SPRAY FORMED ALLOY 625 TUBULARS

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Abstract

Two series of Alloy 625 tubular preforms were spray deposited to evaluate the sensitivity of the alloy to spray deposition parameters. Variations in the metal flow rate, atomization pressure, substrate translation velocity, and melt size were made to determine their effect on the resulting preform microstructure, distribution and percent porosity, and tensile properties. All preforms were evaluated in the as-sprayed condition. Changing the gas to metal ratio (metal flow rate/atomizing gas pressure) did not result in significant variation in microstructure but did affect the amount of porosity in the preform. Porosity levels were generally greatest near the inner diameter of the preform and diminished through the remainder of the thickness. Preliminary tensile results from specimens located away from the inner diameter exhibited consistently high tensile strength. Bulk density measurements were calculated for the second series of preforms and demonstrated a dependence on processing parameters.

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Introduction

Alloy 625 is being used extensively in ship machinery system construction by the U.S. Navy. Because of its high material cost, its high manufacturing cost and its resistance to hot/cold forming, it is highly desirable to produce Alloy 625 components in net-shape or near-net shape preforms that require minimal working and/or machining. DTRC evaluated Alloy 625 piping produced by Osprey Metals, Ltd., to determine the feasibility of using spray forming in place of conventional ingot processing. The results of this earlier study (1) indicated that quality preforms could be produced and 10 and 20 cm piping successfully formed by standard and accelerated cold working schedules using the Roll Extrusion™ Process. To further evaluate the spray forming characteristics of Alloy 625, a series of 10 cm inner diameter cylindrical preforms were produced to determine the sensitivity of the microstructure to spray deposition parameters. Evaluation of the material in the as-sprayed condition was elected to establish properties and characteristics of the preform prior to a solution anneal heat treatment applied to samples in the earlier study.(1) Solution heat treatment of the preforms in the earlier work had little effect on the tensile properties and imparted a slight increase in tensile ductility.

Experimental Details

Thirteen different preforms of Alloy 625 were sprayed at the Osprey spray forming facility at David Taylor Research Center, Annapolis, MD. Starting material was bar stock purchased to ASTM B446. All starting stock materials met the chemical limits of this specification. For each run nitrogen was used to purge the chamber and the crucible during the melt cycle. Melt temperature was monitored using a type B thermocouple in an alumina sheath mounted to the side wall of the crucible. Prior to the beginning of the pour the high pressure atomizing gas and substrate rotation were started. For these runs the substrate was a mild steel mandrel with an OD of 10.2 cm and wall thickness of 1.5 mm. In the last run of the first series the mandrel was plasma coated with alumina to reduce heat conduction from the first material to be deposited. The mandrel was translated under the spray at a speed between 1.1 to 4.5 mm/s, while rotation speed was held at 240 rpm. In addition, the cover gas pressure was increased to maintain a near-constant metal flow rate during spraying. Heat-up times were approximately 12 minutes and spray times ranged from 40 seconds to 66 seconds. General spray forming parameters for the first series of preforms are exhibited in Table I. Run A was produced to establish baseline parameters for evaluation purposes. During this run, atomizing gas pressure was varied at the operator's discretion to establish a visually satisfactory spray and smooth preform deposit. The spray collector's translation motion was also controlled to establish a satisfactory and uniform deposit thickness. Runs B, C, and D were sprayed at increasingly higher temperatures to improve upon microporosity levels and cold conditions seen in run A. After producing a quality preform in run D, the metal deposition rate was reduced to evaluate parameter sensitivity in run E. Run F attempted to create additionally cool spraying conditions.

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TABLE I. Spray Deposition Process Parameters for First Series

RUN	MELT SIZE (kg)	ATOMIZING PRESSURE (bar)	DEPOSITION RATE (kg/min)	POUR TEMPERATURE (°C)	GAS/METAL RATIO (kg/kg)
A	20.2	8.2	37.8	1460	0.33
B	19.4	7.5	27.7	1340	0.43
C	19.5	7.5	27.8	1390	0.43
D	18.6	7.5	28.0	1450	0.42
E	18.6	7.5	19.3	1360	0.62
F	18.9	8.8	25.7	1400	0.56
G	26.3	8.8	23.2	1385	0.59
H	26.3	8.8	28.8	1425	0.48

Runs G and H had increased melt size by 40%. This permitted the spraying of a thicker preform in run G and a longer, thin preform in run H. Table II contains the general spray forming parameters for the second set of preforms. In this series of preforms atomization pressure and deposition rate were varied. A wide range of gas-to-metal ratios were investigated.

