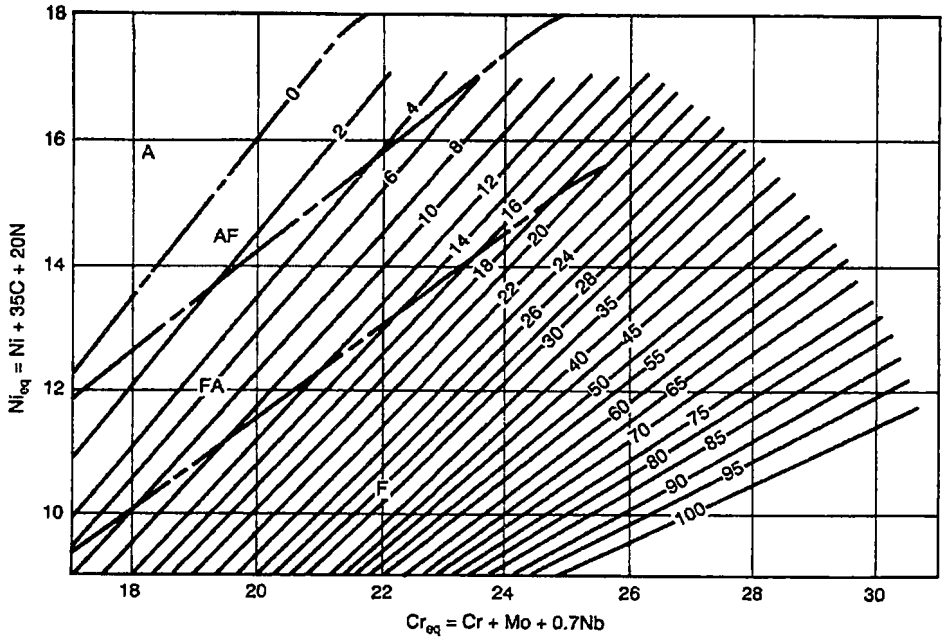

Figure 3. WRC 1988 Diagram

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"WRC 1992 Diagram"

Shortly after the submission of the WRC 1988 diagram, D. J. Kotecki and T. A. Siewert sought to include the effect of copper on the formation of ferrite in duplex stainless steels. While developing the WRC 1988 diagram, a copper coefficient was considered. However, research had not provided sufficient agreement on a universal value. Therefore, as the demand for duplex stainless steels increased, a need was recognized to modify the existing WRC diagram to include the effects of copper on the chromium equivalent.[25]

The resulting WRC 1992 (Figure 4) constitution diagram presented increased accuracy and the ability to extend the chromium and nickel equivalencies to allow dilution calculations incorporating dissimilar base materials and electrode compositions.

As a result of the advent of this diagram, engineers were able to rely on increased accuracy in ferrite prediction for copper-bearing alloys. Alloys with residual copper contents were not adversely affected. Additionally, this diagram allowed for the accountability of dissimilar weld joint configurations, which was a luxury not afforded by previous constitution diagrams.[26]

Magnetic Instrumentation

The use of x-ray diffraction, Mössbauer techniques and magnetic saturation as methods of ferrite measurement were previously described. Experimental

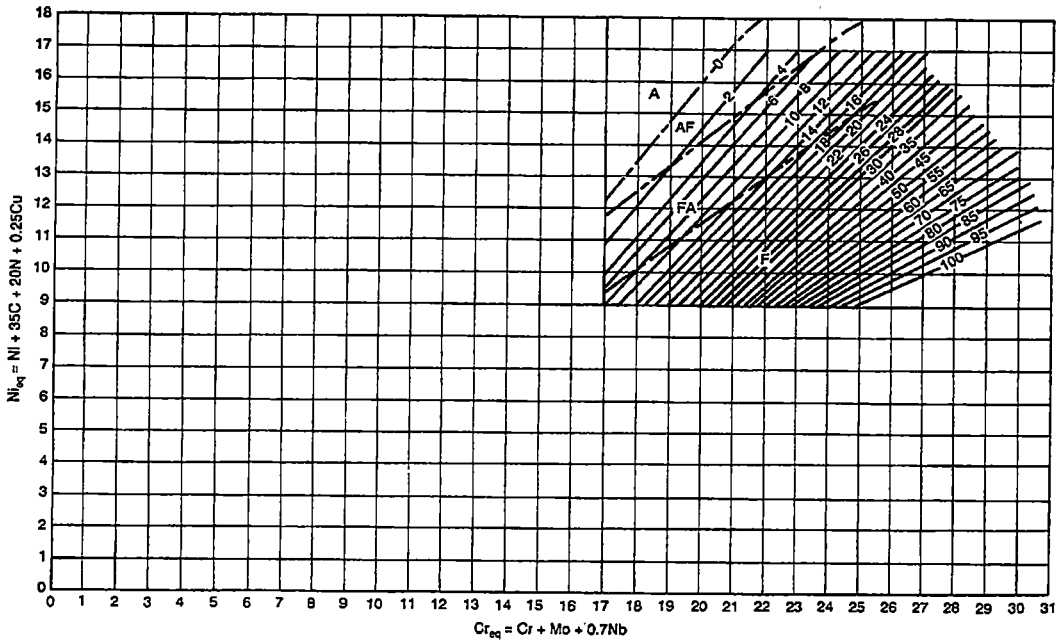

Figure 4. WRC 1992 Diagram

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trials revealed that these practices would not be readily applied to field engineering situations due to the use of laboratory confined equipment or variations in material response to each technique. However, as previously indicated, developments in magnetic instrumentation proved useful in creating reliable, reproducible and user-friendly ferrite measurement equipment. In the following sections, magnetic indicators, attractive force indicators and magnetic permeability instruments are introduced as viable methods for quantifying ferrite content.

The measurement of ferrite content yielding reproducible results was addressed separately by E. Stalmasek, E.W. Pickering, E.S. Robitz and D. M. Vandergriff. Stalmasek investigated the "Measurement of Ferrite Content in Austenitic Stainless Steel Weld Metal Giving Internationally Reproducible Results" (Reference 52) while Pickering, Robitz and Vandergriff concentrated on "Factors Influencing the Measurement of Ferrite Content in Austenitic Stainless Steel Weld Metal Using Magnetic Instruments" (Reference 39). Both articles are contained within Welding Research Council Bulletin 318 (Reference 53). WRC Bulletin 318 is addressed in the following paragraphs as individual magnetic measurement devices are described.

The following items were identified as significant to the development of new ferrite measurement devices by the above authors. [27]

- Ferrite chemistry, distribution, particle shape/size, and degree of transformation were identified as factors which make precise and accurate ferrite measurement difficult.
- The utilization of different measuring techniques does not necessarily yield identical results.

- Sample size and shape must be considered such that its geometry does not affect ferrite measurement due to unwanted edge effects.
- Reproducibility between instruments required the institution of a standard calibration procedure to incorporate all techniques.
- The relationship between ferrite number and ferrite percent is non-linear.

For additional information describing the ferromagnetic properties of ferrite in a duplex microstructure, refer to reference 52.

"Magnetic Indicators (e.g., Severn Gage)"

Having identified ferrite as a ferromagnetic phase, the first efforts to construct a device to assess ferrite content included magnetic indicators. Utilizing a permanent bar magnet, suspended from a lever arm, the substrate ferrite content was compared to a reference magnet. The reference magnet was either a permanent magnet or electromagnet.

R. B. Gunia and G. A. Ratz reviewed the performance of such devices in WRC Research Bulletin 132 (August 1968). Gunia and Ratz differentiated between instruments utilizing a permanent reference magnet (Severn Gage, Tinsley Gage and Elcometer) and those using an electromagnetic reference magnet (Ferrite Tester, Magne-Probe, Magnetoscope and Permascope).[28]

The advantage associated with such devices included ease of use and portability. With the inclusion of reference magnets of varying strength and associated ferrite

content, the user was able to quickly determine a range over which the ferrite content of the subject was contained. This technique eliminated the need for laborious metallography and time consuming analysis. However, the degree to which the ferrite content range could be characterized was governed by the reference magnets. Thus, this technique was only a "quick and dirty" estimation of the substrate ferrite content. No calibration of the instrument was required beyond establishing the ferrite content of the reference magnets.

"Attractive Force (e.g., Magne Gage)"

Building on the characteristics of the magnetic indicators, a device was sought which could directly correlate the force required to separate a magnet from a substrate (tear-off force) to the ferrite content of the substrate. The governing principle was that increasing ferrite content would promote a larger ferromagnetic response, which would result in increasing force required to separate a reference magnet from a substrate. However, no such device existed for that specific purpose.

While Schaeffler was developing his constitution diagram, a device had been constructed to measure the thickness of nonmagnetic coatings on magnetic materials. The principles governing the Magne Gage were easily defined. A permanent magnet, suspended from a lever arm, would be lowered until the magnet was in contact with the substrate. Using a calibrated dial, increasing torque was applied, through a helical spring, until the reference magnet separated from the substrate. The dial reading was recorded and compared to a calibration curve, which revealed the coating thickness or ferrite

content.[29] When properly calibrated, the Magne Gage proved to be a useful tool in assessing ferrite content.[30]

The advantage of the Magne Gage was its capability of directly measuring the ferrite content based upon magnetic response. The operator was no longer limited to a range of possible ferrite contents, as described with the use of the Severn gage. Rather, calibration to coating thickness standards (primary standards) allowed the operator to directly assess the ferrite content as a function of ferrite number. Conversely, the Magne Gage was primarily a laboratory instrument and was sensitive to outside vibrations. The Magne Gage and the primary coating thickness standards are shown in subsequent figures.

Use of the Magne Gage was revised in 1982 by D. J. Kotecki when he proposed the extension of the WRC ferrite number system. Increasing use of duplex stainless steel alloys required that the existing ferrite number system be expanded to include ferrite contents above 28 FN. This new system covered the full range of duplex alloys up to fully ferritic material. The new extended ferrite number (EFN) system proved to be statistically viable, as compared to the original ferrite number system.[31]

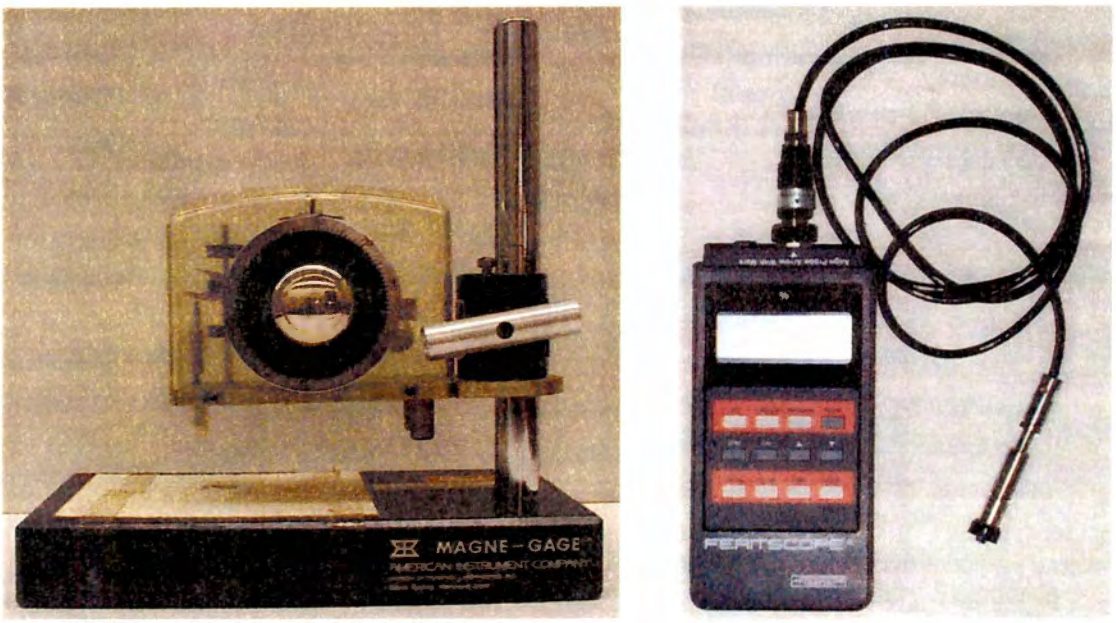
The use of the Magne Gage increased in later years as scientists and engineers sought to determine the relationship between ferrite content and as-welded mechanical properties. Studies by D. J. Kotecki[32] and D. L. Olson[33] further validated the Magne Gage as a useful tool in characterizing the ferrite content of austenitic and duplex alloys.

Increased use of Magne Gages spurred the implementation of the IIW 5th Round Robin of FN Measurements to assess interlaboratory variations in ferrite measurement. The results showed suitable repeatability with proper calibration.[34]

"Magnetic Permeability (e.g.: Feritscope®)"

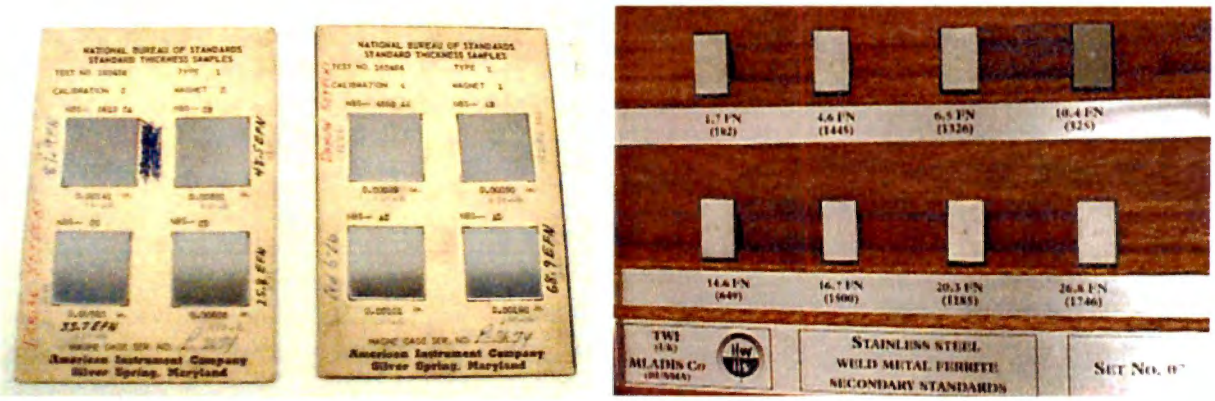
Magnetic permeability has been defined as the ratio of magnetic induction to magnetic field strength. Ferrite measurement, using this technique, required that a magnetic field be induced on a substrate and the resulting field strength be measured to establish the magnetic permeability.[35] This technique, provided by Gunia and Ratz, was later confirmed by E. Stalmasek in WRC Bulletin 318. Stalmasek further commented that "*the overall permeability of a two phase alloy containing one ferromagnetic and one nonferromagnetic phase, depends, at a given strength of the magnetizing field, upon the individual permeability, upon the content and upon demagnetization factor of the ferromagnetic phase*".[36] In short, this established that the strength of the induced field varied with the amount of ferromagnetic phase present.

The Fischer Feritscope® was developed as a hand-held device which utilized magnetic permeability as a method to assess ferrite content. As depicted in Figure 5, the Feritscope® was designed to be portable and provide the operator with a user-friendly interface which readily provides ferrite content on the ferrite number scale. Calibration of the Feritscope® has been performed using cast secondary standards. Cast secondary standards (Figure 6) were developed by NPO CNIIT-MASH (Russia) and produced by Mladis Co. (Russia) under organizational support of the Russian Welding Society. Each set of standards was produced from centrifugally chill cast rings and were distributed to TWI.[37] Cast secondary standards were used exclusively to calibrate Feritscopes®, but may also be used in the calibration of Magne Gages. The volume of



Magne Gage Feritscope®

Figure 5. Magne-Gage and Fischer Feritscope®.



Primary Standards Cast Secondary Standards

Figure 6. Primary and Cast Secondary Standards

ferrite in each standard was controlled through modifying the alloy content, such that a full range of ferrite numbers is attainable.

Additional round robin testing was initiated by D. J. Kotecki, in conjunction with IIW, to assess the reproducibility of Feritscopes® when calibrated using cast secondary standards. An interlaboratory variability of ±14% was established. This value was slightly higher than the variability established for Magne Gages (±10%) in previous round robin test series.[38]

The advantages associated with the advent of the Feritscope® included increased operator efficiency and portability of the device. However, there has not been a significant research effort to characterize the service performance of this gage when applied to a multitude of conditions. Such conditions include analyzing the measurement probe's response to varying surface finishes, surface discontinuities and gage repeatability. As the manufacturer does not currently provide such a database, a study to clarify these operating variables has been introduced as a part of this research effort.

Literature Review - Conclusions

The IIW has remained involved in the implementation of additional round robin testing to further characterize factors which affect ferrite measurement. Such factors include, but are not limited to, the following:

- Substrate Surface Finish
- Measurement Probe Interaction Volumes
- Correlation between Ferrite Number and Ferrite Volume Percent

- Reliability and Repeatability of Available Techniques

Although it has been established that significant accomplishments have been made in the field of ferrite measurement, it remains the belief of researchers and engineers that additional testing, to explore the limitations of current techniques, is required to further develop accurate and repeatable methods of ferrite measurement.

CHAPTER IV

PROCEDURES

The Magne Gage and Feritscope® were the exclusive instruments selected for non-destructive ferrite determination using the FN scale. ASTM E562 was utilized for manual point counting to determine the ferrite/austenite volume fraction. Operational procedures regarding use of the manual point counting, Magne Gage and Feritscope® are defined in the literature review. Metallographic preparation of the cast duplex stainless steels was conducted with standard procedures. Oxalic acid etching was employed to definitively reveal the ferrite/austenite phase morphology

Ferrite Measurement Round-Robin

A ferrite measurement round-robin study was initiated to examine the following issues:

- The repeatability and reproducibility in ferrite measurement, between laboratories, using the Magne Gage and Feritscope® techniques.
- The applicability of manufacturing cast secondary standards from static or centrifugal castings.
- A more defined correlation between different ferrite measurement techniques: manual point counting and measurement by Magne Gage and Feritscope®.

The round-robin process required that a comprehensive data packet be designed to instruct each participant to measure ferrite content using multiple techniques on a standard set of samples. Each participant was provided with detailed instructions, in the form of an operator checklist, to facilitate the data acquisition. Guidelines for proper calibration methods and measurement techniques were also provided to ensure repeatability between participants. A copy of the round-robin protocol is provided in Appendix A. Refer to this appendix for further information regarding the round-robin timetable, instruction set and measurement guidelines.

Each participant was asked to measure ferrite on a specific set of samples and record their determinations using their available ferrite measurement techniques. Twelve round-robin samples, of varying ferrite content, were manufactured. The sample set consisted of a series of austenitic and duplex stainless steels whose chemical composition and ferrite content are documented. Ferrite content measurements are explored in the following sections of this analysis. The chemical composition of each block is presented in Table 1. Using the data recording forms provided, the participants forwarded their results to UTK for analysis and then sent the sample set to the next participant. The total duration of the round-robin was five months. Eight participants from academia and industry volunteered their resources for this study.

Prior to examining the participant responses, repeatability and reproducibility must be defined. For this round-robin, repeatability and reproducibility are defined according to the guidelines of ASTM E1301, "Standard Guide for Proficiency Testing by Interlaboratory Comparisons":

Table 1. Chemical Composition (wt %) of the Round-Robin Test Samples

Sample Code	Alloy	% C	% Mn	% Si	% P	% S	% Cr	% Ni	% Mo	% N
A	CF8	0.058	0.60	1.52	0.012	0.013	18.5	9.98	0.02	0.020
B	CF3M	0.027	1.04	1.02	0.009	0.009	18.8	10.79	2.12	0.030
C	CF8M	0.083	1.20	1.20	0.030	0.013	19.8	9.53	2.21	0.020
D	ASTM A890-4A	0.026	0.38	0.81	0.030	0.010	22.1	6.00	2.91	0.226
E	ASTM A890-4A	0.020	0.95	0.56	0.016	0.007	22.3	5.50	3.00	0.200
F	ASTM A890-4A -CC	0.020	0.95	0.56	0.016	0.007	22.3	5.50	3.00	0.200
G	ASTM A890-5A	0.020	0.78	0.64	0.011	0.008	24.0	7.60	4.50	0.180
H	ASTM A890-5A -CC	0.020	0.78	0.64	0.011	0.008	24.0	7.60	4.50	0.180
I	ASTM A890-5A	0.026	0.51	0.34	0.024	0.002	24.7	7.44	4.53	0.191
J	CD7MCuN -CC	0.030	0.94	0.68	0.023	0.005	24.8	5.50	2.90	0.200
K	CD7MCuN	0.030	0.94	0.68	0.023	0.005	24.8	5.50	2.90	0.200
L	CD7MCuN	0.038	1.00	0.98	0.018	0.008	25.0	5.54	2.92	0.128

"CC" indicates centrifugally cast material. All other alloys are statically cast.

Repeatability: "the closeness of agreement between test results obtained with the same test method, in the same laboratory, by the same operator with the same equipment in the shortest practical period of time using test units or test specimens taken at random from a single quantity of material that is as nearly homogenous as possible"

Reproducibility: "the closeness of agreement between test results obtained with the same test method on identical material in different laboratories"

In order to sufficiently qualify the repeatability of a round-robin sample, a gage repeatability and reproducibility study should be employed. This technique would mandate that multiple round-robin samples, of the same ferrite content, be examined by a single operator, utilizing a specified measurement technique. By isolating the technique and operator, the only remaining source of experimental error is limited to the repeatability of the test blocks. As multiple test blocks of identical ferrite content were not produced for this study, repeatability strictly cannot be characterized. However, "2σ values less than 10% of the mean ferrite content" has been established as criteria to indicate probable repeatability.[40] The 2σ values have been reported for each participant for information.

The reproducibility between laboratories has been expressed in previous round-robins as 2σ/mean, where σ is the standard deviation for a set of measurements and the

mean is the arithmetic average of a set of measurements. The prevailing assumption indicating sufficient reproducibility between participants is $2\sigma \leq 14\%$ of the mean ferrite content of the round-robin sample.[41].

