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High Temperature Corrosion studies on Pulsed Current Gas Tungsten Arc Welded Alloy C-276 in Molten Salt Environment

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Abstract: Alloy C-276 is widely used in the power plant environment due to high strength and corrosion in highly aggressive environment. The investigation on high-temperature corrosion resistance of the alloy C-276 PCGTA weldment is necessary for prolonged service lifetime of the components used in corrosive environments. Investigation has been carried out on Pulsed Current Gas Tungsten Arc Welding by autogenous and different filler wires (ERNiCrMo-3 and ERNiCrMo-4) under molten state of K₂SO₄-60% NaCl environment at 675°C under cyclic condition. Thermogravimetric technique was used to establish the kinetics of corrosion. Weight gained in the molten salt reveals a steady-state parabolic rate law while the kinetics with salt deposits displays multi-stage growth rates. PCGTA ERNiCrMo-3 shows the higher parabolic constant compared to others. The scale formed on the weldment samples upon hot corrosion was characterized by using X-ray diffraction, SEM and EDAX analysis to understand the degradation mechanisms. From the results of the experiment the major phases are identified as Cr₂O₃, Fe₂O₃, and NiCr₂O₄. The result showed that weld fabricated by ERNiCrMo-3 found to be more prone to degradation than base metal and ERNiCrMo-4 filler wire due to higher segregation of alloying element of Mo and W in the weldment

Keyword: Alloy C-276, Pulsed Current Gas Tungsten Arc Welding, High Temperature Corrosion



1. Introduction

Alloy C-276 is a solid solution Ni-Cr-Mo based super alloy widely used in many industrial applications like aerospace, marine and nuclear industries owing to its excellent resistance to corrosion [1]. It is also extensively used in nitric, formic and sulfuric acid processing industries, hot contaminated media, chlorine water and brine solution. Also, it finds it is used to clad the outlet duct in flue gas desulfurization in coal fired power plant environment due to excellent resistance to sulphur compounds and chloride ions encountered in these scrubbers [2]. This excellent corrosion resistance provided by alloy C276 to a wide range of application is due to the ability of nickel matrix to provide accommodation to a large alloying elements, such as Cr, Fe, Cu and Mo while retaining the single phase (FCC) structure [3]. The combination of both Cr and Mo increases resistance to localized corrosion; where Cr provides resistance to oxidizing environments while Mo improves resistance in the reducing environments [4].

Though alloy C-276 offers excellent corrosion resistance, the corrosion resistance of the welded components is of major issue due to formation of the secondary P , μ and carbide phases that could be formed during welding. Segregation of alloying elements during weld pool solidification is largely responsible for the formation of these secondary phases as reported by various researchers [5-10]. Cieslak et al. [5] reported the segregation of alloying elements during welding process has not only reduced the corrosion properties but also the mechanical and corrosion properties of the weldment. Further, author has also reported that the alloy C-276 is susceptible to hot cracking. The weld corrosion resistance can be substantially improved by suppressing the formation of P , μ and carbide phases by selecting the suitable welding process and parameters [11]. In this regard, many researches [12-18] have reported that pulsed current welding process can reduce the effect of micro-segregation and will improve the weldment resistance. Hot corrosion is the major issue in the power plants operating with high temperature in the aggressive environments [19]. During combustion, a large amount of chlorine and sulfur along with volatile alkali metals such as potassium and sodium are released as vapors in the flue gases. These subsequently gets deposited on the metallic surface or on the already formed oxide layer which further accelerates the oxidation process leading to heavy metal loss [20]. Many researchers confirmed that these failures were due to corrosion, in which there was extensive penetration of sulfur, chlorine, oxygen and carbon, leading to the formation of metal sulfides, chlorides, oxides and carbides resulting in reduced life of the components and catastrophic failures [21]. Arivazhagan et al. [22] has demonstrated the high temperature corrosion on dissimilar weldments of low alloy steel and stainless steel exposing the weldments to synthetic salt mixture of K_2SO_4 -60%NaCl under cyclic condition in lab scale. They observed that the weld zone and weld interfaces encountered severe corrosion. The aggressive hot corrosion environment is provided by salt mixture of K_2SO_4 -60%NaCl with a low melting point of 507 °C [23, 24].

However, very limited study has been conducted on the corrosion behavior weldment of alloy C-276 in the high temperature service environment. It is very important to investigate the alloy C-276 weldments in high temperatures of 675 °C, where it finds its practical application. The present work investigates to study about the hot corrosion behavior of pulsed current gas tungsten arc (PCGTA) weldments (with and without filler) of alloy C-276 in molten salt environment of K_2SO_4 -60% NaCl under cyclic conditions at 675 °C. A cyclic regime of 50 cycles (Cycle of 1 h heating and 20 min cooling) is considered to provide the severe conditions for testing is employed [25-26]. These conditions correspond to the actual industrial environment where breakdown/shutdown occurs frequently.

Thermogravimetric technique was used to establish the kinetics of corrosion. The products of hot corrosion studies had been analyzed by X-Ray diffraction, scanning electron microscopy (SEM), energy dispersive analysis of X-ray (EDAX) techniques to understand the hot corrosion mechanisms operating in these environments.

