FINAL REPORT VOLUME 3

GUIDANCE DOCUMENT FOR THE EVALUATION OF CAST SUPER DUPLEX STAINLESS STEEL

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This is Volume 3 of 5 of the final report for The Department of Energy Grant # DE-FC07-00 ID13975 entitled "Behavior of Duplex Stainless Steel Castings."

FOREWARD

The final report for the DOE Grant DE-FC07-00 IDI13975 consists of five volumes. The volumes provide in depth information on Cast Duplex and Cast Super Duplex Stainless Steels. Volume 1 is entitled "Metallurgical Evaluation of Cast Duplex Stainless Steels and their Weldments" involves comparison of selected grades of Duplex Stainless Steels and their welds with their wrought counterparts regarding corrosion performance, mechanical properties and weldability. Volume 2 entitled "The Development of Qualification Standards for Cast Duplex Stainless Steel" involves interlaboratory testing and Volume 3 "The Development of Qualification Standards for Cast Super Duplex Stainless Steels to ASTM A923. Volume 4 is the "Guidance Document for the Evaluation of Super Duplex Stainless Steel" and involves the applicability of ASTM A923 to the Cast Super Duplex materials. Volume 5 is the data package for the incorporation of ASTM A890-5A material into the ASTM A923.

In volume 1 selected grades of Duplex Stainless Steel castings and their welds, in comparison with their wrought counterparts, were evaluated, regarding corrosion performance, mechanical properties and weldability. Multiple heats of cast duplex stainless steel were evaluated in the as-cast, solution annealed static cast and solution annealed centrifugal cast conditions, while their wrought counterparts were characterized in the solution annealed condition and in the form of as-rolled plate. Welding, including extensive assessment of autogenous welds and a preliminary study of composite welds, Shielded Metal Arc Weld (SMAW), was performed. The evaluations included Critical Pitting Temperature (CPT) testing, Intergranular Corrosion (IGC) testing, ASTM A923 (Methods A, B and C), Charpy impact testing, weldability testing (ASTM A494), ferrite measurement and microstructural evaluations.

Volume 2 deals with the Development of Qualification Standards for Cast Duplex Stainless Steel (A890-4A) which is equivalent to wrought 2205. This volume involves testing of cast Duplex Stainless Steel to several ASTM specifications, formulating and conducting industry round robin tests and studying the reproducibility of the results. ASTM E562 (Standard Test Method for Determining Volume Fraction by Systematic manual Point Count) and ASTM A923 (Standard Test Methods for Detecting Detrimental Intermetallic Phase in Wrought Duplex Austenitic/Ferritic Stainless Steels) were the specifications utilized in conducting this work. An ASTM E562 industry round robin, ASTM A923 applicability study, ASTM A923 industry round robin, and an ASTM A923 study of the effectiveness of existing foundry solution annealing procedures for producing cast Duplex Stainless Steel without intermetallic phases were implemented.

Volume 3 is comprised of the Development of Qualification Standards for Cast Super Duplex Stainless Steel (A890-5A) which is equivalent to wrought 2507. The objective of this work was to determine the suitability of ASTM A923 "Standard Test methods for Detecting Detrimental Intermetallic Phase in Duplex Austenitic-Ferritic Stainless Steels" for 25 Cr Cast Super Duplex Stainless Steels (ASTM A890-5A). The various tests which were carried out were ASTM A923 Test Method A, B and C (Sodium Hydroxide Etch Test, Charpy Impact Test and Ferric Chloride Corrosion Test), ferrite measurement using Feritscope®, ASTM E562 Manual Point Count Method and X-Ray Diffraction, hardness measurement using Rockwell B and C and microstructural analysis using SEM and EDS.

Volume 4 is the guidance document for the evaluation of cast Super Duplex Stainless Steel which deals with the various evaluation methods which were defined and used for the work on volume 3 for the "Development of Qualification Standards for Cast Super Duplex Stainless Steel alloy A890-5A (2507 Wrought Equivalent)". The document explains in detail each test which was conducted. It also includes some of the results which were acquired during this work.

