CHARACTERIZATION OF CURRENT PRODUCTION AOD+ESR

ALLOY 625 PLATE

P. Ganesan, R. H. Boswell, G.D. Smith and J.R. Crum

Inco Alloys International, Inc.
3200 Riverside Drive
Huntington WV 25705

Abstract

INCONEL® alloy 625 has excellent fabricability because of its very high ductility (over 50% elongation) even at yield strengths as high as 414 MPa (60 ksi). Alloy 625 also has the unique combination of strength, ductility and versatile corrosion resistance that it has found many applications in the chemical process industry. It is melted in the electric arc furnace (EAF) and transferred to the argon oxygen decarburization (AOD) vessel for refining. It is further refined by electroslag remelting (ESR). Over the last 25 years many improvements have taken place in the AOD+ESR processing that it was decided to characterize the current production plate produced by the AOD+ESR process for its microstructure and mechanical properties. Also after fabrication it is important to restore the alloy's strength and corrosion resistance to its full potential. In addition to the characterization data, this paper will therefore also cover the results of a study conducted to develop an optimum post-weld heat treatment to retain high strength and corrosion resistance.

Introduction

The development of any new alloy by traditional methods, requires years of lab work to select an optimum composition for scale-up to commercial size ingots. In addition, developing the methods of production from ingot to useful forms, and obtaining physical and mechanical properties to code qualify the material, could be another long and arduous process. The cost of such a development could run into millions of dollars by the time the alloy is successfully marketed. The development of alloys in the 90's has been predominantly related to upgrading the alloys already in existence with specific changes in composition and/or method of production for specific customer driven applications to supply a reliable product on time and at low cost (1). For example INCONEL alloy 601GC® was developed for furnace roller application with grain control for better strength at higher temperatures than alloy 601 (2). INCOLOY® alloy 803 was developed with higher nickel and chromium contents than alloy 800HT® for better stress rupture strength and superior oxidation and carburization resistance and is currently being used as internally finned tubes for ethylene pyrolysis application (3). The

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processing of alloy 617 was modified to obtain good grain size control for better fatigue strength for land based gas turbine hot gas path components (4).

Following alloy 718, alloy 625 is the second largest selling superalloy for a variety of diverse applications from aerospace to petrochemical and chemical process industries. INCONEL alloy 625 is a solid solution strengthened nickel-chromium-molybdenum-niobium alloy that is used for its high strength, excellent fabricability and weldability and outstanding corrosion resistance. General information about the alloy's fabricability, weldability and its physical and mechanical properties can be obtained from reference (5). The versatile corrosion resistance of alloy 625 in various aqueous and chemical processing environments has been presented earlier (6).

The argon-oxygen-decarburization (AOD) process was invented initially for stainless steel making but was quickly adopted by IAII and others for nickel base alloys. Electroslag remelt process (ESR) was introduced for better control of composition and microstructure for high performance materials. The AOD+ESR process has been used at IAII for the last 25 years. Over the years both AOD and ESR processes have improved in many ways. Therefore, it was decided to characterize the current production plate product produced by AOD+ESR process. This paper characterizes the microstructure, and mechanical properties of alloy 625 plates produced by the AOD+ESR processes.

Alloy 625 has a unique combination of high strength (over 60 ksi yield strength) with ductility (over 50% elongation) and versatile corrosion resistance (36 mpy maximum corrosion rate in the standard ASTM G28A test). Its balance of properties led to its wide acceptance in the chemical process industry. Because of its relative ease of fabrication, it is made into a variety of components for plant equipment. Some of the applications which require high strength and corrosion resistance are bubble caps, reaction vessels, distillation columns and heat exchangers. The challenge faced by fabricators is to retain the alloy's high strength and corrosion resistance by giving a suitable post fabrication heat treatment. In addition to the characterization of the microstructure and mechanical properties, this paper includes the results obtained on the development of an optimum post-weld heat treatment for alloy 625 that would maintain its high strength and corrosion resistance.

Experimental

Materials and Composition

Table 1 gives the composition of 18 heats of alloy 625 produced by AOD+ESR process and used for this metallographic evaluation. Of the 18 heats, 12 had an average nitrogen content of 0.02% and the other six heats had a nitrogen content of 0.03%. Over the years, improvements to the AOD process include chemistry control, refining procedures, AOD gas control and teeming practice. Similarly, continuous improvements have been made in the ESR process such as slag/metal chemistry control resulting in improved ingot surface and its metallurgical quality. Table 2 gives the average composition of about 175 heats produced subsequent to the metallographic evaluation study. To get an idea of the statistical variation in different elements, standard deviation is also included, as this should provide an idea of the consistency of the product.

