P/M ALLOY 625 SPECIAL COMPOSITION

FOR REDRAW-STOCK WELDING WIRE

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Abstract

P/M Alloy 625 provides specific advantages for critical service oil and gas industry parts. In this study, two P/M versions were compared with cast and wrought 625 when processed as redraw stock for welding wire. A modified P/M 625 with 11 Mo-0.1 Fe-0.005C is especially suited for overlay-cladding of 4130 steel valve bodies. The low Fe content provides for the dilution of Fe from the steel during welding and the higher Mo content improves the pitting resistance of the 625 overlay. A refined grain size with no precipitation of carbides was observed and tensile properties met requirements. Best corrosion resistance was obtained in 10 pct FeCl₃ • 6H₂O solution and in a simulated oil and gas environment. No cracking was observed in any of the U-bend specimens exposed for 14 days at 232°C (450°F) in a solution containing 25 pct NaCl, 0.5 pct acetic acid and 1 g/l sulfur, pressurized with 100 psi H₂S. Microscopic examination of autogenous welds revealed that the grain size of the CW Alloy 625, in the base metal, was five times larger than the P/M versions. The grain size of the high Mo-low C P/M version was larger than that of the conventional P/M Alloy 625 because of formation of small carbides in the latter which restricted grain growth.

Introduction

The primary benefits of rapidly solidified P/M material^{1, 2} are improved homogeneity and the opportunity for a refined grain size or distribution of dispersoids. These benefits would be particularly appropriate for the processing of Alloy 625 redraw-stock welding wire since conventional cast-wrought (CW) product can be prone to segregation and fabrication problems. For oil and gas industry service, Alloy 625 powder has been an effective way of providing the required corrosion resistance via the HIP cladding of the interior surface of well-head parts such as safety-valve bodies of 4130 steel.³ An alternate procedure is to use CW or P/M 625 wire for overlay welding with a computer-controlled head inside the valve body, and this paper provides a comparison of the CW product, an equivalent P/M composition and a high Mo-low Fe variant of P/M 625. The latter is most appropriate because the dilution of Fe from the 4130 steel during

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welding added onto the usual Fe content of 625 degrades the expected corrosion resistance of the 625 composition. Also, a higher Mo content improves the pitting resistance of the 625 overlay to the corrosive environment passing through the valve body.

Materials and Procedures

The powders were produced by argon atomization of VIM heats, screened to – 150 mesh and consolidated by atmospheric pressure technology, the CAP process, ¹, ² into 5.5 in. or 10 in. diameter billets which were extruded at 1107°C (2025°F) to a reduction of 4.5 in. or 6.5 in. and then hot rolled from 990°C (2100°F) to 2.375 in rcs bar stock. Their chemical compositions, on a wt pct basis, are listed in Table I along with a CW product employed in this study. Coil rolling began at 1163°C (2125°F), with the reduction proceeding to 0.625 in. and then to 0.25 in. diameter and the rolled rod was coil directly. Also, some bar stock from each heat was hot rolled from 1135°C (2075°F) to a nominal thickness of 0.125 in. for the corrosion susceptibility and weldability examinations.

The resistance to crevice corrosion* was characterized by determining the critical crevice temperature (CCT). The CCT is the highest temperature above which the alloy becomes susceptible to crevice corrosion. Corrosion coupons with dimensions $0.75 \times 1 \times 0.25$ in. were machined and polished through 600 grit paper. Crevices were formed with Teflon cylinders that were pressed against the two major faces of the coupons with a rubber band. Specimens susceptible to crevice corrosion were attacked in the regions of contact with the Teflon cylinders on the specimens faces and with the rubber bands at the specimen edges. Crevice specimens were exposed for 24 hours at a controlled temperature in 10% FeCl₃ • 6H₂O solution with pH=1. Tests were performed at intervals of 2.5°C until the critical temperatures were determined.

Stress corrosion cracking resistance* of the three versions of Alloy 625 was evaluated in a simulated sour oil and gas environment. Duplicate specimens were exposed in the autoclave for 14 days at 232°C (450°F) in a solution containing 25 pct NaCl, 0.5 pct acetic acid, 1 g/l sulfur and pressurized with 100 psi H₂S. After 14 days specimens were examined for cracking.

Microscopic examination* of autogenous welds on wrought and powder metallurgy Alloy 625 was conducted to study grain growth in the heat affected zone (HAZ). The welding parameters were 10 volts and 162 amp in one pass at a travel speed of 12 in./min., providing a heat input of 0.32 kJ/min. with Ar shielding gas.

