**FINAL REPORT
VOLUME 5**

**DATA PACKAGE FOR ASTM A923 SUPPORTING
INCLUSION OF A890-5A SUPER DUPLEX
STAINLESS STEEL
(CAST EQUIVALENT OF 2507)**

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This is Volume 5 of 5 of the final report for
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“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 inter-laboratory testing and Volume 3 “The Development of Qualification Standards for Cast Super Duplex Stainless Steel” provides information on the testing of 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 comprises 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

As per the ASTM A923 experimental procedures all test methods were carried out on 11 heats of foundry solution annealed A890-5A Superduplex Stainless Steel. A full scope of heat treatment was carried out on a single heat from Foundry D. The heat treatment schedule was derived from the testing of wrought DSS to establish the original A923 specification.

In order to support the inclusion of ASTM A890-5A material in ASTM A923, eleven heats of 5A material in the form of test bars were received from four SFSA foundries in the foundry solution annealed condition. The foundry solution annealing treatment for all the materials submitted for testing in accordance with A923 is shown in Table 1. The material from the eleven heats was tested in accordance with ASTM A923 Methods A, B and C. While no minimum toughness for the charpy impact testing or test temperature is given, per Method B, for the 2507 wrought alloy (to be agreed upon by purchaser and supplier), a temperature of -50°F was chosen as a temperature that may be representative of an industry typical test temperature. The chemistry for the eleven heats is given in Table 2. Sufficient material from one heat from Foundry D was available for a full scope of heat treatment following the schedule used for the testing of the wrought material in the development of the ASTM A923 specification.

Table 1: Details of the A890-5A Material Procured

Foundry	Heats	Number of Bars per Heat	Solution Annealing by Foundry
A	331F, 285F, 144F	1	Heat to 2200°F, Hold 3 hours, Water Quench
B	S1507, S1508	2	Heat to 2050°F, hold 2 hours, furnace cool to 1950°F, hold 1 hour, Water Quench
C	41544, 41414, 44073, 41374, 45633	1	Heat to 2075°F, cool to 1940°F, Hold 1 hour, Water Quench
D	04J142	10	Heat to 2100°F, Hold 1 hour, Water Quench

Table 2: Chemical Composition of Test Bars from Various Foundries**A890-5A GRADE TEST BARS**

Elements	Foundry A			Foundry B		Foundry C					Foundry D
	Heat 1	Heat 2	Heat 3	Heat 1	Heat 2	Heat 1	Heat 2	Heat 3	Heat 4	Heat 5	Heat 1
	144F	285F	331F	S1507	S1508	45633	44073	41374	41414	41544	04J142
Carbon	0.019	0.024	0.019	0.03	0.03	0.016	0.0194	0.016	0.019	0.03	0.02541
Sulphur	0.006	0.006	0.006	0.025	0.025	0.001	0.0039	0.01	0.009	0.005	0.00689
Phosphor	0.004	0.023	0.004	0.022	0.021	0.021	0.024	0.02	0.02	0.02	0.01157
Nitrogen	0.26	0.25	0.25	0.16	0.13	0.1537	0.1629	0.1615	0.166	0.1532	0.13935
Manganese	0.69	0.78	0.69	0.75	0.95	0.74	0.4	0.46	0.42	0.5	0.59528
Silicon	0.36	0.38	0.36	0.71	0.58	0.43	0.59	0.54	0.48	0.63	0.75715
Chromium	25.3	25.3	25.3	25.6	25.8	24.5	24.15	24.61	24.77	24.54	25.16827
Nickel	7.6	7.8	7.6	7.2	7	8	7.77	7.88	7.76	7.63	6.87326
Molybdenum	4.1	4.6	4.1	4.6	4.7	4.14	4.18	4.29	4.52	4.35	4.52612
Copper	NA	NA	NA	0.94	0.98	0.262	0.174	0.202	0.133	0.195	0.04285
Tungsten	NA	NA	NA	0.12	0.05	0.037	0.027	0.025	0.024	0.026	0.02137
Vanadium	NA	NA	NA	NA	NA	0.086	0.058	0.05	0.05	0.067	0.04186
Cobalt	NA	NA	NA	NA	NA	0.086	0.044	0.037	0.033	0.042	0.04712
Titanium	NA	NA	NA	NA	NA	0.001	0.001	0.001	0.001	0.001	0.00047
Aluminum	NA	NA	NA	NA	NA	0.014	0.023	0.017	0.018	0.018	0.01261
Tantalum	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	0.0019
Niobium	NA	NA	NA	NA	NA	0.013	0.015	0.03	0.017	0.022	0.01735
Tin	NA	NA	NA	NA	NA	0.007	0.007	0.007	0.007	0.007	NA
Boron	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA

The Suitability of ASTM A923 for Detecting the Presence of Intermetallic Phases in Super Duplex Stainless Steel Casting A890-5A

Test Method A

The following tables show the classification of etch structures from Method A based on ASTM A923. Figure 1 – 19 shows the microstructure of the etch structures at 400x taken under the Optical Light Microscope. All the microstructures are labeled with their foundry heat identity followed by the etchant, SA (if foundry solution annealed) and magnification.

Table 3: Classification of Etch Structure for Foundry Solution Annealed Materials from Foundry A, B, C and D

Foundry	Specimen Identity	Foundry Solution Annealed	Classification of Etch Surface
A	331	Heat to 2200°F, Hold 3 hours, Water Quench	Unaffected Structure
	185		Unaffected Structure
	285		Unaffected Structure
B	S1507	Heat to 2050°F, hold 2 hours, furnace cool to 1950°F, hold 1 hour, Water Quench	Unaffected Structure
	S1508		Unaffected Structure
C	41414	Heat to 2075°F, cool to 1940°F, Hold 1 hour, Water Quench	Unaffected Structure
	41544		Unaffected Structure
	44073		Unaffected Structure
	41374		Unaffected Structure
	45633		Unaffected Structure
D	04J142	Heat to 2050°F minimum, Hold for 1 hour, Furnace cool to 1950°F	Unaffected Structure

Table 4: Classification of Etch Structure for Materials Heat Treated as shown from Foundry D

Foundry	Specimen Identity	Heat Treatment	Classification of Etch Surface
D	04J142 - A	A Heat to 1950°F, Hold 30 min., Water Quench	Unaffected Structure
	04J142 - B	B Heat to 1950°F, Hold 30 min., Water Quench, Heat to 1550°F, Water Quench	Affected Structure
	04J142 - C	C Heat to 1950°F, Hold 30 min., Water Quench, Heat to 1550°F, Hold 5 min., Water Quench	Affected Structure
	04J142 - D	D Heat to 1950°F, Hold 30 min., Water Quench, Heat to 1550°F, Hold 10 min., Water Quench	Affected Structure
	04J142 - E	E Heat to 1950°F, Hold 30 min., Water Quench, Heat to 1550°F, Hold 15 min., Water Quench	Affected Structure
	04J142 - F	F Heat to 1950°F, Hold 30 min., Water Quench, Heat to 1550°F, Hold 20 min., Water Quench	Affected Structure
	04J142 - G	G Heat to 1950°F, Hold 30 min., Air Cool	Affected Structure
	04J142 - H	H Heat to 1950°F, Hold 30 min., Slow Cool	Affected Structure