2. Experimental Procedure

2.1 Welding of Alloy C-276

The material selected for the study was alloy C-276 which was procured in the rolled sheet form of 4mm thickness. Welding was carried out in Pulsed Current Gas Tungsten Arc Welding (PCGTA) mode with and without filler wire. The chemical composition for base metal and filler wire (ERNiCrMo-3 and ERNiCrMo-4) are reported in Table 1. The various welding parameter employed for this studies are in Table 2.

Table 1 Chemical composition of base metal and filler wires.

Base/Filler Metal	Chemical Composition (% Wt.)								
	Ni	Mo	Cr	W	Co	Mn	Fe	Nb	Others
Alloy C-276	Bal	16.36	15.83	3.45	0.05	0.41	6.06	--	0.17 (V), 0.005(P), 0.002 (S), 0.02 (Si), 0.005(C)
ERNiCrMo-3	Bal	10.0	22.0	---	---	0.5	1.0	4.5	0.015 (P), 0.015 (S), 0.5 (Si) 0.5 (Cu), 0.4 (Al), 0.4 (Ti), 0.1(C)
ERNiCrMo-4	Bal	17.00	16.5	4.5	2.50	1.0	7.0	--	0.04 (P), 0.03 (S), 0.5 (Cu), 0.02(C)

Table 2 Process parameters used for welding.

Parameters	Autogenous PCGTAW	PCGTA ERNiCrMo-3	PCGTA ERNiCrMo-4
Peak Current (Amps)	238	213	200
Background current (Amps)	160	145	100
Pulse Frequency (Hz)	6Hz	6Hz	6Hz
Pulse on time	50%	50%	50%
Voltage (Volt)	17	13	11.7
Argon Gas (lit/min)	15	15	15

2.2 Sample preparation for hot corrosion test

Composite (30 mm × 10 mm × 4 mm) employed for the hot corrosion test were sliced from the welded plates by keeping weld as center using wire cut electrical discharged machining. The composite region consists of Weld Zone (WZ), Heat Affected Zone (HAZ) and Base Metal (BM). The coupon used for corrosion test were mirror polished down to 1 μm by SiC paper and alumina powder before the corrosion test. The hot corrosion studies were conducted for autogenous and filler wire case and the results are compared.

2.3 Molten salt corrosion test

The experiment was conducted for 50 cycles of which each cycle consists of 1 h heating at 675 °C in the silicon carbide tube furnace followed by 20 min of cooling in air. The coating of uniform thickness with 3–5 mg/cm² of K₂SO₄–60% NaCl applied with a camel hair brush on the preheated sample (250 °C). The alumina boats were used to keep the samples inside the tube furnace. The hot corrosion environment was

simulating to create a real case for hot corrosion testing. During the corrosion tests, the weight change measurements were taken at the end of each cycle using an electronic weighing balance machine, which has a sensitivity of 1 mg. The spalled scale was also retained during the measurement of the weight change to determine the total rate of corrosion. Efforts were made to formulate the kinetics of corrosion and the same is presented in this paper. The samples after corrosion tests were subjected to characterization studies by SEM/EDAX, XRD for a surface of the scale.

3. Results

3.1 Visual Examination and Thermo-gravimetric analysis

Figure 1(a-d) show the macrograph of hot corrosion samples (i) as salt coated condition (ii) 5th cycle (iii) 25th cycle (iv) 50th cycle of autogenous, ERNiCrMo-3 and ERNiCrMo-4 PCGTA weldments. During cyclic hot corrosion study after a few cycles the color of the weldment in all the cases (autogenous, ERNiCrMo-3 and ERNiCrMo-4) was turned to be black color from the white color salt coated. Yellow color small spots are found in the autogenous weldment (Fig. 1b (ii)) after a few cycle and remained same for the end of 50th cycles.

