

High temperature air oxidation studies on Inconel 625 weldments

A. Vaibhav Krishna, K. Pavan Kumar, Movva Bhushan, P. Prabakaran
K. Devendranath Ramkumar*, N. Arivazhagan

*Corresponding Author, E-mail: deva@vit.ac.in
Telephone No. 091-416-2202184

Abstract

High temperature corrosion studies on the Ni based superalloys are the thrust areas of research nowadays. In this work, an attempt has been made to investigate the weldability, mechanical and metallurgical properties of the GTA weldments of Inconel 625 superalloy. Tensile studies In addition to mechanical characterization, the various zones of the weldments were subjected to cyclic air oxidation studies at 800 °C. Further the hot corroded samples were characterized using the combined techniques of optical microscopy, SEM/EDAX analysis.

Keywords: Gas tungsten arc welding; Inconel 625; Mechanical characterization; SEM/EDAX analysis; hot corrosion

1. Introduction

Inconel 625, a nickel based superalloy which is widely used in marine, chemical plants due to its properties such as higher tensile strength and high corrosion resistance [Sims et al. (1987)]. The presence of Nb and Mo in the nickel chromium matrix provide the solid solution strengthening and offer the strength to this alloy, hence no precipitation hardening heat treatment is required for these materials. Kuk Hyun Song et al. (2009) studied the mechanical properties of friction stir welded of Inconel 625 having 2 mm thick. The authors reported the formation of MC carbides such as NbC and (Ti,Nb)C were distributed in the grains and grain boundaries. It was also noticed that the weld strength of Inconel 625 joints was found to be higher as compared to the base metals owing to the grain refinement.

Zheng et al. (2010) investigated the performance of Inconel 600 and Inconel 625 for liquid droplet erosion (LDE), which often occurs in bellows made of nickel-based alloys, threatens the security operation of the nuclear power plant. It was reported from the studies that Overall, the resistance to cavitation erosion and jet impingement erosion of Inconel 625 is much superior to that of Inconel 600. Boser (1979) conducted the studies on the creep behavior of silver diffused Inconel 625 samples which was used for the construction of heater tubes for the stirling engine operated at 700 - 800 °C. The studies concluded that silver did not diffuse neither by bulk nor by grain boundary diffusion into Inconel 625 at temperatures up to 900 °C. Preventing high temperature oxidation and hot corrosion at elevated temperatures due to oxidation resistance of alloy 625 is the main purpose for such a broad range of applications [Sidhu et al. (2005)].

High chromium content provides good resistance to oxidation at high temperatures mainly due to formation of a protective chromium-rich oxide layer. It was reported that the Chromium is one of the

most effective alloying elements to act against corrosion, necessitating at least 18 to 24 wt.% to optimize its effectiveness [Philip et al. (2007), Wang et al. (2003), Grabke et al.(2004)]. Significant content of molybdenum makes alloy 625 resistant to pitting and crevice corrosion; whereas a combined nickel and molybdenum content makes it resistant to non-oxidizing environments and crevice corrosion [Martin et al. (2003)]. The presence of niobium protects it against sensitization during welding, thereby preventing subsequent inter-granular attack [20,21]. Also, niobium and tantalum stabilization makes this alloy suitable for corrosion service in as-welded conditions. Furthermore, alloy 625 has an appropriate resistance to chloride corrosion cracking [Philip et al. (2007)]. The presence of high concentrations of Cr, Mo and Nb could cause formation of protective oxides of Cr_2O_3 , NiO , Nb_2O_5 and NbCrO_4 which could normally prevent the high temperature corrosion in superalloy.[Otero et al.(1991)].

Mohammadi Zahrani et al. (2012) conducted the studies on hot corrosion behavior of alloy 625 in the molten salt environment consisting of $47\text{PbSO}_4\text{--}23\text{ZnO--}13\text{Pb}_3\text{O}_4\text{--}10\text{Fe}_2\text{O}_3\text{--}7\text{PbCl}_2$ (wt.%) at 600, 700 and 800 °C. The authors reported that the corrosion product layer mainly consists of chromium, oxygen, and nickel. General surface corrosion, intergranular corrosion, formation of voids and a network of distributed pores on the surface and the cross-section, as well as internal oxidation and sulfidation were identified as different modes of corrosion attack. Mohammadi Zahrani et al. (2010) reported that Inconel 625 tubes were employed in the construction of water wall tubes where the deposits of lead, zinc and Iron form the salt mixture at high temperature causing the degradation by corrosion.