Figure 1: Microstructure of 144, NaOH, SA, 400x

Figure 2: Microstructure of 285, NaOH, SA, 400x

Figure 3: Microstructure of 331, NaOH, SA, 400x

Figure 4: Microstructure of S1507, NaOH, SA, 400x

Figure 5: Microstructure of S1508, NaOH, SA, 400x

Figure 6: Microstructure of 41374, NaOH, SA, 400x

Figure 7: Microstructure of 41414, NaOH, SA, 400x

Figure 8: Microstructure of 41544, NaOH, SA, 400x

Figure 9: Microstructure of 44073, NaOH, SA, 400x

Figure 10: Microstructure of 45633, NaOH, SA, 400x

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Figure 14: Microstructure of 04J142 – C, NaOH, 400x

Figure 15: Microstructure of 04J142 – D, NaOH, 400x

Figure 16: Microstructure of 04J142 – E, NaOH, 400x

Figure 17: Microstructure of 04J142 – F, NaOH, 400x

Figure 18: Microstructure of 04J142 – G, NaOH, 400x

Figure 19: Microstructure of 04J142 – H, NaOH, 400x

In the case of Foundry D materials which were subjected to the full course of heat treatment schedule it was possible to compare the microstructure of solution annealed sample as received from the foundry with the microstructure of the samples which were subjected to different heat treatments. The as received solution annealed sample had an acicular structure¹. After subjecting to different heat treatments the samples were rendered in an equiaxed structure as it can be observed from the microstructures (Figures 1 – 19).

Test Method B

Table 5 shows the -50°F Charpy Impact Toughness of all samples that were submitted by Foundry A, B, C and D. All materials were foundry solution annealed. Figure 20 is a graphical presentation of the average impact values for each lot.

Table 6 shows the Charpy Impact Energy of the samples that were made from material sent by Foundry D. Figure 21 is a graphical presentation of the average impact value for the Foundry D heat following the heat treatment schedule for wrought material used in the initial development of A923.

Table 5: Charpy Impact Toughness for Foundry Solution Annealed Material at -50°F

Foundry	Specimen Identity	Impact Ft-Lbs
A	285	65.5
	285	76.5
	285	81.5
	285	75.5
	331	72
	331	61.5
	331	79
	331	77
	144	92
	144	135
	144	100
	144	91.5
	S1507	94.5
	S1507	63
	S1507	78
B	S1507	78.5
	S1508	65.5
	S1508	64
	S1508	78.5
	S1508	63
	45633	99
	45633	136.5
C	45633	146
	45633	129
	41414	123.5
	41414	113
	41414	138.5
	41374	134.5
	41374	142
	41374	131
	41374	113
	41544	145
	41544	135
	41544	144.5
	41544	146.5
	44073	148
	44073	155.5
	44073	149
	44073	157.5
	04J142	69.5
D	04J142	72
	04J142	75.5
	04J142	70

Solution Annealing by Foundry

Foundry	Solution Annealing by Foundry
A	Heat to 2200°F, Hold 3 hours, Water Quench
B	Heat to 2050°F, hold 2 hours, furnace cool to 1950°F, hold 1 hour, Water Quench
C	Heat to 2075°F, cool to 1940°F, Hold 1 hour, Water Quench
D	Heat to 2100°F, Hold 1 hour, Water Quench

Figure 20: Charpy Impact Toughness of Foundry Solution Annealed Per A923 Test Method B at -50°F

Table 6: Charpy Impact Toughness at -50°F for Heat Treated Materials from Foundry D

Heat Treatment Schedule	Absorbed Energy (ft-lbs)
A Heat to 1950°F, Hold 30 min., Water Quench	90 71
B Heat to 1950°F, Hold 30 min., Water Quench, Heat to 1550°F, Water Quench	2.5 2
C Heat to 1950°F, Hold 30 min., Water Quench, Heat to 1550°F, Hold 5 min., Water Quench	1.5 1
D Heat to 1950°F, Hold 30 min., Water Quench, Heat to 1550°F, Hold 10 min., Water Quench	8 7.5
E Heat to 1950°F, Hold 30 min., Water Quench, Heat to 1550°F, Hold 15 min., Water Quench	2 1.5
F Heat to 1950°F, Hold 30 min., Water Quench, Heat to 1550°F, Hold 20 min., Water Quench	1.5 1.5
G Heat to 1950°F, Hold 30 min., Air Cool	11 9 9.5 10
H Heat to 1950°F, Hold 30 min., Slow Cool	2.5 2 2.5 2.5

Charpy Impact Toughness at -50°F

Figure 21: Charpy Impact Toughness at -50°F of Foundry D bars exposed to the Heat Treatment shown (As Per Background data for A923 wrought material)

Test Method C

Table 7 summarizes the results from the corrosion test on the materials procured from Foundry A, B, C and D. All of these materials were foundry solution annealed. Figure 22 shows the graphical view of corrosion rate of various heats.

Table 8 shows the corrosion test results of the material which was sent by Foundry D and subjected to the heat treatment schedule for wrought material in A923. Figure 23 shows the graphical view of the corrosion rate and the heat treatment schedule.

Table 7: Corrosion Rates for Foundry Solution Annealed Materials from Foundry A, B, C and D

Foundry	Specimen Identity	Area of the Specimen	Weight of the Specimen	Weight after Testing	Corrosion Rates	Corrosion Results
A	331	195.312	15.85025	15.84881	0.073728	Pass
	144	185.546	14.95525	14.95519	0.003234	Pass
	285	224.515	17.99977	17.99811	0.073937	Pass
B	S1507	204.984	15.26527	15.26455	0.035125	Pass
	S1508	195.218	15.54399	15.54348	0.026125	Pass
C	41414	185.546	14.70874	14.70714	0.086232	Pass
	41544	195.593	16.12668	16.12647	0.010737	Pass
	44073	185.734	15.41645	15.41623	0.011845	Pass
	41374	195.312	15.65445	15.65424	0.010752	Pass
	45633	214.937	17.55149	17.54949	0.093051	Pass
D	04J142	175.874	14.53285	14.53261	0.013646	Pass

Solution Annealing by Foundry

Foundry	Solution Annealing by Foundry
A	Heat to 2200°F, Hold 3 hours, Water Quench
B	Heat to 2050°F, hold 2 hours, furnace cool to 1950°F, hold 1 hour, Water Quench
C	Heat to 2075°F, cool to 1940°F, Hold 1 hour, Water Quench
D	Heat to 2100°F, Hold 1 hour, Water Quench

Figure 22: Corrosion Rate of Foundry Solution Annealed material from Foundry A, B, C and D (Acceptance Criteria – Weight loss $\leq$ 10 milligrams/decimeter/day (10 mdd))

Table 8: Corrosion Rates for Heat Treated Materials from Foundry D

Foundry	Specimen Identity	Area of the Specimen	Weight of the Specimen	Weight after Testing	Corrosion Rates	Corrosion Results
D	04J142 - A	185.359	14.81757	14.81754	0.001618	Pass
	04J142 - B	166.11	13.8771	13.42999	26.9165	Fail
	04J142 - C	156.156	12.65836	12.23875	26.87121	Fail
	04J142 - D	175.781	14.27541	14.20991	3.726228	Pass
	04J142 - E	175.875	14.75958	14.54034	12.46567	Fail
	04J142 - F	175.781	14.63388	14.21803	23.65728	Fail
	04J142 - G	175.875	14.5804	14.54974	1.743284	Pass
	04J142 - H	195.594	16.55008	15.11975	73.1275	Fail