Results and Discussion

The microstructure of the as rolled 2.375 in. rcsbars of the P/M materials contained a fine and uniform ASTM9.5 grain size. After hot rolling to .25 in. diameter, then solutioning at 1093°C (2000°F) and water quenching, the grain size of KR694, per Figure 1, was uniform and very fine at ASTM10.5, in both L and T directions, with very fine carbides evenly distributed throughout the structure. With no carbides present in V220 because of its miniscule carbon content, the

^{*} Corrosion test procedures and autogenous welding parameters employed in AMAX Materials Research Center Report ISJ-1464 by A. Poznansky and results provided therein.

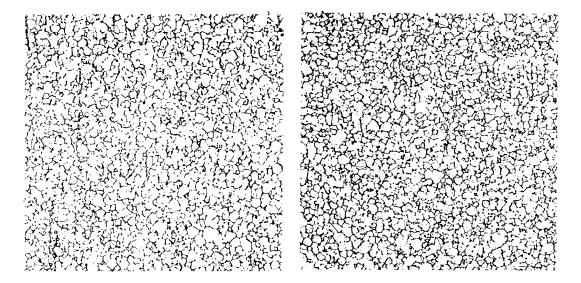


Figure 1. Structure in longitudinal and transverse direction of hot rolled 0.25 in. diameter coil of KR694 heat of P/M Alloy 625. ASTM 10.5 grain size. X200.

grain size was coarser at ASTM7–8. The precipitation of carbides and intermetallic phases would be dependant upon composition and hence their appearance would vary with time at a particular temperature. The advantage of the low 0.005 pct carbon V220 composition with respect to structural stability and corrosion resistance was observed in the TEM examination of a specimen held for one hour at 800°C (1472°F) which revealed no evidence of $M_{23}C_6$ (nor intermetallic precipitates). Wardle and Eriksson⁴ obtained the same result for an Osprey spraydeposited 0.014 pct carbon P/M 625 and published a complete precipitation diagram over the 750 to 1000°C range.

 $\frac{{\tt Table} \ 1}{{\tt Chemical} \ {\tt Compositions-Alloy} \ {\tt 625}}$

	C&W	P/M	P/M		
Element	C2248	KR694	V220		
С	0.042	0.055	0.005		
Mn	0.04	0.09	0.24		
Sì	0.060	0.30	0.42		
s	0.001	0.0004	0.002		
P	0.010	0.007	0.005		
Cr	22.13	21.43	21.57		
W	0.07	0.02	-		
ν	<0.02	0.03	-		
Mo	8 98	9.03	11.05		
Co	0.18	0.16	0.11		
Cu	0.040	0.06	-		
Sn	0.0070	-	-		
Pb	<0.001	-			
Al	0.300	0.29	0.28		
Ti	0.350	0.40	0.30		
Fe	3.38	4.42	0.12		
В		0.0023	0.0002		
Zr	0.40	0.03	-		
Cb+Ta	3.740	3.64	3.66		
N	0.270	0.091	0.001		
Ni	Balance	Balance	Balance		
02	N/A	0.015	0.017		

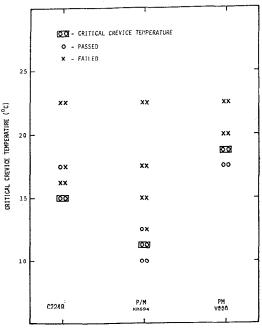


Figure 1 Crevice Corrosion Resistance of Wrought and P/M Versions of Alloy 625 in 108 FeCl $_3$ -6 $\rm H_2O$, 24 Hours Test.

Table II Tensile Test Results at Room Temperature

		Diam.	0.2%	Yield	<u>Ultir</u>	<u>nate</u>	<u>E1.</u>	R.A.
Ident	Type	Inch	MPa	<u>ksi</u>	<u>MPa</u>	<u>ksi</u>	<u>8</u>	8
C2248	CW	2 3/8	478	69.5	958	139.0	42	49
KR694	P/M	2 3/8	553	80.3	989	143.6	41	48
V220	P/M	2 3/8	352	51.1	755	109.5	40	45
C2248	CW	1/4	469	68.2	965	140.0	43	48
KR694	P/M	1/4	485	70.4	1016	147.3	49	45
V220	P/M	1/4	347	50.3	744	105.2	48	43