Cyclic air oxidation studies were demonstrated on Inconel 625 in order to evaluate its oxidation resistance. It was reported by the authors that Long-term exposure leads to microstructural modification and seemed to be detrimental. The chromia scale losses its protective properties and then IN-625 exhibits lower cyclic-oxidation resistance when exposed to 900 and 1000 °C [N'dah et al. (2006)].

As evident from the existing literatures, Inconel 625 offers good weldability and higher corrosion resistance and operating at high temperatures. However no specific emphasis has been given on the corrosion behavior of the weldments at high temperature environments hitherto. This study investigates the weldability, metallurgical and mechanical properties of the gas tungsten arc welded Inconel 625 plates having 5 mm thickness. Furthermore cyclic hot corrosion studies were performed under air oxidation at 800 °C. Thermogravimetric analysis was carried out to study and interpret the weight gain / loss on the various zones of the weldments. The corrosion products were systematically characterized using the combined techniques of SEM/EDAX analysis.

2. Experimental Work

2.1 Candidate Metals and welding procedure

The chemical composition of the candidate metal Inconel 625 and the filler metal ERNiCrMo-3 is represented in Table 1. The dimensions of the candidate metals employed in this research work has the dimensions of 120 mm x 50 mm x 5 mm. Standard V-groove butt configurations having root face of 1 mm and an included angle of 60° was employed to weld these similar metals by GTAW process using ERNiCrMo-3 filler metal. A specially designed fixture employing the copper back plate was used to avoid while welding to avoid bending and distortions. The process parameters employed in the GTA welding of Inconel 625 is represented in Table 2. The welded samples were characterized for flaw detection using X-Ray radiography NDT inspection techniques. Further the weldments were

sliced to various coupons using wire-EDM after NDT analysis for further studies which are outlined below.

2.2 Metallurgical and mechanical characterization

Metallographic examination was carried out on the composite region [parent metal + weld + HAZs] of the weldment as shown in Fig.1(c). The composite region of the weldments were polished using the emery sheets of SiC with grit size varying from 220 to 1000 and followed by disc polishing using alumina to obtain a mirror finish of 1μ on the weldments. Electrolytic etching (10% oxalic acid solution; 6V DC supply and 1 A / Cm^2) was employed to examine the microstructure of Inconel 625. Further the samples were sliced to different dimensions to conduct various mechanical tests and assess the properties under room temperature. Tensile studies were performed on the ASTM E8 standard samples of the weldments. Three trials on each weldment were conducted to check the reproducibility of the results. The fractured samples were characterized to understand the mode of fracture by SEM analysis. Hardness studies were conducted on the composite region of the weldment by keeping weld as center using Vicker's Microhardness tester employing a load of 500 gf and 10 s dwell time at the regular intervals of 0.25 mm. After assessing the mechanical properties, hot corrosion studies were performed on the various zones of the weldments and are outlined below.

2.3 Hot Corrosion Test

Before corrosion run, the samples were mirror polished down to 1μ finish employed for corrosion studies. Cyclic air oxidation studies were conducted on the various zones of the weldments at 800°C . These corrosion studies were performed on the different regions of GTA welded Inconel 625 coupons each measuring $10 \times 10 \times 5\text{ mm}$; also on the composite region [Base Metal + HAZ + Weld] measuring $30 \times 10 \times 5\text{ mm}$ to estimate the corrosion behaviour for 50 cycles (each cycle constitutes 1 hour heating followed by 20 minutes of cooling to room temperature). During the course of corrosion, the color changes on the coupons were noticed and recorded. Also the weight changes had been measured for all regions for each cycle using electronic weighing balance with a sensitivity of 1 mg. The weight gain or loss of the spalled scale was also included at the time of measurement to determine the rate of corrosion. The hot corroded samples were characterized using XRD, SEM/EDAX analysis. Also SEM/EDAX analysis was employed on the various zones of the weldments to determine the presence of various elements which is helpful to assess the structure - property correlations. The following chapter addresses the results and discussions of the experimental work.