* Heat Treatment “A” sample showed corrosion rate equivalent to zero (≈0)

Figure 23: Corrosion Rate of material from Foundry D (Acceptance Criteria – Weight loss ≤ 10 milligrams/decimeter/day (10 mdd))

Evaluation and Correlation between the Test Methods of A923

All the materials from Foundry A, B, C and D were subjected to the test methods of A923. Test Method A was the Sodium Hydroxide Etch Test (visual identification of intermetallic phases). One half of fractured Charpy sample was used to carry out the Sodium Hydroxide Etch Test. Solution Annealed samples from Foundry A, B, C and D was considered as unaffected structures. The heat treatment “A” sample from Foundry D (solution annealed in The University of Tennessee) also revealed an unaffected structure. The samples which underwent heat treatment schedules “B”, “C”, “D”, “E”, “F”, “G” and “H” were all identified as affected structures. Heat Treatments “B” to “F” were subjected to two stages of heat treatment which is Heat to 1950 °F, hold for 30 minutes and water quench followed by heat to 1550°F, hold for different time periods before water quenching them. Heat Treatment “G” is the air cool sample which is heated to 1950 °F, held for 30 minutes and then air cooled. Heat treatment “H” is the slow cool sample which is heated to 1950 °F, held for 30 minutes and then furnace cooled.

From the Charpy Impact Test (Test Method B) it was found that foundry solution annealed samples from Foundries A, B, C and D had toughness as greater than 60ft-lbs (Figure 20). The material from Foundry C had the highest toughness. The Charpy impact energy was greater than 100ft-lbs for all the samples that were tested. There were two heats from Foundry B. Heat S1507 had porosity in the Charpy samples that were machined but had a higher impact value than the other heat S1508. This may be because the porosity was not in the notched region of the sample. Out of the three materials from Foundry A, heat 144 had a comparatively greater toughness than the other heats 331 and 285. The heat treatment “A” sample from Foundry D (solution annealed at The University of Tennessee) also showed a toughness value greater than 60ft-lbs.

The samples from Foundry D were subjected to the full scope of heat treatment. After heat treatment the Charpy Impact Test was carried out. From Figure 21 it can be observed that the toughness dropped from 60ft-lbs to 1-10ft-lbs when the second stage of heat treatment was carried out (Heat to 1550°F and hold for different time periods before

water quenching). Air cooled and slow cooled samples also showed a similar drop in toughness.

The Ferric Chloride Corrosion Test (Test Method C) was carried out on these fractured Charpy samples. It was seen that the foundry solution annealed samples from Foundry A, B, C and D passed. All these samples passed the acceptance criteria of weight loss ≤ 10 mdd by a wide margin (Figure 22). The heat treatment “A” sample from Foundry D (solution annealed at The University of Tennessee) also passed by a similar margin. The heat treatment “D” and “G” samples also passed. All the samples which underwent other heat treatment schedules failed the corrosion test (Figure 23).

From the results of the three test methods can be derived a correlation between the results. Also one can predict the best method which could be used to detect the presence of detrimental intermetallic phases. Test Method A can be used to visually detect the presence of an affected structure but cannot identify the type of intermetallic phases present. It was possible to visually deduce whether a sample is affected with a greater percentage of intermetallic phases when compared with another sample. The estimated percentage of intermetallic phases present in air cooled (Figure 18) sample (heat treatment “G” sample from Foundry D) was less when compared with slow cooled (Figure 19) sample (heat treatment “H” sample from Foundry D). It was visually confirmed that heat treatment “D” sample (Figure 15) had a lesser percentage of intermetallic phase when compared to heat treatment “B”, “C”, “E” and “F” samples (Figure 13-14, 16-17) which had similar percentage of intermetallic phase.

The Charpy Impact Test indicated that all the solution annealed materials (Figure 20) have high toughness. There was no precipitation of detrimental intermetallic phases. In these foundry solution annealed samples Figure 21 indicates that the second stage of the heat treatment process (heat treatment “B”, “C”, “D”, “E” and “F”) precipitated intermetallic phases and reduced the impact toughness considerably. During the second stage of heat treatment when the samples are heated to 1550°F, the heating process takes approximately an hour for the samples to reach this temperature after they

were water quenched from 1950°F. It may be concluded that the intermetallic phases were precipitated during the heating stage (to 1550°F). There was an increase in the toughness of heat treatment “D” sample as compared to the toughness of heat treatment “B” and “C” samples. The reason for this observation is unknown at this time.

There were no intermetallic phases for the samples from heat treatment schedule “A” (solution annealed at the University of Tennessee). This demonstrates that solution annealed samples do not contain any intermetallic phases but the subsequent heat treatment schedules result in the precipitation of intermetallic phases as noted in Table 4. The heat treatment “G” and “H” samples which were heated to 1950°F and then air cooled and slow cooled respectively also precipitated intermetallic phases. The air cooled sample contained lesser amount of intermetallic phases as compared to slow cool sample though both had low toughness (1-10ft-lbs). This indicates that precipitation of small amount of intermetallic phase reduce the toughness considerably (1-10ft-lbs). Air cooling and slow cooling precipitates intermetallic phases but when similar material is heated to the same temperature and water quenched no intermetallic phases are precipitated which indicates that solution treatment (1950°F) dissolves all the intermetallic phases which have been precipitated during heating. This is true only for first stage of heat treatment which is heat treatment “A” (Heating to 1950°F followed by water quenching). It can be concluded that heat treatment “A”, intermetallic phases did not precipitate when the samples were water quenched, however intermetallic phases developed in the air to slow cooled samples.

The sample from Foundry D which underwent heat treatment schedule “D” had low toughness from the Charpy Impact Test (Test Method B) and also an affected structure in the Sodium Hydroxide Etch Test (Test Method A) but still passed the Ferric Chloride Corrosion Test (Test Method C). This demonstrates the ineffectiveness of Test Method C in detecting the presence of detrimental intermetallic phases.

The sample from Foundry D which underwent heat treatment schedule “G” also passed the Ferric Chloride Corrosion Test though it revealed an affected structure and

low toughness. But the amount of intermetallic phase which was determined from the OLM image was less when compared to samples which had undergone other heat treatment schedules. In previous work A890-4A grade samples which had undergone heat treatment schedule “G” did not reveal the presence of intermetallic phase though they had similar toughness². From this it may be concluded that air cooling is not rapid enough to prevent the precipitation of intermetallic phases in A890-5A material.

From the results it can be concluded that Charpy Impact Test (Test Method B) is the best method out of the three to indicate the presence of detrimental intermetallic phase. From the toughness it is possible to predict the Sodium Hydroxide Etch Test (Test Method A) and Ferric Chloride Corrosion Test (Test Method C) results. The trend which was observed was that if the toughness is greater than 60ft-lbs then the samples had an unaffected structure and a high corrosion resistance (passed the acceptance criteria of weight loss ≤ 10 mdd by a wide margin). If the toughness was below 15ft-lbs then the samples revealed an affected structure and low corrosion resistance (weight loss ≥ 10 mdd). There was a 1:1 correlation between Test Method A and B. Though Test Method A is a direct method of detecting the presence of intermetallic phases, it cannot be confirmed whether the intermetallic phases present are detrimental or not. The lowering of toughness from Test Method B confirms whether the intermetallic phase present is detrimental. However, the toughness value of the material may also decrease if the percentage of ferrite is high. But this can be determined using Feritscope.