Having defined repeatability and reproducibility, attention is now focused on the individual characterization of the set of ferrite content samples by each of the participants. For each participants data, the mean and 2σ values were calculated. Repeatability of measurements can be assessed for each participant while reproducibility characterization is discussed for each ferrite measurement technique.

CHAPTER V

RESULTS AND DISCUSSION

Participant Responses

The University of Tennessee

Prior to initiating the round-robin, the University of Tennessee-Knoxville (UTK) was responsible for designing the round-robin protocol. Additionally, the sample set was manufactured and characterized by the UTK Materials Joining Research Group prior to the initiation of the study. Characterization included ferrite measurement by Magne Gage and Feritscope®. Additionally, metallographic point counting was employed to define volume percent ferrite and thus the relationship between ferrite volume percent and ferrite number.

UTK characterization of the sample set included measurements by Magne Gage and Feritscope®. Calibration of each instrument was performed using AWS A4.2, per the round-robin protocol instructions. Tables 2 and 3 summarize the results of measurements by Magne Gage and Feritscope®. Each table illustrates the number of

Table 2. University of Tennessee Magne Gage Results

Sample Code	Determination Set 1 (Highest FN)	Determination Set 2 (Highest FN)	Determination Set 3 (Highest FN)	Determination Set 4 (Highest FN)	Determination Set 5 (Highest FN)	Mean FN	Standard Deviation (2σ)	Repeatability 2σ≤10%Mean (Yes or No)
A	3.1	3.4	3.1	3.4	2.8	3.2	0.5	No
B	9.8	11.2	9.5	10.3	14.0	11.0	3.6	No
C	11.2	12.6	11.7	11.2	15.1	12.4	3.3	No
D	63.2	68.6	62.6	63.5	62.6	68.6	5.1	Yes
E	62.5	62.2	65.5	62.2	62.2	65.5	2.9	Yes
F	63.2	62.2	62.6	66.6	64.6	64.8	3.6	Yes
G	70.6	71.2	75.5	69.2	74.9	72.3	5.5	Yes
H	62.9	61.8	61.8	62.3	63.2	62.4	1.3	Yes
I	77.5	70.6	75.5	69.2	74.9	73.5	7.0	Yes
J	76.9	76.6	76.0	75.8	75.5	76.2	1.2	Yes
K	80.6	82.6	84.0	83.5	78.6	81.9	4.5	Yes
L	95.5	96.9	93.2	95.2	95.5	95.3	2.7	Yes

Table 3. University of Tennessee Feritscope® Results

Sample Code	FN 1	FN 2	FN 3	FN 4	FN 5	FN 6	FN 7	FN 8	FN 9	FN 10	Mean FN	Standard Deviation (2σ)	Repeatability 2σ≤10%Mean (Yes or No)
A	3.0	2.7	3.1	3.0	3.0	3.1	3.0	2.9	3.0	3.1	3.0	0.2	Yes
B	6.0	9.3	8.8	11.4	6.3	11.7	7.6	5.6	10.2	9.4	8.6	4.4	No
C	11.5	11.1	12.4	12.7	11.7	13.0	11.2	12.7	12.3	13.2	12.2	1.5	No
D	45.4	58.3	51.1	57.1	51.4	55.7	50.5	53.4	57.4	57.0	53.7	8.2	No
E	63.4	45.7	49.1	49.2	45.8	45.6	45.2	45.5	45.7	61.2	49.6	13.7	No
F	56.5	59.5	56.4	64.4	59.8	56.5	62.5	58.3	65.2	56.6	59.6	6.8	No
G	65.2	67.5	68.7	63.9	67.0	66.8	67.5	73.3	65.8	71.7	67.7	5.7	Yes
H	64.8	65.3	62.6	64.1	60.1	61.3	62.6	62.1	63.3	61.8	62.8	3.2	Yes
I	71.2	71.0	71.9	72.0	71.2	65.3	74.2	71.1	74.9	69.8	71.3	5.2	Yes
J	74.0	73.1	71.7	72.0	75.0	74.2	75.2	74.5	67.5	74.6	73.2	4.7	Yes
K	74.0	72.5	77.4	74.5	75.1	76.2	77.1	75.8	78.0	74.4	75.5	3.4	Yes
L	93.3	98.0	90.0	91.8	94.6	94.2	97.9	92.4	92.4	89.4	93.4	5.8	Yes

ferrite determinations, followed by the mean ferrite number for each sample. The standard deviation (2σ) was also calculated and incorporated into the data.

Analysis of the data set reveals that the samples ranged in ferrite content from approximately 3 FN to 95 FN with minimal disparity (<2% of the mean FN) between the two techniques in measuring ferrite number. However, ferrite measurement using Magne Gage and Feritscope® techniques identified samples A, B, C, D, E and F with the 2σ values greater than 10% of the mean ferrite content. This statistic indicates insufficient repeatability for this group of samples utilizing either the Magne Gage or Feritscope® techniques. This indicates that this group of samples cannot be used as cast secondary standards. Samples G, H, I, J, K and L exhibited 2σ values less than 10% of the mean ferrite content, indicating acceptable repeatability for use as cast secondary standards.

The Lincoln Electric Company

The Lincoln Electric Company was the second participant in this round-robin. Lincoln Electric characterized the sample set using both the Magne Gage (Serial Number: P-6459) and Feritscope® (Model MP-3). Each gage was calibrated using AWS A4.2, as prescribed in the round-robin protocol. Tables 4 and 5 summarize the Lincoln Electric results of measurement by Magne Gage and Feritscope®.

Analysis of this data set reveals that the samples ranged in ferrite content from approximately 3 FN to 95 FN with minimal disparity between the two techniques in measuring ferrite number. Ferrite measurement using either the Magne Gage or

Table 4. The Lincoln Electric Company Magne Gage Results

Sample Code	Determination Set 1 (Highest FN)	Determination Set 2 (Highest FN)	Determination Set 3 (Highest FN)	Determination Set 4 (Highest FN)	Determination Set 5 (Highest FN)	Mean FN	Standard Deviation (2 Sigma)	Repeatability $2\sigma \leq 10\%$Mean (Yes or No)
A	3.4	3.3	3.2	3.4	3.4	3.3	0.2	Yes
B	11.8	12.6	12.4	12.5	11.3	12.1	1.1	Yes
C	14.1	14.6	15.3	15.0	14.6	14.7	0.9	Yes
D	59.3	63.5	61.4	61.9	61.2	61.5	3.0	Yes
E	62.6	67.8	68.3	66.6	60.2	65.1	7.1	No
F	59.7	59.3	64.7	65.7	65.0	62.9	6.2	Yes
G	71.1	74.7	73.5	70.9	71.1	72.3	3.5	Yes
H	61.4	62.8	62.1	62.1	62.1	62.1	1.0	Yes
I	74.0	70.9	74.5	77.8	73.1	74.1	5.0	Yes
J	76.9	73.5	73.3	77.1	76.4	75.4	3.8	Yes
K	79.7	84.5	80.2	75.2	82.8	80.5	7.1	Yes
L	92.7	91.0	92.7	91.7	94.3	92.5	2.5	Yes

Table 5. The Lincoln Electric Company Feritscope® Results

Sample Code	FN 1	FN 2	FN 3	FN 4	FN 5	FN 6	FN 7	FN 8	FN 9	FN 10	Mean FN	Standard Deviation (2 Sigma)	Repeatability 2σ≤10%Mean (Yes or No)
A	2.8	2.8	2.9	2.8	2.8	2.8	2.8	2.8	2.8	2.8	2.8	0.1	Yes
B	10.0	8.2	7.8	9.3	8.9	8.6	10.0	11.0	9.3	8.9	9.2	1.9	No
C	12.0	11.0	12.0	11.0	12.0	12.0	13.0	12.0	14.0	12.0	12.1	1.8	No
D	60.0	59.0	59.0	59.0	48.0	53.0	60.0	57.0	57.0	48.0	56.0	9.4	No
E	49.0	51.0	63.0	47.0	59.0	46.0	46.0	52.0	47.0	59.0	51.9	12.5	No
F	50.0	60.0	60.0	59.0	65.0	48.0	48.0	61.0	64.0	49.0	56.4	13.7	No
G	66.0	66.0	64.0	63.0	68.0	65.0	66.0	72.0	66.0	70.0	66.6	5.4	Yes
H	61.0	65.0	64.0	64.0	63.0	63.0	65.0	63.0	64.0	63.0	63.5	2.4	Yes
I	71.0	69.0	72.0	76.0	72.0	73.0	75.0	74.0	65.0	74.0	72.1	6.4	Yes
J	74.0	74.0	74.0	74.0	73.0	73.0	73.0	75.0	75.0	73.0	73.8	1.6	Yes
K	75.0	74.0	76.0	76.0	76.0	76.0	77.0	76.0	77.0	75.0	75.8	1.8	Yes
L	87.0	86.0	86.0	87.0	85.0	85.0	84.0	82.0	86.0	87.0	85.5	3.2	Yes

Feritscope® revealed that samples B, C, D, E and F exhibited the 2σ values greater than 10% of the mean ferrite content, indicating insufficient repeatability for use as a cast secondary standard. Samples A, G, H, I, J, K and L exhibited acceptable repeatability for use as cast secondary standards.

The ESAB Company

The ESAB Company was the third participant in this round-robin. ESAB characterized the sample set using the Magne Gage (Serial Number: 18032-106). ESAB does not currently utilize the Feritscope®. Therefore, this data was unavailable. The Magne Gage was calibrated using AWS A4.2, as prescribed in the round-robin protocol. Table 6 summarizes the results of investigation by Magne Gage.

Preliminary analysis of this data set reveals that the samples ranged in ferrite content from approximately 3 FN to 87 FN. Ferrite characterization of the sample set at ESAB was consistent with the scope of the round-robin. Magne Gage ferrite measurement identified samples A and E with a 2σ value greater than 10% of the mean ferrite content, indicating insufficient repeatability for use as a cast secondary standard. Samples B, C, D, F, G, H, I, J, K and L exhibited 2σ value less than 10% of the mean ferrite content, indicating acceptable repeatability for use as a cast secondary standards.

Table 6. The ESAB Company Magne Gage Results

Sample Code	Determination Set 1 (Highest FN)	Determination Set 2 (Highest FN)	Determination Set 3 (Highest FN)	Determination Set 4 (Highest FN)	Determination Set 5 (Highest FN)	Mean FN	Standard Deviation (2 Sigma)	Repeatability $2\sigma \leq 10\%$Mean (Yes or No)
A	3.2	3.2	3.6	3.2	3.2	3.3	0.4	No
B	11.7	11.7	13.0	12.5	12.1	12.2	1.1	Yes
C	15.0	15.0	15.0	14.1	15.0	14.8	0.8	Yes
D	58.1	58.8	55.4	57.0	58.1	57.5	2.7	Yes
E	47.2	52.2	55.2	63.6	63.6	56.4	14.4	No
F	60.2	60.9	57.0	57.7	60.4	59.2	3.5	Yes
G	63.7	63.5	65.8	64.1	61.1	63.6	3.4	Yes
H	56.6	55.3	55.5	54.0	56.8	55.6	2.3	Yes
I	66.0	69.6	72.5	72.7	69.2	70.0	5.5	Yes
J	70.5	72.5	70.5	71.7	68.6	70.8	3.0	Yes
K	75.9	76.5	74.2	78.4	76.3	76.3	3.0	Yes
L	89.4	88.6	85.1	84.6	87.6	87.1	4.2	Yes

The Hobart Brothers Company

The Hobart Brothers Company was the fourth participant in this round-robin. The Hobart Brothers Company characterized the sample set using both the Magne Gage (Serial Number: P-6712) and Feritscope® (Model MP-30). Each gage was calibrated using AWS A4.2, as prescribed in the round-robin protocol. Tables 7 and 8 summarize the results of inspection by Magne Gage and Feritscope®. Sample L was not able to be characterized using a Magne Gage, as its ferrite content was beyond the limits of calibration. All other samples were fully characterized.

Analysis of this data set reveals that the samples ranged in ferrite content from approximately 3 FN to 95 FN with minimal disparity (<10% of the mean) between techniques in ferrite number. Ferrite measurement revealed that samples A, B, C and E had 2σ values greater than 10% of the mean ferrite content. This indicates insufficient repeatability for samples A, B, C, and E, when characterized using either a Magne Gage or Feritscope®, for use as a cast secondary standard. The remaining samples exhibited suitable repeatability for use as cast secondary standards.

NIST

The National Institute of Standardization and Testing (NIST) was the fifth participant for this round-robin. NIST characterized the sample set using the Magne Gage (Serial Number: 3814). Currently, NIST does not utilize the Feritscope®; therefore, this data was unavailable. The Magne Gage was calibrated using AWS A4.2,

Table 7. The Hobart Brothers Company Magne Gage Results

Sample Code	Determination Set 1 (Highest FN)	Determination Set 2 (Highest FN)	Determination Set 3 (Highest FN)	Determination Set 4 (Highest FN)	Determination Set 5 (Highest FN)	Mean FN	Standard Deviation (2 Sigma)	Repeatability 2σ≤10%Mean (Yes or No)
A	3.3	3.5	3.3	3.1	3.1	3.3	0.3	Yes
B	11.3	12.6	11.9	10.6	11.5	11.6	1.5	No
C	14.4	15.0	14.4	14.4	14.1	14.5	0.7	Yes
D	54.4	54.3	57.7	52.4	56.5	55.1	4.1	Yes
E	54.0	53.5	53.0	61.0	57.0	55.7	6.7	No
F	61.3	61.5	61.3	61.5	61.3	61.4	0.2	Yes
G	65.0	63.7	63.5	63.5	63.5	63.8	1.3	Yes
H	59.0	59.0	57.3	56.5	58.6	58.1	2.3	Yes
I	67.0	66.5	66.7	67.0	67.5	66.9	0.8	Yes
J	68.8	68.6	71.5	68.6	71.3	69.8	3.0	Yes
K	71.3	75.5	74.3	75.0	76.8	74.6	4.1	Yes
L	NT	NT	NT	NT	NT	NT	NT	NT

NT = Not Tested

Table 8. The Hobart Brothers Company Feritscope® Results

Sample Code	FN 1	FN 2	FN 3	FN 4	FN 5	FN 6	FN 7	FN 8	FN 9	FN 10	Mean FN	Standard Deviation (2 Sigma)	Repeatability 2σ≤10%Mean (Yes or No)
A	3.0	2.7	2.7	2.9	3.1	3.0	3.3	3.0	2.9	2.7	2.9	0.4	No
B	9.9	9.0	8.7	11.6	11.5	10.9	9.9	11.5	10.8	11.2	10.5	2.1	No
C	12.6	13.9	14.2	9.2	11.6	12.2	12.0	12.1	12.5	12.8	12.3	2.7	No
D	56.5	53.0	54.1	54.3	57.7	56.7	57.0	55.6	53.1	53.4	55.1	3.5	Yes
E	56.8	55.2	56.8	51.5	58.8	52.8	51.4	52.9	51.9	55.3	54.3	5.2	Yes
F	54.1	56.2	55.6	59.4	58.8	59.2	59.2	55.3	58.4	55.2	57.1	4.1	Yes
G	68.9	72.5	70.5	66.1	68.7	71.5	71.3	66.8	64.3	72.6	69.3	5.7	Yes
H	60.0	55.8	62.3	65.0	58.5	62.4	64.8	61.9	63.8	62.2	61.7	5.7	Yes
I	73.2	76.5	73.6	81.5	82.1	71.0	75.1	75.2	75.4	74.1	75.8	7.0	Yes
J	74.9	72.3	78.2	78.7	74.8	73.2	71.2	78.7	74.8	77.1	75.4	5.4	Yes
K	74.2	75.7	80.6	84.8	77.7	76.9	80.9	76.3	83.9	79.7	79.1	7.0	Yes
L	97.0	92.7	95.4	95.8	92.5	97.2	99.2	96.7	95.8	97.1	95.9	4.1	Yes

as prescribed in the round-robin protocol. Table 9 summarizes the Magne Gage results. Analysis of this data set reveals that the samples ranged in ferrite content from approximately 3 FN to 90. NIST's characterization of the sample set was consistent with the scope of the round-robin. Magne Gage measurements revealed that samples B and E exhibited a 2σ value greater than 10% of the mean round-robin sample ferrite content. This indicates insufficient repeatability for use as a Magne Gage cast secondary standard. The remaining samples exhibited 2σ values less than 10% of the mean round-robin sample ferrite content, indicating that the remaining samples are suitable for use as cast secondary standards.

Foster Wheeler Inc.

Foster Wheeler Inc. was the sixth participant for this round-robin. Foster Wheeler characterized the sample set using the Feritscope® (Model MP-3 / 122-13088A). Foster Wheeler does not currently utilize the Magne Gage; therefore, this data was unavailable. The Feritscope® was calibrated using AWS A4.2, as prescribed in the round-robin protocol. Table 10 summarizes the results utilizing the Feritscope®.

Analysis of this data set reveals that the samples ranged in ferrite content from approximately 3 FN to 92. Ferrite measurement at Foster Wheeler was consistent with the scope of the round-robin. Ferrite measurement, using the Feritscope®, revealed that samples A, B, C, D and E exhibited 2σ values greater than 10% of the mean round-robin sample ferrite content. This indicates insufficient repeatability for the above samples when characterized with a Feritscope®.

Table 9. The N.I.S.T. Magne Gage Results

Sample Code	Determination Set 1 (Highest FN)	Determination Set 2 (Highest FN)	Determination Set 3 (Highest FN)	Determination Set 4 (Highest FN)	Determination Set 5 (Highest FN)	Mean FN	Standard Deviation (2 Sigma)	Repeatability $2\sigma \leq 10\%$Mean (Yes or No)
A	3.3	3.5	3.3	3.5	3.5	3.4	0.2	Yes
B	10.6	10.7	12.6	12.1	12.2	11.6	1.8	No
C	14.3	14.8	14.4	14.8	14.9	14.6	0.5	Yes
D	60.7	60.3	60.1	58.6	58.3	59.6	2.2	Yes
E	58.1	66.4	63.5	58.8	61.4	61.6	6.8	No
F	60.9	62.0	60.4	60.3	63.3	61.4	2.5	Yes
G	69.0	67.7	66.9	65.6	66.7	67.2	2.5	Yes
H	58.8	58.8	57.5	58.3	59.6	58.6	1.5	Yes
I	73.5	74.0	71.6	71.4	72.1	72.5	2.3	Yes
J	73.2	75.0	70.7	71.6	72.1	72.5	3.3	Yes
K	76.3	72.7	81.5	79.2	79.2	77.8	6.8	Yes
L	91.2	90.4	88.1	89.4	87.0	89.2	3.4	Yes

Table 10. Foster Wheeler Inc., Feritscope® Results

Sample Code	FN 1	FN 2	FN 3	FN 4	FN 5	FN 6	FN 7	FN 8	FN 9	FN 10	Mean FN	Standard Deviation (2 Sigma)	Repeatability 2σ≤10%Mean (Yes or No)
A	3.7	3.6	3.4	3.6	3.8	4.1	3.7	4.0	3.8	3.7	3.7	0.4	No
B	8.4	8.3	12.0	12.0	8.8	8.7	10.0	12.0	7.9	12.0	10.0	3.6	No
C	13.0	11.0	13.0	12.0	12.0	8.2	12.0	14.0	13.0	13.0	12.1	3.2	No
D	60.0	62.0	57.0	64.0	63.0	54.0	60.0	56.0	61.0	57.0	59.4	6.5	No
E	63.0	57.0	62.0	54.0	62.0	63.0	64.0	59.0	59.0	61.0	60.4	6.3	No
F	62.0	65.0	62.0	63.0	65.0	64.0	63.0	64.0	66.0	65.0	63.9	2.7	Yes
G	70.0	70.0	72.0	66.0	74.0	68.0	73.0	68.0	74.0	71.0	70.6	5.4	Yes
H	67.0	66.0	69.0	65.0	62.0	64.0	68.0	64.0	69.0	68.0	66.2	4.8	Yes
I	76.0	74.0	76.0	77.0	78.0	78.0	73.0	75.0	74.0	77.0	75.8	3.5	Yes
J	74.0	78.0	77.0	76.0	74.0	77.0	73.0	74.0	74.0	75.0	75.2	3.4	Yes
K	79.0	78.0	77.0	78.0	79.0	79.0	80.0	79.0	79.0	80.0	78.8	1.8	Yes
L	89.0	92.0	94.0	91.0	92.0	92.0	91.0	95.0	90.0	96.0	92.2	4.4	Yes

Stainless Foundry Inc.

Stainless Foundry Inc. was the seventh participant for this round-robin. Stainless Foundry characterized the sample set using the Feritscope® (Model MP-30 / 078-17838A). Stainless Foundry does not currently utilize the Magne Gage; therefore, this data was unavailable. The Feritscope® was not calibrated using AWS A4.2. Rather, this Feritscope® used the guidelines of AWS A4.2 as a reference but proceeded with a calibration according to the Feritscope® manufacturer's guidelines. This entailed the use of Fischer calibration standards, rather than the secondary standards, required by AWS A4.2. This data is invaluable as it provides insight into ferrite measurement interlaboratory variance among participants who use different calibration procedures. Table 11 summarizes the results of determinations by Feritscope®.

Analysis of this data set reveals that the samples ranged in ferrite content from approximately 3 FN to 104. Stainless Foundry's characterization was consistent with the scope of the round-robin. Ferrite measurement, utilizing the Feritscope®, revealed that samples B, E, F, I and J exhibited 2σ values greater than 10% of the mean round-robin samples ferrite content. This indicates insufficient repeatability for these samples utilizing the Feritscope® technique, calibrated under a manufacturer's procedure. These samples are not adequate for use as Feritscope® cast secondary standards. The remaining samples, A, C, D, G, H, K and L exhibited 2σ values less than 10% of the mean round-robin samples ferrite content, indicating suitable repeatability for use as cast secondary standards.