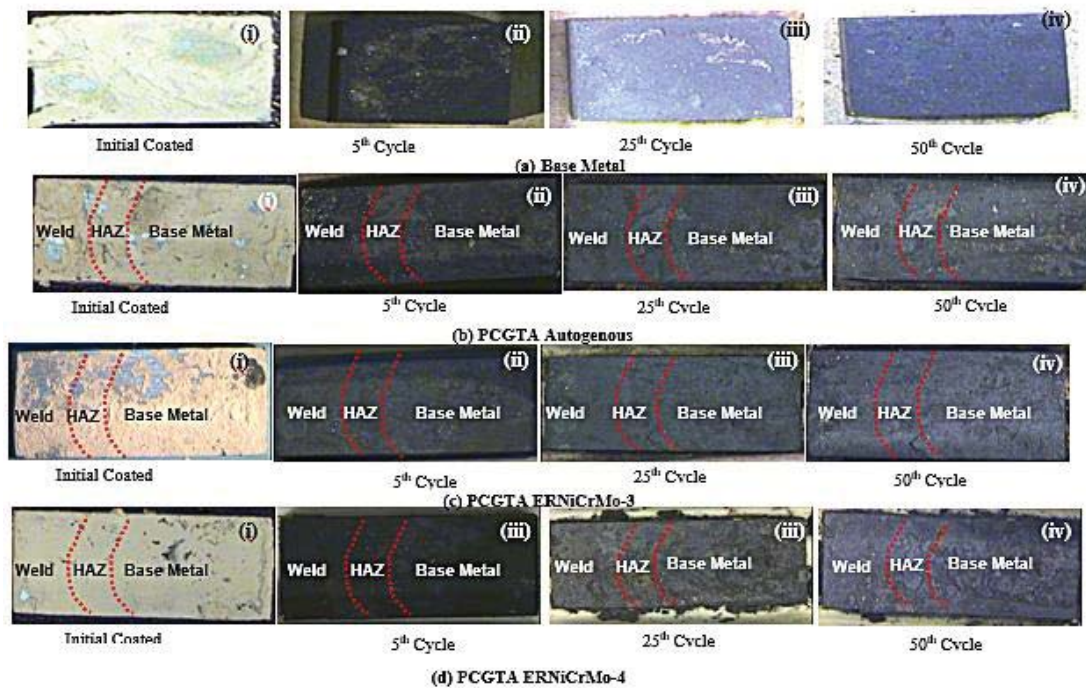


Fig. 1 Macro Photograph after exposure in Molten salt mixture of K_2SO_4 -60% NaCl at 675 °C (a) Base Metal (b) PCGTA Autogenous (c) PCGTA ERNiCrMo-3 (d) PCGTA ERNiCrMo-4

After 18th cycle the color of the scale become thicker and start to spalled out with increase in further cycles and color also changed to blue color and remain same for the end of cycles. Similarly, in ERNiCrMo-4 the color become blue after 10th cycles and also the thicker scale starts to form after the 10th cycle and the scale starts to spalled out further increasing the cycles. The scale formed on ERNiCrMo-3

weldment is very high compared to the autogenous and ERNiCrMo-4 and scale spalled also extensively high from the 20th cycle. The comparative thermo-gravimetric analysis results are shown in Fig. 2. The weight gained in ERNiCrMo-3 is found to be very high as compared to the ERNiCrMo-4 and autogenous weldments. Very negligible weight gain found to be in base metal as compare to the other weldments. Figure 3 shows the parabolic rate constants, K_p for base metal, autogenous weld, ERNiCrMo-3 and ERNiCrMo-4 weld are 0.02, 0.05, 0.17 and 0.16 $\text{mg}^2/\text{cm}^4/\text{h}$ respectively.

3.2 XRD analysis.

Figure 4 shows the XRD analysis of weldments. XRD analysis was carried out for all the specimens at the end of the 50th corrosion cycles. The major phases formed in the weldments are Cr_2O_3 , NiCr_2O_4 , NiO , Fe_2O_3 , FeS and MnO_2 .

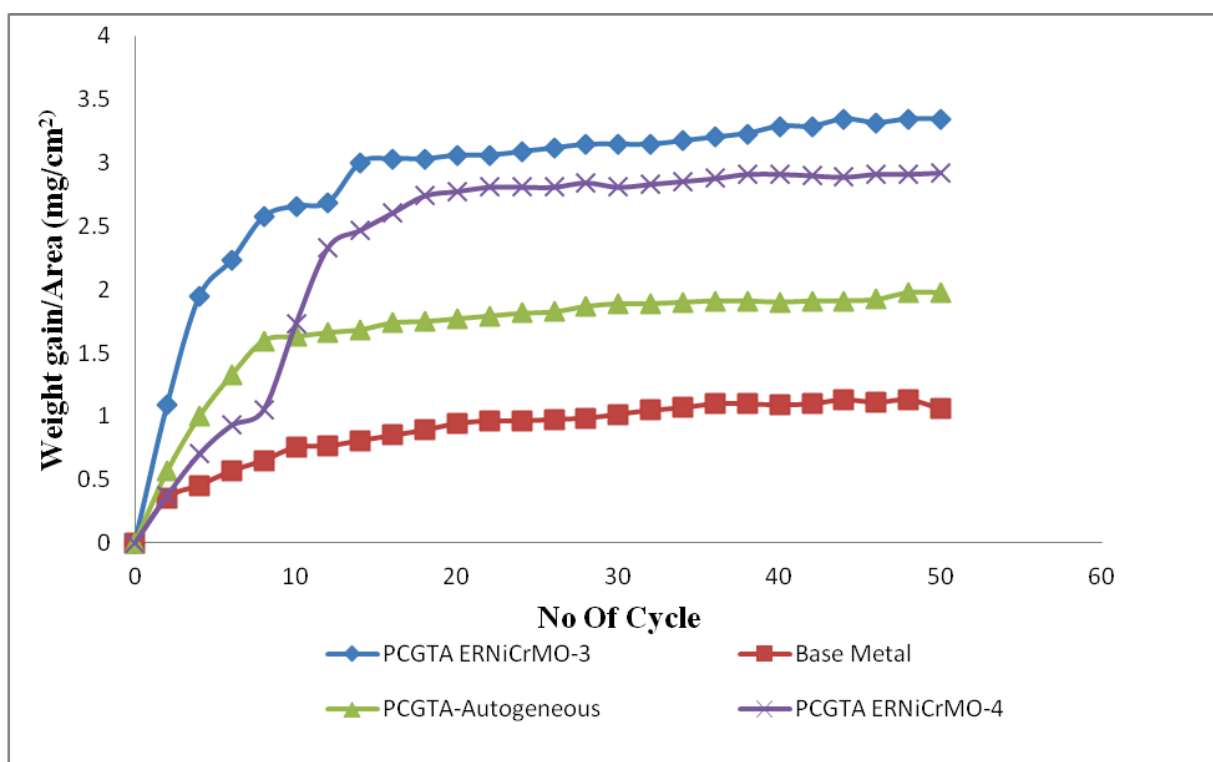


Fig. 2. Plots of cumulative weight gain (mg/cm^2) as a function of time (number of cycles).