It can be concluded that the micrographs from A890-4A grade included in ASTM A923 are applicable for A890-5A grade to compare and detect the presence of detrimental intermetallic phase. The micrographs can be used to verify whether the A890-5A sample has an unaffected, affected or a possibly affected structure. It was also observed that when compared to A890-4A grade A890-5A grade is more sensitive to heat treatment. The A890-4A grade when subjected to Charpy Impact Test showed a gradual decrease in toughness with increasing hold time at 1550°F. But the A890-5A grade material showed a significant drop in toughness from heat treatment schedule “A” to “B” when the second stage of the heat treatment process was followed (Heat to 1550°F and

hold for different time periods before water quenching). Qualitatively a greater amount of intermetallic phase is precipitated in the A890-5A material when compared to A890-4A grade for the same heat treatment schedule.

Ferrite Measurement

The Feritscope was used to measure the ferrite content in all the Charpy samples before carrying out toughness tests. Table 9 shows the average of volume ferrite in percentage in every heat calculated from the ferrite number.

Table 9: Ferrite Content in Volume Percentage

Foundry	Specimen Identity	Ferrite Number	Volume % Ferrite
A	285	66.2	46.9
	144	53.7	40.2
	331	61.3	44.3
B	S1507	84	56.8
	S1508	65.1	46.4
C	45633	75.3	52.1
	41414	71.1	49.7
	41374	74.3	51.5
	41544	73.8	51.2
	44073	64.7	46.2
D	04J142	100.6	65.9
	04J142 - A	95.5	63.1
	04J142 - B	59.2	43.2
	04J142 - C	39.1	32.1
	04J142 - D	80.4	54.8
	04J142 - E	41.6	33.5
	04J142 - F	35.9	30.3
	04J142 - G	79.7	54.5
	04J142 - H	3.8	12.7

From the above data it can be seen that all the foundry solution annealed heats from Foundry A, B and C which had high toughness showed volume percentage of ferrite to be approximately 50.

The foundry solution annealed material from Foundry D showed a significant variation in ferrite content with heat treatment schedules. The foundry solution annealed and heat treatment “A” samples (solution annealed at the University of Tennessee) showed ferrite content of approximately 65%, higher than the foundry solution annealed samples from other foundries even though they had similar toughness. The samples which underwent heat treatment “B”, “D” and “G” showed an approximate volume percent of ferrite of 50 though they had low toughness. Heat treatment “B”, “D” and “G” samples contained intermetallic phases. The other heat treatment samples had low ferrite content especially heat treatment “H” sample which showed the volume percent of ferrite to be approximately 12.7%. This was evident from the affected microstructures which showed that a large proportion of ferrite was converted into intermetallic phases.

Though there were few exceptions it could be concluded that with increasing presence of intermetallic phases in the microstructure the volume percentage of ferrite content is reduced as most of the ferrite is converted into intermetallic phases. Since heat treatment “B” samples exhibited ferrite of approximately 50% but had low toughness (Test Method B) and failed the Sodium Hydroxide Etch Test (Test Method A) and Ferric Chloride Corrosion Test (Test Method C) ferrite percent is not a good indicator of the absence of detrimental intermetallic phases.

ASTM E562 Ferrite Measurement – Standard Test Method for Determining Volume Fraction by Systematic Manual Point Count

Ferrite measurement was also carried out using a manual point count method stated in ASTM E562. Table 10 shows the average volume ferrite in percentage calculated using point count method and also Feritscope®.

Figure 24 shows a comparison between the volume percentage of ferrite measured using the Feritscope® and ASTM E562 Manual Point Count method.

Figure 24: Comparison of Volume Percentage of Ferrite from the Feritscope® and per ASTM E562 Manual Point Count

Table 10: Volume Ferrite in Percentage

Sample Identity	Number of Fields (n)	% of Grid Points	Average volume % ferrite	Standard Deviation (s)	95% Confidence Limit	Volume % Ferrite using Point Count	Ferrite Number (Feritscope ®)	Volume % Ferrite from Feritscope ®
144	1	42.9	41.2	1.5	1.1	40.1 – 42.3	53.7	40.2
	2	39.7						
	3	41.9						
	4	42.4						
	5	43.7						
	6	39.3						
	7	40.3						
	8	41.9						
	9	40.7						
	10	39.6						
45633	1	55.2	51.6	1.7	1.2	50.4 – 52.8	75.3	52.1
	2	52.3						
	3	51.1						
	4	52.9						
	5	51.6						
	6	49.5						
	7	52.1						
	8	51.1						
	9	50.3						
	10	49.6						
S1508	1	46.2	45.9	1.8	1.3	44.6 – 47.2	65.1	46.4
	2	45.4						
	3	47.6						
	4	49.1						
	5	44.8						
	6	45.1						
	7	45.6						
	8	46.9						
	9	43.9						
	10	44.1						
04J142	1	66.7	65.9	2.1	1.4	63.8 - 68	100.6	65.9
	2	69.1						
	3	64.9						
	4	63.4						
	5	65.9						
	6	65.6						
	7	67.1						
	8	64.2						
	9	63.5						
	10	68.9						
04J142 - D	1	55.2	53.6	2.5	1.8	51.8 – 55.4	80.4	54.8
	2	57.3						
	3	53.8						
	4	53.5						
	5	51.7						
	6	53.7						
	7	56.6						
	8	54.5						
	9	50.5						
	10	49.6						

From the table we can infer that the volume percentage of ferrite calculated using Feritscope® falls within the 95% confidence limit for volume percentage of ferrite calculated using ASTM E562. The standard deviation measured from ASTM E562 ranged from 1.5 – 2.5%, which indicates that ferrite measurement by Point Counting per ASTM E562 shows similar results when compared with the Feritscope®.

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

1. Operator bias plays 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.

It can be concluded that the volume percentage of ferrite measurement from either method produces similar results but Feritscope® is more robust because it is easy to operate, eliminates operator bias and there are minimal requirements for specimen preparation^{3,4}.

Hardness Measurement

The hardness was measured on all the samples from various heats using both Rockwell B and C. The following table shows the hardness of all the heats and heat treatments.