Table 11. Stainless Foundry Inc., Feritscope® Results

Sample Code	FN 1	FN 2	FN 3	FN 4	FN 5	FN 6	FN 7	FN 8	FN 9	FN 10	Mean FN	Standard Deviation (2 Sigma)	Repeatability 2σ≤10%Mean (Yes or No)
A	2.9	3.0	3.0	2.9	3.0	2.9	2.9	2.9	2.9	2.9	2.9	0.1	Yes
B	11.3	11.9	9.4	7.4	10.0	8.7	10.1	7.8	8.6	11.4	9.7	3.1	No
C	12.7	12.7	12.9	12.7	12.0	13.0	13.4	12.9	12.2	13.0	12.8	0.8	Yes
D	56.0	52.6	59.8	54.8	55.7	52.2	53.2	56.1	57.9	51.2	55.0	5.4	Yes
E	58.6	56.5	55.1	50.8	63.4	54.2	51.5	58.4	52.6	64.1	56.5	9.3	No
F	66.1	61.3	60.1	65.5	58.0	58.9	57.6	58.0	62.7	67.9	61.6	7.5	No
G	64.9	65.2	68.6	66.9	69.7	67.3	67.4	67.1	62.7	67.0	66.7	4.0	Yes
H	66.4	62.0	61.2	58.8	59.7	59.8	59.6	62.3	63.7	59.1	61.3	4.8	Yes
I	65.0	78.0	64.7	72.5	79.2	74.0	65.2	70.9	74.0	74.0	71.8	10.5	No
J	69.2	70.7	76.8	75.7	76.7	69.6	65.6	74.7	68.0	77.4	72.4	8.6	No
K	77.3	73.4	77.0	74.2	81.4	79.1	78.9	81.1	81.9	77.0	78.1	5.8	Yes
L	98.1	104.0	110.0	105.0	104.0	103.0	104.0	104.0	103.0	102.0	103.7	5.8	Yes

Fristam Pumps Inc.

Fristam Pumps Inc. was the eighth and final participant for this round-robin Fristam Pumps characterized the sample set using the Feritscope® (Model MP-30 / 058-17469A). Fristam Pumps does not currently utilize the Magne Gage; therefore, such data was unavailable. The Feritscope® was not calibrated using AWS A4.2. Rather, this Feritscope® used the guidelines of AWS A4.2 as a reference but proceeded with a calibration according to the Feritscope® manufacturer's guidelines. This entailed the use of Fischer calibration standards, rather than the secondary standards, required by AWS A4.2. This data is also invaluable, as it provides insight into ferrite measurement interlaboratory variance among participants who use different calibration procedures. Table 12 summarizes the results of inspection by Feritscope®.

Analysis of this data set reveals that the samples ranged in ferrite content from approximately 3 FN to 102. Ferrite characterization of the sample set, at Fristam Pumps, was consistent with the scope of the round-robin. Ferrite measurement revealed that samples B, C, D, E, F, G, H, I, K and L exhibited 2σ values greater than 10% of the mean round-robin ferrite content. This indicates insufficient repeatability for use as cast secondary standards, when calibrated under a manufacturer's procedure. Samples A and J exhibited 2σ values less than 10% of the mean round-robin ferrite content, indicating suitable repeatability for use as cast secondary standards.

Table 12. Fristam Pumps Inc., Feritscope® Results

Sample Code	FN 1	FN 2	FN 3	FN 4	FN 5	FN 6	FN 7	FN 8	FN 9	FN 10	Mean FN	Standard Deviation (2 Sigma)	Repeatability 2σ≤10%Mean (Yes or No)
A	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.1	2.9	2.9	3.0	0.1	Yes
B	8.7	8.4	7.9	9.7	10.0	11.0	7.1	10.3	9.4	9.9	9.2	2.4	No
C	12.8	13.5	12.2	14.0	13.2	13.3	14.1	12.1	12.5	12.2	13.0	1.5	No
D	59.0	62	52.9	52.9	61.3	51.8	63.2	60.4	50.2	50.8	56.5	10.3	No
E	55.9	50.2	61.4	59.3	55.9	54.1	48.7	49.7	57.6	58.6	55.1	8.7	No
F	61.3	60.7	50.9	51.5	50.8	57.8	53.0	58.2	58.8	62.4	56.5	9.1	No
G	62.1	73.1	73.1	64.3	68.1	66.0	70.8	72.5	72.3	72.9	69.5	8.2	No
H	63.4	61.9	57.3	57.0	69.9	64.6	63.4	63.5	64.7	61.6	62.7	7.4	No
I	77.1	74.5	76.1	68.4	76.9	69.0	72.8	78.4	78.6	68.6	74.0	8.2	No
J	74.3	73.2	71.3	74.1	68.8	74.9	78.1	71.8	74.1	74.5	73.5	5.0	Yes
K	78.4	73.3	81.9	84.5	86.7	82.1	82.1	70.3	78.0	79.8	79.7	9.9	No
L	98.5	93.9	111.0	106.0	101.0	102.0	99.1	112.0	103.0	97.5	102.4	11.6	No

Observations on Participant Data

The following observations are based upon the data returned by each of the above participants in the round-robin study:

- All participants identified sample E as unsuitable for use as a cast secondary standard, regardless of calibration method.
- In general, participants using a manufacturer's calibration identified more noncompliant samples than participants utilizing an AWS A4.2 calibration.
- For those participants who calibrated to AWS A4.2, 5 of 6 participants identified sample B as unsuitable for use as a cast secondary standard. Four of six participants identified samples A and C as unsuitable and three of six identified sample D as unsuitable for use as a cast secondary standard. Samples A, B, C, and D are statically cast austenitic and duplex alloys.

Two of six participants identified sample F (centrifugally cast duplex) as non-compliant. However, this behavior is not considered conclusive. Note that the two participants who identified this sample utilized the same Feritscope secondary calibration standards. Participants utilizing other AWS A4.2 sanctioned secondary standards did not identify sample F as unsuitable. All other centrifugally cast duplex samples (H and J) demonstrated suitable repeatability for use as a cast secondary standard. Thus, it can be concluded that, in general, centrifugally cast materials exhibit improved repeatability over the statically cast materials.

Ferrite Measurement by Point Counting

As previously stated, the round-robin test samples were metallographically characterized prior to the initiation of the measurements. The first aspect of characterization was a systematic point count of ferrite content utilizing the techniques outlined in ASTM specification E562. This specification is the "Practice for Determining Volume Fraction by Systematic Manual Point Count." Prior to analysis, each of the twelve round-robin samples was metallographically polished to a uniform 0.05μ surface finish. The samples were then electro-etched in oxalic acid (10V, 0.05A for 20-60 seconds) and viewed under an optical light microscope. Five locations, within a prescribed measurement region, were selected and photographed to obtain 200x micrographs. These micrographs were then utilized to perform the manual point count (grid method).

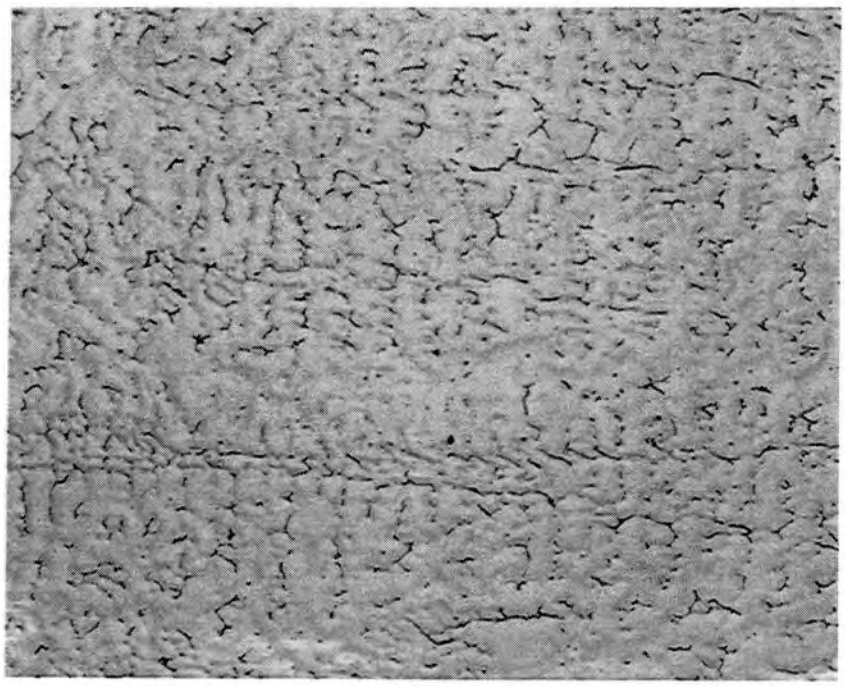
Ten point count determinations were employed for each micrograph location. In total, 600 individual determinations (50 determinations per sample) were employed to characterize the sample set. The average ferrite content and 2σ standard deviation were calculated for each sample and are summarized in Table 13. Photomicrographs representative of the round-robin samples (A-L), are provided in Figures 7-18.

The results of the point counting analysis indicate that the ferrite content of the sample set ranges from 3.4 to 60.1 volume percent ferrite. The average 2σ

Table 13. Ferrite Content (Volume %) of the Round-Robin Sample Set by Systematic Manual Point Count.

Sample Code	Sample Identification	Mean Ferrite Content (Vol %)	2σ
A	CF-8	3.4	0.9
B	CF-3MHF	12.5	1.9
C	CF-8M	14.1	1.5
D	ASTM A890-4A	35.1	3.0
E	ASTM A890-4A	37.7	2.1
F	ASTM A890-4A (CC)	35.7	2.7
G	ASTM A890-5A	48.0	3.2
H	ASTM A890-5A (CC)	40.7	3.0
I	ASTM A890-5A	52.2	3.1
J	CD7MCuN (CC)	52.9	2.7
K	CD7MCuN	57.4	2.4
L	CD7MCuN	60.1	2.4

"CC" indicates centrifugally cast material. All other alloys are statically cast.



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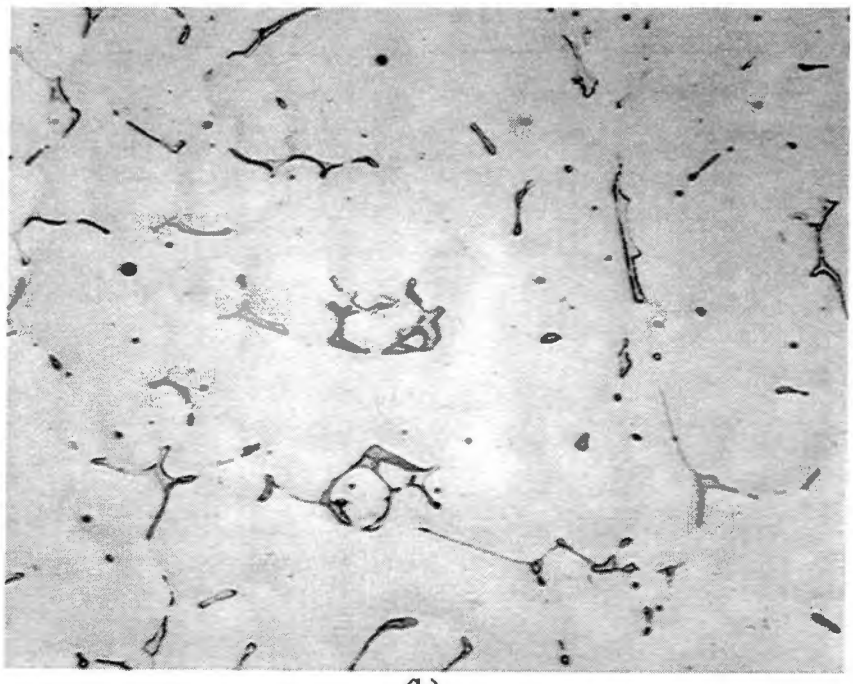
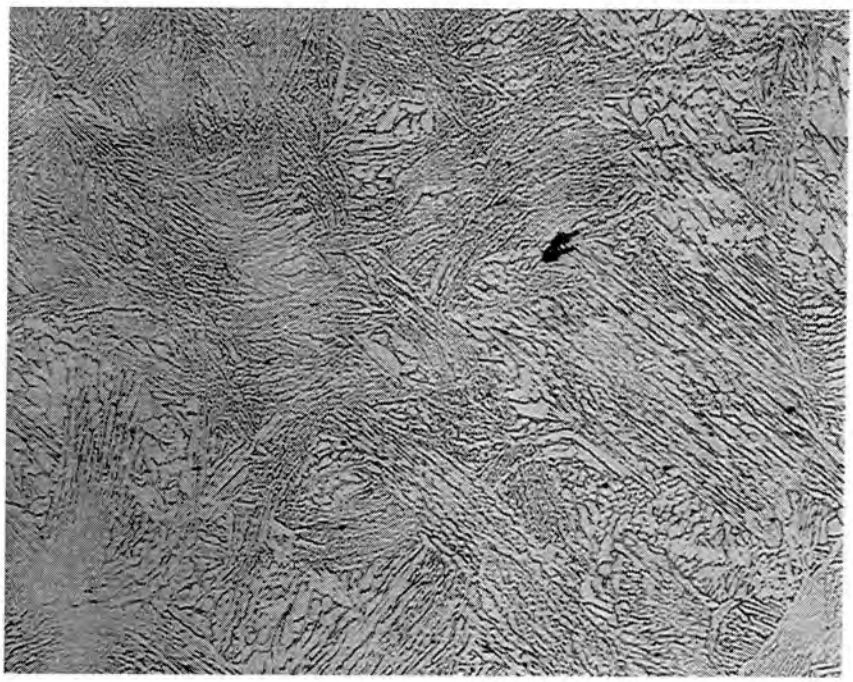


(b)

Figure 7. Round-Robin Sample A (CF8 – 3.4% Ferrite). (a) 50x and (b) 200x

Etchant: Oxalic Acid



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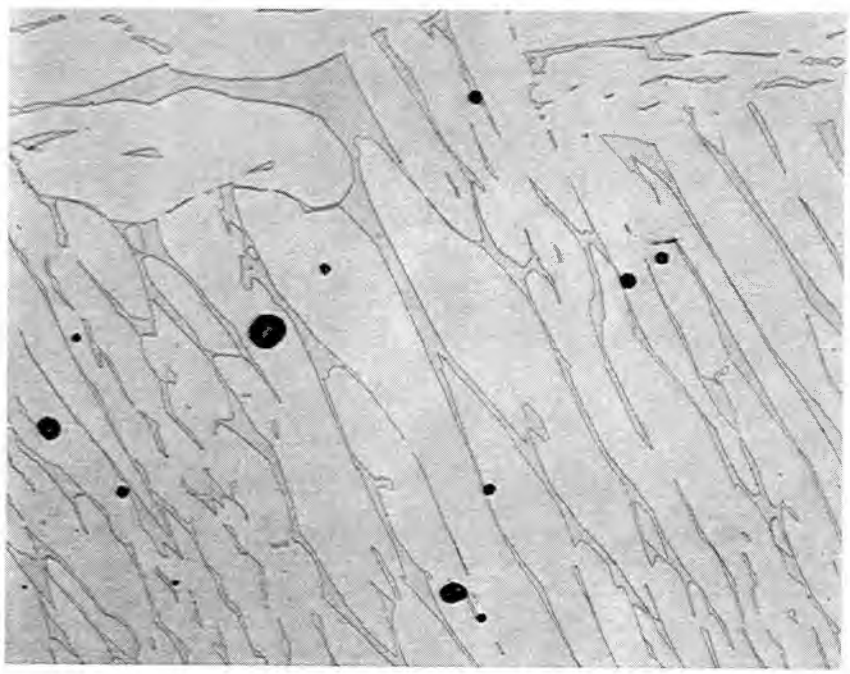
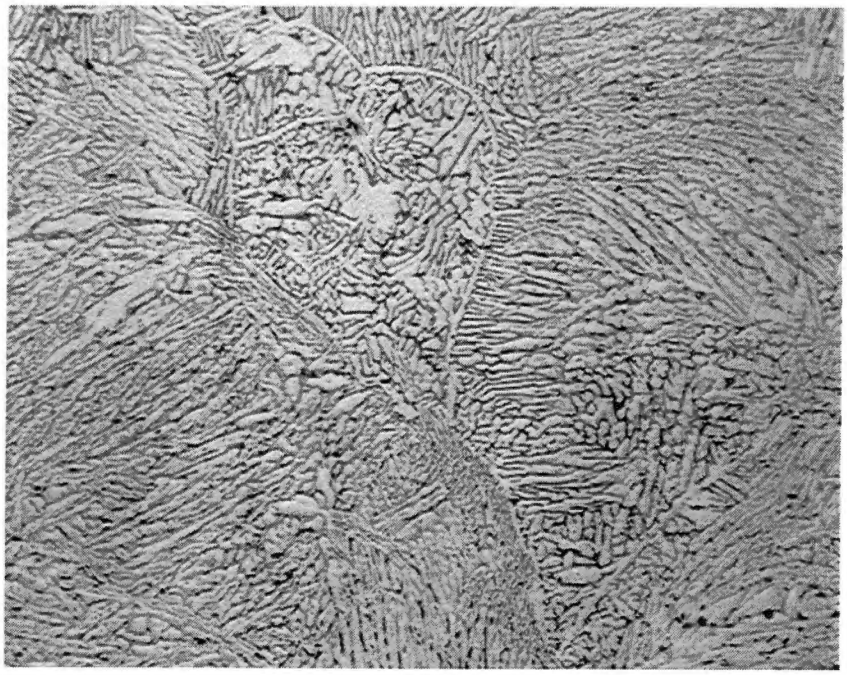


Figure 8. Round-Robin Sample B (CF3M – 12.5% Ferrite). (a) 50x and (b) 200x

Etchant: Oxalic Acid



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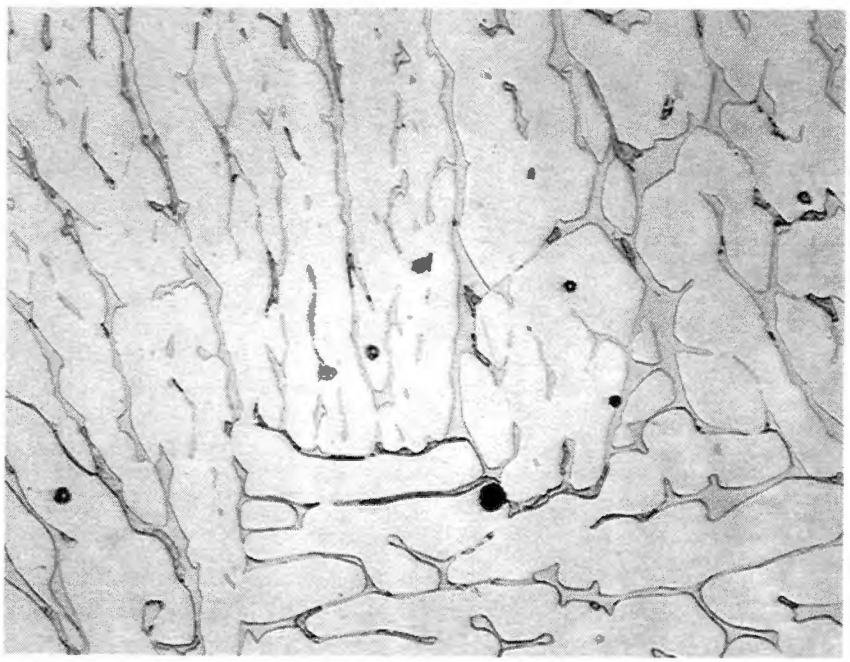
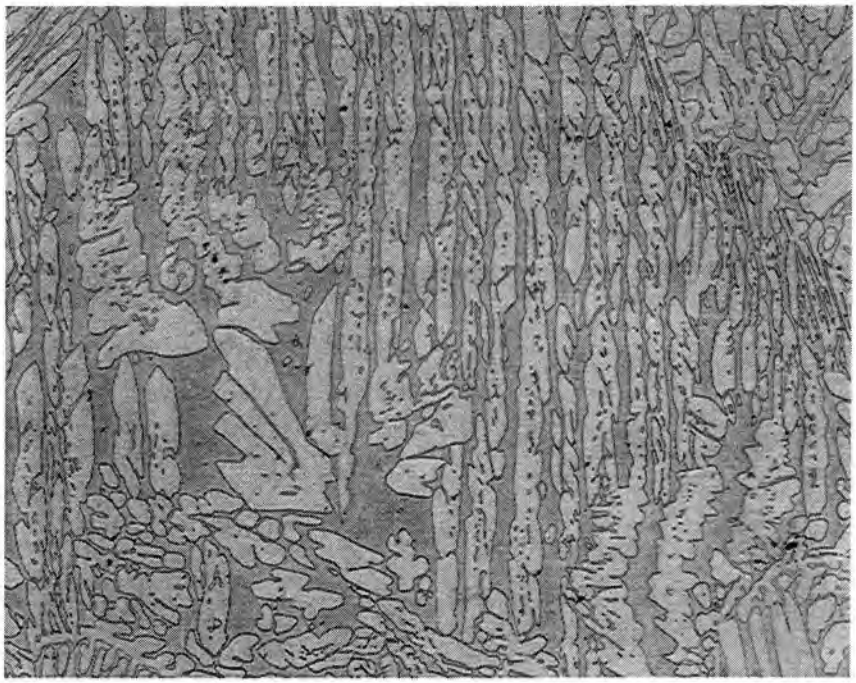


(b)