Volume 5 is the Data Package for the evaluation of Super Duplex Stainless Steel Castings prepared at the end of work comprised in volumes 3 and 4. The document deals with the various evaluation methods used in the work documented in volume 3 and 4. This document covers materials regarding evaluation of the A890-5A material in terms of inclusion in ASTM A923. The various tests which were conducted on the A890-5A material are included in this document.

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INTRODUCTION

Super Duplex Stainless Steel is an alloy with at least 25% chromium which contains two phases – Ferrite and Austenite (Figure 1). These phases are present in approximately equal proportion of 50% each. A standard Super Duplex Stainless Steel is wrought 2507, the cast version of which is A890-5A. Applications of wrought and Cast Super Duplex Stainless Steel have been on the increase. Currently, there are no nationally recognized standards available for performance related evaluation of the cast Super Duplex Stainless Steels¹.

Specifications related to the corrosion resistance and fabrication of Super Duplex Stainless Steel is becoming increasingly important from the industry prospective. It is important to identify the presence of detrimental intermetallic phases in the cast Super Duplex Stainless Steel in order to evaluate its performance. These phases have a major impact on the various physical and mechanical properties of the material². The program of "Development of Qualification Standards for Cast Super Duplex Stainless Steel alloy A890-5A (2507 Wrought Equivalent)" aimed to examine the suitability of the ASTM A923 specification for this material. The evaluation methods that were defined and used in this program are the following:

a) The ASTM A923-03 Specification has three test methods (Methods A, B and C), all of which are applicable to cast A890-4A and wrought 2205 Duplex Stainless Steels and wrought 2507 Super Duplex Stainless Steels. Test Method A is the "Sodium Hydroxide Etch Test for Classification of Etch Structures of Duplex Stainless Steels," Test Method B is the "Charpy Impact Test for Classification of Structures of Duplex Stainless Steels," and Test Method C is the "Ferric Chloride Corrosion Test for Classification of Structures of Duplex Stainless Steels." Test results indicated that Cast Super Duplex Stainless Steel can be added into the specification. It showed that the A890-4A Grade (Cast Duplex Stainless Steel) micrographs can be applied to A890-5A Grade (Cast Super Duplex Stainless Steel) in order to compare and identify the presence of intermetallic phases. It was

concluded that the Chary Impact Test could be carried out at -50°F and yield toughness upon which to compare equivalent heats. Test Method C was found suitable for the cast Super Duplex material without any modification.

- b) Charpy Impact Test was conducted on all the heats per ASTM A370, "Standard Methods and Definitions for Mechanical Testing of Steel Products," and ASTM E23, "Notched Bar Impact Testing of Metallic Materials." The test was carried out at -50°F which may be representative of the industry typical test temperature.
- c) Ferrite determination using the Fisher Model MP-3C Feritscope® was applied on all the heats. Feritscope® is considered to be most suitable for calculating the volume percentage of ferrite because it is easy to operate, eliminates operator bias and there are minimal requirements for specimen preparation.
- d) Volume percentage of ferrite was also calculated using "ASTM E562-02: Standard Test Method for Determining Volume Fraction by Systematic Manual Point Count." This method was employed in order to compare the results with the Feritscope[®] and to determine which method is more suitable.
- e) Hardness Measurement was carried out using Rockwell B and Rockwell C.
 "ASTM E18-03: Standard Test Method for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials" was used.
- f) Microstructural Characterization was carried out using OLM, SEM and EDS.

Based on the above test methods, this guidance document has been developed for the evaluation of Cast Super Duplex Stainless Steel.

TEST METHODS AND PROCEDURES

1) <u>Recommended Procedures for ASTM A923 Test Methods A, B and C</u>

1.1) <u>Test Method A: Sodium Hydroxide Etch Test for Classification of Etch</u> <u>Structures of Duplex Stainless Steels</u>

1. Test Apparatus

The apparatus includes epoxy resin, epoxy hardener, grinding papers, polishing clothes, a suitable power supply and a metallurgical microscope.