Metallography

The plates of alloy 625 from the heats in Table 1 were evaluated by obtaining samples from the middle of all the four sides of the plate. Longitudinal sections were mounted and polished, and were evaluated for microcleanliness (oxides, nitrides and banding) in the unetched and etched condition. A 0.5% bromine plus methanol solution was used for etching. The etching procedure involved initial cleaning of the sample by immersion in full strength reagent grade hydrochloric acid for about 10 seconds. Samples were then rinsed in methanol and immersed in 0.5% bromine methanol mixture for about 30 seconds. After etching, all samples were cleaned in methanol ultrasonically for about 3 minutes and air dried.

Mechanical Properties

Samples for mechanical properties evaluation were obtained from the plates evaluated for microstructure. Full size specimens were machined for room temperature tensile tests. In addition to room temperature tensile tests, bend tests were done to check the bend ductility of the AOD+ESR processed alloy 625. For comparison mechanical properties data obtained from current production alloy 625 is also presented in the results section.

Determination of Optimum Post-weld Heat Treatment

Alloy 625 is normally produced to a final anneal of 1038°C (1900°F) having a minimum of 414 MPa (60 ksi) yield strength and 36 mpy corrosion resistance in the ASTM G28A (Streicher Test) corrosion test. The 1038°C (1900°F) anneal stabilizes the alloy by the formation of NbC and reduces the formation of M_6C or $M_{23}C_6$ type carbides in subsequent exposure in the sensitization range of 649- 871°C (1200-1600°F). A typical practice used by fabricators is to lower the annealing temperatures, 927-954°C, (1700-1750°F) to maintain mechanical strength; however, corrosion properties can be compromised by using a lower annealing temperature. Therefore, to determine the optimum post-weld heat treatment, post-weld annealing temperatures ranging from 954-1177°C (1750-2150°F) were evaluated, followed by a 650°C (1200°F) post-weld aging temperature for times ranging from 0 to 24 hours. Welded corrosion coupons and mechanical test specimens were produced by butt welding two 19 mm (0.75") alloy 625 plates together using the gas metal arc welding process. The experimental details of the weld parameters and the welding process have been presented elsewhere (7).

Results

Composition

Table 1 shows the minimum, maximum, average and standard deviation of pertinent elements obtained from the compositions of 18 heats that were used for metallographic evaluation. Of the 18 heats evaluated, 12 had a nitrogen content of 0.02% and six had a nitrogen content of 0.03%. Table 1 statistics indicates that the heat to heat variation in all elements is very minimal. A statistical evaluation of over 175 heats produced by the AOD+ESR process over a period of time were conducted to get a quantitative information on the variation in different elements. Table 2 gives the average composition of 175 heats for all the elements. The standard deviation also shown in Table 2 indicates the heat to heat variation to be minimal for all the elements with the exception of iron. An assessment of the process capability potential can be obtained for all the elements by the calculation of a statistic called Cp (8). The process capability index Cp, assuming the process follows a normal distribution, is calculated as: (USL-LSL)/6s where USL and LSL are upper and lower specification limits and s is the standard deviation. As examples, assuming the limiting composition for chromium, niobium and molybdenum to be 21 - 23%, 3.15-4.15% and 8.0-10.0% respectively and using the standard deviation for the 3 elements from Table 2, the Cp values can be calculated to be 2.8, 2.1 and 3.0 respectively. Cp values of over 1.3 are considered good.

Table 1 Average Composition of AOD+ESR Processed Alloy 625 Heats Produced for Metallographic Analysis

	C	Ni	Fe	Cr	M0	Nb	Si	Al	Ti	N
MAX	.027	60.81	4.57	21.67	9.08	3.49	0.14	0.38	0.34	0.031
MIN	.018	60.31	4.32	21.54	9.01	3.42	0.05	0.23	0.20	0.020
AVG	.023	60.59	4.45	21.62	9.05	3.46	0.10	0.30	0.27	0.024
STD	.003	0.16	0.09	0.03	0.02	0.02	0.04	0.04	0.04	0.004