Figure 9. Round-Robin Sample C (CF8M – 14.1% Ferrite). (a) 50x and (b) 200x

Etchant: Oxalic Acid



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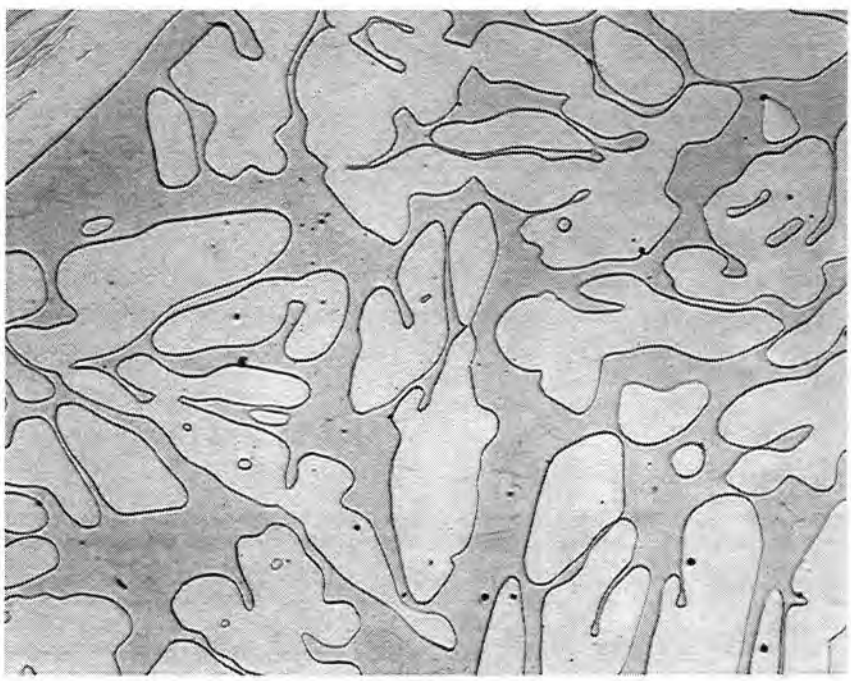
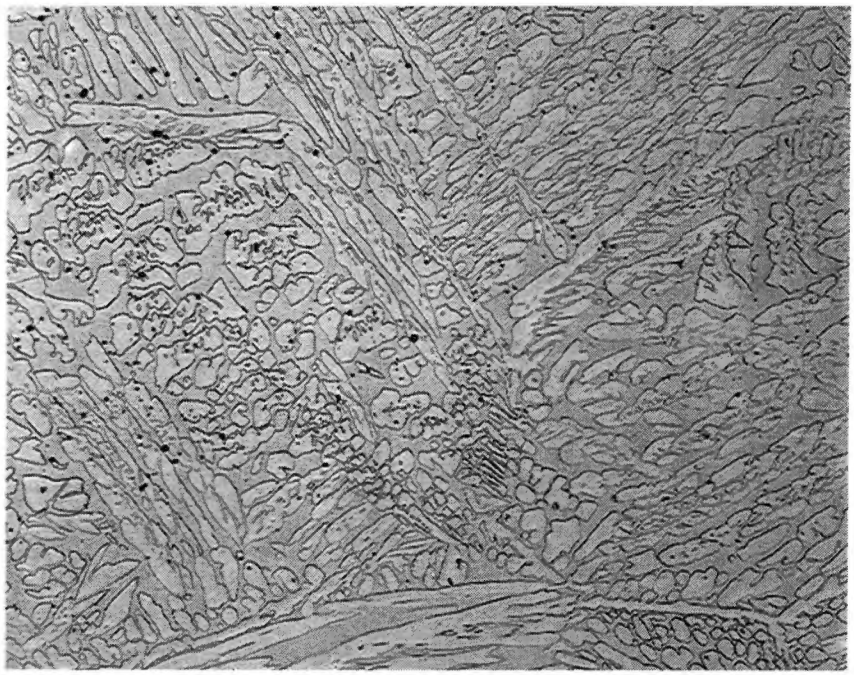


(b)

Figure 10. Round-Robin Sample D (ASTM A890-4A – 35.1% Ferrite). (a) 50x and (b) 200x; Etchant: Oxalic Acid



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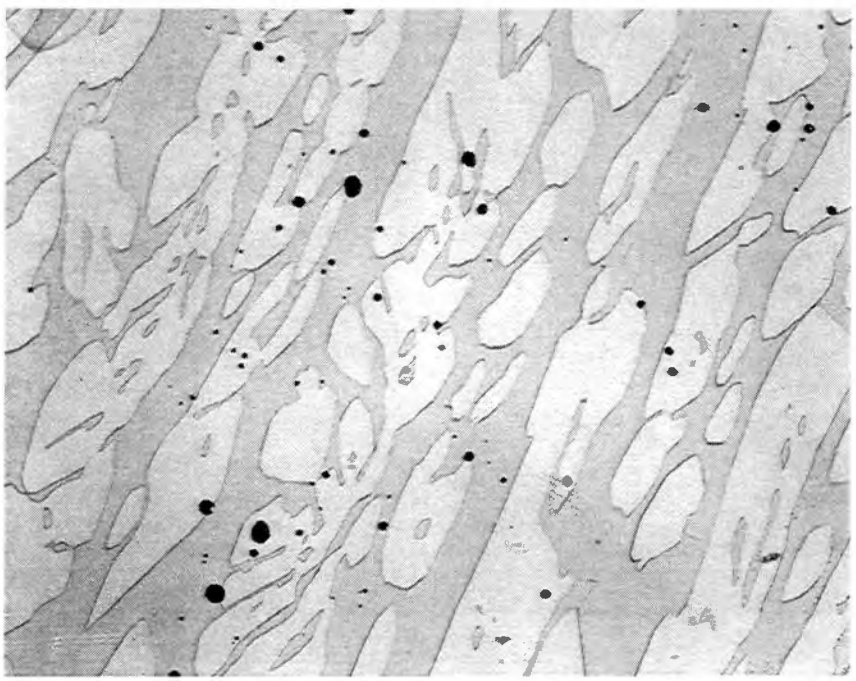
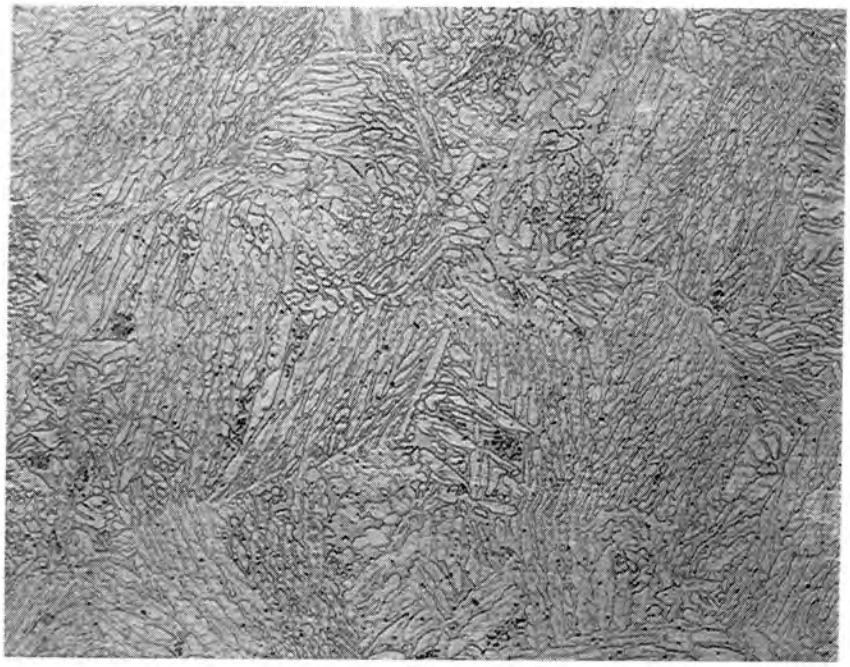


(b)

Figure 11. Round-Robin Sample E (ASTM A890-4A – 37.7% Ferrite). (a) 50x and (b) 200x; Etchant: Oxalic Acid



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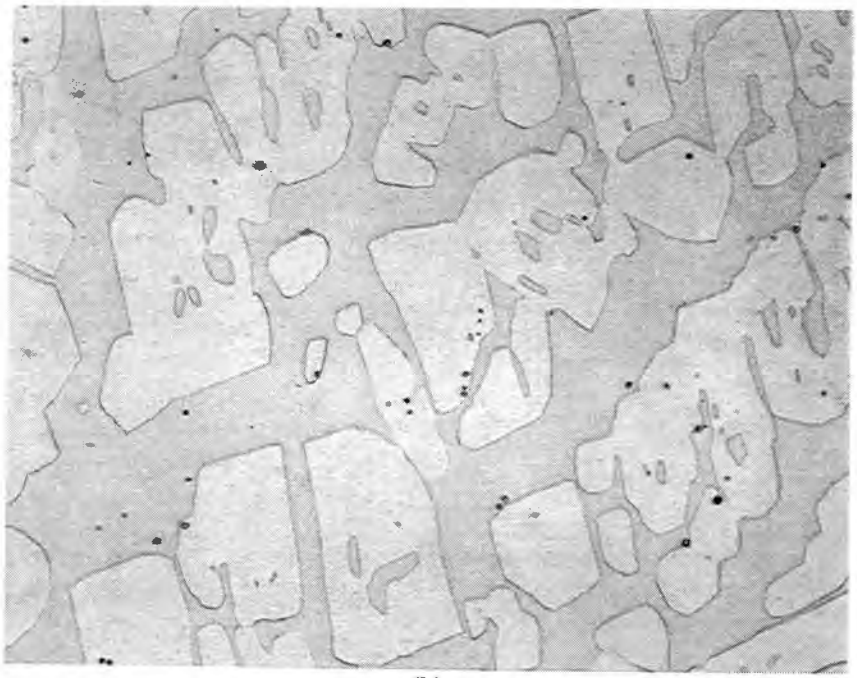
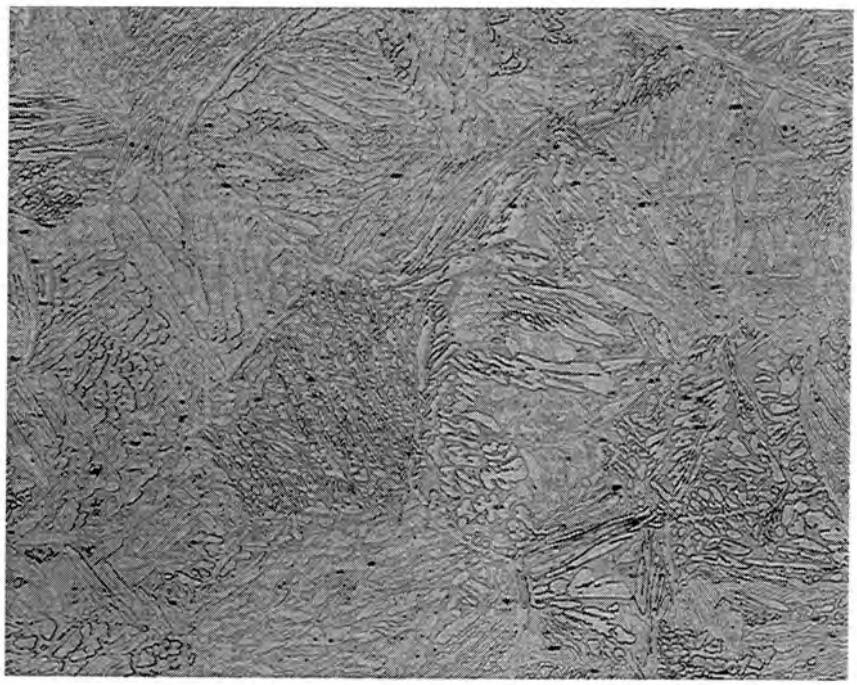


(b)

Figure 12. Round-Robin Sample F (ASTM A890-4A-CC – 35.7% Ferrite). (a) 50x and (b) 200x; Etchant: Oxalic Acid



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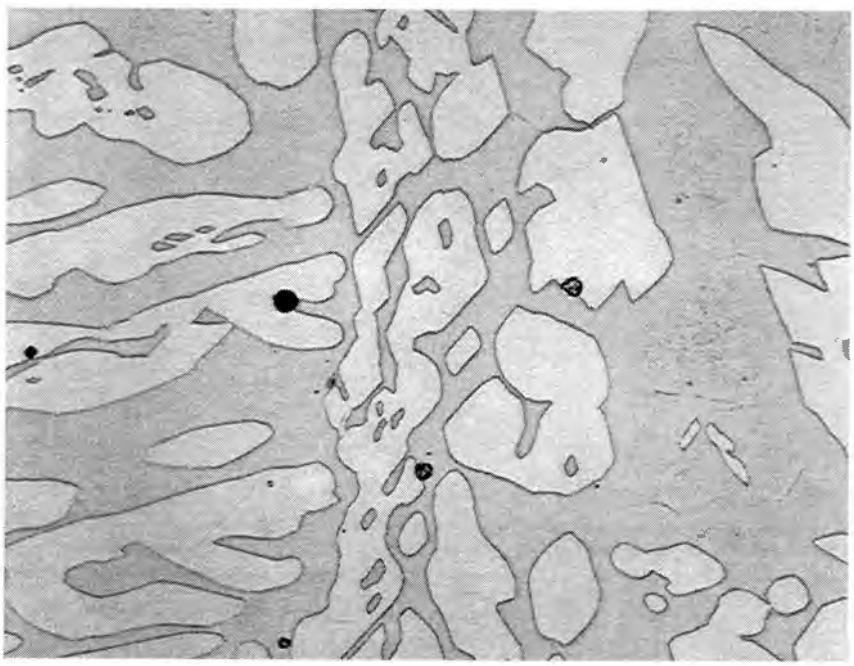
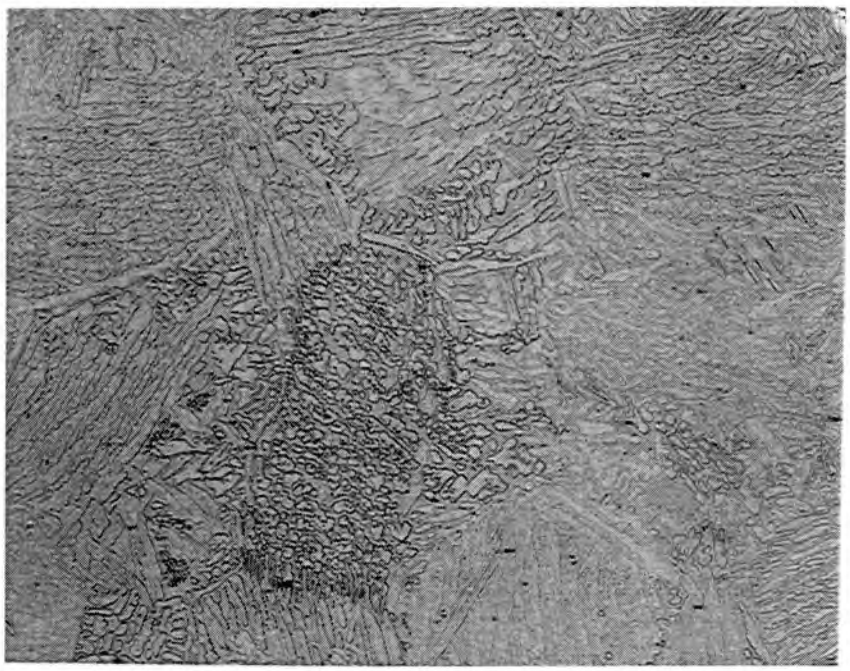


(b)

Figure 13. Round-Robin Sample G (ASTM A890-5A – 48.0% Ferrite). (a) 50x and (b) 200x; Etchant: Oxalic Acid



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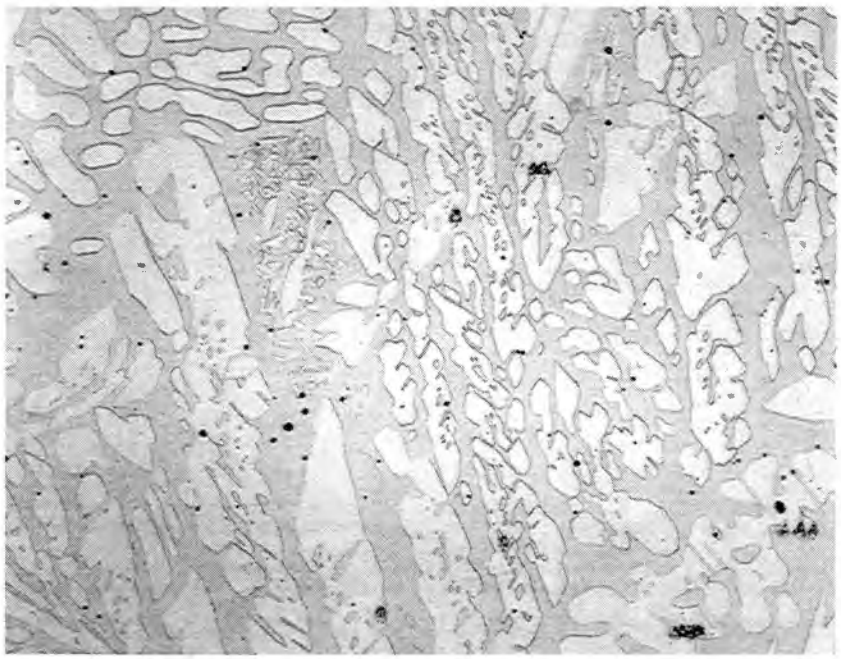
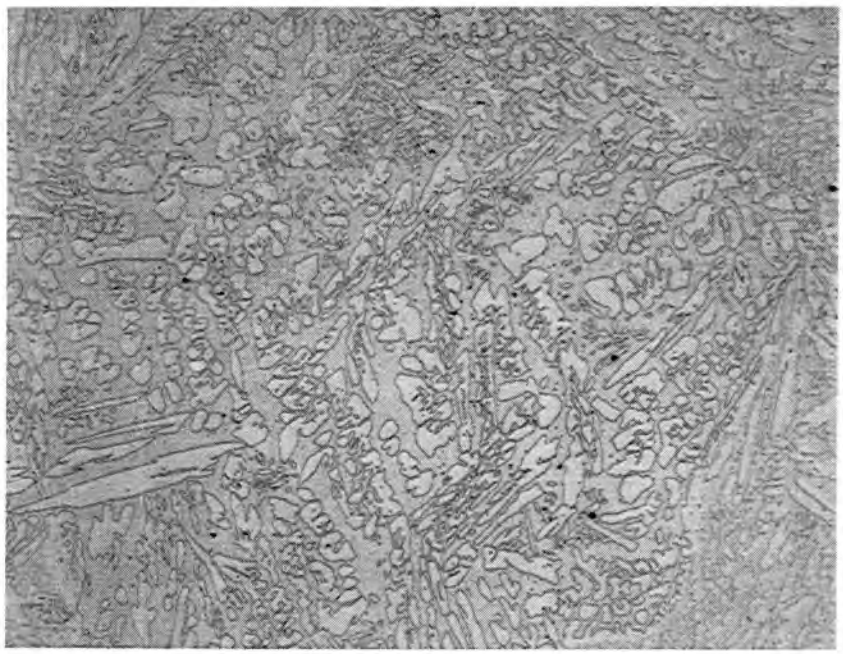
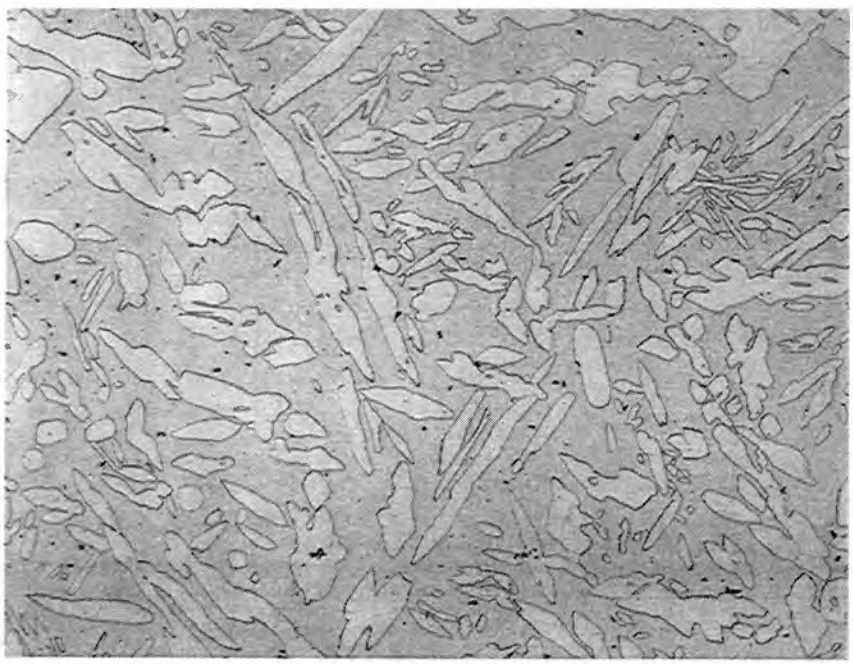


(b)

Figure 14. Round-Robin Sample H (ASTM A890-5A-CC – 40.7% Ferrite). (a) 50x and (b) 200x; Etchant: Oxalic Acid



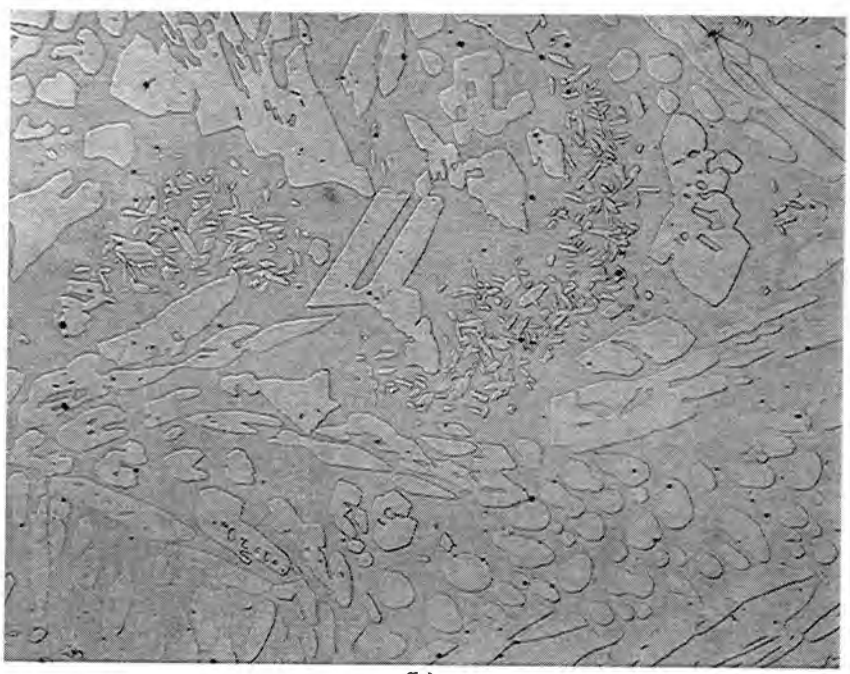
(a)