TABLE II. Spray Deposition Process Parameters for Second Series

RUN	MELT SIZE (kg)	ATOMIZING PRESSURE (bar)	DEPOSITION RATE (kg/min)	POUR TEMPERATURE (°C)	GAS/METAL RATIO (kg/kg)
I	19.0	8.6-11.7	15.8	*	0.81-1.11
J	19.2	9.3	30.3	1460	0.45
K	19.2	9.0	24.5	1476	0.56
L	18.6	9.0	35.0	*	0.39
M	18.8	9.0	32.3	*	0.43

* Indicates thermocouple failure

Each preform was sectioned and subjected to metallographic examination to determine grain size, porosity levels and microstructural features. Samples removed from one quarter of the distance from each end of the tubular preform were used to determine the porosity level and grain size. A LECO 2001 automatic image analyzer was used to make grain size and porosity level measurements. Grain size was measured in accordance with ASTM E112 while porosity area measurements were made on adjacent fields from the inner diameter to the outer diameter using the image analyzer on three random locations around the circumference. Mechanical properties were determined from 0.91 cm diameter tensile specimens removed from the midsection of the preform approximately one

half the distance from the inner and outer diameter. Bulk density measurements were performed on the second series of preforms using the technique specified in ASTM B311-58.

Results and Discussion

For each of the runs the major alloying elements were within the specified chemistry for Alloy 625 under ASTM B446 with only minor changes in percentages as a result of the induction melting and spray deposition process. There were more significant chemistry changes in tramp element levels, although none exceeded specification maximums. Chemical analysis for tramp elements of runs A through H appear in Table III. Nitrogen levels in the preforms generally ranged from five to ten times that found in the starting melt stock. Such a high level of nitrogen in an alloy system containing elements with a strong affinity for nitrogen is expected to have some impact on the microstructure and properties. Similarly, the oxygen content in the preforms were found to be three to five times that of the starting materials. However, in the worst case the oxygen level never exceeded 60 ppm and therefore was not considered a detriment. Other tramp element levels remained mostly unchanged.

TABLE III - Tramp Element Chemical Analysis of Alloy 625 Preforms

		PREFORM IDENTIFICATION							
	SPEC. (Weight %)	A	B	C	D	E	F	G	H
C	<0.10	0.031	0.028	0.027	0.054	0.055	0.046	0.032	0.026
Mn	<0.50	0.13	0.14	0.14	0.063	0.063	0.096	0.15	0.15
Si	<0.50	0.16	0.12	0.16	0.15	0.19	0.14	0.26	0.18
Al	<0.40	0.29	0.32	0.32	0.26	0.27	0.26	0.29	0.30
Ti	<0.40	0.18	0.18	0.19	0.25	0.25	0.22	0.19	0.21
S	<0.015	<0.001	<0.001	<0.001	<0.001	<0.001	0.007	0.003	0.004
P	<0.015	0.005	<0.002	0.003	0.011	0.012	0.004	0.009	0.010
N	--	0.088	0.121	0.126	0.090	0.083	0.061	0.094	0.055
O	--	0.0025	0.0038	0.0052	0.004	0.004	0.003	0.006	0.003

Each preform was dimensionally measured to determine its average thickness along its length. These results appear in Table IV. Generally the section thickness averaged approximately 2.5 cm, although there was some significant variation in run F and G. Run F had a slow translation speed resulting in a short thicker preform while run G had a longer thick section because of the greater charge weight. The thickest deposit was produced in run G where the maximum thickness of 3.48 cm was recorded. No significant metallurgical change was evident in the thicker preforms.