3. Results

3.1 Macro and Microstructure Examination

Macro-structure examination shown in Fig.1 clearly revealed that proper fusion had occurred and sound welds of Inconel 625 could be obtained on employing the process parameters in GTA welding. NDT analysis confirmed that there were no weld defects including lack of fusion, undercuts and porosities etc. Microstructure on the GTA weldments of Inconel 625 shown in Fig.2 clearly indicates the formation of precipitates at the weld

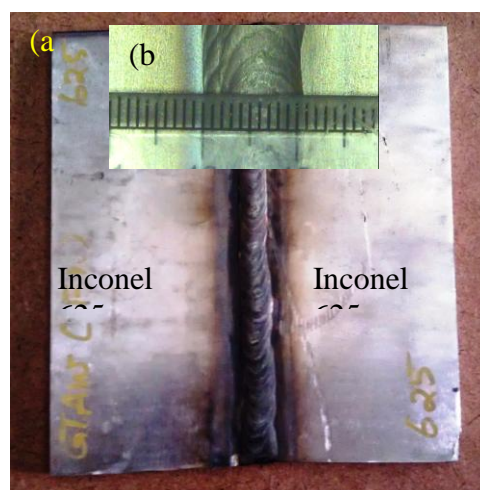


Fig. 1 (a) GTA welded Inconel 625 samples (b) Closer view of the weld head

zone and in the heat affected zone (HAZ). Dendritic growth is observed at the weld zone.

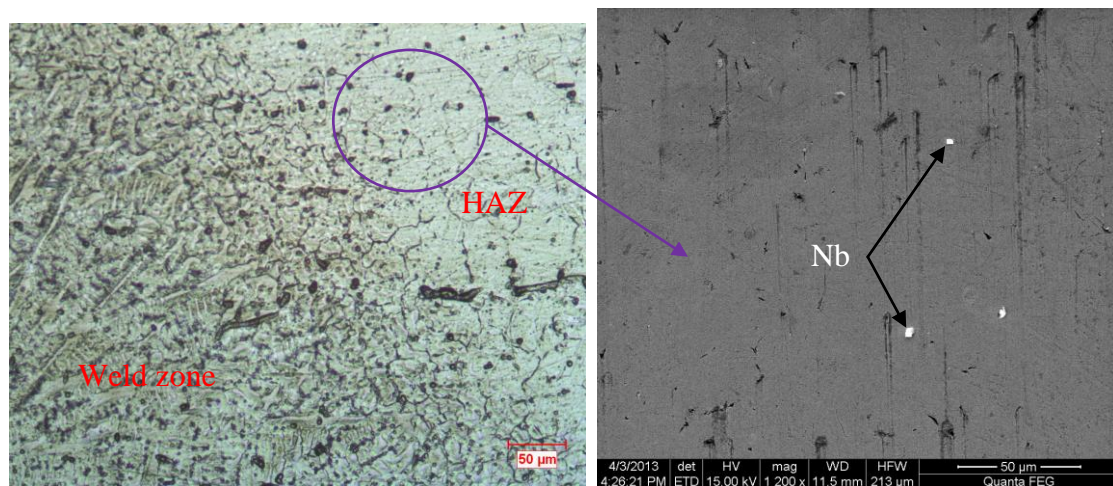


Fig. 2(a) Microstructure showing the weld - HAZ of Inconel 625 weldments **(b)** SEM image representing the HAZ of Inconel 625