(b)

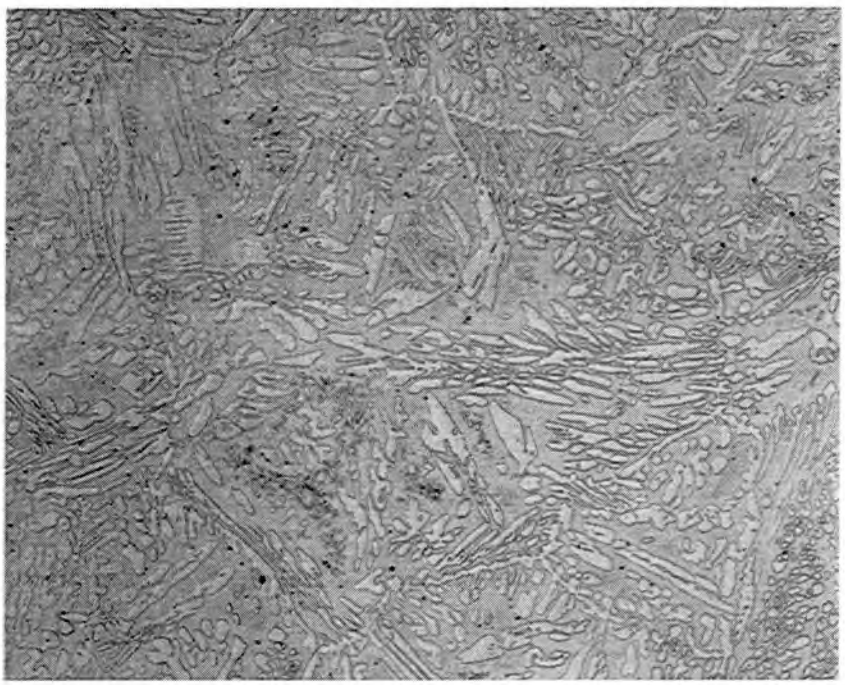
Figure 15. Round-Robin Sample I (ASTM A890-5A – 52.2% Ferrite). (a) 50x and (b) 200x; Etchant: Oxalic Acid

(a)

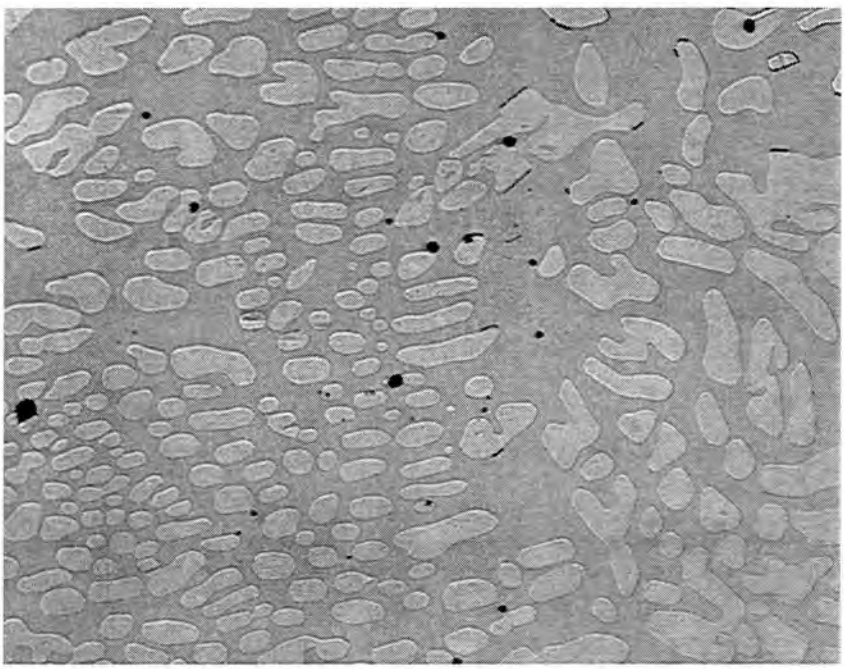


(b)

Figure 16. Round-Robin Sample J (CD7MCuN-CC – 52.9% Ferrite). (a) 50x and (b) 200x; Etchant: Oxalic Acid

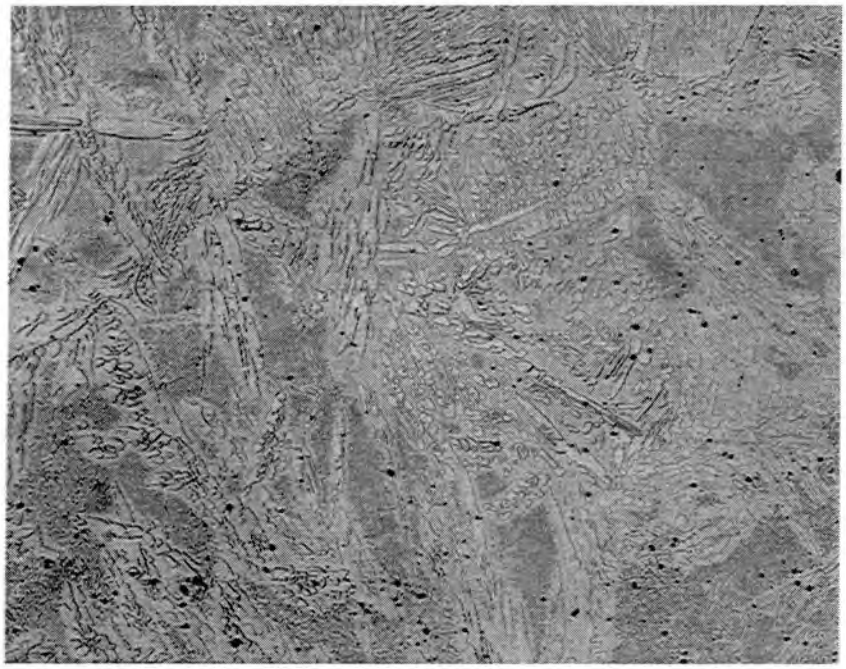


(a)

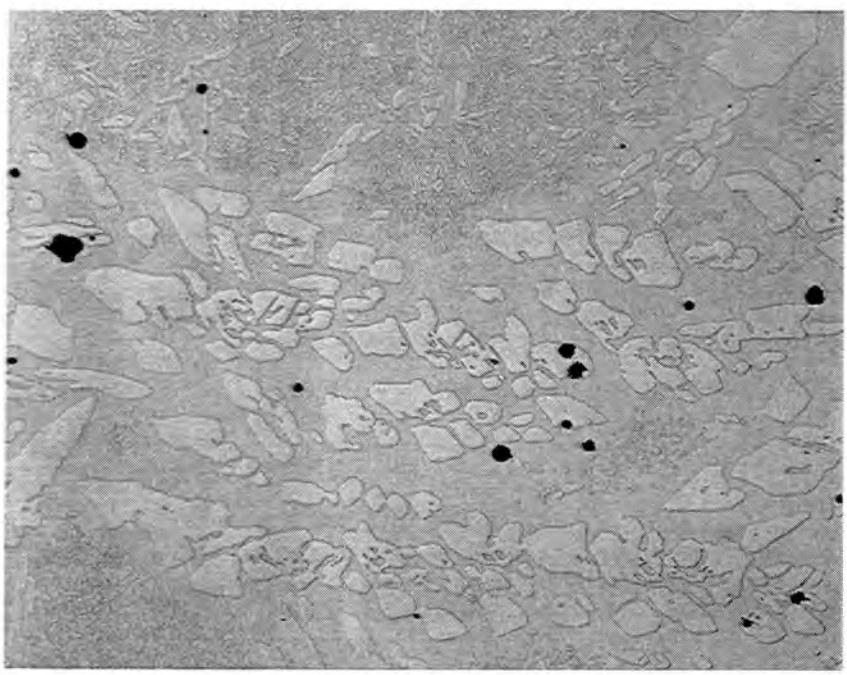


(b)

Figure 17. Round-Robin Sample K (CD7MCuN – 57.4% Ferrite). (a) 50x and (b) 200x; Etchant: Oxalic Acid



(a)



(b)

Figure 18. Round-Robin Sample L (CD7MCuN – 60.1% Ferrite). (a) 50x and (b) 200x; Etchant: Oxalic Acid

value, for the entire sample set, was 2.4, ranging from 0.9 to 3.2. The samples were selected from a series of austenitic and duplex stainless steel castings.

Ferrite Measurement by Magne Gage

Ferrite measurement, using the Magne Gage, was reported for the five round-robin participants who utilized this technique. Table 14 is a summary of round-robin ferrite content utilizing the Magne Gage, as determined by the five participants. Analyzing the entire data set, encompassing all five participants, the round-robin samples are characterized by a mean FN value and interlaboratory reproducibility. Summarizing the Magne Gage trials, Table 14 reveals that the average ferrite content of the round-robin samples ranges from 3.3 to 91 FN.

Ferrite measurement using the Magne Gage technique, properly calibrated to AWS A4.2, identified samples C, D and E with 2σ values greater than 14% of the mean. The significance of this correlation is as follows:

- Utilizing previous round-robin studies as a reference, a 2σ variance greater than 14% of the mean indicates that the corresponding round-robin sample does not exhibit sufficient interlaboratory reproducibility, for use as a Magne Gage cast secondary calibration standard.

All other 2σ values were less than 13% for this data set, indicating sufficient interlaboratory reproducibility for samples A, B, F, G, H, I, J, K and L.

Table 14. Summary of Round-Robin Ferrite Content utilizing the Magne Gage, as Determined by Participants.

Sample Code	University of Tennessee (FN)	Lincoln Electric (FN)	ESAB (FN)	Hobart Brothers Co. (FN)	NIST (FN)	Round-Robin Mean FN	Standard Deviation (2σ)	Reproducibility
A	3.2	3.3	3.3	3.3	3.4	3.3	0.2	6%
B	11.0	12.1	12.2	11.6	11.6	11.7	1.0	9%
C	12.4	14.7	14.8	14.5	14.6	14.2	2.1	15%
D	68.6	61.5	57.5	55.1	59.6	60.4	10.3	17%
E	65.5	65.1	56.4	55.7	61.6	60.9	9.3	15%
F	64.8	62.9	59.2	61.4	61.4	61.9	4.1	7%
G	72.3	72.3	63.6	63.8	67.2	67.8	8.6	13%
H	62.4	62.1	55.6	58.1	58.6	59.4	5.7	10%
I	73.5	74.1	70.0	66.9	72.5	71.4	5.9	8%
J	76.2	75.4	70.8	69.8	72.5	72.9	5.6	8%
K	81.9	80.5	76.3	74.6	77.8	78.2	6.0	8%
L	95.3	92.5	87.1	N/A	89.2	91.0	7.2	8%

Reproducibility (%) = 2σ/Mean FN * 100 Reproducibility less than 14% is typical of previous WRC round-robins.

Ferrite Measurement by Feritscope®

Ferrite measurement, using the Feritscope®, was reported by six round-robin participants. However, prior to summarizing these results, it is necessary to recount that of the six participants who returned Feritscope® data, four calibrated according to AWS A4.2 while the remaining two participants calibrated their Feritscopes® using the manufacturer's calibration. Table 15 documents round-robin ferrite content (FN) utilizing the Feritscope®, as determined by participants who calibrated according to AWS A4.2

Summarizing these AWS A4.2 calibrated Feritscope® trials, Table 15 reveals that the mean ferrite content of the round-robin samples ranges from 3.1 to 91.8 FN. Ferrite measurement using an AWS A4.2 calibrated Feritscope® reveals that sample B exhibited a 2σ value greater than 14% of the mean. As previously stated, this value indicates that sample B does not exhibit suitable interlaboratory reproducibility for use as a cast secondary standard. All 2σ values, for the remaining samples, were less than 11%, indicating sufficient interlaboratory reproducibility.

Summarizing Feritscope® trials utilizing a modified AWS A4.2 calibration, Table 16 reveals that the average ferrite content of the round-robin samples ranges from 3.0 to 103.1 FN. Ferrite measurement, using this modified calibration procedure, demonstrated that sample A exhibited a 2σ values greater than 14% of the mean. The remaining samples exhibited 2σ values less than 14% for this data set, indicating sufficient interlaboratory reproducibility.

Table 15. Summary of Round-Robin Ferrite Content utilizing the Feritscope®, as Determined by Participants who Calibrated According to AWS A4.2.

Sample Code	University of Tennessee	Lincoln Electric	Hobart Brothers Co.	Foster Wheeler	Mean FN	Standard Deviation (2σ)	Reproducibility
A	3.0	2.8	2.9	3.7	3.1	0.2	6%
B	8.6	9.2	10.5	10.0	9.6	1.9	20%
C	12.2	12.1	12.3	12.1	12.2	0.2	2%
D	53.7	56.0	55.1	59.4	56.1	2.3	4%
E	49.6	51.9	54.3	60.4	54.1	4.7	9%
F	59.6	56.4	57.1	63.9	59.3	3.3	6%
G	67.7	66.6	69.3	70.6	68.6	2.7	4%
H	62.8	63.5	61.7	66.2	63.5	1.8	3%
I	71.3	72.1	75.8	75.8	73.7	4.8	7%
J	73.2	73.8	75.4	75.2	74.4	2.3	3%
K	75.5	75.8	79.1	78.8	77.3	4.0	5%
L	93.4	85.5	95.9	92.2	91.8	10.9	11%

Reproducibility (%) = 2σ/Mean FN * 100 Reproducibility less than 14% is typical of previous WRC round-robins.

Table 16. Summary of Round-Robin Ferrite Content utilizing the Feritscope®, as Determined by Participants who Calibrated According to a Modified AWS A4.2 procedure.

Sample Code	Stainless Foundry	Fristam Pumps	Mean FN	Standard Deviation (2σ)	Reproducibility
A	2.9	3.0	3.0	0.9	29%
B	9.7	9.2	9.5	0.8	8%
C	12.8	13.0	12.9	0.9	7%
D	55.0	56.5	55.7	4.5	8%
E	56.5	55.1	55.8	5.5	10%
F	61.6	56.5	59.1	7.5	13%
G	66.7	69.5	68.1	4.0	6%
H	61.3	62.7	62.0	5.1	8%
I	71.8	74.0	72.9	4.1	6%
J	72.4	73.5	73.0	2.8	4%
K	78.1	79.7	78.9	1.6	2%
L	103.7	102.4	103.1	12.6	14%

Reproducibility (%) = 2σ/Mean FN * 100 A variance less than 14% is typical of previous WRC round-robins.

Examining Feritscope® data and discriminating between calibration procedures, the following observations are evident:

- In general, the reproducibility associated with calibration to AWS A4.2 was approximately equal to the reproducibility associated with the modified calibration. This indicates that both calibrations provide sufficient reproducibility for the assessment of ferrite content using a Feritscope® and Magne Gage.
- Utilization of a modified AWS A4.2 calibration procedure will not promote sufficient repeatability when characterizing round-robin samples. Examining the results of participants who calibrated to AWS A4.2 and comparing this with participants who used a manufacturer's calibration, it was found that participants using a manufacturer's calibration reported a significantly larger number of non-compliant samples (2σ > 10% of the mean ferrite content).

  An example of this is clearly illustrated by the response of Fristam Pumps, where nearly the entire round – robin sample set was outside of the 2σ window, for use as cast secondary standards based upon repeatability measurements. This was not the case for those participants using an instrument calibrated to the industry accepted standard, AWS A4.2. Additionally, ferrite measurement on sample L indicated that participants utilizing a modified calibration were not able to establish accurate ferrite measurement for all FN>90. This is due to the fact that a manufacturer's calibration or modified AWS A4.2 calibration procedure, can not calibrate the Feritscope® for use over the entire FN range. (calibration

is only valid over the FN range of the standards provided)

FN vs. Percent Ferrite

The literature review indicated that engineers in academia and industry have struggled to correlate ferrite number (FN) to a volumetric estimation of ferrite content (percent ferrite). The completion of the round-robin allows a correlation to be drawn between the FN evaluations, obtained from Magne Gage and Feritscope® surveys, and the volumetric determinations obtained from manual point counting. Utilizing the data sets provided in Tables 15 and 16, a correlation can be drawn to relate ferrite number to percent ferrite when the appropriate instrumentation is calibrated to AWS A4.2.

Figure 19 illustrates the correlation between FN and volume percent, as determined by the round-robin test data. Only data which was obtained from a proper AWS A4.2 calibration was utilized to compose this chart. Note that the chart contains data obtained from both the Magne Gage and Feritscope®. The results show that the correlation, between FN and volume percent ferrite for round robin samples A, B and C, is 0.9:1. The correlation between FN and volume percent ferrite, for round-robin samples D-L, is 1.5:1. This result clearly shows a disparity between the correlation factors over the full FN scale. It is important to note that the correlation between ferrite number and volume percent ferrite is not uniform over the full FN range and the proper correlation factor should be chosen when transposing ferrite number and volume percent ferrite.

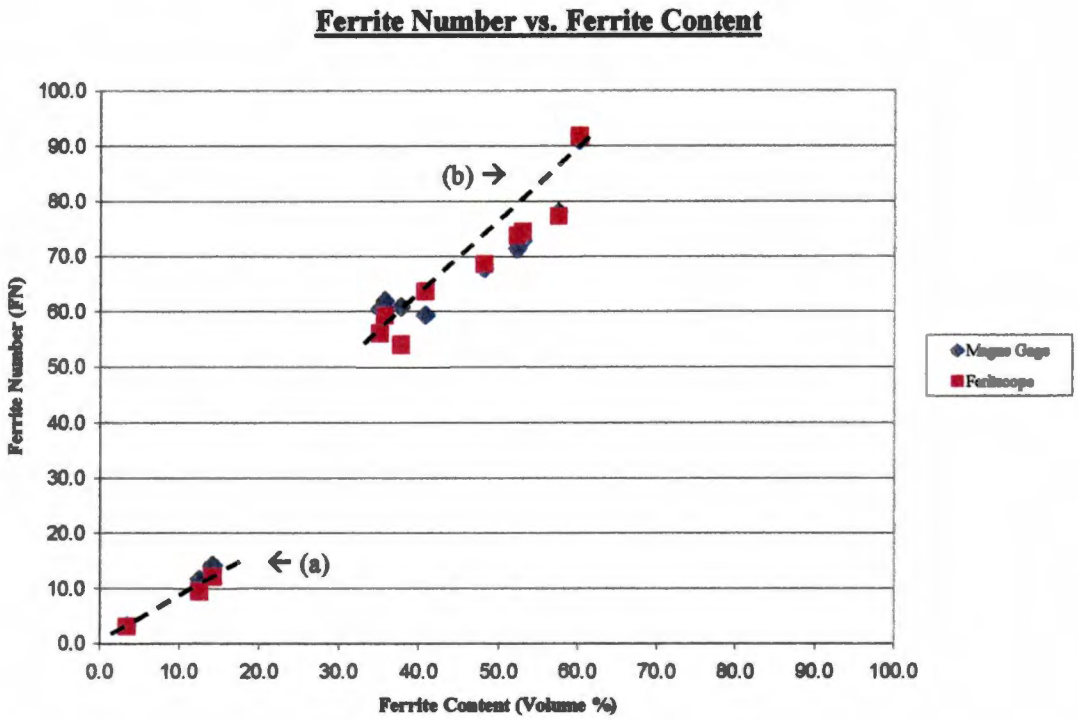


Figure 19. Ferrite Number vs. Ferrite Content, as determined by AWS A4.2 Calibration of Magne Gage and Feritscope® Instruments. (a) Slope = 0.9; (b) Slope = 1.5

Round-Robin – Conclusions

The primary goals of the round-robin study were defined as follows:

- Assess the repeatability and reproducibility of ferrite measurement data, between laboratories, using Magne Gage and Feritscope® techniques.
- Determine the applicability of manufacturing cast secondary standards from static and centrifugal castings.
- Determine a more defined correlation between measurement techniques, including ferrite measurement by manual point counting, Magne Gage and Feritscope®.

The following conclusions can be drawn:

1. Round Robin participant measurements of samples A, B, C, D, and E repeatedly exhibited a 2σ value which indicate probable insufficient repeatability when used with a Magne Gage and/or a Feritscope®. Samples A, B, C, D, and E are statically cast austenitic and duplex alloys whose 2σ repeatability is greater than 10% of the mean FN of the respective round-robin sample. Data obtained from all five participants, who calibrated to AWS A4.2 and used a suitable application method, confirmed this conclusion.

2. Samples H and J exhibited a 2σ repeatability less than 10% of their mean FN values, as determined by all participants using proper calibration and application techniques. Sample F was identified as unsuitable for utilization as a cast secondary standard. However, this behavior could not be conclusively confirmed. In general, the improved repeatability of the centrifugal castings was independent of ferrite measurement technique. Improved repeatability is attributed to the centrifugal casting process, which generally results in a more uniform ferrite/austenite phase morphology. This microstructure is a key in producing a cast secondary standard with little ferrite content variation. Thus, it is to be concluded that cast secondary standards should be manufactured using the centrifugal casting process.

3. Instrument calibration, utilizing AWS A4.2, demonstrated improved repeatability. It is recommended that AWS A4.2 be utilized for the calibration and operation of Magne Gage and Feritscope® instruments to maintain optimum repeatability. Interlaboratory reproducibility was unaffected by calibration procedure.

4. A comparison of point count, Magne Gage and Feritscope® techniques revealed that a suitable correlation could be drawn between ferrite number (FN) and volume percent. For FN values ranging from 0-15, this correlation factor is 0.9:1 (FN:Volume Percent). For FN values ranging from 55-90 FN, this correlation factor is 1.5:1 (FN:Volume Percent).

Depth Profile Characterization

Producers and users of cast stainless steels require the ability to accurately assess the ferrite content of a casting. Ideally, a non-destructive test, designed to assess ferrite content, is desired to characterize a solution annealed and finished casting. Differences in cooling rates between the surface and center of a casting can affect its ferrite content as well as the potential for mold-liquid metal interaction. The goal of the depth profile study was to determine at what depth below a cast surface, a uniform level of ferrite representative ferrite content representative of the casting occurs.