Table IV also presents a summary of the metallographic examination on runs A through H. The average grain size of the spray deposited preforms remained between 11 and 16 microns. Although there was not a large difference in average grain size among these runs, there were some notable microstructural differences. Runs B, C and D, those with the higher deposition rate and lower

TABLE IV - Dimensional and Metallographic Examination Results

RUN NUMBER	PREFORM THICKNESS (cm)	AVERAGE GRAIN SIZE (microns)	AVERAGE PERCENT POROSITY TOTAL	INNER (25%)	MIDDLE (50%)	OUTER (25%)
A	2.38	16	4.43	10.54	1.25	0.02
B	2.36	11	4.58	6.85	4.73	0.95
C	2.74	12	3.23	3.84	1.69	4.62
D	2.71	12	0.20	0.05	0.06	0.66
E	2.69	14	3.25	7.93	2.46	0.14
F	3.22	14	1.66	5.53	0.18	0.01
G	3.48	14	1.65	3.84	1.15	0.30
H	2.51	16	0.44	0.32	0.05	0.01

atomizing pressure, showed a wider variation in grain sizes and scattered locations in the inner half of the preform where solid spherical particles had embedded themselves in the solidifying microstructure. The percent area of porosity was calculated for each preform using a complete ring section removed from the preform. As exhibited in Table IV, the porosity level averaged less than 5% for the worst run and was as small as 0.2% in the best case. Porosity in the spray deposited runs generally was worst on the inner diameter of each preform, where the initial spray contacted the room temperature steel collector but improved as the deposition continued (See Figure 1). For example, runs A, F and H, which exhibited this trend, had at least 75% of their total porosity located in the inner 25% of their thickness. Porosity levels improved with increasing superheat (runs B, C, D) and with the use of a ceramic coated collector (run H vs.G) for an unheated collector condition.

Tensile results are incomplete due primarily to difficulties in removing and machining tensile specimens from each preform. On many of the finished tensile specimens either one side of the thread or the radius outside the round test section contained surface porosity defects and either could not be tested or failed outside of the test gage (including in the threads). These high porosity areas in the specimen grips occurred because the grips were machined from ends of the preform which would normally be removed before further processing. Table V contains the results of the tensile specimens that were found visually acceptable and produced an in-gage failure during tensile testing.

The yield and ultimate tensile strengths were quite uniform among the samples with respective averages of 405 and 881 MPa. These as-deposited strengths met the minimum Alloy 625 piping tensile properties. Ductility measurements of percent elongation and reduction in area showed more variation

than the tensile properties, however they met minimum piping draft specifications. In some specimens this variation may be attributed to porosity evident on fracture surfaces.

TABLE V - Tensile Test Results from As-sprayed Preforms

SAMPLE	0.2% OFFSET YIELD STRENGTH (MPa)	ULTIMATE TENSILE STRENGTH (MPa)	PERCENT ELONGATION	REDUCTION IN AREA (%)
B/1	420	882	37	30
C/1	413	889	49	37
C/2	407	868	48	58
D/1	400	882	45	45
D/1-1	407	896	47	46
D/2	400	882	37	36
E/1	407	875	51	45
H/2	386	854	51	42
AVERAGE	405	881	46	43
DRAFT SPEC.	379	758	30	--

General spray forming parameters for the second set of preforms were provided in Table II. These preforms were not sectioned and examined microstructurally, but instead were evaluated in terms of their bulk density values. This information is provided in Table VI. From these results it appears that higher deposition rates yield higher densities and that for Alloy 625 gas-to-metal ratios of around 0.5 are optimum.

TABLE VI. Bulk Density Measurements for Second Series

RUN	DENSITY (g/em3)	PERCENT DENSITY
I	7.562	89.8
J	8.318	98.7
K	8.377	99.7
L	8.337	99.1
M	8.292	98.7
WROUGHT	8.416	--

Conclusions

Variations of atomizing gas pressure in the range of 7.5 to 8.8 bar either independently or in combination with variations in deposition rates from 19.3 to 37.8 kg per minute did not have a dramatic affect on the resulting microstructure or tensile properties in regions of Alloy 625 preforms away from the inner-diameter porosity layer. Porosity appears to be the preform characteristic most sensitive to minor variations in processing parameters. Porosity levels in the as-deposited preform can be maintained at levels that have minimal effect on properties. Generally the greatest porosity appears adjacent to the inner diameter of the preform. This porosity appears to be controllable to percentages less than 1% with appropriate attention to processing details and collector condition. Nitrogen levels in nitrogen atomized Alloy 625 preforms are significantly higher than starting melt stock. This nitrogen pickup likely has a positive effect on the tensile strength exhibited in these materials.

References

1. Angela L. Moran and William A. Palko, J. Metals, Vol. 40, No. 12, December 1988, p. 12 - 15.

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