2. Procedure

- 2a) Extract the specimen from the castings at location representing typical microstructure of the casting. These specimens are mounted using epoxy resin and epoxy hardener.
- 2b) Grind and polish the specimens to a metallographic surface finish $(0.05\mu m)$.
- 2c) Add 40g of reagent grade sodium hydroxide (NaOH) to 100ml of distilled water to prepare the etching solution. Eye protection is necessary for this operation.
- 2d) A polished specimen should be etched at approximately 1 to 3V dc, for 5 to 60s. Note: It is to be noted that the proper etching period for the optimum results varied depending upon SDSS alloy system. When etching is performed within the period of 5 to 60s, the intermetallic phases are revealed by yellow, then brown staining followed by staining of the ferrite. Etching time also plays an important role. Insufficient etching will not reveal the microstructure and over etching will result in a loss of contrast, which will cause difficulties in distinguishing the phases.
- 2e) Following etching, the specimen should be rinsed thoroughly in hot water and in acetone or alcohol, followed by air drying.
- 2f) Observation Examine the etched surface under a metallurgical microscope at 400x or 500x magnification.

- 2g) Classification of Etched Structures The etched microstructures are classified into the following types:
 - a) Unaffected Structure (Figure 2) The sample has been etched and the microstructure is without the revelation of any intermetallic phase. The austenite-ferrite boundaries are smooth.
 - b) Possibly Affected Structure (Figure 3) The sample has been etched and isolated indications of possible intermetallic phase are noted. The austenite ferrite boundaries show a fine waviness.
 - c) Affected Structure (Figure 4) The indication of an intermetallic phase is readily revealed upon etching.
 - d) Centerline Structure (Figure 5) The intermetallic phase is observed as a continuous or semi-continuous phase in the mid-thickness region of the sample, with or without the affected structure outside of the mid-thickness region, indicative of segregation. (This structure is only applicable to wrought materials).
- 2h) Figures 6 and 7 shows the microstructures of unaffected and affected structures of Cast Super Duplex Stainless Steel⁵.

1.2) <u>Test Method B: Charpy Impact Test for Classification of Structure of Duplex</u> <u>Stainless Steels</u>

1. Charpy Sample Extraction

Charpy samples are extracted from the materials submitted for test. The samples should be extracted remote from the edges (atleast 3 mm away for the edges).

2. Charpy Test Procedure

- 2a) The Charpy Impact test should be conducted per ASTM A370, "Standard Methods and Definitions for Mechanical Testing of Steel Products," and ASTM E23, "Notched Bar Impact Testing of Metallic Materials." The test may be conducted at a temperature of -50°F because this temperature is representative of the industry typical test temperature.
- 2a) Report the test temperature and the energy absorbed.

3. ASTM A923 Test Method B Acceptance Criteria

- 3a) No acceptance criteria is stated in the ASTM A923 for Cast Super Duplex Stainless Steel. However, from the Charpy Impact testing it was found that all solution annealed materials which showed an unaffected structure had a toughness greater than 60ft-lbs at -50°F. Hence, this can be taken as potentially an acceptance criterion.
- 3b) If a test specimen shows an impact value below the specified acceptance criteria, one retest of two specimens is permitted. For acceptance, both retested specimens shall show a value at or above the acceptance criteria.

1.3) <u>Test Method C: Ferric Chloride Corrosion Test for Classification of</u> <u>Structure of Duplex Stainless Steels</u>

1. Test Apparatus

The apparatus includes glass beaker of at least 1000ml, glass cradle to hold the sample, weighing dish, balance and water bath.

2. Procedure

- 2a) The specimens can be of various sizes and shapes.
- 2b) All the surfaces of the specimen should be ground to a uniform surface finish. Sharp edges and corners should be rounded.
- 2c) The dimensions of the test specimens and the total exposed surface area should be calculated
- 2d) The specimen should be weighed to the nearest 0.001g or better.
- 2e) The test solution is prepared by dissolving 100g of ferric chloride, FeCl₃.6H₂O, in 900ml of distilled water (approximately 6% FeCl₃ by weight). The solution is filtered into the glass beaker using a filter paper or a glass wool to remove insoluble particles.

Note: The glass beakers should be clean. This can be done by boiling a solution of 10% hydrochloric acid in the beaker.