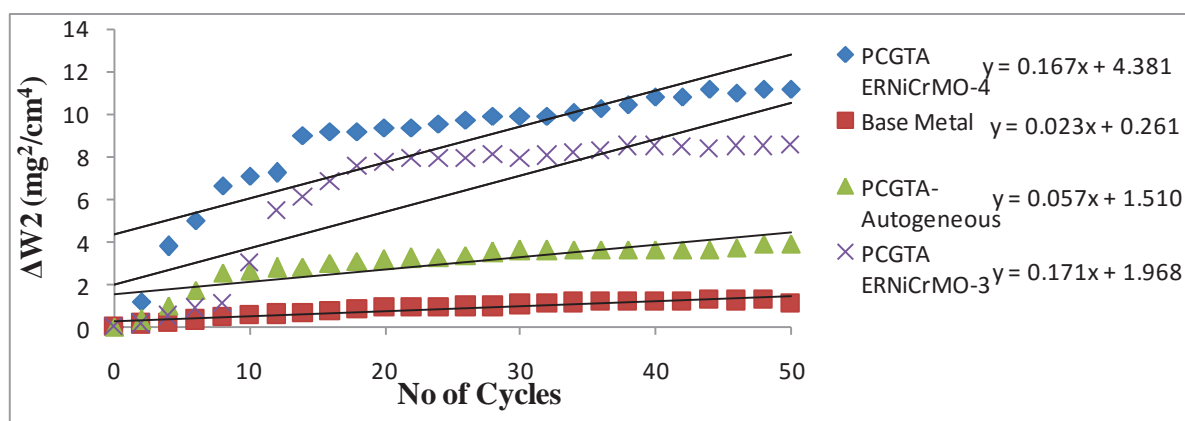


Fig. 3. Plot for $(\text{Cumulative weight gain}/\text{area})^2$ vs number of cycles.

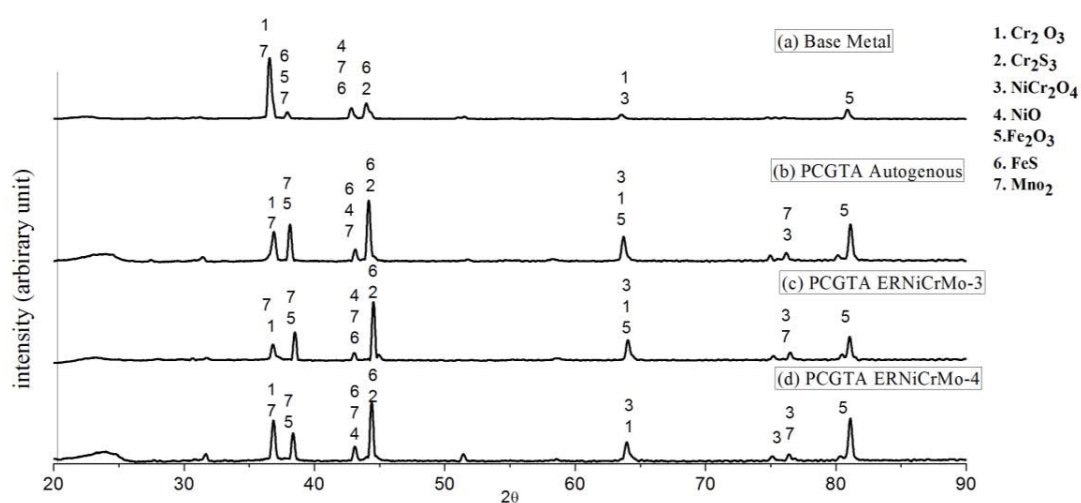


Fig. 4 XRD Analysis of Molten Salt Mixture of $K_2SO_4 - 60\%$ NaCl at $675\text{ }^\circ\text{C}$ (a) Base Metal; (b) PCGTA Autogenous; (c) PCGTA ERNiCrMo-3; (d) PCGTA ERNiCrMo-4

3.3 SEM/EDAX

The SEM/EDS analysis of autogenous welded zone as well as weld interface indicate the presence of continuous and smooth scale crystals dispersed throughout their scales (Fig 5). In the both cases, the scale consists mainly of NiO and contains small quantities of oxides of Fe and Cr.