Table 2 Average Composition of about 175 Heats of AOD+ESR Processed Alloy 625 Heats

HEAT #	C	Ni	Fe	Cr	Mo	Nb	Si	Al	Ti	N
Average	0.022	61.12	3.56	21.65	9.05	3.44	0.17	0.18	0.26	0.022
Std dev	0.003	0.72	0.62	0.12	0.11	0.08	0.05	0.04	0.03	0.004

Microstructure

The metallographic samples were first evaluated for nitrides per ASTM E45 test method. Number of nitride particles in a field of view (0.51 mm^2 at 100X) is given a "D" rating. According to E45 test procedure if the number of particles are 1, 4, 9, 16, 25, 36, 49 the "D" rating would be 0.5, 1.0, 1.5, 2.0, 2.5, 3.0 and 3.5 respectively. In general the nitride rating varied between D2 to D3.5. If there are stringers longer than 0.5 mil at 100X then they are normally given B rating in addition to D rating. Stringer length of less than 3 mils is given as B0.5 and over 3 mils but less than 7 mils the rating is B1.0 and so on (see ASTM E45 test procedure). Figures 1 and 2 show examples of microstructures before and after etch for plate samples containing 0.02 and 0.03% nitrogen respectively. It is clear that the higher nitrogen containing heat tends to form nitrides as stringers.

Detailed analysis of metallographic data is shown in Table 3. The results are shown for AOD+ESR heats with different melt rates, fluxes, nitrogen levels and number of samples evaluated for each melt rate and flux used. Oxide type inclusions were not seen and therefore all the samples were given a rating of 0.5 and not reported in the Table. The evaluation was done in terms of stringer like nitrides, nitride particles and banding. For a given flux (a) and melt rate, A, the higher nitrogen containing heats showed relatively higher number of samples with stringer rating of B1.5 and a nitride rating as high as 3.5

Mechanical Properties

Table 4 shows maximum, minimum, the average and standard deviation of room temperature tensile data obtained from 48 plates of alloy 625 heats that were evaluated for microstructure. Cpk index, another process control statistic similar to Cp and considered as a measure of the process performance, can be calculated as $(X_{avg}\text{-LSL})/3s$ where the numerator is the difference between the average of the measurements and the lower specification limit and s is the standard deviation. For the data in Table 4, Cpk-1 using the minimum limits of 414 MPa (60 ksi), 827 MPa (120 ksi) and 30% for yield strength, tensile strength and elongation respectively have been calculated and shown in Table 5. As before a value of 1.0 indicates that the process is capable and a value of 1.3 is desirable. In this case the values of Cpk-1 for YS, TS and El were 0.40, 0.83 and 1.73 indicating that either the process was not centered or the process was

Table 3 Metallographic Evaluation Of Alloy 625 Plates Produced By AOD + ESR Processes

N_2	Increasing Melt Rate (flux)	Total Samples Evaluated	Stringer			Nitride								Banding				
			B0.5	B1	B1.5	D0.5	D1	D1.5	D2	D2.5	D3	D3.5	D0	D1	D2	D3	Cl
0.02	A (a)	34	21	12	1	0	0	0	3	7	24	0	28	6	0	0	0
	B (a)	27	12	13	2	0	0	0	5	4	18	0	14	8	1	0	1
	B (b)	58	45	13	0	0	0	0	2	9	38	9	48	10	0	0	0
	B (c)	4	4	0	0	0	0	0	0	0	2	2	3	1	0	0	0
0.03	A (a)	35	22	12	1	0	0	0	0	3	23	9	22	13	0	0	0
	B (b)	3	1	2	0	0	0	0	0	1	2	0	0	2	0	0	0