Table 11: Hardness Value Measured using Rockwell B and C

Foundry	Specimen Identity	HRB	HRC
A	285	103.9	22.5
	144	94.1	18.9
	331	103	21.8
B	S1507	96.1	24.4
	S1508	96.1	21.9
C	45633	102.7	20.8
	41414	98.6	20.4
	41374	102.2	20.1
	41544	98.4	20.9
	44073	100.9	18.9
D	04J142	102.8	25.5
	04J142 - A*	99.1	20.4
	04J142 - B	101.9	24.9
	04J142 - C	106.1	30.8
	04J142 - D	101.6	20.1
	04J142 - E	106.8	29.9
	04J142 - F	108.6	29.9
	04J142 - G	102.7	22.1
	04J142 - H	109.9	35.2

* Refer to heat treatment schedule in Table 4

From the above data it was observed that the hardness for all the heats from Foundry A, B and C was approximately between 94 to 103 and 18 to 26 on HRB and HRC scales respectively. For the samples from Foundry D the solution annealed and heat treatment “A” showed similar hardness. Heat treatment “B”, “D” and “G” also showed similar hardness though heat treatment “B”, “D” and “G” samples contained intermetallic phase. This may be because from the OLM micrographs it was possible to determine visually that there was only a small amount of intermetallic phases present as compared to samples which had undergone other heat treatment schedules. Samples from heat treatment schedules “C”, “E”, “F” and “H” showed greater hardness on both the scales because the presence of intermetallic phase increases the hardness of the material.

These results were similar to the results that were obtained for the ferrite content measurement. It may be possible to conclude that there was a direct relationship between

the ferrite content and the hardness. As the ferrite content decreased due to the transformation of intermetallic phases there was an increase in the hardness. If the volume percent of ferrite content is approximately 50, then the hardness is approximately between 94 to 103 and 18 to 26 in HRB and HRC scales respectively.

Evaluation of Fracture Surfaces from Test Method B

SEM examination was carried out on the fracture surfaces of one sample from each heat. The following figures show SEM images of the fracture surfaces of the samples. All these samples are labeled with their specimen identity followed by its impact toughness and magnification.

Figure 25: Fracture Surface of 04J142, 72ft-lbs, 500x

Figure 26: Fracture Surface of 04J142 – A, 80.5ft-lbs, 500x

Figure 27: Fracture Surface of 04J142 – B, 2.5ft-lbs, 500x

Figure 28: Fracture Surface of 04J142 – C, 1.5ft-lbs, 500x

Figure 29: Fracture Surface of 04J142 – D, 7.5ft-lbs, 500x

Figure 30: Fracture Surface of 04J142 – E, 1.5ft-lbs, 500x

Figure 31: Fracture Surface of 04J142 – F, 1.5ft-lbs, 500x

Figure 32: Fracture Surface of 04J142 – G, 9.5ft-lbs, 500x

Figure 33: Fracture Surface of 04J142 – H, 2.5ft-lbs, 500x

Figure 34: Fracture Surface of 331, 72.5ft-lbs, 500x

Figure 35: Fracture Surface of 144, 104ft-lbs, 500x

Figure 36: Fracture Surface of 285, 75ft-lbs, 500x

Figure 37: Fracture Surface of 41374, 130ft-lbs, 500x

Figure 38: Fracture Surface of 41414, 125ft-lbs, 500x

Figure 39: Fracture Surface of 41544, 143ft-lbs, 500x

Figure 40: Fracture Surface of 45633, 126.5ft-lbs, 500x

Figure 41: Fracture Surface of 44073, 152.5ft-lbs, 500x

Figure 42: Fracture Surface of S1507, 78.5ft-lbs, 500x

Figure 43: Fracture Surface of S1508, 68ft-lbs, 500x

The fractures can be categorized into two types – Ductile and Brittle. A ductile fracture is characterized by a dimpled surface. It is associated with high energy release. On the other hand brittle fracture is characterized by rapid crack propagation with low energy release. Brittle fractures display either cleavage (transgranular) or intergranular fracture. The fracture may have a bright granular appearance. The materials which undergo brittle fracture have greater hardness as compared to those which undergo ductile fracture.

From the SEM images it was observed that all the foundry solution annealed samples from Foundry A, B, C and D showed ductile fracture. All these samples had large dimpled ductile fracture surfaces. These samples had a high toughness. The heat treatment “A” sample (solution annealed at the University of Tennessee) from Foundry D also showed ductile fracture. The samples from other heat treatment schedules generally showed brittle fracture. Cleavage was evident on the fracture surface of heat treatment “G” sample. These samples had low toughness. Most of the samples which underwent brittle fracture had hardnesses greater than the solution annealed samples which showed ductile fracture.

Corrosion Analysis of Test Method C Samples using SEM

SEM was carried out on all of the ASTM A923 Test Method C samples before and after the corrosion test was performed. The following Figures show SEM images of all samples:

Figure 44a: 04J142-SA, Before Corrosion, 1000x

Figure 44b: 04J142-SA, After Corrosion, 1000x

Figure 45a: 04J142-A, Before Corrosion, 1000x

Figure 45b: 04J142-A, After Corrosion, 1000x

Figure 46a: 04J142-B, Before Corrosion, 1000x

Figure 46b: 04J142-B, After Corrosion, 1000x

Figure 47a: 04J142-C, Before Corrosion, 1000x

Figure 47b: 04J142-C, After Corrosion, 1000x

Figure 48a: 04J142-D, Before Corrosion, 1000x

Figure 48b: 04J142-D, After Corrosion, 1000x

Figure 49a: 04J142-E, Before Corrosion, 1000x

Figure 49b: 04J142-E, After Corrosion, 1000x

Figure 50a: 04J142-F, Before Corrosion, 1000x

Figure 50b: 04J142-F, After Corrosion, 1000x

Figure 51a: 04J142-G, Before Corrosion, 1000x

Figure 51b: 04J142-G, After Corrosion, 1000x

Figure 52a: 04J142-H, Before Corrosion, 1000x

Figure 52b: 04J142-H, After Corrosion, 1000x

Figure 53a: 144, Before Corrosion, 1000x

Figure 53b: 144, After Corrosion, 1000x

Figure 54a: 285, Before Corrosion, 1000x

Figure 54b: 285, After Corrosion, 1000x

Figure 55a: 331, Before Corrosion, 1000x

Figure 55b: 331, After Corrosion, 1000x

Figure 56a: S1507, Before Corrosion, 1000x

Figure 56b: S1507, After Corrosion, 1000x

Figure 57a: S1508, Before Corrosion, 1000x

Figure 57b: S1508, After Corrosion, 1000x

Figure 58a: 41374, Before Corrosion, 1000x

Figure 58b: 41374, After Corrosion, 1000x

Figure 59a: 41414, Before Corrosion, 1000x

Figure 59b: 41414, After Corrosion, 1000x

Figure 60a: 41544, Before Corrosion, 1000x

Figure 60b: 41544, After Corrosion, 1000x

Figure 61a: 45633, Before Corrosion, 1000x

Figure 61b: 45633, After Corrosion, 1000x

Figure 62a: 44073, Before Corrosion, 1000x

Figure 62b: 44073, After Corrosion, 1000x

After carrying out SEM on the samples which were tested for corrosion resistance it was possible to correlate the results from the ASTM A923 Test Method C with the images that were obtained.

All the foundry solution annealed samples from Foundries A, B, C and D had been subjected to corrosion tests, passed Test Method C and when the SEM was carried out on these samples before and after they had subjected to corrosion, only pits, depressions and surface irregularities were found on these samples. Sample 331, from Foundry A showed pits and surface irregularities. Some surface irregularity was observed in this sample before the corrosion test was carried out but it was not well defined. The samples from heat S1507 and S1508 of Foundry B showed the presence of pits after the corrosion test was carried out. In the samples from Foundry C pits were present in sample 41414 and depressions in samples 45633 and 44073.