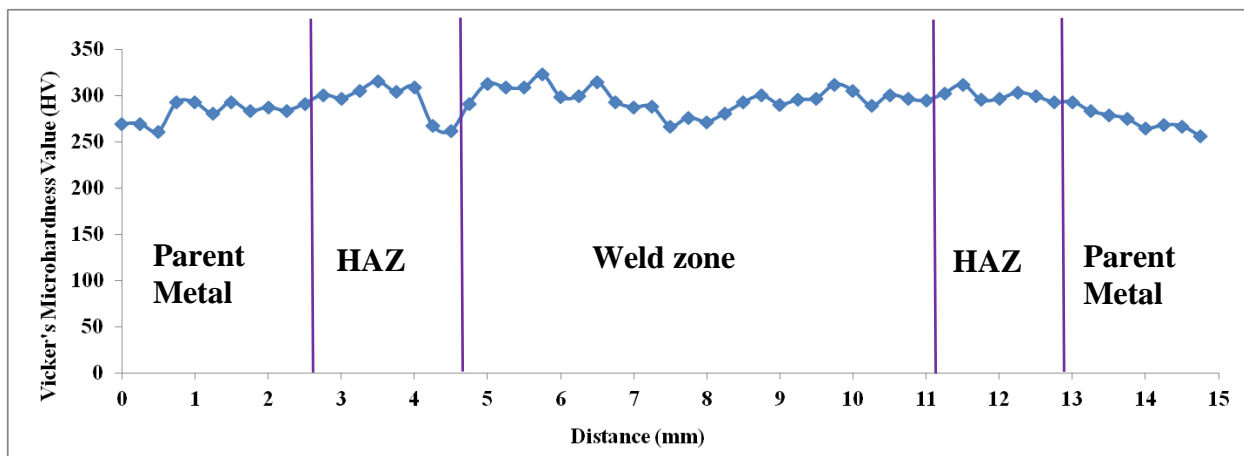


Fig. 3 Hardness profile representing the various zones of Inconel 625 weldments

3.2 Mechanical properties of the weldments

Hardness profile represented in Fig.3 had shown that the hardness values are almost equivalent along the entire width of the weldment. However in a closer perspective, the hardness values at the weld zone are slightly lower than that of the other zones. Tensile studies shown in Fig. 4(a) revealed that the fracture obtained at the weld zone for all three trials. The average ultimate tensile strength, young's modulus and the ductility obtained from these trials were 874 MPa, 61.76 GPa, and 46.7% respectively. This could be confirmed by the SEM fractographs (Fig. 4(b)) which give an insight on the mode of fracture.

3.3 SEM/EDAX analysis on the as-welded sample

SEM/EDAX analyses were performed on the various zones of the GTAW Inconel 625 weldments to understand the presence of elements and also to get the better understanding the structure-property relationships (Fig. 5). On examining the SEM/EDAX results of GTA weldments, it is well understood that all the zones of the weldment has maximum amounts of Ni, Cr, Mo, Nb. However the Nb content in the weld zone is slightly lower than the heat affected zone and parent metal.

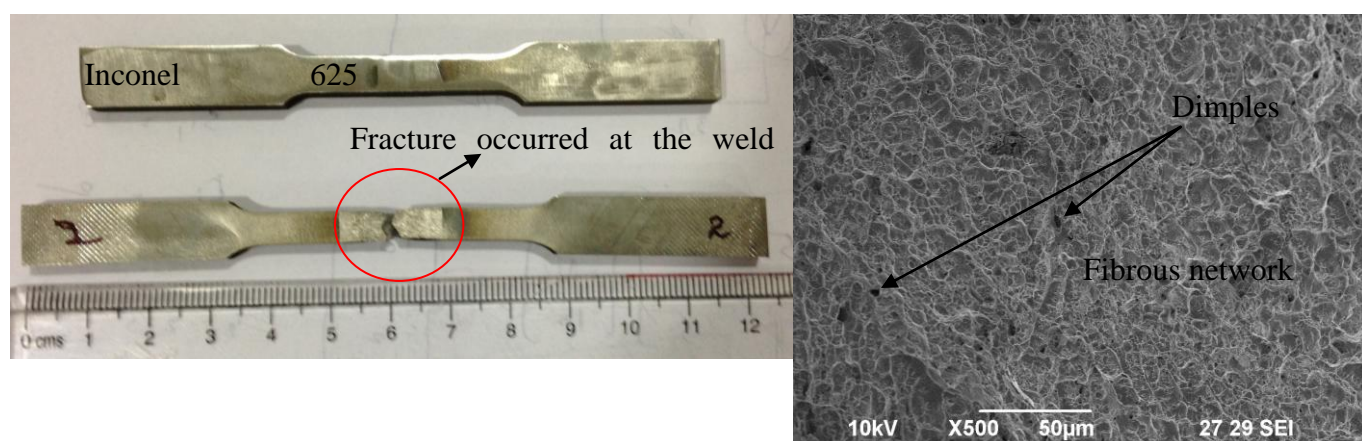


Fig. 4 (a) Fractured tensile sample (b) SEM fractograph showing the dimples and micro-voids