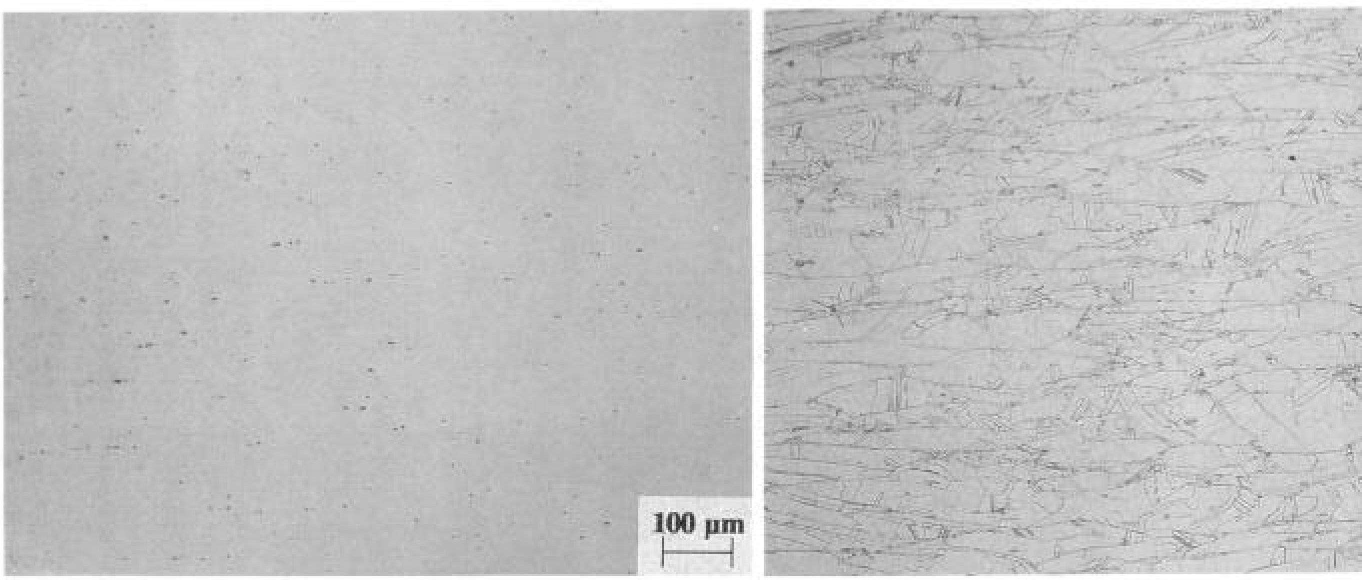

Figure 1 . Microstructures from a longitudinal section of alloy 625 plate from heat containing 0.02% nitrogen in th
etched condition (100X).