The heat treatment “A” sample (solution annealed at the University of Tennessee) passed the Test Method C as revealed by the SEM images which do not show any defects. The SEM images of heat treatment “B”, “C”, “E”, “F” and “H” showed crack-like surface irregularities in the samples after corrosion testing. These samples failed the corrosion test. The sample from heat treatment “H” which showed a high corrosion rate had crack-like discontinuities throughout the surface of the specimen. Though heat

treatment “D” and “G” passed the corrosion test, SEM images revealed pits and oxides on the corrosion surface.

It was observed that although the samples passed the corrosion Test Method C pits, oxides, surface irregularities and depressions were observed on the sample surface after the corrosion test was carried out. All these indications were highly localized. They were not distributed throughout the samples surface. It has been shown by Arthur H Tuthill⁵ that the assumption of uniform weight loss over the full area of specimen in calculating the corrosion rate may be reasonable for carbon steel, however in the case of stainless steels it is misleading due to the fact that corrosion of stainless steel is highly localized as seen in the case of these samples also. This also indicates that Test Method C is a not a good method for determining the presence of detrimental intermetallic phase when compared to Test Method A and B.

Since these samples were not polished and etched to reveal the microstructure, as it was not required for Test Method C according to ASTM A923, it was not possible to determine which phases were present when indications were observed.

Verification of the type of Secondary Phases present in Foundry D samples using Scanning Electron Microscopy and Energy Dispersive Spectroscopy

SEM and EDS was carried out on the samples from Foundry D which were subjected to heat treatment schedule derived from the testing of wrought DSS. SEM and EDS was also carried out on the foundry solution annealed sample from Foundry D in order to compare its EDS results with the heat treatment “A” (Solution Annealed at the University of Tennessee) sample results. The following figures show the SEM images of these samples as well as the EDS results.

Figure 63a: Secondary Electron SEM image of 04J142 (Foundry Solution Annealed), 1000x

Figure 63c: EDS on Austenite Phase (γ)

Figure 63c: EDS on Ferrite Phase (δ)

Figure 64a: Secondary Electron SEM image of 04J142-A (Heat Treatment “A”), 1000x

Figure 64b: EDS on Austenite (γ)

Figure 64c: EDS on Ferrite Phase (δ)

Figure 65a: Back Scatter SEM image of 04J142-B, 1000x

Figure 65b: EDS on Secondary Phase (S)

Figure 66a: Back Scatter SEM image of 04J142-C, 1000x

Figure 66b: EDS on Secondary Phase (S)

Figure 67a: Secondary Electron SEM image of 04J142-D, 1000x

Figure 67b: EDS on Secondary Phase (S)

Figure 68a: Secondary Electron SEM image of 04J142-E, 1000x

Figure 68b: EDS on Secondary Phase (S)

Figure 69a: Secondary Electron SEM image of 04J142-F, 1000x

Figure 69b: EDS on Secondary Phase (S)

Figure 70a: Secondary Electron SEM image of 04J142-G, 1000x

Figure 70b: EDS on Secondary Phase (S)

Figure 71a: Back Scatter SEM image of 04J142-H, 1000x

Figure 71b: EDS on Secondary Phase (S)

From the SEM micrographs the presence of intermetallic phases can be identified. All the samples which had undergone heat treatment schedule “B”, “C”, “D”, “E”, “F”, “G” and “H” had intermetallic phases. Back scatter was carried out on some of the

samples in order to more clearly identify the intermetallic phases as it is easier to differentiate between different phases using back scatter image compared to secondary electron image. By carrying out EDS on all these samples it was possible to identify the intermetallic phase as well as the composition. The solution annealed and the heat treatment “A” sample contained only austenite and ferrite phase. From EDS it was possible to identify the ferrite and austenite phase respectively. The austenite phase contained a lower percentage of chromium and molybdenum and higher nickel content as compared to the ferrite phase. Both the samples had similar chemical composition (Figure 63 and 64).

In the heat treatment sample “B”, “C”, “D”, “E” and “F” $\gamma_2 + \sigma$ (Secondary austenite + Sigma) phases were identified. All these samples had undergone the second stage of heat treatment. The second stage is heating the sample to 843°C (1550°F) and holding for different time periods before water quenching. The $\gamma_2 + \sigma$ phases are precipitated between 700°C to 900°C⁶. Growth of these phases into the δ phase can be observed in the SEM images. The precipitate of carbides could also be observed in samples from heat treatment “B”, “C” and “D” samples along the γ/δ and γ/γ_2 interphase boundaries⁷ as observed from the SEM images.

SEM images revealed that the heat treatment “D” sample contained lower amounts of intermetallic phase when compared to heat treatments “B”, “C”, “E” and “F”. Also in contrast there was higher toughness for heat treatment “D” samples as evident in Figure 21. The heat treatment “G” sample which was air cooled also contained lesser amounts of intermetallic phases as evident from its SEM image (Figure 70). The SEM images of heat treatment “A”, “G” and “H” samples indicated that as the toughness of these samples decreased in the order of “A” > “G” > “H”, intermetallic phases increased in the same order from zero in heat treatment “A” samples to a higher amount in heat treatment “H” samples.

In heat treated samples “C”, “E”, “F”, “G” and “H” σ (Sigma) phase was observed on the γ/δ interphase boundaries. From the SEM image of heat treatment “H”

sample it was observed that the amount of sigma present was high. This also explains the higher value of hardness of this sample on the Rockwell B and C scale because presence of sigma phase increases hardness. The amount of sigma present in the heat treatment “G” sample was less compared to heat treatment “H” sample. From this it can be concluded that slow cooling tends to transform more of ferrite into sigma as compared to air cooling.

There does not appear to be a correlation between volume percent ferrite and the precipitation of detrimental intermetallic phases. With the precipitation of intermetallic phase the volume percent of ferrite should decrease. This was observed only in heat treatment “C”, “E”, “F” and “G” samples. The volume percentage of ferrite was approximately 50 for heat treatment “B”, “D” and “G” (Table 9) samples though intermetallic phases were observed in these samples. This suggests that volume percent of ferrite is not a good indicator of amount of detrimental intermetallic phases.

SEM was also carried out on the foundry solution annealed samples from Foundry A, B and C. The images are included in Appendix D.

Conclusions

I. Data Package

- 1a) A data package was developed based on the results which were acquired from various tests performed on the ASTM A890-5A cast Super Duplex Stainless Steel material procured from various foundries.

II. 1. The Suitability of ASTM A923 for Detecting the Presence of Intermetallic Phases in Super Duplex Stainless Steel Castings

- 1a) Since ASTM A923 Methods adequately identifies the presence of intermetallic phases in A890 – 5A grade Cast Super Duplex Stainless Steel A890 – 5A can be directly included in ASTM A923.
- 1b) The micrographs from the A890-4A grade (now in ASTM A923) are applicable for the A890-5A grade to compare and detect the presence of intermetallic phases. Using these micrographs one can verify whether an A890-5A sample has an unaffected, affected or a possibly affected structure.