- 2f) The pH of the test solution should be adjusted to approximately 1.3 prior to the beginning of the test by the addition of HCl or NaOH, as required.
- 2g) Place the specimen in the glass cradle and suspend the cradle from the stop cock into the test solution.
- 2h) These beakers are kept in a water bath (Figure 8) at a temperature of 40°C (107°F) for 24 hours.
- 2i) At the end of 24 hours the samples should be removed and rinsed with water, scrubbed with a soft bristle to remove corrosion products, dipped in acetone or alcohol and dried in air.

2a) The specimens should be again weighed to 0.001g or better.

3. ASTM A923 Test Method C Acceptance Criteria

3a) The corrosion rate should be calculated in accordance with the weight loss and total surface area using the equation

Corrosion rate = Weight loss/[(Specimen Area) x (Time)]

- 3b) The calculated corrosion rate should not exceed 10milligrams/decimeter/day (mdd).
- 3c) If the specimen shows a corrosion rate in excess of 10mdd, one retest on two new specimens from the same product is permitted. No retest specimen should exhibit a corrosion rate in excess of 10mdd.

2) <u>Ferrite Measurement using Feritscope®</u>

Ferrite measurement using Fisher Feritscope® (Model MP – 3C or later model), as shown in figure 9, is recommended for the ferrite measurement in Cast Super Duplex Stainless Steel. The Feritscope® is an easy-to-use practical field instrument. The Feritscope® measurements are carried by bringing the probe into contact with the surface of the specimen and holding it in place until an audible sound is heard. This instrument can be calibrated to measure the Ferrite Number (FN). Individual readings using the Ferritscope® require no more than three seconds to accomplish and the operator can take readings in rapid succession.

1) Specimen Preparation

- 1a) Samples removed from castings should be extracted at a sufficient depth below the as-cast surface to ensure uniform ferrite content. For a typical solution annealed and water quenched castings, a depth of 0.125" is recommended for accurate ferrite measurement. The appropriate depth can be obtained as a result of any finishing process, which results in sufficient material removal and adequate surface finish. Careful attention should be taken to ensure that the finishing process does not induce microstructural transformations at the surface of the substrate, which could influence the ferrite content (i.e. rough milling)³.
- 1b) Ferrite measurement should be employed on a substrate of a minimum 120-grit surface finish. Measurements taken on the as-cast, grit blasted or machined surface finishes may yield a ferrite content which is not indicative of the casting. The substrate surface should be sufficiently flat to promote accurate measurement.

2) Measurement Procedures

Prior to performing ferrite measurements, the appropriate surface finish should be imparted to the material, as described in the previous section. The instrument should be calibrated as indicated below and its calibration should be checked prior to measurement. A sufficient number of measurements should be employed to ensure statistical characterization of the material. In all cases, the operational instructions, provided by the manufacturer, should be employed to ensure accurate ferrite measurement.

Ferrite measurement procedure using a Feritscope®:

- 2a) Calibrate the Feritscope® according to the procedure outlined in the specification AWS A4.2-Section 5 whenever possible to provide traceability or a procedure agreed by the material producer and purchaser.
- 2b) Record the background information as to material, specimen number and applied application number of the Feritscope® and ID of the instrument.
- 2c) Perform 10 successive measurements at the same spot by lowering the probe perpendicular to the specimen³.
- 2d) Record each measurement and report the average Ferrite Number (FN) value. The volume percentage of ferrite is calculated using the equation

Volume % Ferrite = 0.55(EFN) + 10.6

This equation is not applicable if the measured Ferrite Number is in the range of 0-28.

Note: The appropriate specification for the calibration and implementation of ferrite measuring equipment is AWS A4.2.

3) Ferrite Measurement using ASTM E562 Manual Point Count

Though Feritscope® is the most suitable for measuring the ferrite content, Manual Point Count method can also be used for calculating the volume percentage of ferrite in Cast Super Duplex Stainless Steels.

1) Specimen Preparation

- The specimen on which the ferrite measurement has to be carried out should be mounted, ground and polished to metallographic surface finish (0.05μm).
- 1b) Polished samples are etched with solute-sensitive etchants like 40% sodium hydroxide solution to clearly reveal the microstructure.
- The etched structures should be observed under the Optical Light Microscope at an appropriate magnification. A 100x magnification is typical.