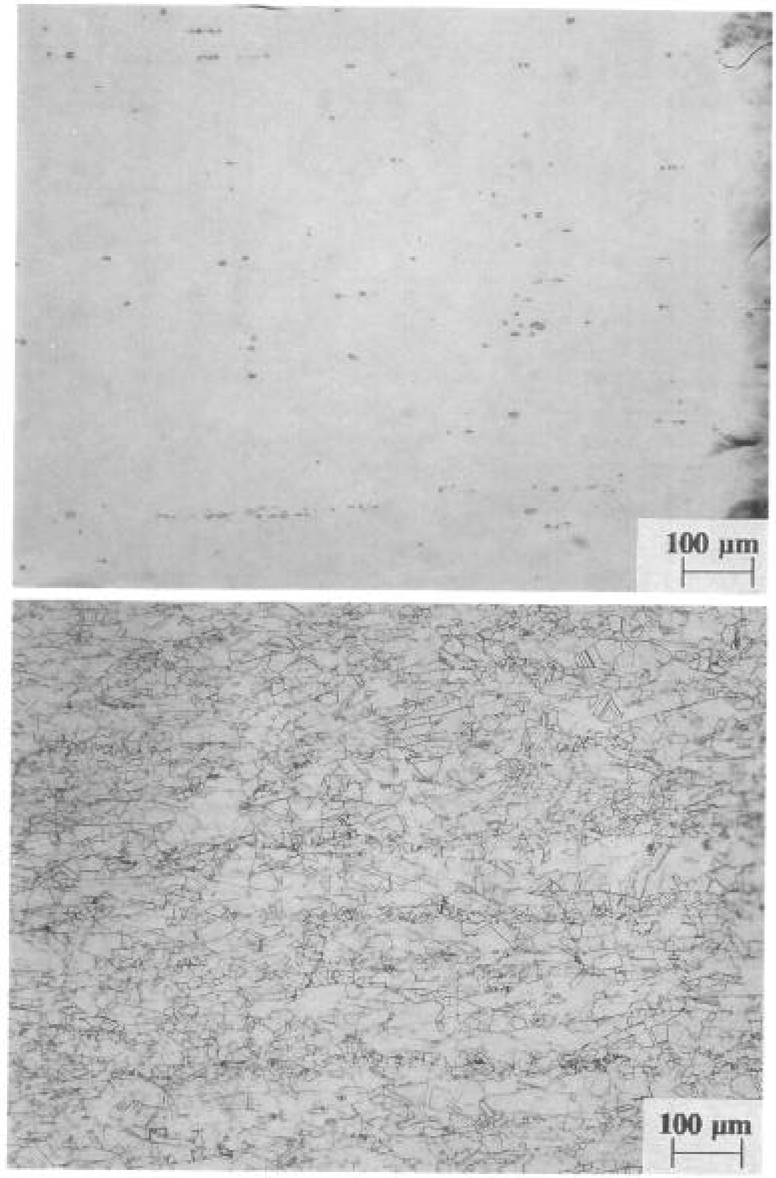

Figure 2. Microstructures from a longitudinal section of alloy 625 plate from heat containing 0.03% nitrogen in the unetched and etched condition (100X).

not in statistical control with values of Cpk for YS and TS of <1. Note that there was only one point below 60 ksi minimum yield strength limit; however, there were several unusually high yield strength values. Several unusually high yield strength points indicate that the process was more likely not centered. If we assume the limits to be 55 ksi YS, 110 ksi TS and 30% elongation, Cpk-2 values for YS, TS and El were 0.59, 1.58 and 1.73 respectively. For these lower limits, only YS is out of control. Note that there are no values that are below the limits. Now one can examine the data and if any assignable causes can be found the abnormal data can be removed and the data set reanalyzed. In this case a few abnormally high data points were removed as these were believed to be due to a number of reasons from annealing furnace malfunctions to a problem encountered during rolling. The Cpk-1 and Cpk-2 were

Table 4 Average Room Temperature Tensile Properties Of 48 Plates From Heats Evaluated For Microstructure

	MPa	ksi	Mpa	ksi	El %
Maximum	660.5	95.8	986.0	143.0	51.2
Minimum	408.9	59.3	861.9	125.0	36.5
Average	488.32	70.82	903.91	131.10	46.15
Std Dev	61.51	8.92	30.73	4.46	3.12

Table 5 Cpk Indices Calculated From the Average Of Room Temperature Tensile Properties (48 samples)

	MPa	ksi	Mpa	ksi	El %
Average	488.32	70.82	903.91	131.10	46.15
Std Dev	61.51	8.92	30.73	4.46	3.12
Limits 1	414	60	827	120	30
Cpk-1	0.40	0.40	0.83	0.83	1.73
Limits 2	379	55	758	110	30
Cpk-2	0.59	0.59	1.58	1.58	1.21
Evaluation of the Averages After Removing Data Points With Assignable Causes					
Average	471.27	68.35	896.69	130.05	46.95
Std Dev	43.64	6.33	24.89	3.61	2.22
Cpk-1	0.44	0.44	0.93	0.93	2.54
Cpk-2	0.70	0.70	1.85	1.85	2.54

recalculated and are shown in Table 5. The indices for YS, TS and El were all improved. Cpk value for YS was still low. The reason for Cpk to be low is because the process spread is high as indicated by the large standard deviation, especially for YS. After removing the high yield strength points the standard deviation reduces some what for all the three measurements namely YS, TS and El. Thus the Cpk analysis forces one to look at the data and look for different directions to find assignable causes.

Based on the analysis of the tensile data obtained from plates used for microstructural evaluation, several process oriented actions were taken to reduce the standard deviation (process spread). The tensile data obtained for current production plates are shown in Table 6. At the bottom of the Table 6, the Cpk-1 and Cpk-2 values for YS, TS and elongation are also shown. Note that the standard deviation for YS, TS and elongation have been improved from the initial values of 8.92, 4.46, 3.12 to 4.03, 2.34 and 1.81 for current production plates. Now there are no values below 60 ksi yield strength and no unusually high values for yield strength either. This improvement has resulted in much better Cpk indices for current production plates versus earlier production (Tables 4, 5 and 6). To obtain a value of one or greater for Cpk of yield strength with 60 ksi limit (Cpk-1) we need to further reduce the standard deviation to at least a value of 2 or increase the average yield strength to about 72 ksi for a standard deviation of 4. For a structure sensitive property, such as yield strength, it may be difficult to achieve and an average yield strength of over 70 ksi; this YS level could result in plate levelability problems. Currently efforts are continuing to improve from both fronts.