2. Evaluation of the ASTM A923 Test Methods A, B and C

- 2a) All of the eleven foundry solution annealed heats from foundries A, B, C and D passed all the ASTM A923 Test Methods A, B and C. In addition, the heat treatment “A” samples (solution annealed at The University of Tennessee) from Foundry D also passed all three ASTM A923 Test Methods.
- 2b) The samples which were subjected to heat treatment schedules “B”, “C”, “D”, “E” and “F” (Heating to 1950°F, hold for 30 minutes, Water Quench and then heat to 1550°F, Water Quench after holding for 0, 5, 10, 15 and 20 minutes respectively) did not pass the ASTM A923 Test Methods. These samples underwent the second stage of heat treatment (Heating to 1550°F, holding for different time intervals respectively, water quenching after water quenching from 1950°F). The heating

process took approximately an hour for the samples to reach the temperature of 1550°F. Intermetallic phases are expected to precipitate during the heating process to 1550°F as well as during the hold time at 1550°F. These heat treatment schedules were used for the testing of the wrought material and A890-4A grade cast Duplex Stainless Steel for the formulation of the ASTM A923 specification.

- 2c) During the first stage of heat treatment (Heating to 1950°F, hold for 30 minutes and water quench) the amount of precipitated detrimental intermetallic phases is virtually zero as anticipated.
- 2d) It was also observed that when compared to the A890-4A grade A890-5A grade is more sensitive to heat treatment. The A890-4A grade when subjected to Charpy impact test showed a gradual decrease in there toughness with increasing hold time at 1550°F. But the A890-5A grade material showed a significant drop in toughness from heat treatment schedule “A” to “B” when second stage of heat treatment process was followed (Heat to 1550°F and hold for different time periods before water quenching).
- 2e) It was observed that from Test Method A one can visually detect the presence of an affected structure but cannot adequately identify the type of intermetallic phases present. However it is possible to visually deduce whether a sample is affected with a greater percentage of intermetallic phases when compared with another sample.
- 2f) The air cooled sample (from 1950°F) contained a lesser amount of intermetallic phases as compared to slow cool sample though both had low toughness (1-10ft-lbs). This indicates that precipitation of even a small amount of intermetallic phase can reduce the toughness considerably (1-10ft-lbs).
- 2g) The heat treatment “G” samples (air cooled from 1950°F) revealed an affected structure though the amount of intermetallic phase which was visualized from the OLM image was less as compared to samples which had undergone other heat treatment schedules. They had low toughness. This was similar to ASTM A890-4A grade (previous test results) which indicated that air cooling was not sufficiently rapid in preventing the intermetallic phase precipitation in A890-4A material. The same is true for ASTM A890-5A material.

- 2h) The samples from Foundry D which underwent heat treatment schedules “D” (Heat to 1950°F, hold 30 minutes, water quench, heat to 1550°F, hold 10 minutes, water quench) and “G” (air cooled from 1950°F) had low Charpy toughness (Test Method B) and also an affected structure in the Sodium Hydroxide Etch Test (Test Method A) but still passed the Ferric Chloride Corrosion Test (Test Method C). This explains the ineffectiveness of Test Method C in detecting the presence of detrimental intermetallic phase.
- 2i) SEM images of corrosion samples proved that corrosion is localized; hence, the assumption of uniform weight loss over the full area of the specimen is not appropriate for calculating the corrosion rate. This indicates that possibly ASTM A923 Test Method C is not an appropriate method for predicting the presence of detrimental intermetallic phases in cast super duplex stainless steel.
- 2j) There was an increase in the toughness of heat treatment “D” (Heat to 1950°F, hold 30 minutes, water quench, heat to 1550°F, hold 10 minutes, water quench) samples as compared to the toughness of heat treatment “B” (Heat to 1950°F, hold 30 minutes, water quench, heat to 1550°F, water quench) and “C” (Heat to 1950°F, hold 30 minutes, water quench, heat to 1550°F, hold 5 minutes, water quench) samples. The reason for this observation may be related to the heating time to 1550°F.

3. Determination of the best ASTM A923 Test Method for Detecting the Presence of Detrimental Intermetallic Phases

- 3a) A correlation was determined between the ASTM A923 Test Methods A, B and C in determining the presence of detrimental phases in the material.
- 3b) It was observed that if the toughness is greater than 60ft-lbs then all samples had an unaffected structure and a high corrosion resistance (passed the acceptance criteria of weight loss ≤ 10 mdd by a wide margin). If the toughness was below 15ft-lbs then the samples always revealed an affected structure and exhibited low corrosion resistance (weight loss ≥ 10 mdd) except for heat treatment “D” and “G” samples which passed the Ferric Chloride Corrosion Test (Test Method C) indicating the

ineffectiveness of Test Method C in determining the presence of detrimental intermetallic phases.

- 3c) There was a 1:1 correlation between Test Method A and B. Though Test Method A is a direct method of detecting the presence of intermetallic phases, it cannot be confirmed whether the intermetallic phases present are detrimental or not. The lowering of toughness from Test Method B confirms whether the intermetallic phase present is detrimental. However, the toughness of a material may also decrease if the percentage of ferrite is high. This can be determined using the Feritscope®.
- 3d) ASTM A923 Test Method B (Charpy Impact Test) was determined to be the most suitable method out of the three test methods for predicting the presence of detrimental intermetallic phase in the material.

4. Evaluation by Ferrite and Hardness Measurement

- 4a) With increasing presence of intermetallic phase in the microstructure the volume percentage of ferrite content is reduced as most of the ferrite is converted into intermetallic phases.
- 4b) Since heat treatment “B” (Heat to 1950°F, hold 30 minutes, water quench, heat to 1550°F, water quench) samples exhibited ferrite of approximately 50% but had low toughness (Test Method B) and failed the Sodium Hydroxide Etch Test (Test Method A) and Ferric Chloride Corrosion Test (Test Method C), ferrite percent is not a good indicator of the presence or absence of detrimental intermetallic phase. Heat treatment “D” (Heat to 1950°F, hold 30 minutes, water quench, heat to 1550°F, hold 10 minutes, water quench) and “G” (Air cooled from 1950°F) samples also contained intermetallic phases and had low toughness but still had volume percentage ferrite of approximately 50%.
- 4c) As the ferrite content decreased there was an increase in the hardness. This is because as the ferrite content decreased as ferrite was converted into intermetallic phases which increased the hardness of the material. In general, the ferrite content

decreases with an increase in the hardness because the ferrite is converted into intermetallic phases which have a higher hardness than ferrite.

5. Evaluation by SEM and EDS

- 5a) In the Charpy Impact Test the entire sample which showed brittle fracture had low toughness and all samples with ductile fracture showed high toughness. The solution annealed samples from Foundry A, B, C and D which had high toughness showed ductile fracture. The heat treated samples from Foundry D underwent brittle fracture except for heat treatment “A” (Heat to 1950°F, hold 30 minutes, water quench) sample which was solution annealed at the University of Tennessee.
- 5b) The chemical composition of the intermetallic phase was determined by carrying out EDS on the material from Foundry D after subjecting it to different heat treatment schedules.
- 5c) The type of intermetallic phase present in the samples was identified by examining the microstructure under SEM.
- 5d) The SEM images of heat treatment “A” (solution annealed at the University of Tennessee), “G” (air cooled from 1950°F) and “H” (furnace cooled from 1950°F) samples indicated that as the toughness of these samples decreased in the order of “A”>“G”>“H”, intermetallic phase volume increased in the same order from zero in heat treatment “A” sample to a higher amount in heat treatment “H” sample. This result is directly influenced by the cooling rate.