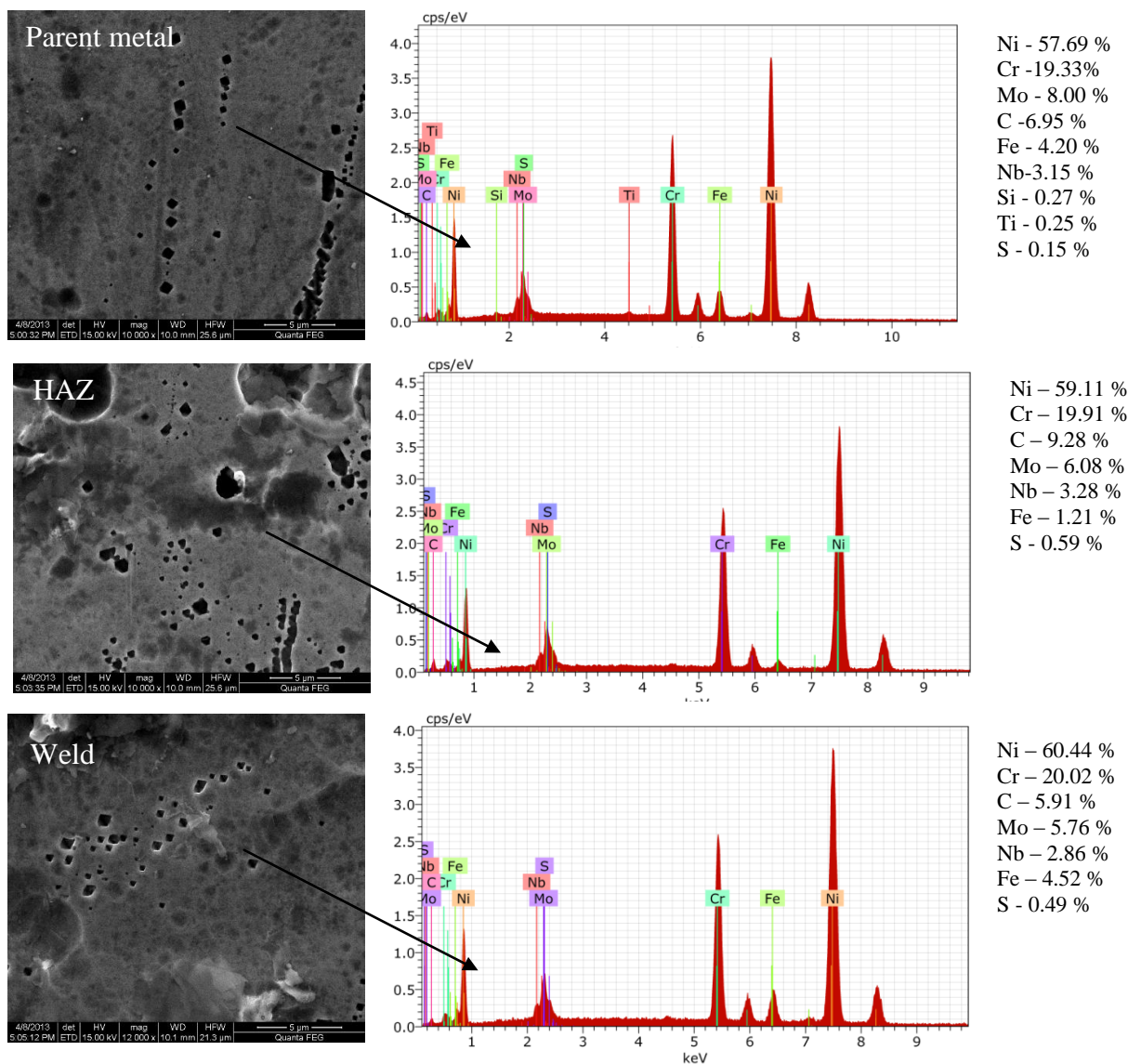


Fig. 5 SEM/EDAX analysis on the various zones of the Inconel 625 in as-welded conditions