Figure 3 shows the bend test results for AOD+ESR processed samples from plates that were descaled by pickling after anneal. 4.7 mm (0.187") thick samples, they all passed the 2-T

Table 6 Tensile Properties of Current Production Plate Samples

	Yield Strength		Tensile Strength		Elongation
	MPa	ksi	MPa	ksi	%
	444.7	64.5	870.1	126.2	52.6
	444.7	64.5	874.3	126.8	52.2
	436.5	63.3	868.8	126.0	53.3
	442.0	64.1	874.3	126.8	53.6
	475.1	68.9	900.5	130.6	50.8
	485.4	70.4	902.6	130.9	50.5
	442.0	64.1	855.0	124.0	52.6
	464.7	67.4	899.8	130.5	52.6
	413.7	60.0	876.4	127.1	55.4
	484.7	70.3	878.4	127.4	51.8
	470.2	68.2	912.2	132.3	52.8
	527.5	76.5	894.3	129.7	49.0
	464.7	67.4	899.1	130.4	51.3
	502.6	72.9	889.5	129.0	51.0
	439.2	63.7	879.8	127.6	52.0
	473.0	68.6	886.7	128.6	49.2
	434.4	63.0	873.6	126.7	51.0
	488.2	70.8	878.4	127.4	48.6
	426.1	61.8	881.2	127.8	51.6
	451.6	65.5	926.0	134.3	47.4
	426.1	61.8	890.8	129.2	50.6
Average	458.91	66.56	886.27	128.54	51.42
Std Dev	27.78	4.03	16.10	2.34	1.81
Cpk-1	0.54	0.54	1.22	1.22	3.95
Cpk-2	0.96	0.96	2.65	2.65	3.95

(samples bent to a radius equivalent to twice the thickness of the plate) bend tests indicating that the presence of nitrides did not affect bend ductility.

Post-Weld Heat Treatment Study

Figure 4 shows the effect on the ASTM G28A corrosion rates of various annealing temperatures followed by an aging treatment at 650°C (1200°F) for times ranging from 0 to 24 hours. The results show that the ASTM G28A intergranular corrosion rates of the annealed material are lower than material in the as-welded condition except for the lowest annealing temperature, 950°C (1750°F), which had a significantly higher corrosion rate of >3.1 mm/yr (120 mpy). For a given annealing temperature, the corrosion rate increases with aging time at 650°C (1200°F).

Figure 5 shows the variation in yield strength with annealing temperature followed by aging at 650°C (1200°F). Without aging, the yield strength decreases with increasing annealing temperature. For a given annealing temperature the yield strength increases with aging time.

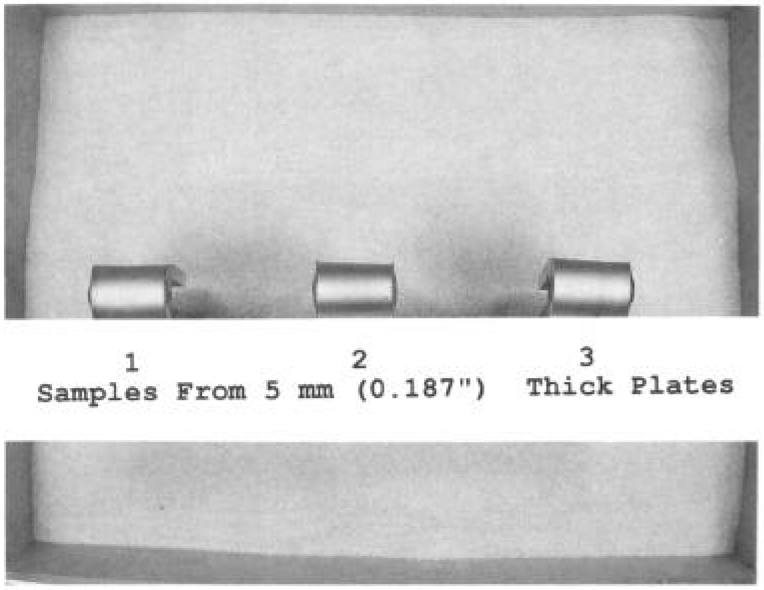

Figure 3. 2T U-bend photomacrograph of plate samples from different heats of AOD+ESR processed alloy 625.