SEM micrograph along with EDAX measurements at some selected points for the oxide scale of ERNiCrMo-3 weldment exposed to hot corrosion in the molten salt at $675\text{ }^\circ\text{C}$ after 50 cycles is depicted in Fig. 6. However, the SEM micrograph of scale on weld reveals a uniform fine grained crystals consisting mainly of NiO, the scale on weld interface consists of distorted porous and non-protective granules with comparatively higher amount oxides of Cr, Mo and Fe. The presence of small quantities of these oxides in the top scale indicates the diffusion of these elements from the substrate.

SEM micrographs for ERNiCrMo-4 weld and weld interface after exposure to $K_2SO_4-60\%$ NaCl environment at $675\text{ }^\circ\text{C}$ are shown in Fig. 7 along with EDS results. SEM micrographs indicate non-protective nature of scale in the weld as well as weld interface. The main constituent of the scales for these two cases is NiO as can be seen from the EDS results at certain points in the respective cases. The white crystalline phase in the weld is rich in NiO with small oxide quantities of Fe, Cr and Mo. A massive and dense scale has been observed for weld interface which is rich in NiO (black matrix).

4. Discussion

Thermogravimetric data has been compiled in Fig. 2 in the form of a plot between weight gain per unit area expressed in mg/cm^2 and number of cycles for hot corroded base metal and all the weldments. Further, the weight gains square (mg^2/cm^4) data were plotted as a function of time (number of cycles) as shown in Fig. 3 to establish the rate law for the hot corrosion. It is clear that in spite of some fluctuations in the data, the data conforms to the parabolic rate law to an acceptable limit for all the weldments and base metal. The curve showing that the initial rate of weight gained is higher in all the cases which slowly decrease.

It has been observed that the rate of weight gain was relatively high in all the cases during the initial period of exposure, which may partially be attributed to the formation of some catalyst which serves as

oxygen carrier to the specimen, therefore it will lead to rapid oxidation of the basic elements of the alloy C-276 to form protective oxide scales. Simultaneously, the protective scale is destroyed or eliminated by molten salts and consequently the metal surface is exposed to direct action of aggressive environment as has been indicated in the current investigation also. Arivazhagan et al. [22] as well as Devendranath et al. [27] have suggested simultaneous growth of oxides and their dissolution in molten salt in their study. After a period of high corrosion rate it has been observed that the rate of corrosion tends to be almost uniform with the further progress of study. This can be ascribed to the slower growth rate of Ni and Cr scale. Furthermore, the formation of NiCr_2O_4 in the scales with the progress of study might have also contributed to slower oxidation rate as has been suggested by Arivazhagan et al. [22].

The parabolic rate constant of ERNiCrMo-3 shows higher compared to the other weldments. It is also well evident from the thermogravimetric analysis ERNiCrMo-3 obtained the higher weight gained compared to the other weldments. As reported in the Introduction section the proper selection of welding process and parameter may reduce the microsegregation. The same authors [28-29] also reported that PCGTA ERNiCrMo-3 shows the higher segregation compared to autogenous and ERNiCrMo-4 PCGTA weldment. The higher segregation is caused by large difference between chemical compositions of base metal and filler metal. Alloy C-276 has the nominal chemical composition Ni-16Cr-16Mo-5Fe-4W, whereas nominal chemical composition of ERNiCrMo-3 is Ni-22Cr-10Mo-1Fe. The variation in the chemical composition between the base metal and filler wire (ERNiCrMo-3) leads to higher segregation with higher corrosion compared to other weldments. It is observed from the present study weight gain autogenous weldment has proved to be the most corrosion resistant in the aggressive environment under study. It is also noted that the autogenous weldment has slightly less corrosion resistance as compared to base metal in the same environment.