Three depth profile blocks were manufactured. One each from two different heats of ASTM A890-4A and one from a single heat of ASTM A890-6A. The 1" cubic blocks were removed perpendicular to the cast surface. Initial ferrite measurement included a profile of each block, which entailed utilizing the Feritscope® to characterize the ferrite content of the cube on each of four mutually orthogonal sides. Each side evaluated was perpendicular to the cast surface. After establishing the ferrite content as a function of depth from the cast surface, material was removed, using a ceramic grinding disk, from the cast surface of the block, proceeding perpendicularly into the casting, until a uniform ferrite content was established. The ferrite content was determined, using a Feritscope®, at five separate locations on the measurement face (parallel to the cast surface), as material was removed from the cast surface. A uniform ferrite level was considered attained when successive ferrite measurements remained relatively unchanged (±5 FN) with increasing depth below the cast surface.

ASTM A890-4A – Heat 1

ASTM A890-4A is a common duplex grade alloy which has been employed by the United States Navy for marine service. Its widespread acceptance in the European community and increasing use in the United States makes it an ideal candidate for extensive characterization.

As previously described, a cube of ASTM A890 (Heat 1) was extracted perpendicular to the cast surface. As material was successively removed from the cast surface and the ferrite content recorded, a relationship was defined between ferrite content and the depth below the cast surface. Figure 20 illustrates this relationship for ASTM A890-4A (Heat 1).

A ferrite survey on the cast surface revealed that the surface ferrite content equals 40 FN. However, after 0.025" of material removal, the depth profile sample reaches a uniform ferrite content of 62 FN. Figure 20 illustrates that removal of more than 1/8" of material is more than adequate to establish a uniform ferrite content for the bulk of the casting.

ASTM A890-4A – Heat 2

In order to assess any variation between heats, a second heat of ASTM A890-4A was selected for similar analysis. Using the same technique, ASTM A890-4A (Heat 2) was characterized to establish the relationship between ferrite content and depth below a

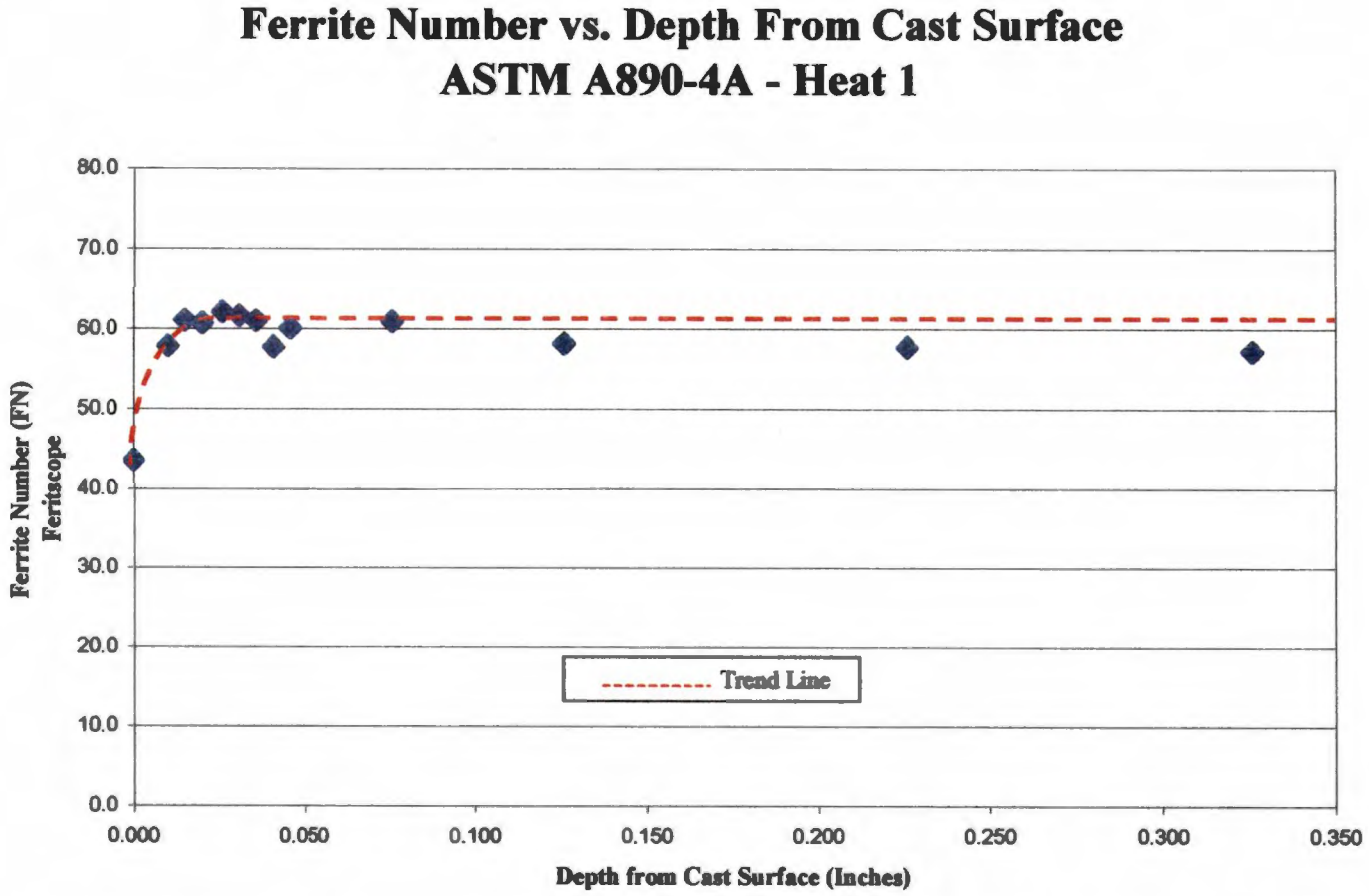


Figure 20. Ferrite Number vs. Depth from Cast Surface for ASTM A890-4A – Heat 1.

cast surface. Figure 21 illustrates this relationship for ASTM A890-4A (Heat 2). A ferrite survey on the cast surface revealed a surface ferrite content of 22 FN. However, after 0.050" of material removal, the depth profile sample reaches a uniform ferrite content of 48 FN. Thus, removal of more than 1/8" of material is sufficient to establish a uniform ferrite content for the bulk of the casting with a reasonable degree of certainty.

ASTM A890-6A

To compare depth profile data between alloys, a heat of ASTM A890-6A was selected for analysis. Using the same technique, ASTM A890-6A was characterized to further establish the relationship between ferrite content and depth below a cast surface.

Figure 22 illustrates this same relationship for ASTM A890-6A. A ferrite survey on the cast surface revealed a surface ferrite content of 42 FN. However, with only 0.025" of material removed, the ferrite content reaches a uniform ferrite level of 45 FN. Figure 22 further illustrates that removal of 1/8" of material is more than sufficient to establish a uniform ferrite content for the bulk of the casting with a reasonable degree of certainty.

Probe Interaction Volume

An inherent factor which affects the accuracy of ferrite measurement, using a Feritscope®, is the measurement probe interaction volume. Recall from the literature

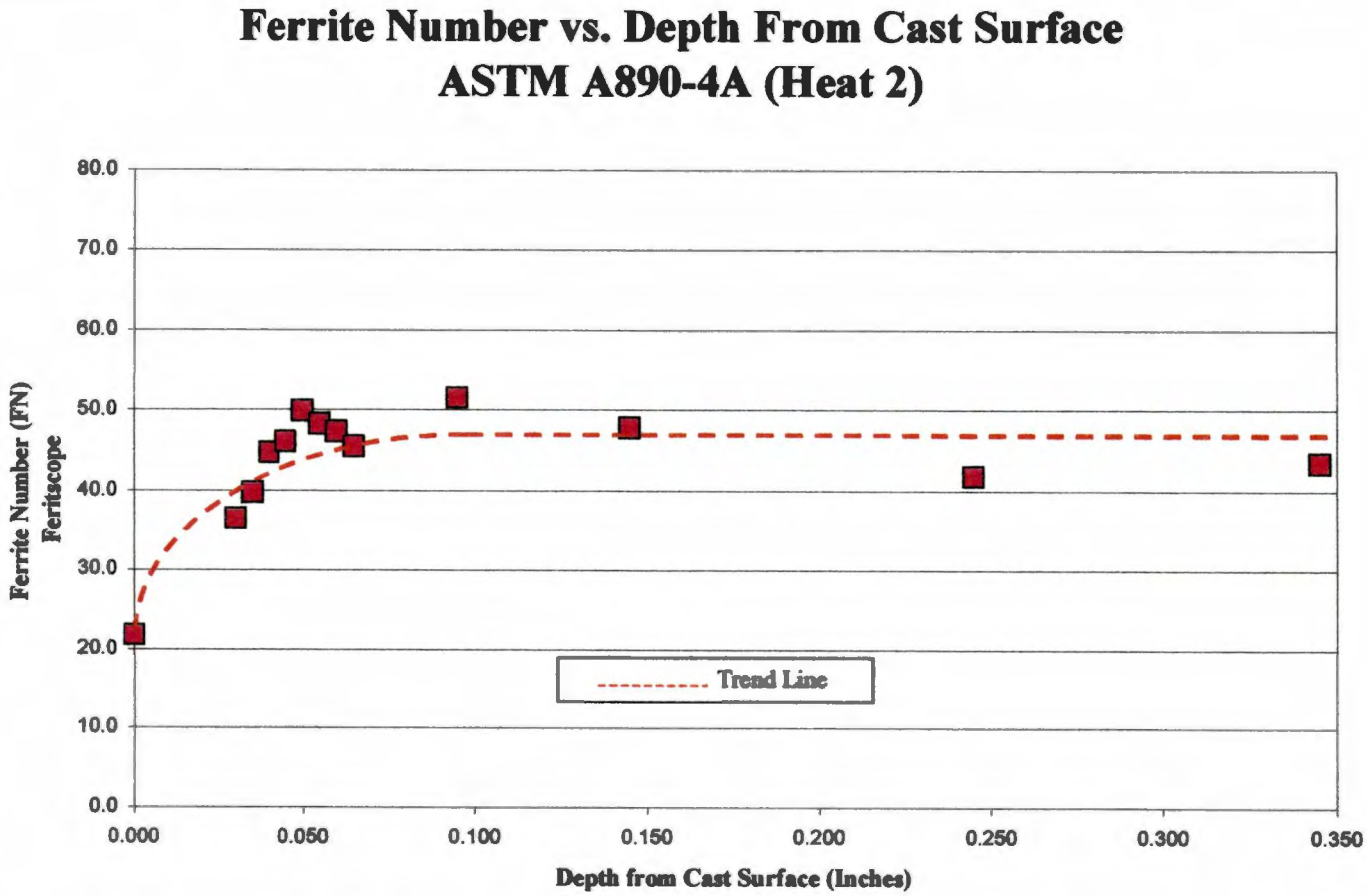


Figure 21. Ferrite Number vs. Depth from Cast Surface for ASTM A890-4A – Heat 2.

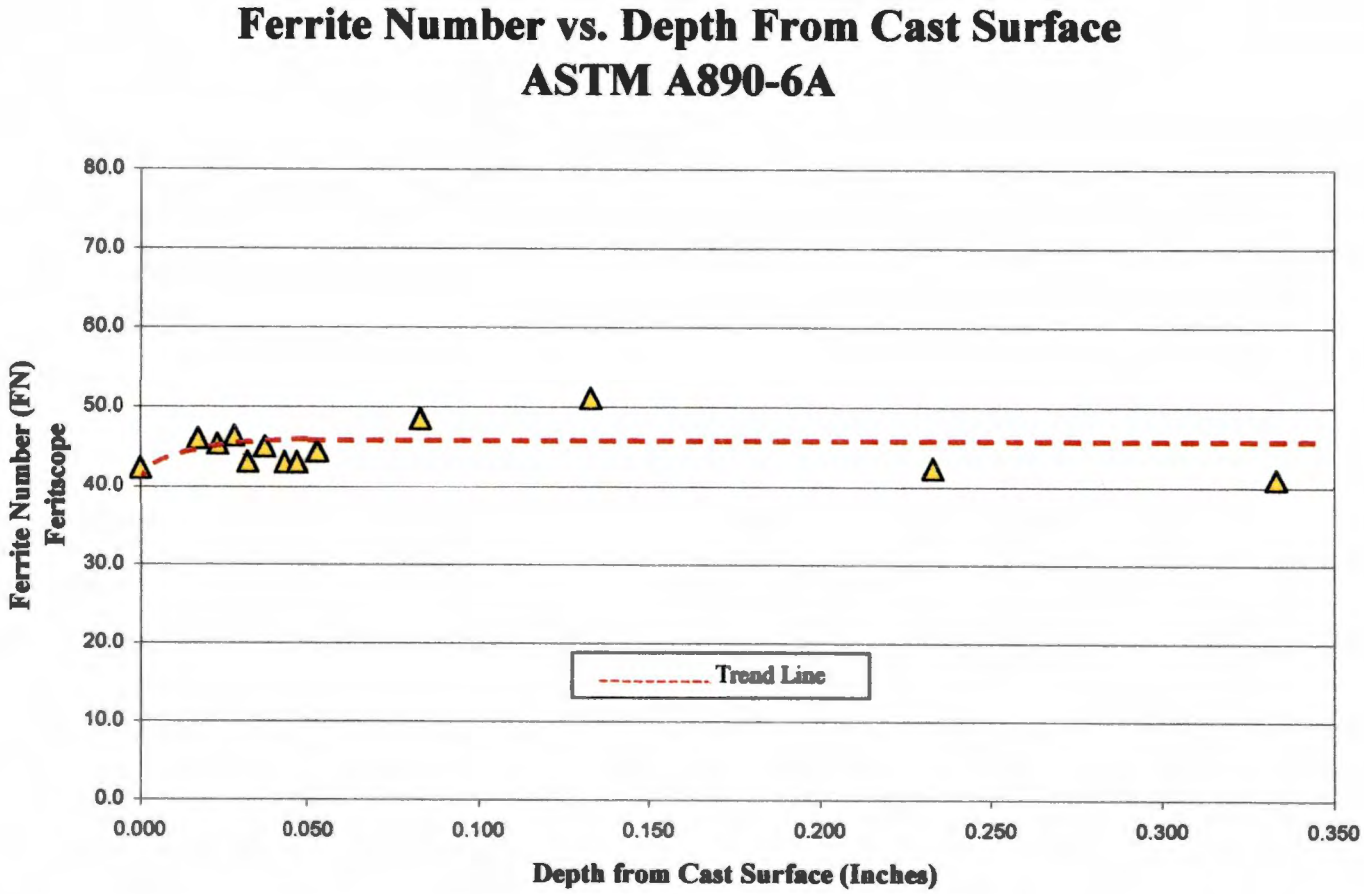


Figure 22. Ferrite Number vs. Depth from Cast Surface for ASTM A890-6A.

review that the measurement probe induces a magnetic field in the substrate and compares the magnetic response to the calibration set stored in memory. It is logical to assume that an interruption in the induced magnetic field would adversely affect the accuracy of ferrite measurement. Initial work on the depth profile study required that edge profiles be conducted to estimate the ferrite content of the block. The initial characterization served as a guideline for material removal, establishing changes in ferrite content with increasing depth below a cast surface.

An increase in ferrite content, as a function of depth below the cast surface, was noted for each depth profile block, as demonstrated in Figures 20-22. However, since ferrite measurement proceeded from the edge, adjacent to the cast surface, towards the interior of the casting, it was proposed by the UTK Materials Joining Research Group that the magnetic field induced by the Feritscope® was influenced by the proximity of the measurement probe to the edge of the block. This suggestion was based upon preliminary work with the Feritscope® prior to the institution of this program.

To fully characterize this phenomenon, a standard sample, consisting of a 1" cube of statically cast ASTM A890-6A, was prepared for analysis. Ferrite surveys showed that the ferrite content remained virtually unchanged as function of position within the block. The block was then placed on a calibrated measurement stage and the Feritscope® probe was centered on the edge of the block. Precisely one half of the probe was positioned within the sample. Ferrite measurement then proceeded in 0.005" increments until a uniform ferrite content was achieved. Uniform ferrite content is defined as three or more successive ferrite determinations whose FN values are relatively unchanged (±5 FN). The results are illustrated in Figure 23.

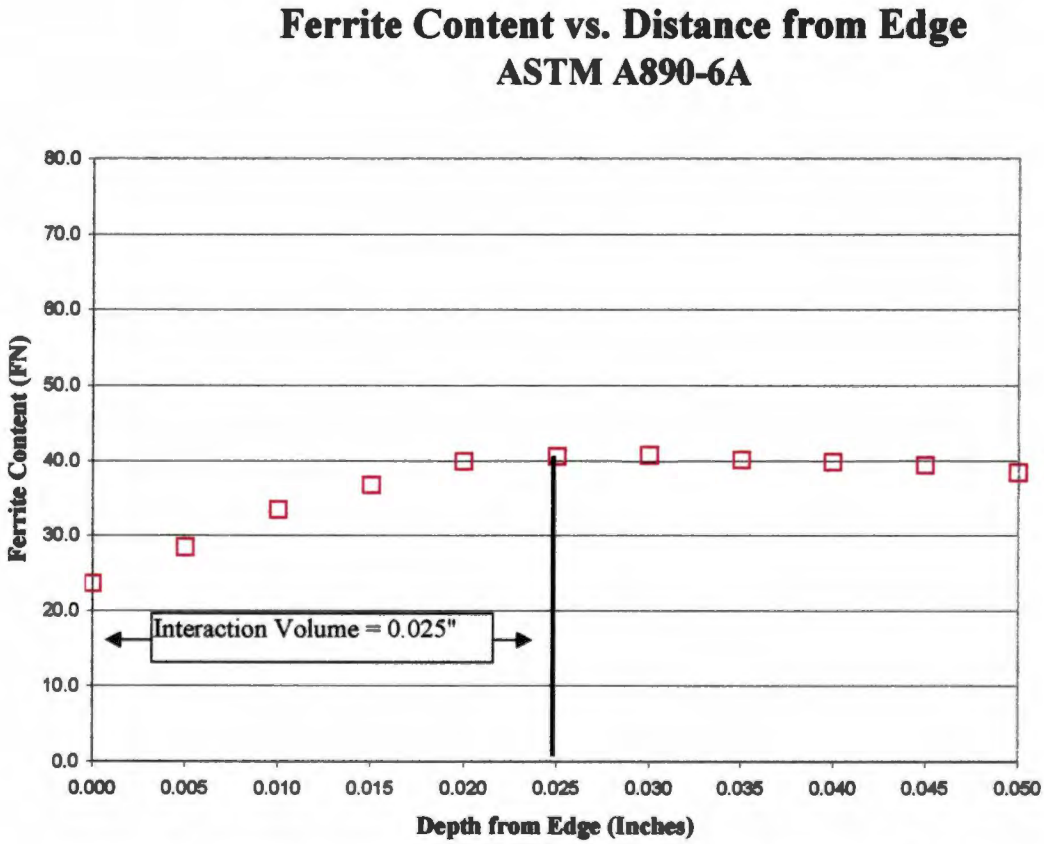


Figure 23. Ferrite Content vs. Distance from Edge for ASTM A890-6A.

As depicted in Figure 23, the ferrite content at the edge of the sample is approximately 24 FN, however, after incrementing to 0.025", the ferrite content reaches a uniform value of 40 FN. This suggests that measurements taken at least 0.025" below a surface discontinuity or edge will reveal an accurate ferrite content. Note that 0.025" is also the radius of the Feritscope® probe. This indicates that the radius of the full interaction volume can be approximated by the probe diameter.

Depth Profile Characterization – Conclusions

Based upon the data obtained for the depth profile characterization study, the following conclusions can be derived:

1. Removal of 1/8" of material from the cast surface will result in a ferrite content most characteristic of the bulk of the finished casting. Trials using two alloy systems and two heats of one alloy system confirmed this behavior.
2. Ferrite measurements, utilizing a Feritscope®, taken directly on a cast surface are not indicative of the true ferrite content of the casting. Producers and users of cast products are encouraged to measure ferrite content at a subsurface location, preferably at a depth greater than 1/8" below the cast surface. Removal of 1/8" of material eliminates changes in ferrite content due to cooling rate/microstructure/mold interaction immediately on or below the cast surface.
3. The interaction volume of the Feritscope® probe is defined as 0.050", which is the diameter of the probe. Ferrite measurement performed, with an uninterrupted interaction volume, will promote accurate ferrite measurement. Thus, care should

be taken to ensure a full interaction volume, free of edge effects and surface finish discontinuities, as previously discussed.

Effect of Surface Roughness on Ferrite Measurement

It has been indicated that surface finish can affect the accurate ferrite measurement. Recognizing that producers and users of stainless steel castings wish to characterize the ferrite content of the cast product in the solution annealed and machined forms, a study was implemented to assess ferrite content as a function of surface finish.

Five standard surface finish test blocks, of uniform ferrite content, were prepared from a "CD7MCuN" duplex stainless steel centrifugal casting. CD7MCuN was chosen due to its uniform ferrite content as a function of depth. Each 1"x 3/4"x 3/4" block was designed such that the measurement face was radially oriented in the centrifugal casting.. The measurement face was initially prepared to a uniform surface finish of 0.05μ utilizing metallographic polishing techniques.

Five specific locations were examined on each block using optical light microscopy. Each location was then documented photographically. Next, each specific region was located on a Feritscope® measurement stage and ferrite measurement was performed using a Feritscope®. After metallographic and Feritscope® characterization, a specific surface finish was imparted. The blocks were then placed on the measurement stage and ferrite measurement was performed at the identical locations to directly correlate any change in ferrite content. The results of this work effort are presented in the following sections.