2) Test Procedure

- 2a) A grid should be prepared such that the size of the ferrite pool is one half of the spacing between grid points. This grid should be then superimposed on the microstructure.
- 2b) The number of points falling within the ferrite should be counted. Any point falling completely within the ferrite should be counted as one. Any point falling on the phase boundary or any that could be deemed questionable should be counted as one half.
- 2c) The following equations should be used to calculate the volume percentage of ferrite per ASTM E562 :

 P_T = Total number of points in the test grid

 P_i = Point count on the ith field

 $P_P(i) = P_i / P_T * 100 =$ Percentage of grid points in the ferrite on the ith field

n = number of fields counted

 $P_{P} = 1/n \sum P_{P}(i) = \text{Arithmetic average of } P_{P}(i)$ s = [1/(n-1) $\sum [P_{P}(i) - P_{P}]_{2}$]1/2 = Estimate of standard deviation (σ) 95% CI = \pm ts/ \sqrt{n} = 95 % confidence interval

t = Multiplier related to the number of fields examined and used in conjunction with the standard deviation n of the measurements to determine the 95% CI, see (Table 1 of ASTM E562).

 $V_V = P_P \pm 95\%$ CI = Volume fraction of ferrite as a percentage

% RA = (95% CI / P_P) = % Relative accuracy, a measure of statistical precision

From the comparison that was made between the results which were acquired from the Feritscope® and Manual Point Count method in the research program it was found that the volume percentage of ferrite calculated from both the methods were similar remarkably(Figure 10).

There are few disadvantages of manual point count, which affect the accuracy of the results from ASTM E562:

- 1. Operator bias pays a role in counting the phases falling inside the grid.
- 2. Specimen Preparation: Surface defects or abnormalities due to polishing or etching can lead to difficulty in distinguishing between the phases.
- 3. Grid Preparation: Thickness of grid lines can cause difficulty in determining whether a phase actually lies at the intersection or not^{4,5,7}.

It can be concluded that the volume percentage of ferrite measurement from either method produces similar results but the Feritscope® is more robust because it is easy to operate, eliminates operator bias and there are minimal requirements for specimen preparation⁵.

Figure 11 shows the microstructure of a specimen at 100x on which Manual Point Count was done.

4) <u>Hardness Measurement</u>

Where hardness measurements are of interest they shall be conducted using either Rockwell B or Rockwell C scale. "ASTM E18-03: Standard Test Method for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials" should be used.

1) Specimen Preparation

- 1a) The specimen on which the hardness measurement should be carried out should be ground to a smooth finish. The surface should be made flat.
- 1b) The specimen should not be mounted as that could lead to an error in hardness determintation.

2) Test Procedure

- 2a) The Rockwell Hardness Tester should be calibrated for Rockwell B or Rockwell C using standard test blocks.
- 2b) After the calibration is done, the specimen should be placed on the sample holder. Then the indenter should be brought down to touch the specimen and the reading should be noted.
- 2c) An average of three readings should be taken.

From the research results it was concluded that as the ferrite content decreased there was an increase in the hardness. This is because the ferrite content decreased as ferrite was converted into non-ferromagnetic intermetallic phases which increase the hardness of the material⁵.

5) Microstructural Evaluation

In addition to the Sodium Hydroxide Etch Test (Test Method A) microstructural characterization can provide for enhanced understanding of the internal behavior of the various phases and the precipitates. An explanation of experimental results can usually be defined when the microstructure is clearly revealed and understood.

1) Optical Light Metallographic Evaluation

- 1a) Specimens for metallographic evaluation extracted from the castings are mounted, ground and polished to metallographic surface finish (0.05μm).
- 1b) Polished samples are etched with solute-sensitive etchants like 40% sodium hydroxide solution to clearly reveal the microstructure.
 Note: A properly selected etching technique is very important for best revealing of the microstructure. The most appropriate etchant for Cast Super Duplex Stainless

Steel is 40% Sodium Hydroxide (NaOH). The specimen should be electrolytically etched at a voltage of about 1-3V DC for about 5-60s. This etchant reveals intermetallic phases also in addition to the ferrite and austenite phase.