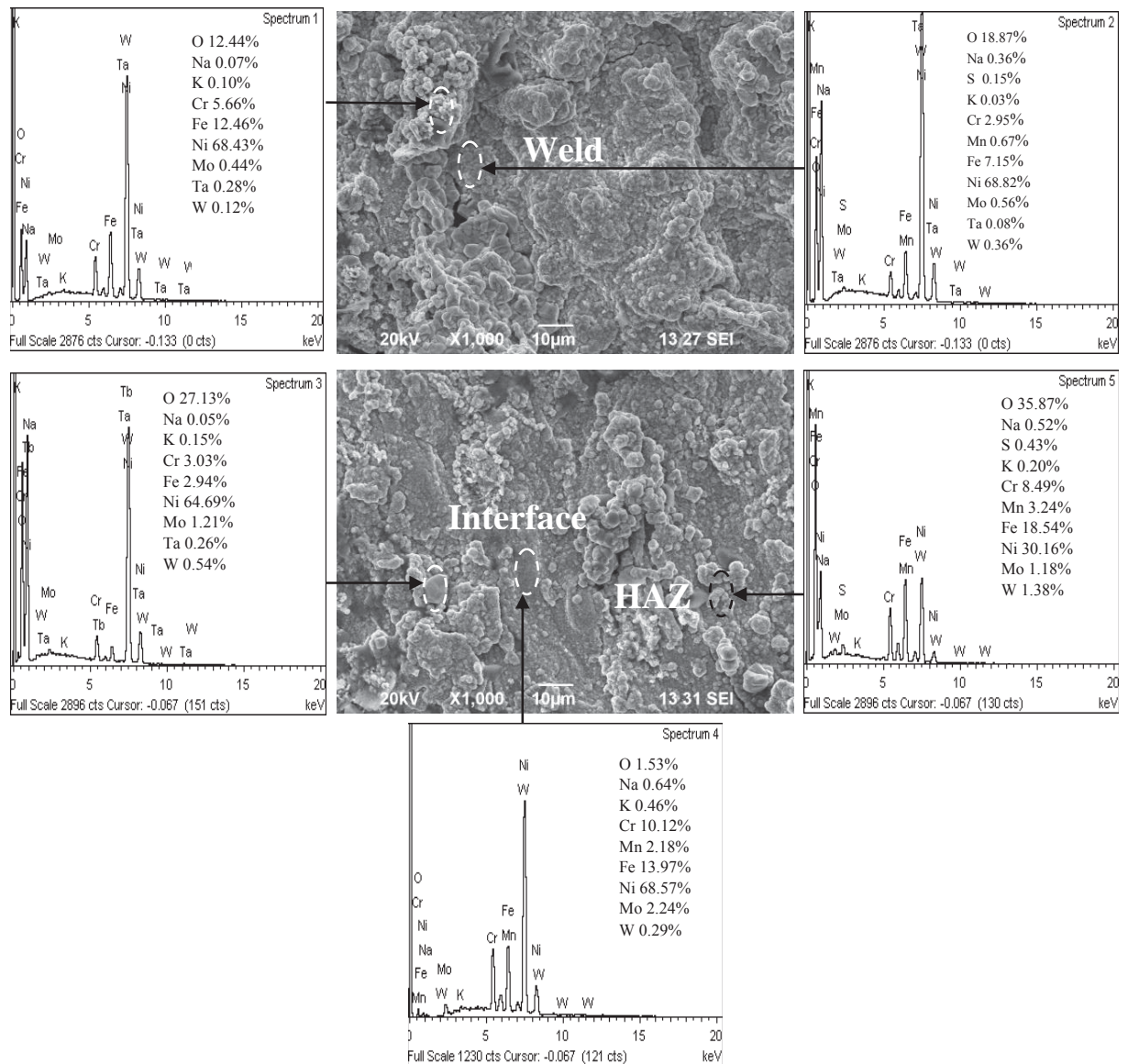


Fig. 5. SEM/EDAX analysis of hot corroded alloy C-276 Autogenous PCGTAW subjected to molten salt environment $K_2SO_4 + NaCl$ (60%) at 675°C

X-ray diffractograms of the scales for base metal and all the weldments are given in Fig. 5. XRD analysis shows the major predominant phases inferred from all the three weldments are Cr_2O_3 , $NiCr_2O_4$, NiO , Fe_2O_3 , FeS and MnO_2 . The presence of spinel $NiCr_2O_4$ has also been confirmed in all the specimens and these X-ray diffraction results are well supported by SEM/EDAX results. The studies conducted by Arivazhagan et al. [22] and Devendranath Ramkumar et al. [27] on similar studies also endorse formation of identical phases.

It is evident from the diffraction patterns that autogenous, ERNiCrMo-3 and ERNiCrMo-4 have shown formation of similar phases after hot corrosion in the given environment. NiO and $NiCr_2O_4$ are indicated as the main phases in all the weldments. The protection of weldments and base metal from hot corrosion

might partially be due to the oxides of nickel and chromium, and spinel of Ni and Cr present in the top scales as revealed by the surface XRD analysis