Tensile test results are listed in Table II. In a comparison between CW C2248 and P/M KR694, having comparable carbon contents, the much finer, more uniform ASTM10 grain size in the latter compared to the ASTM5–6 in the former resulted in higher yield and ultimate strength while ductility was maintained in both bar and wire. When the carbon content was reduced by a factor of 10 and the iron content reduced to a residual in the case of V220, the yield and ultimate strengths were reduced significantly but the ductility was maintained with an ASTM7–8 grain size. The reduction in strength properties of V220 compared to KR694 is attributed to the absence of carbides, the low Fe content and coarser grain size. However, the lower values assure fabricability and not be a factor in the overlay produced from the wire. Also, the introduction of a critical amount of cold work into the material during final wire production would improve strength properties.

The 10 fold reduction in carbon content to 0.005 pct in P/M V220 reduces the possibility of deleterious effects of M₂₃C₆ arising from improper welding or processing heat treatments. Finally, the lower strength properties of V220 are analogous to those obtained by Wardle and Eriksson⁴ on P/M 625 tubes formed by the Osprey process analyzing 0.014C-1.3Fe-8.2Mo. These authors confirm that tensile properties and toughness of Alloy 625 can be correlated with M₂₃C₆ precipitation and they have shown that a 0.014 pct carbon P/M 625 is resistant to change with increasing time between 750 and 1000°C.

The results* of the crevice corrosion test in 10 pct FeCl₃ · 6H₂O solution are shown in Figure 2. P/M V220 exhibited the corrosion. No crevice attack was observed up to 27.5°C (82°F). The improved crevice corrosion resistance of this version of Alloy 625 is attributed to its higher molybdenum concentration (11 pct) and its low carbon and nitrogen content (<0.005 pct). The other two versions of P/M and wrought Alloy 625 exhibited lower critical crevice temperatures. The wrought product C2248 had a critical crevice temperature of 20°C (68°F) compared to 12.5°C (55°F) for the P/M KR694.

All three versions of Alloy 625 were not susceptible to stress corrosion cracking in a solution containing 25 pct NaCl, 0.5 pct acetic acid and 1 g/l sulfur pressurized with 100 psi H₂S at 232°C (450°F). The plastically deformed U-bend specimens of the three versions of Alloy 625 were covered with a dark layer, probably of sulfides, after the fourteen days of exposure in the simulated gas and oil environment. It should be noted that most stainless steel grades are susceptible to severe cracking in this environment.

The microstructures of autogenously welded specimens* of the three versions of Alloy 625 are shown in Figure 3. The grain size, in the heat affected zone, of the

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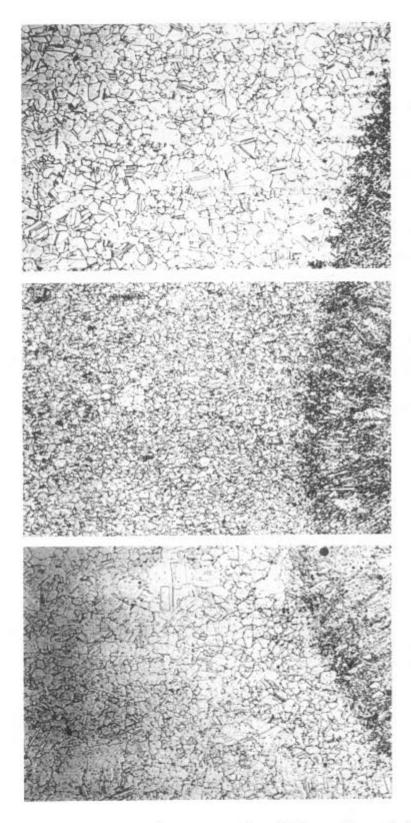


Figure 3. Microstructures of autogenously welded versions of Alloy 625. (a) Wrought C224B. (b) P/M KR694. (c) PM V220. X200

wrought product C2248 is approximately 50μ (ASTM5–6) while the grain size of the P/M versions V220 and KR694 are 25 and 10μ (ASTM7–8 and ASTM10), respectively. Grain growth in the heat affected zone was insignificant in all three versions and therefore should not affect mechanical properties. The grain size of the wrought product, in the base metal, is five times larger than that of the power metallurgy product, even though both have the same composition and both were solution annealed for 1 hour at 982°C (1800°F). A smaller grain size usually results in improved mechanical properties. The grain size of the P/M high Mo-low C version V220 was larger than that of the P/M KR94 because of formation of small carbides in the latter which tend to restrict grain growth.

Acknowledgement

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