3.4 Hot Corrosion

Hot corrosion studies were performed on the various zones of the weldments, subjected to cyclic air oxidation at 800 °C. The weight changes were recorded at the end of every cycle. It is well understood from thermogravimetric analysis shown in Fig. 6 that the weight loss was observed to be meager on the various zones of the weldment from beginning to the end of the corrosion cycle. Black spots were appeared on the parent metal from 1st cycle and these spots were spread and tarnished throughout the sample from 5th cycle till 45th cycle. Green color spots with brown tarnish color were formed on the surface of the parent metal and persistent till the corrosion run. White spots with grey color tinges were formed on the HAZ of the weldment and this grey tinges layer grew in middle and the corners of the HAZ sample turned to blackish color till the corrosion run. Multicolored tinges were appeared on the weld zone at till 5th cycle. This multicolored tinges layer was transformed to greenish yellow spots from 6th cycle till the end of corrosion.

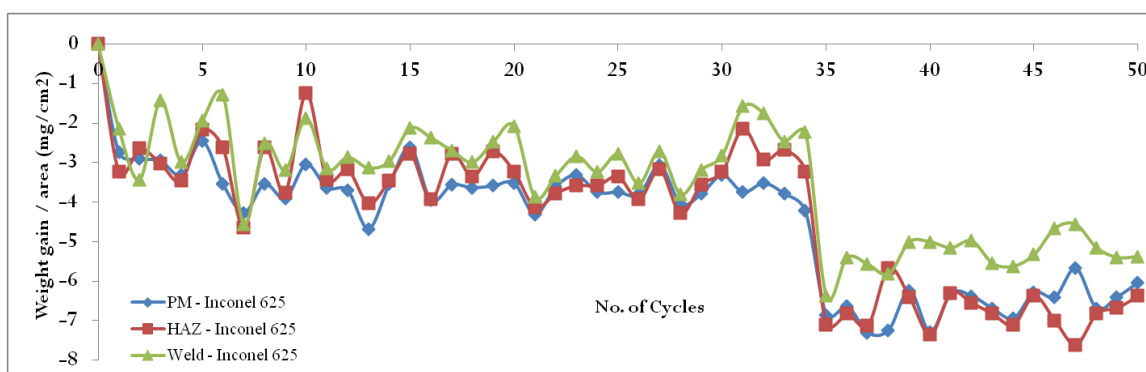


Fig. 6 Thermogravimetric analysis on the various zones of the hot corroded Inconel 625 weldments subjected to air oxidation at 800 °C

A closer observation on the thermodynamic analysis clearly indicates that the weld zone had shown better corrosion resistance as compared to other zones of the weldment. SEM/EDAX analysis was performed on the various zones of the hot corroded samples after the corrosion studies as shown in Fig.8. The analysis clearly indicates the formation of NiO, Cr₂O₃, MoO, NbO across the various zones of the weldments as predominant phases. The oxides at the weld zone appeared to have hemispherical globules.

4. Discussions

Inconel 625 plates could be successfully welded by GTA welding process. Macrostructure and NDT analysis confirmed the same. Microstructure examination revealed the formation of grain boundaries at the HAZ of Inconel 625 and the dark patches were witnessed at this zone. These dark patches would be M₂₃C₆ as reported by other researchers. As evident from the SEM/EDAX analysis, the formation of NbC, (Nb,Ti)C contributed for the better mechanical properties. Lower values of hardness and strength at the weld zone could be probably due to the presence of lesser amounts of Nb as evident from the SEM/EDAX analysis.

From the tensile and hardness plots, it is well understood that as the hardness at the weld region was found to be lower as compared to other zones, the fracture had occurred at the weld zone. It is inferred from the SEM results that the presence of dimples, micro-voids which coalesce together which was interconnected by fibrous network contributed for the ductile mode of fracture.

Further hot corrosion studies, it is understood that the weight loss was observed on all the zones of the weldments. This would be probably due to the chromium evaporation and/or spallation followed by reoxidation as reported by N'dah et al. (2007). The same phenomenon was explained by other researchers that the reoxidation can lead to rapid chromium consumption which can result in breakaway oxidation. The presence of Mo and its derivative oxides probably could have protected the surface to undergo oxidation to the minimal extent at high temperatures. This is also inferred by the presence of Mo in the SEM/EDAX analysis on the hot corroded samples of parent metal and weld zone could probably resist the oxidation. The absence of Nb on the hot corroded samples clearly conveys the spallation nature at 800 °C.