250 Microinch Surface Finish

A 250 microinch milled surface finish was imparted on Surface Finish Sample 1. Total material removed by milling was 0.025". Prior ferrite measurement on the 0.05μ as-polished surface, using a Feritscope®, revealed a mean ferrite content of 70.1 FN with a 2σ standard deviation of 0.5 FN. After the 250 microinch milled surface finish was imparted, the average ferrite content recorded was 68.0 FN with a 2σ standard deviation of 0.2 FN. The disparity between measured ferrite content is not significant in this case. It is apparent that imparting a 250 microinch finish did not significantly influence the measurement of ferrite content in this sample, although, the mean milled surface finish FN falls outside of the 2σ variance established for the metallographically polished surface. A photograph of Sample 1 is shown in Figure 24.

64 Microinch Surface Finish

A 64 microinch milled surface finish was imparted on Surface Finish Sample 2. Total material removal by milling was 0.025". Ferrite measurement on the 0.05μ as-polished surface, using a Feritscope®, revealed an average ferrite content of 76.0 FN with a 2σ standard deviation of 0.0 FN. After the 64 microinch milled surface finish was imparted, the mean ferrite content recorded was 68.0 FN with an average 2σ standard deviation of 0.2 FN. It is apparent that imparting a 64 microinch finish significantly

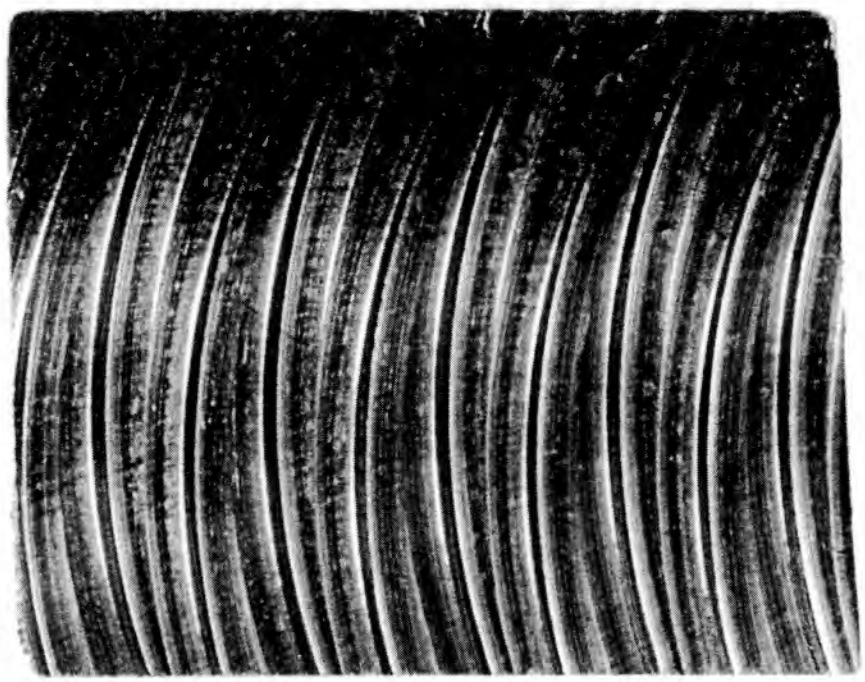


Figure 24. Surface Finish Sample 1 – 250 Microinch Finish

Magnification = 4.5x

influenced the measurement of ferrite content in this sample. A 10 FN reduction in ferrite content was noted after the 64 microinch surface finish was imparted. This reduction is well below the 2σ variance established for the metallographically polished surface finish, indicating significant surface finish effects due to milling. Additionally, regardless of the surface finish, the 2σ value is small when compared to the mean ferrite content. This indicates suitable grouping of the experimental data about the mean ferrite content. A photograph of Sample 2 is shown in Figure 25.

16 Microinch Surface Finish

A 16 microinch milled/ground surface finish was imparted on Surface Finish Sample 3. This was accomplished by milling the sample surface to obtain 0.025" of material removal, including 320 grit sanding to impart the final surface finish. Ferrite measurement on the as-polished 0.05μ surface, using a Feritscope®, revealed an average ferrite content of 72.2 FN with a 2σ standard deviation of 0.1 FN. After the 16 microinch milled surface finish was imparted, the mean ferrite content recorded was 74.6 FN with an average 2σ standard deviation of 0.1 FN. The disparity between ferrite content is not significant in this case. It is apparent that imparting a 16 microinch finish did not significantly influence the measurement of ferrite content in this sample.

Further analysis of the 2σ values for both surface finish conditions indicates excellent grouping of the experimental data about the mean ferrite contents. Also, note that the mean ferrite content of the 16 microinch surface finish is above the 2σ variance associated with a metallographically polished surface. This type of deviation is typical of

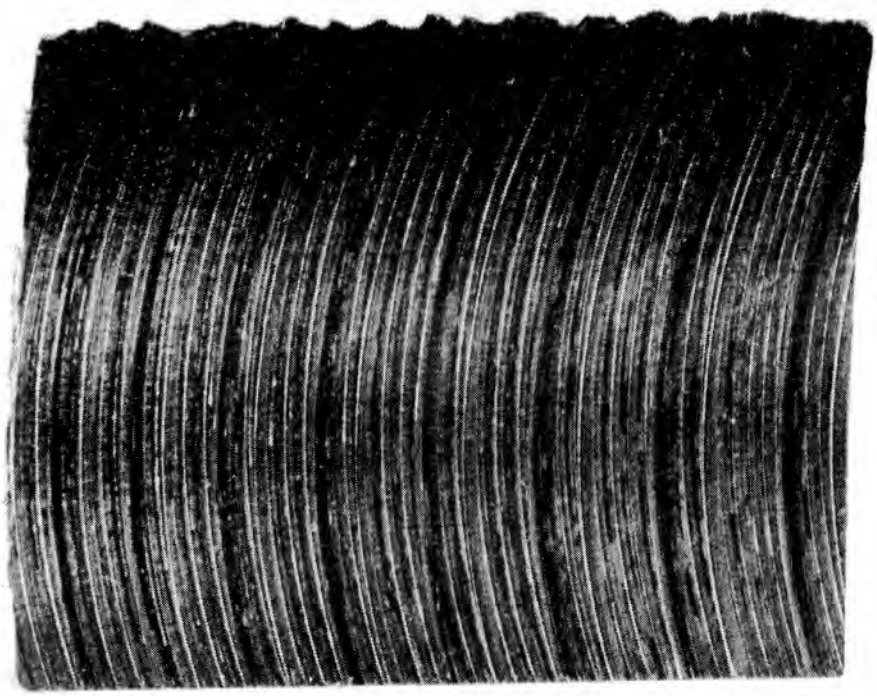


Figure 25. Surface Finish Sample 2 – 64 Microinch Finish

Magnification = 4.5x

this alloy system, therefore, it is warranted that the ferrite content could be elevated after milling. This further illustrates that the impartment of a 16 microinch surface finish did not significantly affect ferrite measurement. A photograph of Sample 3 is shown in Figure 26.

Ground Finish

Using a 4 ½" general purpose 24-grit, angle grinding wheel, a surface finish was imparted on Surface Finish Sample 4. 0.025" of material was removed. Ferrite measurement on the as-polished 0.05μ surface, using a Feritscope®, revealed an average ferrite content of 73.5 FN with an average 2σ standard deviation of 0.1 FN. After the ground surface finish was imparted, the mean ferrite content recorded was 62.7 FN with a 2σ standard deviation of 0.4 FN. The disparity between ferrite content is significant in this case. It is apparent that imparting an angle ground finish significantly influenced the measurement of ferrite content. It is apparent that the utilization of an angle grinder, to impart a surface finish, resulted in an approximate 10 FN reduction in ferrite number. A photograph of Sample 4 is shown in Figure 27. Again, 2σ variations are small, as compared to mean ferrite content of either the metallographically polished or ground surface finish. This further illustrates that the ferrite determinations are well grouped about the mean ferrite contents for each surface finish.

Additionally, the 10 FN reduction is below the 2σ variance established for the polished surface finish, indicating significant surface finish effects due to angle grinding. Metallographic specimens were prepared in a direction transverse to the imparted surface

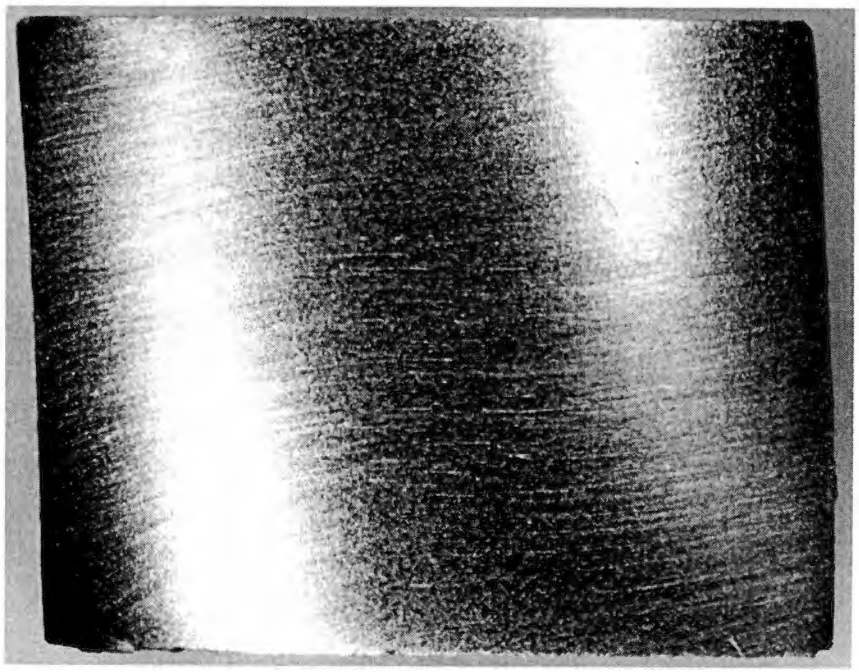


Figure 26. Surface Finish Sample 3 – 16 Microinch Finish

Magnification = 4.5x

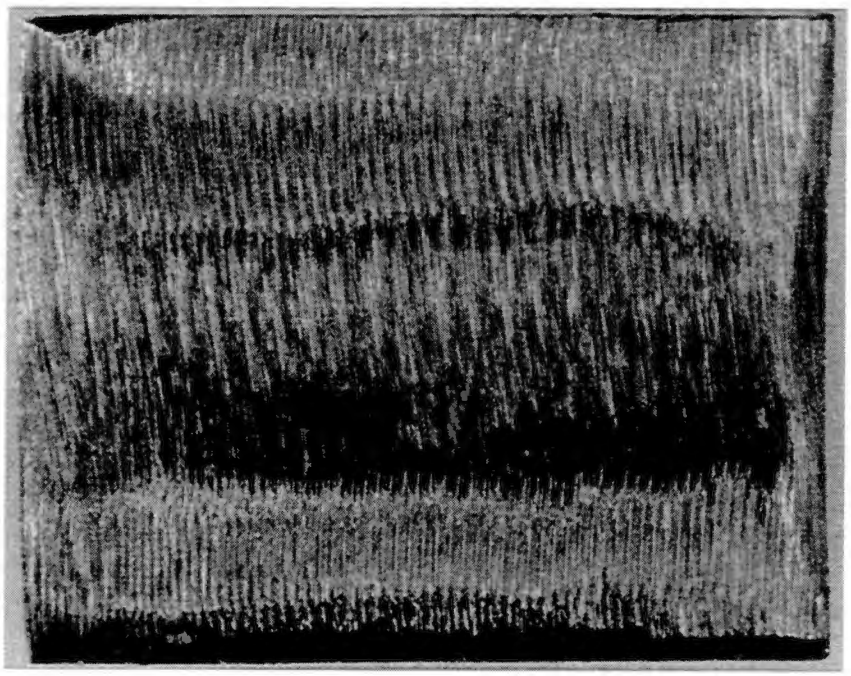


Figure 27. Surface Finish Sample 4 – 24 Grit Ground Finish

Magnification = 4.5x

finish. The results showed that no microstructural changes occurred due to angle grinding.

Additional characterization revealed that removal of the ground surface finish with 120 grit sandpaper (or equivalent) will restore the original ferrite content, as measured on the metallographically polished surface. Grinding with 120 grit sandpaper requires a minimum 0.005" of material removal to eliminate the angle ground surface finish.

#14 Bastard Mill File Finish

Using #14 Bastard Mill File, an as-filed surface finish was imparted on Surface Finish Sample 5. This was accomplished by filing the sample surface to obtain 0.025" of material removal. Ferrite measurement on the as-polished 0.05μ surface, using a Feritscope®, revealed a mean ferrite content of 73.1 FN with a 2σ standard deviation of 0.1 FN. After the #14 Bastard Mill file surface finish was imparted, the average ferrite content recorded was 71.4 FN with an average 2σ standard deviation of 0.2 FN. The disparity between ferrite content is not significant in this case, although the mean ferrite content of the imparted surface finish is below the variance associated with the 2σ value of the metallographically polished surface. The tight grouping of the ferrite determinations is characterized by 2σ values, which are small when compared to the mean ferrite content of the block. Thus, it is apparent that imparting an as-filed surface finish did not significantly influence the measurement of ferrite content in this sample. A photograph of Sample 5 is shown in Figure 28.

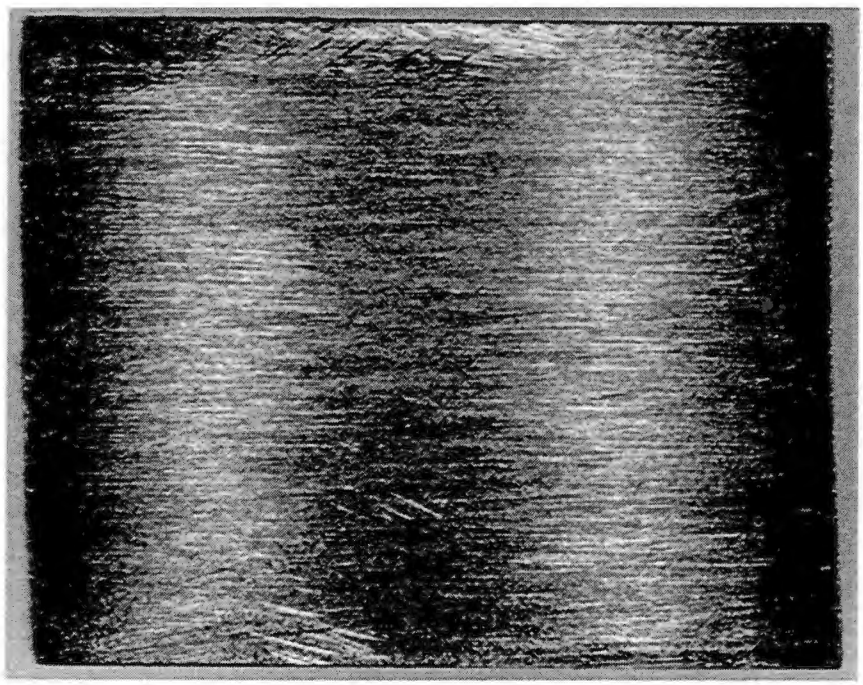


Figure 28. Surface Finish Sample 5 – #14 Bastard Mill File Finish

Magnification = 4.5x

Effect of Surface Finish on Ferrite Measurement – Conclusions

The goal of the surface finish study was to correlate ferrite measurement performed on a machined surface finish to the actual ferrite content of the component. The component ferrite content was simulated using a metallographically polished surface finish sample,. Based upon the experimental data obtained, the following conclusions are reached:

1. Impartment of a 250 microinch, 16microinch or #14 Bastard Mill file surface finish did not adversely affect ferrite measurement, as compared to a metallographic polish. The difference in ferrite measurement between the metallographically polished surface and the imparted surface finish was < 2 FN. The standard deviation associated with ferrite measurement is sufficiently small to assume that the data supporting these surface finishes, surrounds the mean (74 FN). The largest standard deviation encountered was 0.50. A ±3.0 FN variation in ferrite content is considered acceptable at this ferrite content, based upon guidelines established in AWS A4.2. Because impartment of the above surface finishes did not initiate a change greater than 3 FN, the effect of the above surface finishes, on ferrite determination, is not considered significant.

2. The imparting of a 64 microinch surface finish, on a sample with nominally 76 FN, adversely affected ferrite measurement. The 64 microinch surface finish resulted in a 10 FN reduction in measured ferrite content. This change in measured ferrite content is greater than the ±3 FN variation, which is expected.

A reduction in ferrite content can be attributed to either a decrease in ferrite content, or an interruption of the probe interaction volume. The 64 microinch finish, like all milled/ground finishes, provides a series of ridges on/in the surface of the sample. In the case of the 250 microinch or 16 microinch, the spacing and depth of the machined marks did not adversely affect the probe interaction volume, promoting adequate contact between the probe and sample surface. The spacing between 250 microinch machine marks is approximately 0.070". This value is larger than the 0.050" interaction volume established by previously discussed measurements. Conversely, the 16 microinch surface finish exhibits depth and width of machined marks that promote uniform measurement by optimizing the probe interaction volume.

The 64 microinch surface finish exhibits a distance between machined grooves approximately equal to 0.020". As this distance is smaller than the probe interaction volume, it is likely that the magnetic field induced by the Feritscope® probe is interrupted by the 64 microinch surface finish. This resulted in a marked reduction in measured ferrite content because the probe is not making sufficient contact with the sample to promote accurate measurement. The associated interruption in the interaction volume denotes the reduction in measured ferrite content.

3. Impartment of an angle grinder ground surface finish (24 grit) adversely affects ferrite measurement. The angle ground surface finish resulted in a 10 FN

reduction in measured ferrite content. This change in measured ferrite content is greater than the 3 FN variation expected.

Metallographic characterization of a section transverse to the angle ground surface revealed no microstructural. Surface finish topography remains the only interruption in the probe interaction volume affecting ferrite measurement. Additional characterization illustrated that modification of the angle ground surface finish, by removal of 0.005" of material, using a 120 grit abrasive, results in a ferrite determination equivalent to that of the polished surface. It is recommended that in the measurement of cast duplex stainless steel, a two step procedure, employing 120 grit grinding to remove the angle ground surface effects, prior to performing ferrite measurements, be utilized.

4. Producers and users of duplex stainless steel castings should be sensitive to the effects of surface finish on ferrite determinations using a Feritscope®. It is suggested that a #14 Bastard Mill File or angle ground, followed by a 120 grit surface finish, be utilized to provide the optimum surface finish for accurate ferrite measurement. This work also suggests that 250 and 16 microinch surface finishes may also be employed.

5. A limited amount of inspection, using the Magne Gage, on the surface finish samples revealed that the determination of ferrite content is generally unaffected.

Operator Error vs. Instrument Error

Prior to concluding this program, an endeavor was made to characterize the error associated with operation of the Feritscope® and of the instrument itself. Using a fixture and calibrated stage, ferrite content was measured on round-robin sample J using a semi-automated technique. 100 ferrite determinations were conducted and the mean ferrite content and 2σ standard deviation were recorded. Another series of 100 ferrite determinations were then performed manually, on the same sample, at the same location, to assess any change in the mean ferrite content and 2σ standard deviation due to operator error.

Utilizing the fixture and stage, the mean ferrite content was 76.9 FN with a 2σ standard deviation of 0.80 FN. In comparison, the mean ferrite content associated with manual Feritscope® use was 74.7 FN with a 2σ standard deviation of 2.56 FN. Based upon the 2σ standard deviations associated with each methodology, it is apparent that removing variances associated with an individual operator's ferrite measurement technique significantly reduced the magnitude of 2σ. The reduction in 2σ, resulting from a semi-automated technique, implies that there is a larger variance in ferrite measurement associated with an operator technique.

CHAPTER VI

CONCLUSIONS

Utilizing a series of round-robin tests, this program was able to characterize the capabilities of metallographic, magnetic and magnetic permeability methods of ferrite measurement. Depth profile studies further documented the change in ferrite content as a function of depth below a cast surface, providing casting producers and users with guidelines for machining and finishing. Finally, an analysis of surface finish and its effect on ferrite measurement served to further document the proper methods of characterizing the ferrite content of finished castings. Highlighting the important issues from this program, the following program conclusions are presented:

1. Round-robin testing demonstrated that cast secondary standards can be produced from duplex stainless steel castings. It is recommended that centrifugal castings be utilized for this purpose, as their repeatability, when subjected to three ferrite measurement techniques, was more favorable than statically cast materials. The reproducibility of measurements between participants was uniform, regardless of ferrite measurement technique.

A standard procedure for manufacturing cast secondary standards can be described as follows:

(a) Select an alloy (austenitic/duplex) whose ferrite content matches a desired ferrite range.

(b) Produce a centrifugal cast ring. Static castings should not be used.

(c) Remove a 1" x ¾" x ¾" cube from the ring such that the primary ferrite measurement surface is oriented perpendicular to the radial direction of the ring and at least 1/8" below the cast surface, as shown in Figure 29.

(d) Metallographically polish the measurement face to a 0.05μ surface finish. Etch appropriately using an oxalic acid electro-etch technique (10V, 0.05A for 20-60 seconds). Photographically document a region of interest and perform a manual point count (ASTM E562) to assess the ferrite content. This region of interest will later be utilized as the measurement location during calibration.

(e) Permanently scribe the border of the region of interest on the polished surface.

(f) Measure ferrite in the region of interest using a Magne Gage or Feritscope®, which has been calibrated to AWS A4.2. Perform 10 determinations within the region of interest and calculate the mean ferrite content and standard deviation.

(g) Mark the mean ferrite content and standard deviation permanently on the block.

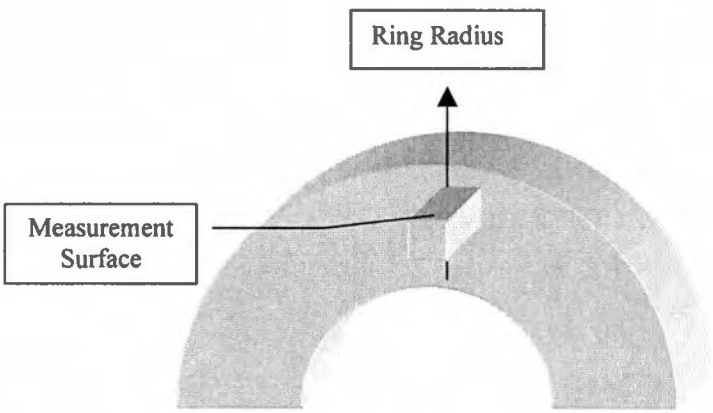


Figure 29. Cast Secondary Standard Extraction

(Ensure that the measurement face is 1/8" below the cast surface)

(h) Perform several additional determinations within the scribed region and compare the data to the mean ferrite content of the block. If 10 successive individual determinations are within 5% of the mean ferrite content, the block is suitable for calibration. Larger values shall be cause for rejection of the block as a calibration standard.