6. Comparison of Volume Percentage Ferrite measured using the Feritscope® and ASTM E562 Manual Point Count

- 6a) From the comparison made between the Feritscope® and ASTM E562 Manual Point Count it was concluded that the Feritscope® is the most suitable method from calculating the volume percentage of ferrite followed by ASTM E562 Manual Point Count method.

6b) Feritscope® is more robust compared to ASTM E562 Manual Point Count which also gives similar results because it is easy to operate, eliminates operator bias and there are minimal requirements for specimen preparation.

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Appendices

Appendix A - ASTM Specifications

1. **ASTM A 890M – 99:** “Standard Specification for castings, Iron-Chromium-Nickel-Molybdenum Corrosion Resistant, Duplex (Austenitic/Ferritic) for General Application”
2. **ASTM A800/A 800M – 91:** “Standard Practice for Steel Casting, Austenitic Alloy, Estimating Ferrite Content”
3. **ASTM A799/A 799 M – 91:** Standard Practice for Steel Castings, Stainless, Instrument Calibration, for Estimating Ferrite Content”
4. **ASTM A781 M – 94a:** “Standard Specification for Castings, Steel and Alloy, Common requirements for General Industrial Use”
5. **ASTM A 370:** “Standard Methods and Definitions for Mechanical Testing of Steel Products”
6. **ASTM A923 – 94:** “Standard Test for Determining the Detrimental Intermetallic Phase in Wrought Duplex (Austenitic/Ferritic) Stainless Steels”
7. **ASTM G48 – 92:** “Standard Test Method for Pitting and Crevice Corrosion Resistance of Stainless Steels and Related Alloys by use of Ferric Chloride Solution”
8. **ASTM A 262 – 93a:** “Standard Practice for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels”
9. **ASTM G 108 – 94:** “Standard Test Method for Electrochemical Reactivation (EPR) for Detecting Sensitization of AISI Type 304 and 304L
10. **ASTM E18 – 03:** “Standard Test Method for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials

11. **ASTM E562 – 02:** “Standard Test Method for Determining Volume Fraction by Systematic Manual Point Count

Appendix B

Table 12: Volume % Ferrite of Foundry Solution Annealed Samples from Foundry A, B, C and D

Foundry	Specimen ID	Ferrite Number	Volume % Ferrite
Foundry A	285	66.6	47.2
		68.7	48.4
		63.1	45.3
		66.3	47.1
	331	62	44.7
		59.4	43.3
		62.4	44.9
		61.2	44.3
	144	92	61.2
		54	40.3
		52.9	39.7
		54.9	40.8
Foundry B	S1507	77.3	53.1
		87.1	58.5
		83.1	56.3
		88.5	59.3
	S1508	66.6	47.3
		64.3	45.9
		64.4	46.1
		64.8	46.2
Foundry C	45633	71.8	50.1
		76.8	52.8
		76	52.4
		76.5	52.7
	41414	70.2	49.2
		69.6	48.9
		73.4	50.9
		71.4	49.9
	41374	70.9	49.6
		78.1	53.5
		76.7	52.8
		75.1	51.9
	41544	76.6	52.7
		70.3	49.3
		73.2	50.9
		63.7	45.6
	44073	69	48.5
		62.4	44.9
		63.8	45.7
Foundry D	04J142	102.5	66.9
		105	68.3
		96.6	63.7
		98.3	64.6

Table 13: Volume % Ferrite of Heat Treated Samples from Foundry D

Foundry	Specimen ID	Ferrite Number	Volume % Ferrite
Foundry D	04J142 - A	99	65.1
		91.9	61.1
	04J142 - B	57.5	42.2
		60.9	44.1
	04J142 - C	34.4	29.5
		43.7	34.6
	04J142 - D	79	54.1
		81.8	55.6
	04J142 - E	45	35.3
		38.3	31.6
	04J142 - F	36.2	30.5
		35.6	30.2
	04J142 - G	81	55.1
		77.8	53.4
		83.4	56.5
		76.9	52.9
	04J142 - H	3.6	12.5
		3.9	12.8
		4.1	12.8
		3.6	12.6

The volume percentage of ferrite was calculated using the formula:

$$\text{Volume \% of Ferrite} = 0.55 * (\text{Ferrite Number}) + 10.6$$

The ferrite number was measured using Feritscope® on all the samples which had undergone charpy impact test. The values in Table 14 are the average of the ferrite number measured in all the samples.

Appendix C

Table 14: Hardness Values measured using Rockwell B and Rockwell C on Foundry Solution Annealed samples from Foundry A, B, C and D

Foundry	Specimen ID	HRB	HRC
Foundry A	285	104.1	22.9
		104.1	22.3
		103.6	22.3
	331	103.3	21.2
		103.2	22.1
		102.5	22.1
	144	83.1	19.7
		96.5	17.8
		102.6	19.1
Foundry B	S1507	84.7	24.1
		100.2	24.6
		103.5	24.5
	S1508	89.4	21.8
		96.9	21.7
		102.6	22.3
Foundry C	45633	102.4	20.8
		103.3	21.2
		102.5	20.5
	41414	72.6	19.3
		91	20.7
		102.2	21.1
	41374	101.9	19.4
		102.2	19.5
		102.5	21.5
	41544	92.9	20.4
		100.3	20.2
		101.9	22.2
	44073	100.8	18.1
		101	19.2
		101	19.6
Foundry D	04J142	100.9	25.1
		103.7	26.6
		103.9	24.7

Table 15: Hardness Values measured using Rockwell B and Rockwell C on Heat Treated samples from Foundry D

Foundry	Specimen ID	HRB Scale	HRC Scale
Foundry D	04J142 - A	97.6	20.9
		99.9	20.2
		99.6	20
	04J142 - B	98.5	22.1
		101.7	28.3
		105.7	24.4
	04J142 - C	106.3	30.7
		107.1	30.4
		104.9	31.4
	04J142 - D	101.1	20.9
		102.6	20.4
		101	19.1
	04J142 - E	100.1	25.7
		103.1	27.3
		105.1	27.8
	04J142 - F	109.4	30.7
		107.3	28.5
		109.1	30.5
	04J142 - G	104.4	29.7
		106.9	28.5
		107.4	27.8
	04J142 - H	105.5	33.5
		108.5	34.4
		109.3	34.6

Three values were measured on HRB and HRC scale on all the samples that were selected to undergo the hardness test. The values in Table 15 are the average of the hardness values measured on Rockwell B and Rockwell C scale.

Appendix D

SEM Images on Foundry Solution Annealed Samples from Foundry A, B and C

Figure 72: Backscatter SEM image of 285, 500x

Figure 73: Backscatter SEM image of 331, 500x

Figure 74: Backscatter SEM image of 144, 500x

Figure 75: Backscatter SEM image of 41374, 500x

Figure 76: Backscatter SEM image of 41544, 500x

Figure 77: Backscatter SEM image of 44073, 500x

Figure 78: Backscatter SEM image of 41414, 500x

Figure 79: Secondary Electron SEM image of 45633, 1000x

Figure 80: Secondary Electron SEM image of S1508, 1000x

Figure 81: Secondary Electron SEM image of S1507, 1000x