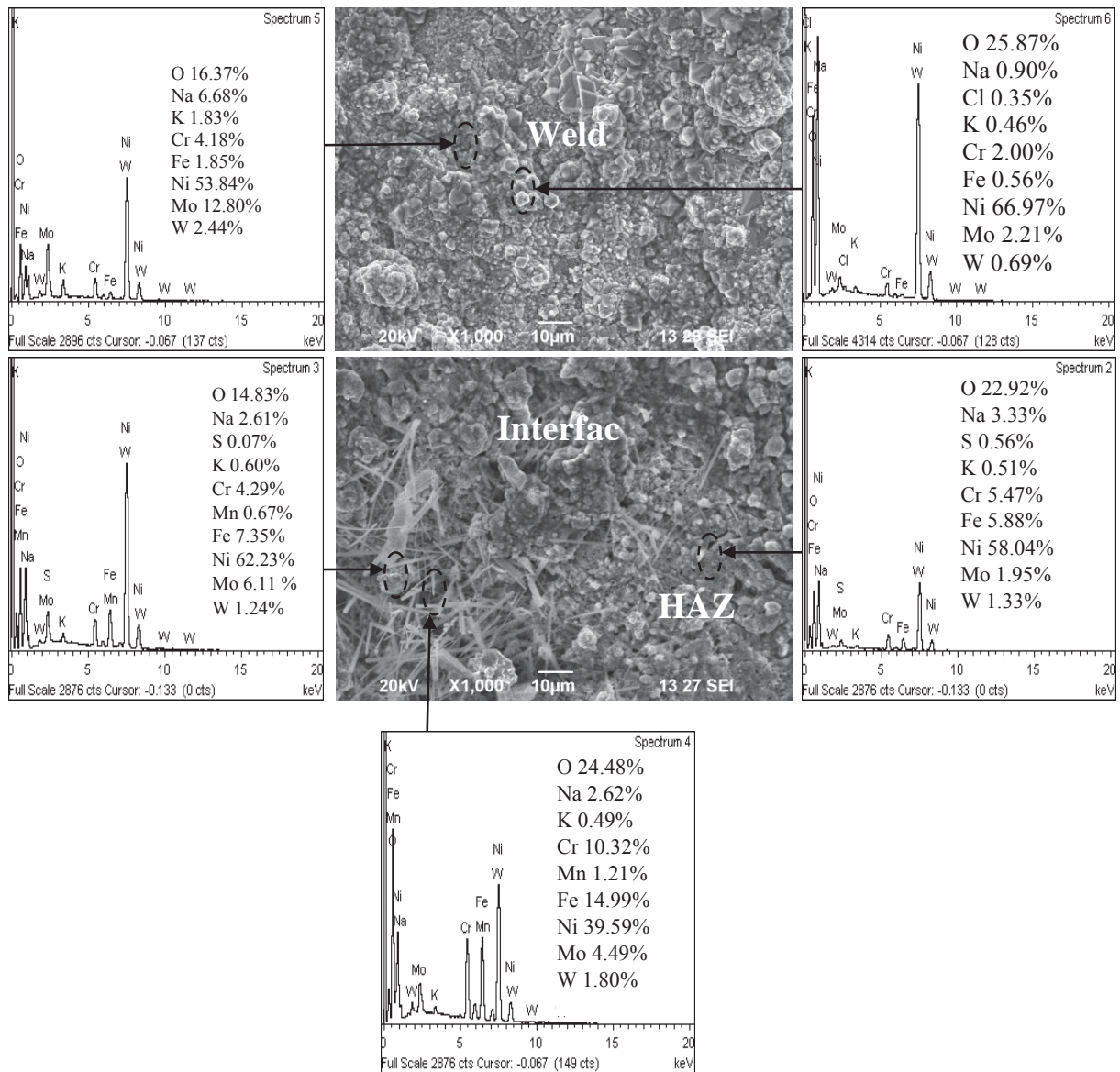


Fig. 6. SEM/EDAX analysis of hot corroded alloy C 276 PCGTAW weld with ERNiCrMo-3 subjected to molten salt environment $K_2SO_4 + NaCl$ (60%) at 675°C