Note: This procedure has not been sanctioned by any standardization organization and is subject to review.

2. The round-robin further demonstrated that instrument calibration, utilizing AWS A4.2, produced improved repeatability, as compared to other calibration methods. It is recommended that AWS A4.2 be utilized for the calibration and operation of Magne Gage and Feritscope® instruments to maintain repeatability. Interlaboratory reproducibility was unaffected by differing calibration methods.

3. Additionally, it was found that a suitable correlation could be drawn between ferrite content and ferrite number. This correlation was established as 0.9:1 (FN:Volume Percent Ferrite) for the low ferrite range (0-15 FN). A second correlation factor was established as 1.5:1 (FN:Volume Percent Ferrite) for the upper ferrite range (55-90 FN). These correlations were comprised of ferrite measurements using metallographic, Magne Gage and Feritscope® techniques.

4. Depth profile studies revealed that removal of 1/8" of material from the cast surface will establish a uniform ferrite content for the finished casting. Trials using two alloy systems and three heats of material confirm this behavior.

5. Ferrite measurements, utilizing a Feritscope®, taken directly on a cast surface are not indicative of the ferrite content of the entire casting. Producers and users of cast products are encouraged to measure ferrite content at a subsurface location, at a depth greater than 1/8" below the cast surface.

6. The interaction volume of the Feritscope® probe is defined as 0.050", which is the diameter of the probe. Ferrite measurement performed, such that the measurement probe is not contained within a discontinuity or edge, will promote accurate ferrite measurement.

7. Surface finish analysis revealed that the impartment of a 250 microinch, 16 microinch or #14 Bastard Mill file surface finish did not adversely affect ferrite measurement. The difference in ferrite measurement between the metallographically polished surface and the imparted surface finish was acceptable.

8. Further surface finish analysis concluded that impartment of a 64 microinch or angle ground surface finish did adversely affect ferrite measurement. Impartment of a 64 microinch or angle ground surface finish (24 grit) resulted in a 10 FN

reduction in measured ferrite content. Ferrite measurement should not be employed directly on either surface finish. However, the effects of angle grinding can be removed by grinding the casting with a 120 grit wheel (or equivalent). A minimum of 0.005" of removal is recommended to free the measurement surface of any pre-existing angle grinding marks.

9. A standard practice for measuring ferrite using either a Magne Gage or Feritscope® is as follows:

   (a) Calibrate the Magne Gage or Feritscope® according to AWS A4.2.

   (b) Examine the surface finish of the sample to ensure that it is free of curvature. Samples exhibiting significant curvature require the use of a conversion factor which accounts for surface curvature effects on ferrite measurement accuracy.

   (c) Examine the surface finish of the sample. This study has shown that a suitable surface finish is required for accurate ferrite determinations. Beyond the recommendations previously discussed, surface finish effects will be left to the discretion of the operator.

   (d) Following the measurement procedure outlined by the manufacturer, perform ferrite measurement determinations using either the Magne Gage or Feritscope®. Ensure that the instrument probe is not contained within a surface discontinuity or sufficiently near an edge to promote inaccurate measurement. Edge/distance effects have been summarized in conclusion (6).

10. It was found that the greatest source of error, when comparing Feritscope® operator technique and instrument variations, was associated with the operator. 2σ analysis revealed that the largest variation in ferrite content, for a given sample, is associated with the operator's technique and not the instrument.

For additional information relating to this program, feel free to contact the University of Tennessee – Knoxville, Materials Joining Research Group.

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3. ASTM A799, "Standard Practice for Steel Castings, Stainless, Instrument Calibration, for Estimating Ferrite Content", ASTM International, West Conshohocken, Pennsylvania, USA, 1992

4. ASTM A800, "Standard Practice for Steel Casting, Austenitic Alloy, Estimating Ferrite Content Thereof", ASTM International, West Conshohocken, Pennsylvania, USA, 1991

5. ASTM A890, "Standard Specification for Castings, Iron-Chromium-Nickel-Molybdenum Corrosion-Resistant, Duplex (Austenitic/Ferritic) for General Application", ASTM International, West Conshohocken, Pennsylvania, USA, 1991

6. ASTM E562, "Practice for Determining Volume Fraction by Systematic Manual Point Count", ASTM International, West Conshohocken, Pennsylvania, USA, 1997

7. ASTM E1301, "Standard Guide for Proficiency Testing by Interlaboratory Comparisons", ASTM International, West Conshohocken, Pennsylvania, USA, 1995

8. ISO 8249-85, "Welding – Determination of Ferrite Number in austenitic weld metal deposited by covered Cr-Ni steel electrodes."

APPENDIX

Ferrite Measurement in Stainless Steel Castings

"A Round Robin Study"

Initiated by

Dr. Carl D. Lundin
William J. Ruprecht

Materials Joining Research Group
Department of Materials Science and Engineering
The University of Tennessee – Knoxville

in conjunction with

The Welding Research Council

and

The Steel Founders' Society of America

1.0 Introduction:

The UT Materials Joining Research Group is initiating a Ferrite Measurement Round Robin study to examine the following issues:

- The reproducibility of ferrite measurement data, between laboratories, using Magne Gage and Feritscope® techniques
- The applicability of manufacturing cast secondary standards from static and centrifugal castings
- A more defined correlation between ferrite measurement techniques will be established. These techniques include manual point counting and measurement by Magne Gage and Feritscope®.

2.0 Round Robin Timeline:

In an effort to minimize the work effort, the timeline described in Table 1 has been established. The primary goal is to send the round robin samples between the Welding Research Council (WRC) committee members prior to the WRC High Alloys Committee meeting in May. The sample set will then proceed to Steel Founders' Society of America (SFSA) participants before returning to UT.

Table 1: UT Ferrite Measurement Round Robin Schedule

Program Launch Date:	February 24, 1999
Samples Arrive / D. Kotecki:	March 1, 1999
D. Kotecki Evaluation Period:	March 1 - 10, 1999
Samples Shipped to Participant 2:	March 11, 1999
Samples Arrive / F. Lake:	March 15, 1999
F. Lake Evaluation Period:	March 15 - 24, 1999
Samples Shipped to Participant 3:	March 25, 1999
Samples Arrive / S. Jana:	March 29, 1999
S. Jana Evaluation Period:	March 29, 1999 through April 7, 1999
Samples Shipped to Participant 4:	April 8, 1999
Samples Arrive / T. Siewert:	April 12, 1999
T. Siewert Evaluation Period:	April 12 - 21, 1999
Samples Shipped to Participant 5:	April 22, 1999
Samples Arrive / J. Feldstein:	April 26, 1999
J. Feldstein Evaluation Period:	April 26, 1999 through May 5, 1999
Samples Shipped to Participant 6:	May 6, 1999
WRC High Alloys Meeting:	**May 10 – 12, 1999**
Samples Arrive / R. Bird:	May 10, 1999

Table 1 (Continued): UT Ferrite Measurement Round Robin Schedule

R. Bird Evaluation Period:	May 10 - 19, 1999
Samples Shipped to Participant 7:	May 20, 1999
Samples Arrive / C. Richards:	May 24, 1999
C. Richards Evaluation Period:	May 24, 1999 through June 2, 1999
Samples Shipped to UT:	June 3, 1999
Publication of Results:	June 30, 1999

Note: This timetable establishes 9 business days for experimental evaluation and 1 business day is provided to ship the samples to the next participant. Shipping will be provided. We anticipate that the WRC members will likely require less analysis time, as they are adequately equipped to measure ferrite on a routine basis. Should the Round Robin progress ahead of (or behind) schedule, each participant will be appropriately notified.

3.0 Requests of the Participants:

The Materials Joining Group is grateful for your participation in this study. We value your time and seek to minimize your work effort. However, the following requests are made to project your success.

3.1 Adherence to the Timetable:

Should a participant, for any reason, be unable to adhere to the timetable outlined in Table 1, please notify the Materials Joining Research Group. UT contacts are listed as follows:

Dr. Carl D. Lundin
Director, Materials Joining Research
Phone: (423) 974-5310
FAX: (423) 974-0880
E-Mail: lundin@utk.edu

William J. Ruprecht III
Graduate Research Assistant
Phone: (423) 974-5299
FAX: (423) 974-0880
E-Mail: ruprecht@utk.edu

In the event of such an occurrence, a quick scheduling response will facilitate the implementation of this round robin.

3.2 Questions regarding the Work Request:

If at any point in this investigation, there is a question with regard to experimental techniques, calibration procedures, reporting of data or scheduling, feel free to contact our office.

3.3 Suggestions from the Participants:

If you have any suggestions to improve the implementation of further studies, please submit them with your data package.

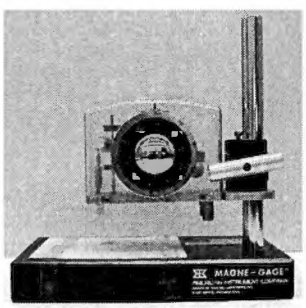
Immediate suggestions which would require a modification to your individual test procedure should be forwarded immediately.

Comments, are always appreciated.

4.0 Work Request:

5.1 Ferrite Measurement:

Participants are asked to measure ferrite (FN) on the sample set provided. Acceptable methods of ferrite measurement include, but are not limited to, the following:



Magne Gage

Feritscope® MP3 (MP3-C)

Using the attached checklist and the provided forms, participants will be asked to calibrate (or report their current calibration) according to AWS A4.2 prior to taking measurements.

5.1 Reporting of Data:

Using the attached forms, participants are asked to record their ferrite measurements and return them to the Materials Joining Group. A mailing envelope is included for the return of the entire package.

A Federal Express mailer has been included so that you may forward the cast standards to the next participant. Please use a Federal Express Box and utilize suitable packing material to prevent damage during shipping.

5.0 Cast Sample Set:

5.1 Contents:

The sample set provided contains 12 rectangular blocks which have been fabricated from austenitic and duplex stainless steels. They are labeled on the ends with a sample code. The following table correlates the sample code with the alloy type.

Sample Code	Alloy Type
A	CF8
B	CF3MN
C	CF8M
D	ASTM A890-4A
E	ASTM A890-4A
F	ASTM A890-4A*
G	ASTM A890-5A
H	ASTM A890-5A*
I	ASTM A890-5A
J	CD7MCuN*
K	CD7MCuN
L	CD7MCuN

* Indicates that the material was centrifugally cast, as opposed to a static casting.

5.2 Condition of Samples:

Each sample has been prepared, on the measurement face, with a surface finish equal to a metallographic polish. This was done so that a microstructural evaluation could be performed prior to initiating the round-robin. Note the presence of a scribed circle on the measurement face. No ferrite measurements are to be taken outside of this circle. This is done so that we may directly compare ferrite measurements with metallographic point counting techniques.

6.0 Ferrite Measurement Instruction Set:

6.1 Magne Gage:

Appendix 1 contains an operator checklist and instruction set for performing ferrite measurements with a Magne Gage.

6.2 Feritscope®:

Appendix 2 contains an operator checklist and instruction set for performing ferrite measurements with a Feritscope®

6.3 Other:

Should a participant wish to utilize other methods of ferrite measurement, please contact the Materials Joining Group as indicated in Item 3.1 of this manual.

7.0 Completion of your Work Effort:

7.1 Forwarding the Sample Set to the Next Participant:

A Federal Express invoice has been provided (pre-addressed / pre-paid). Please use a standard Federal Express Box to ship the sample set to the next participant.

7.2 Returning your Data to the University of Tennessee:

A return envelope (pre-addressed) has been provided. Please seal this manual, containing your data, charts, graphs and comments in the envelope and forward it to the University of Tennessee (c/o The Materials Joining Research Group).

8.0 Acknowledgements:

We would like to acknowledge the following individuals for their guidance and support in performing this round robin study.

Dr. Damian Kotecki – Lincoln Electric
Mr. Frank Lake – ESAB
Mr. Sushil Jana – Hobart Brothers Co.
Mr. Tom Siewert – N.I.S.T.
Mr. Ron Bird – Stainless Foundry
Mr. Joel Feldstein – Foster Wheeler
Mr. Christopher Richards – Fristam Pumps Inc.

Appendix 1: Ferrite Measurement Using a Magne Gage

Please follow the checklist (below) to assure proper measurement and documentation of ferrite content for each sample. You may check the boxes, located before each item number, as you proceed through this study.

☐ 1. Review AWS A4.2-91, Section 4, pp. 4-6, to familiarize yourself with the proper methods of calibrating a Magne Gage instrument. A copy of AWS A4.2-91 has been included for your convenience and is located at the end of this manual.

☐ 2. Calibrate your Magne Gage according to the specifications outlined in AWS A4.2-91 (Section 4).

Please include all graphs and tables used to calibrate your Magne Gage and report whether you are calibrating to Primary Thickness Standards or Secondary Weld Metal Standards. Calibration to Primary Thickness Standards is preferred. Examples of suitable calibration curves are located in the AWS specification on Page 6 and are illustrated by Figure 1.

☐ 3. The data recording sheet is presented on Page 3 of this appendix. Please provide the Instrument Type / Serial Number, Operator Name and Date, as indicated.

☐ 4. Utilize the samples submitted and reference the characteristics of each block, as described in Item 5 of this manual. Perform 5 "sets" of determinations as described below. Each "set" must contain 6 separate determinations. Only the highest FN measured will be reported for each "set" of determinations.

Lower the plastic "magnet guard" until it is in contact with the sample and is wholly contained within the scribed circle. Perform 6 successive determinations without moving the plastic "magnet guard". This will constitute a single "set" of determinations. Ferrite determinations taken outside the scribed circle must be considered invalid.

Record only the highest FN, achieved from each of the 6 determinations, in the space provided. After each "set" of 6 determinations, raise the plastic "magnet guard" and lower it again, within the scribed circle, prior to performing the next "set" of determinations. The highest determined FN should be recorded for each individual "set" of determinations.

Review the data for each sample. For each sample, your data sheet should reflect five FN determinations, which are the highest FN's observed in each of the measurement "sets". (Each "set" should be composed of 6 individual measurements, obtained at one location within the scribed circle, with the plastic "magnet guard" in contact with the sample.)

☐ 5. Upon completion of the successive ferrite determinations, return the samples to their plastic cases and proceed to Appendix 2, *Ferrite Measurement using a Feritscope®.*

Data Sheet 1: Ferrite Measurement Using a Magne Gage

Part 1: Background Information:

Instrument Type / Serial Number: _____
Operator Name: _____
Date: _____

Part 2: Recording of Data:

Record your ferrite measurements in the following table.

Sample Code	Determination Set 1 (Highest FN)	Determination Set 2 (Highest FN)	Determination Set 3 (Highest FN)	Determination Set 4 (Highest FN)	Determination Set 5 (Highest FN)
A					
B					
C					
D					
E					
F					
G					
H					
I					
J					
K					
L					

Appendix 2: Ferrite Measurement Using a Feritscope®

Please follow the checklist (below) to assure proper measurement and documentation of ferrite content for each sample. You may check the boxes, located before each item number, as you proceed through this study.

☐ 1. Review AWS A4.2-91, Section 5, p.7, to familiarize yourself with the proper methods of calibrating a Feritscope® instrument. A copy of AWS A4.2-91 has been included for your convenience and is located at the end of this manual.

☐ 2. Calibrate your Feritscope® according to the specifications outlined in AWS A4.2-91 (Section 5). Calibration to secondary cast standards will be the accepted method for this study. Standardized forms have been provided to assist you in recording your calibration and are located on the following pages.

Table 1 is a sample Feritscope® calibration form, provided courtesy of IIS/IIW-1405-98. A blank calibration form is provided, in the form of Table 2 of this appendix. Highlight the measurements which exceed accepted tolerances, as demonstrated (Blue Underlined) in Table 1, on your calibration sheet (Table 2).

If you wish to provide data for multiple Feritscopes® and/or operators, additional copies of calibration forms may be made from this packet.

☐ 3. Locate the data recording sheet (Data Sheet 2) on Pages 4-5 of this appendix. Please provide the Instrument Type / Serial Number, Operator Name and Date, as indicated. If you wish to record data for multiple operators and/or Feritscopes®, additional copies of the data recording sheet should be made, as needed. Please differentiate between Feritscope® model numbers and operators in the "background information".

☐ 4. Utilize the Sample Set and reference the characteristics of each block, as described in Item 5.0 of this manual.

By lowering the probe perpendicular to the sample, perform 10 successive measurements within the scribed circle. Ferrite measurements taken outside the scribed circle must be considered invalid.

Record each measurement on the attached data sheets and report the average FN value observed for each sample.

☐ 5. Upon completion of the ferrite measurements, return the samples to their plastic cases and review your paperwork to ensure that all data has been included. This concludes ferrite measurement by the Feritscope® technique.

Table 1: Sample Calibration Form (Reference IIS/IIW-1405-98)

Calibration Standard	Appl 4	Appl 3	Appl 1	Appl 2	Appl 1	Appl 2	Appl 3	Appl 4	Appl 2	Appl 4
Air	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
1st Certified FN	4.6	31.0	6.5	4.6	4.6	26.8	52.0	67.0	16.7	58.5
2nd Certified FN	16.7	52.0	31.0	10.4	10.4	37.5	58.5	73.5	26.8	73.5
3rd Certified FN	31.0	85.0	85.0	16.7	14.6	47.0	67.0	85.0	37.5	85.0
Certified FN (and Range) per A4.2, Table 4	Average of 10 Check Readings on Standard Using Above Calibration									
1.7 (1.4 - 2.0)	1.4	1.7	1.5	1.4	1.4	1.8	1.8	1.8	1.6	1.8
4.6 (4.3 - 4.9)	4.4	5.5	4.6	4.3	4.3	5.6	5.7	5.6	5.3	5.9
6.5 (6.2 - 6.8)	6.7	7.6	6.4	6.1	6.2	8.0	8.1	8.0	7.5	8.4
10.4 (10.0 - 10.8)	12.1	12.7	12.3	10.6	10.6	13.4	13.6	13.6	12.2	14.3
14.6 (14.2 - 15.0)	14.5	15.2	14.5	13.5	14.5	16.1	16.2	16.3	14.7	17.0
16.7 (16.2 - 17.2)	16.6	17.3	16.8	16.4	16.7	18.4	18.5	18.8	16.7	19.6
20.3 (19.8 - 20.8)	20.3	20.5	20.5	19.6	19.8	21.8	22.1	22.4	20.7	23.5
26.8 (25.5 - 28.1)	25.8	25.3	25.7	24.2	24.3	27.0	27.7	27.7	26.8	29.7
31.0 (29.4 - 32.6)	31.3	29.8	30.6	28.0	28.4	32.0	32.2	32.7	31.3	34.5
37.5 (35.6 - 39.4)	37.9	37.5	37.8	33.2	33.9	37.8	39.4	39.9	37.7	42.5
47.0 (44.6 - 49.4)	46.8	49.1	45.7	41.0	41.2	47.2	49.1	49.4	47.5	54.0
52.0 (47.8 - 56.2)	48.5	51.6	49.0	43.0	43.6	49.1	53.1	53.0	50.1	58.0
58.5 (53.8 - 63.2)	48.6	52.2	47.8	42.2	43.7	49.1	55.1	52.3	48.8	56.8
67.0 (61.6 - 72.4)	61.6	63.9	60.1	53.6	54.2	64.1	67.9	68.6	63.7	68.7
73.5 (67.6 - 79.4)	67.3	69.2	66.5	58.0	58.0	70.2	74.1	73.2	70.1	72.7
85.0 (78.2 - 91.8)	86.9	85.3	85.7	71.9	71.8	89.4	98.8	86.7	90.7	87.7
Decision	dis-card	dis-card	dis-card	dis-card	use for 0-20 FN	dis-card	use for 30-70 FN	dis-card	use for 15-45 FN	use for 60-90 FN

Table 2: Participant Calibration Form

Calibration Standard	Appl	Appl	Appl	Appl	Appl	Appl	Appl	Appl	Appl	Appl
Air										
1st Certified FN										
2nd Certified FN										
3rd Certified FN										
Certified FN (and Range) per A4.2, Table 4	Average of 10 Check Readings on Standard Using Above Calibration									
Decision										

Data Sheet 2: Ferrite Measurement Using a Feritscope®

Part 1: Background Information:

Instrument Type / Serial Number: _____
Operator Name: _____
Date: _____

Part 2: Recording of Data:

Record your ferrite measurements in the following table.

Sample Code	FN 1	FN 2	FN 3	FN 4	FN 5	FN 6	FN 7	FN 8	FN 9	FN 10	Average FN
A											
B											
C											
D											
E											
F											

Data Sheet 2: Ferrite Measurement Using a Feritscope® - Continued

Sample Code	FN 1	FN 2	FN 3	FN 4	FN 5	FN 6	FN 7	FN 8	FN 9	FN 10	Average FN
G											
H											
I											
J											
K											
L											

Notes:

AWS A4.2-91
(Insert Copy)

VITA

William J. Ruprecht III was born in Connecticut on June 24, 1974. He attended grade school in southern Pennsylvania before moving to Erie, Pennsylvania in 1982. A graduate of McDowell High School, Bill entered the University of Missouri-Rolla in the fall of 1992, majoring in Metallurgical engineering. Prior to achieving his Bachelor of Engineering degree in the spring of 1997, he spent summers pursuing his professional career at GE Transportation Systems, located in Erie, Pennsylvania.

He entered the University of Tennessee-Knoxville graduate program in 1997, under the direction of Dr. Carl D. Lundin. Bill spent two years pursing his engineering Masters in Science degree at UTK prior to his graduation in December of 1999. While working in the Materials Joining Research Group, he was exposed to a number of academic and professional challenges which have significantly molded his career.

He presently works for GE Transportation Systems (Erie, Pa), a manufacturer of commercial locomotives and off-highway vehicle systems.