Discussion

The microstructural analysis of alloy 625 samples showed that the higher nitrogen levels in the alloy result in higher amounts of nitrides and they are distributed in the form of stringers more frequently. It is important to control nitrogen to as low a level as possible as a higher level of nitrogen resulting in stringers of nitrides could act as stress raisers in the material and limit the fatigue strength of the alloy. It has been shown that the fatigue strength improves by 2 orders of magnitude in VIM processed alloy 625 compared to the air melted version (9). This is important for fatigue limited applications such as bellows. The improvements made in the AOD and ESR processes as mentioned earlier have resulted in better ingot surfaces and improved the overall metallurgical quality of the ingots. This has resulted in an alloy 625 plate product with a microstructure that has fewer stringers.

The current AOD+ESR practice is capable of producing heats with extremely low heat to heat variations in compositions of all the elements with the exception of iron which is a common contaminant in IAII melt system. It has also been demonstrated that heats with nitrogen content $<0.03\%$ can be produced with current practice. An analysis of the mechanical properties of samples from heats used for microstructural evaluation indicated wide variation in yield and tensile strengths resulting in high standard deviation. A program was undertaken to reduce the variation by tighter process control. To get an idea of the improvement in the process, for a 379 MPa (55 ksi) yield strength limit an increase in the Cpk value from 0.70 before any control to a Cpk of 0.96 after the tighter control (Tables 4 and 5) is equivalent to a 10 fold decrease in the reject rate (10). The substantial improvement in the mechanical properties especially the decrease in standard deviation of yield strength by 50% indicates that the changes introduced in the process control are taking effect. Additional improvements are on the way to reduce the process spread still further.

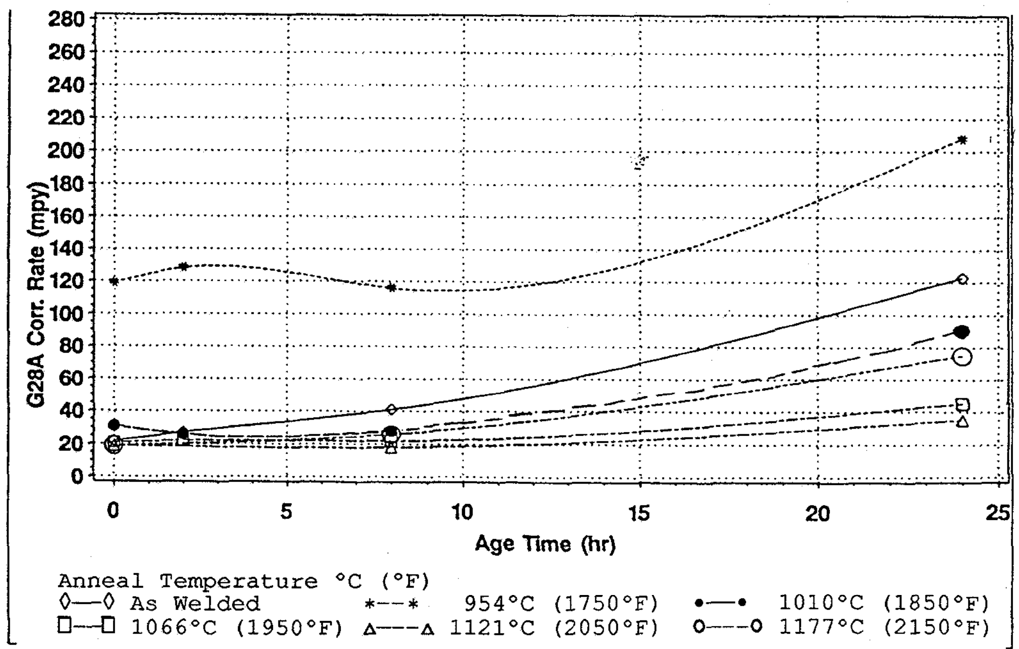

Figure 4. Alloy 625 post-weld anneal and aging study showing ASTM G28A corrosion rate versus aging time at an aging temperature of 650°C (1200°F) after annealing at indicated temperatures for 1 h.