The etched structures should be observed under the Optical Light Microscope at 400x-500x.

2) SEM and EDS

Detailed microstructural evaluation can be conducted using Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) on metallographically prepared samples. Using SEM (Figure 12) one can observe the microstructure at a higher magnification. It is easier to identify the type of intermetallic phases present using SEM with regard to location and shape, coupled with EDS one can determine the chemical composition of the phases present. SEM on the fracture surfaces from the Charpy Impact test (Test Method B) were studied. It was able to clearly distinguish between the ductile and brittle fracture (Figure 13 and 14). SEM was also carried out on the corrosion samples before and after they were subject to the Ferric Chloride Corrosion Test (Test Method C). From the SEM images one can infer that corrosion is highly localized (Figure 15 and 16). Due to the local nature of corrosion one should be cautious in using the uniform weight loss assumption for calculating the corrosion rate as stated in the Test Method C^5 .

SEM was accomplished on the specimens from one foundry which was subjected to the full course of heat treatment which is stated in the ASTM A923 for the wrought material. Using SEM it was possible to identify the type of intermetallic phases (Figure 17). EDS was also carried out on these specimens and the chemical composition of the phases was determined. A typical result is shown in figure 18⁵.

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ASTM SPECIFICATIONS

- ASTM A 890M 99: "Standard Specification for castings, Iron-Chromium-Nickel-Molybdenum Corrosion Resistant, Duplex (Austenitic/Ferritic) for General Application"
- ASTM A800/A 800M 91: "Standard Practice for Steel Casting, Austenitic Alloy, Estimating Ferrite Content"
- ASTM A799/A 799 M 91: "Standard Practice for Steel Castings, Stainless, Instrument Calibration, for Estimating Ferrite Content"
- ASTM A781 M 94a: "Standard Specification for Castings, Steel and Alloy, Common requirements for General Industrial Use"
- ASTM A 370: "Standard Methods and Definitions for Mechanical Testing of Steel Products"
- ASTM A923 94: "Standard Test for Determining the Detrimental Intermetallic Phase in Wrought Duplex (Austenitic/Ferritic) Stainless Steels"
- ASTM G48 92: "Standard Test Method for Pitting and Crevice Corrosion Resistance of Stainless Steels and Related Alloys by use of Ferric Chloride Solution"
- ASTM A 262 93a: "Standard Practice for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels"
- ASTM G 108 94: "Standard Test Method for Electrochemical Reactivation (EPR) for Detecting Sensitization of AISI Type 304 and 304L"

- ASTM E18 03: "Standard Test Method for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials"
- ASTM E562 02: "Standard Test Method for Determining Volume Fraction by Systematic Manual Point Count"

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Figure 1: 2507 Microstructure (After Sandvik Steel [6])





500x





500x



Figure 2: Unaffected Structure from ASTM A923









500x





Figure 4: Affected Structure from ASTM A923





Figure 5: Centerline Structure from ASTM A923



Figure 6: Unaffected Microstructure of Solution Annealed Super Duplex Stainless Steel, NaOH Etched, 400x



Figure 7: Affected Microstructure of Air Cooled Super Duplex Stainless Steel, NaOH Etched, 400x



Figure 8: Temperature Controlled Water Bath



Figure 9: Fisher Model MP - 3C Feritscope®

Ferrite Measurement



Figure 10: Comparison of Volume Percentage of Ferrite from the Feritscope® and per ASTM E562 Manual Point Count



Figure 11: Microstructure of Solution Annealed Super Duplex Stainless Steel, NaOH Etched, 100x



Figure 12: Leo 1525 Manufactured by Leo, UK



Figure 13: Ductile Fracture Surface of Solution Annealed Specimen, 500x



Figure 14: Brittle Fracture Surface of Heat Treated Specimen, 500x



Figure 15a: Air Cooled, Before Corrosion, 1000x



Figure 15b: Air Cooled, After Corrosion, 1000x



Figure 16a: Slow Cooled, Before Corrosion, 1000x



Figure 16b: Slow Cooled, After Corrosion, 1000x



Figure 17: Back Scatter SEM image of Slow Cooled Specimen, 1000x



Figure 18: EDS on Secondary Phase (S)