5. Conclusion

From the present investigations, the following conclusions are drawn and reported as follows:

- [1] Successful, defect free welds of Inconel 625 could be obtained from GTA welding processes employing ERNiCrMo-3 filler metal.
- [2] Tensile fracture occurred at the weld zone of Inconel 625 weldments in all the trials.
- [3] Predominant phases formed during the high temperature air oxidation include NiO, Cr₂O₃, MoO etc.
- [4] Presence of Nb in the HAZ has significantly contributed for greater strength

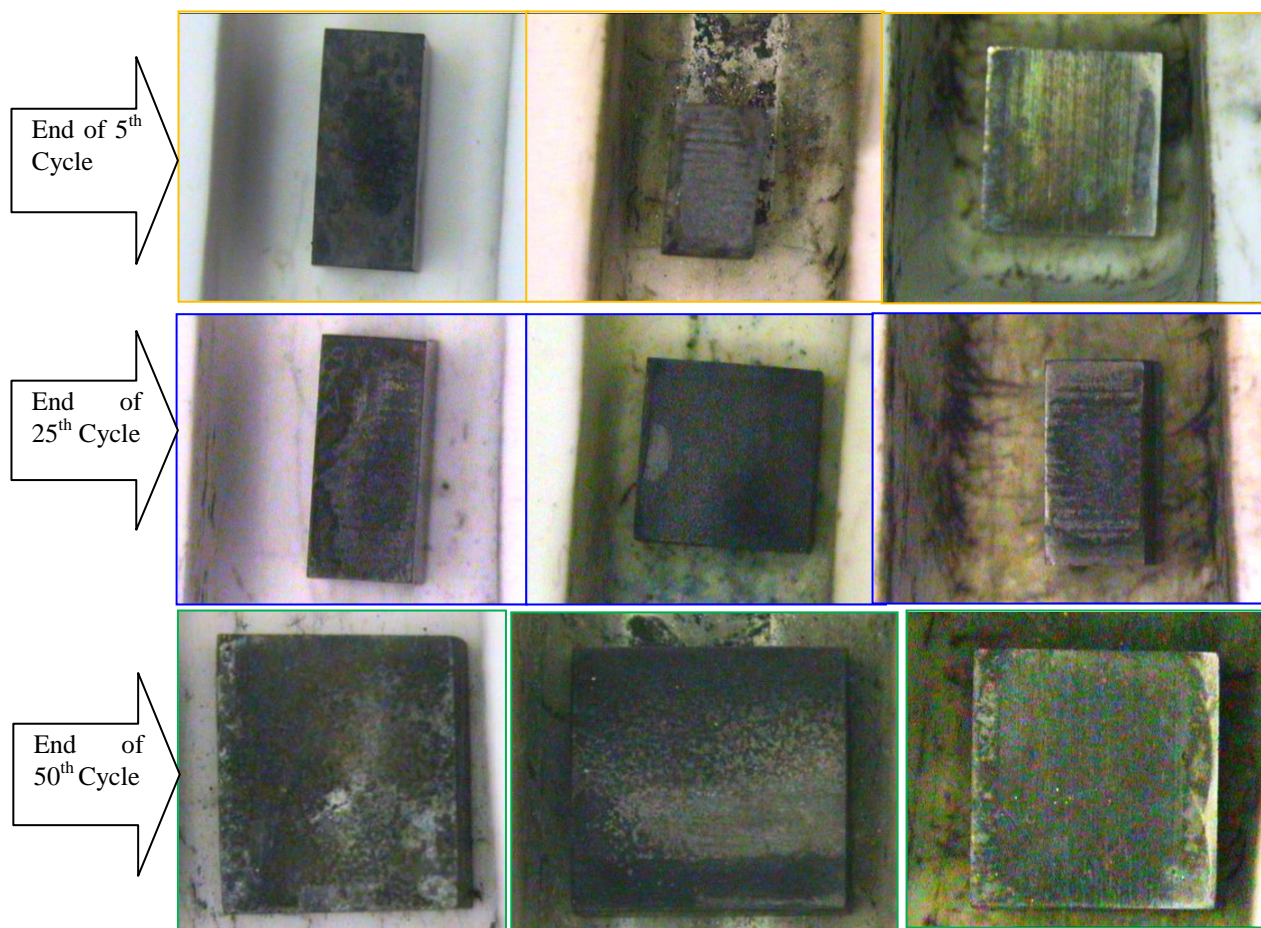


Fig.7 Hot Corroded zones of the Inconel 625 weldments

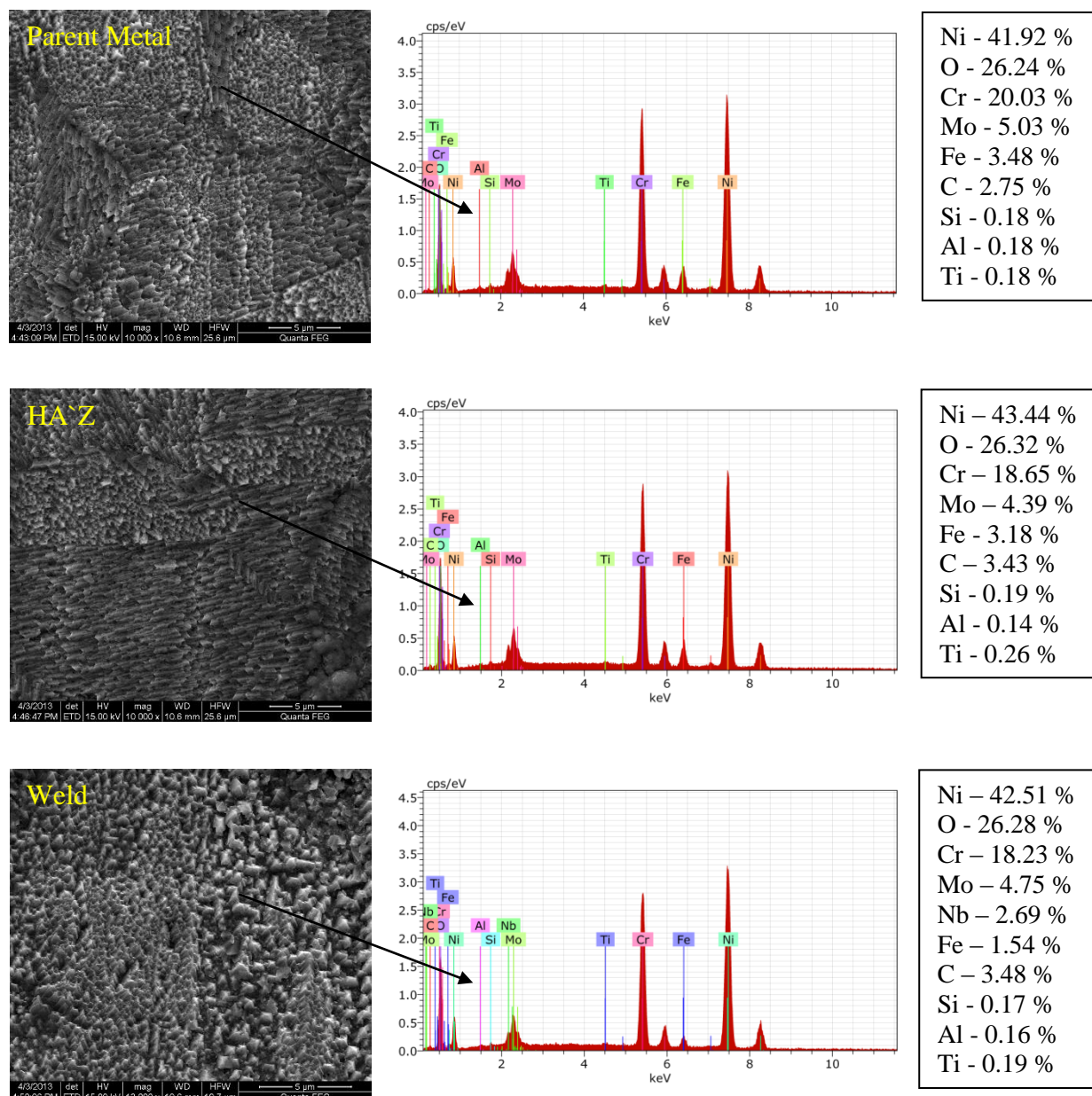


Fig. 8 SEM/EDAX analysis on the hot corroded zones of Inconel 625 weldments

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