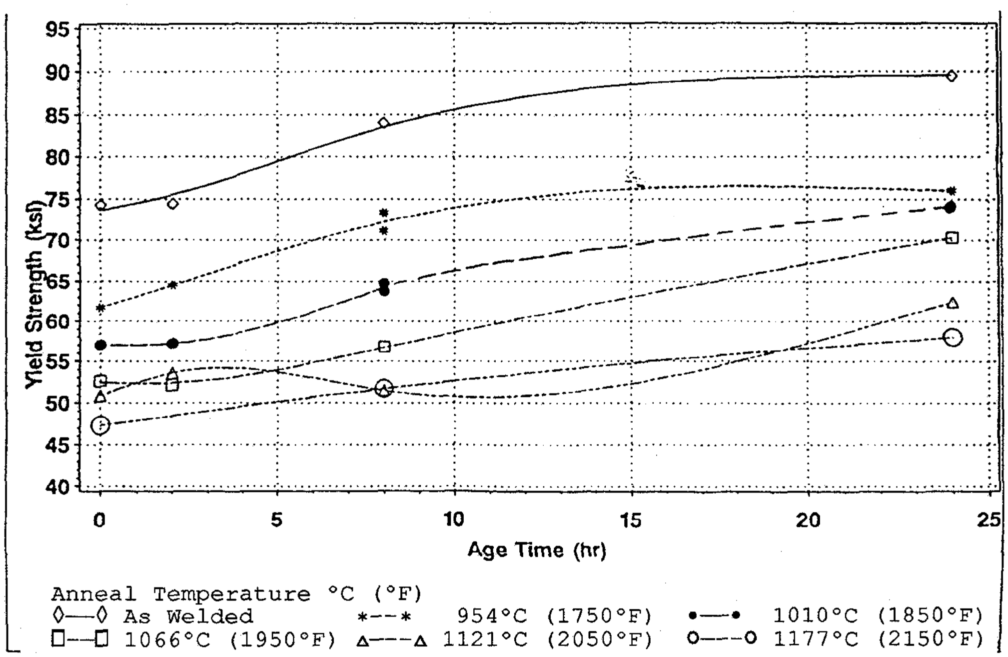

Figure 5. Alloy 625 post-weld anneal and aging study showing yield strength versus aging time at a temperature of 650°C (1200°F) after annealing at indicated temperatures for 1 h.

The results of the post-weld heat treatment study shown in Figures 8 and 9 indicate that the optimum post-weld treatment should be 1010°C (1850°F)/1 h/AC followed by 650°C (1200°F)/8 h/AC to obtain both 414 MPa (60ksi) min yield strength and less than a 0.9 mm/yr (36 mpy) corrosion rate. The lower temperature anneal, 950°C (1750°F), was found to increase the corrosion rate. Similar observations have been made in a welded pipe application where a stabilization anneal at 940°C (1725°F) given to alloy 625 was found to be inadequate to meet the corrosion requirements of NACE MR-0175 (11). The possibility of the formation of Laves during weld solidification is known (12). It is thought that the intermetallic phases such as the Laves phase that is formed during the weld solidification do not dissolve during the 950°C (1750°F) anneal. This leads to a high corrosion rate in the vicinity of these particles as the matrix in these areas is somewhat depleted in niobium which provides resistance to sensitization, and molybdenum and chromium that provide resistance to pitting and general corrosion resistance.

The higher temperature anneal reduces the yield strength to less than the minimum 414 MPa (60 ksi) strength requirement. Recent work by Cortial et. al. (13) on welded alloy 625 plates has shown that exposure to temperatures as high as 950°C (1750°F) can have a negative impact on mechanical properties. Therefore the higher temperature anneal given for corrosion resistance should be followed by a 650°C (1200°F) aging to regain strength by precipitating Ni_3Nb (γ'').

Conclusions

Based on the results obtained, the following conclusions can be drawn about the air melted and electroslag remelted alloy 625 compared to vacuum induction melted and electroslag remelted alloy 625:

1. The microstructure of the AOD+ESR processed alloy 625 show relatively higher amounts of nitrides and carbo-nitride particles for heats containing 0.03% nitrogen compared to 0.02%.

2. With all the improvements in the AOD+ESR processing, it has been possible to produce alloy 625 with minimal heat to heat variations in the composition and also control nitrogen to <0.03% nitrogen.

3. Considerable improvement has been made in the plate processing to reduce the variation in the as-annealed tensile properties resulting in a 50% reduction in the process spread (and the standard deviation).

4. Alloy 625 post-weld heat treatment can be modified by using a 1010°C (1850°F)/1 h/AC anneal followed by an aging treatment of 650°C (1200°F)/8 h/AC to meet both mechanical and corrosion property requirements rather than the 950°C (1750°F) post-fabrication anneal used traditionally for retaining strength but which reduces corrosion resistance.

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