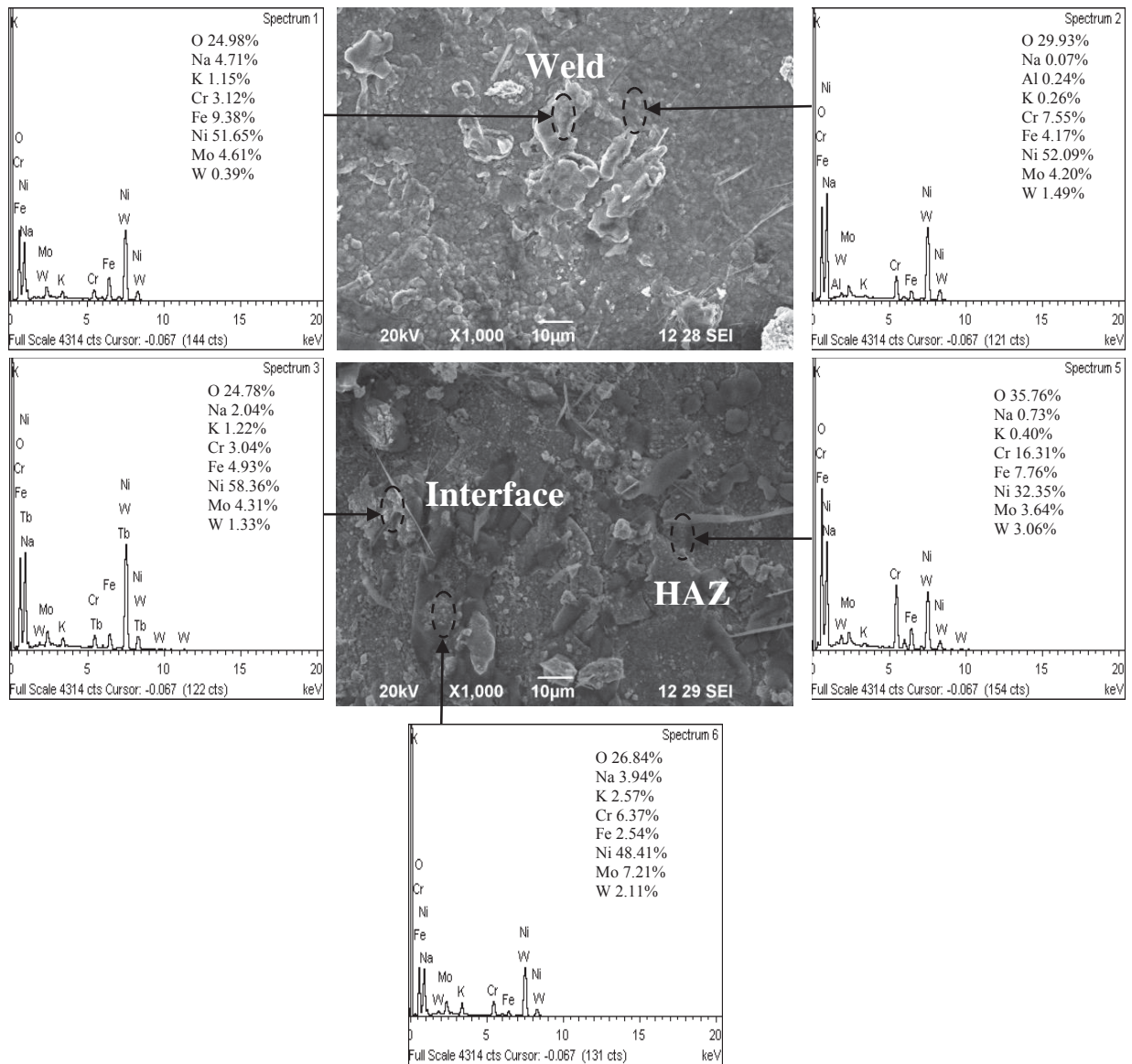


Fig. 7. SEM/EDAX analysis of hot corroded alloy C 276 PCGTAW weld with ERNiCrMo-4 subjected to molten salt environment $K_2SO_4 + NaCl$ (60%) at 675°C

The weldment employed by under alloyed filler has indicated accelerated corrosion in K_2SO_4 -60%NaCl environment at 675 °C in comparison to that of autogenous weldment and base metal. On the basis of cumulative weight gain data for 50 cycles, corrosion rate of the specimen under study can be arranged in the following order:

ERNiCrMo-3 > ERNiCrMo-4> Autogenous > Base C-276

The superior corrosion resistance shown by autogenous weldment as well as base C-276 alloy might be ascribed to the formation of NiO and Cr₂O₃. Whereas ERNiCrMo-3 weldment underwent extensive spalling and sputtering of its scales. In addition to the above mentioned mechanism, the accelerated hot corrosion of this weldment might have also been occurred due to the presence of higher amount of molybdenum as suggested by Peters et al. [30]. This tendency may be attributed to the different values of thermal expansion coefficients of the oxide scale on different zones of the weldment as reported by Arivazhagan et al. [22]. However, as the environment of study in this case contains molten salt, therefore the elements such as Na, Cl and S and oxygen may diffuse along these splats or pores, instead of continuous scale on autogenous and base metal.

EDS analysis for ERNiCrMo-3 and ERNiCrMo-4 weldment indicates that sulphur has penetrated into the substrate alloys and there it is co-existing with Fe and might form FeS. The oxidation of this FeS might yield a protective scale of NiO and Cr₂O₃ as per the views of Arivazhagan et al. [22]. However, the absence of metallic chlorides in the scale, there is traces of Cl in the EDS result might be evaporate die to low melting eutectic. Once sulphide is formed, the diffusion of Ni and Fe through sulphide is faster than in oxide resulting in accelerated attack. The formation of non-protective oxides of Ni and Fe is the cause for introduction of large amounts of sulphur from potassium sulphate into the alloy, which essentially leads to the degradation. The autogenous weldment have been found uniform scaling behavior across the microstructural gradients in weldments when exposed to molten salt environments, whereas the scale forming on the under alloyed weldment was not uniform.

In all the three cases the formation of FeO is found to be absent in the oxide scale irrespective of the test temperature as suggested by Moretimer and Sharp [31]. The absent of FeO is due to the higher concentration of Cr content. From SEM the major concentrated element present is Ni in all the cases. Ni undergoes oxidation preference to alloy C-276. The next major concentrated element to Ni is Cr. The Cr act to protect the surface layer as CrO, CrO₂, Cr₃O. Furthermore, the FeS revealed in the Autogenous PCGTA weldment, which indicates the existence of both oxidation and sulphidation in surface and subsurface reaction. In other cases, the FeS is found to be very small peak observed in XRD, the scale may spall out due to low melting eutectic.

The hot corrosion test was performed for 50 cycles, it is found that the ERNiCrMo-3 weldment was observed as more corrosion than compare to other weldments, which also can be seen in the large degree of scaling and spattering on the weldment (ERNiCrMo-3). It is evident that the thickness of the scale is more in ERNiCrMo-3 than the other two weldments after being exposed to 50 cycles in K₂SO₄-60% NaCl.

5. Conclusion

From the study the major conclusions obtained are listed as follows.

1. The weight gain of salt coated welded specimen follows a parabolic rate law during hot corrosion
2. Major phases are identified as Cr₂O₃ and NiCr₂O₄ on scale over weldment may due to presence of higher amount of Ni and Cr in the weldment and base metal
3. The corrosion rates for the investigated friction welded dissimilar metals based on the overall weight gains after 50 cycles in all the environments could be arranged in the following order: ERNiCrMo-3 > ERNiCrMo-4 > Base metal.
4. PCGTA fabricated by ERNiCrMo-3 shows higher corrosion compared to the other weldment. The higher segregation leads to more corrosion in the weldment compared to base metal and ERNiCrMo-4

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