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Effect of constant current and pulsed current gas tungsten arc welding process on microstructure and mechanical properties of superalloy 59 joints

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Abstract
This research paper investigates the microstructure, microsegregation and mechanical behaviour of Ni-based superalloy 59 which is an important candidate in the pollution control application. The weld joints were produced with continuous current gas tungsten arc welding (CCGTAW) and pulsed current gas tungsten arc welding (PCGTAW) by applying both autogenous mode and filler wire ERNiCrMo-13. Weld flaws and weld aspect ratio of weld joints were identified using a macro analysis. An optical microscope (OM) and scanning electron microscope (SEM) were used to examine the microstructure of the welded joints. PCGTA weldments exposed refined grain structure, reduced heat-affected zone and narrow weld bead compared to CCGTAW. Microsegregation of the alloying elements at the weld center (WC) and weld interface (WI) was examined using Energy Dispersive x-ray spectroscopy (EDS). The findings of the metallurgical characterisation proved that the PCGTA weldments offer minimal microsegregation at the interdendritic region in comparison to CCGTA weldments. X-Ray Diffraction (XRD) examination reveals that there is a 16.7% enhancement in grain refinement in the autogenous mode and a 17.4% improvement in the filler wire ERNiCrMo-13 when switching from CCGTA to PCGTA welding. Tensile, Charpy impact and microhardness tests were used to assess the strength, toughness and hardness of the weld joints. Weld joints fabricated by PCGTAW offers higher tensile strength (~1.4 to 1.6%), higher toughness (~4.4 to 5.4%), and higher hardness (~4.8 to 7.7%) than CCGTAW weld joints.

1. Introduction

Inconel 59 is derived from Ni–Cr–Mo ternary phase diagram, developed by VDM metals and it belongs to solid solution strengthened nickel-based alloy. It has exceptional corrosion resistance as well as great strength at high temperatures. Alloy 59 possess high stability in hot acid and chloride containing environments, due to its high nickel (Ni), chromium (Cr), and molybdenum (Mo) concentration. The thermal stability of alloy 59 is high due to the absence of tungsten (W) and copper (Cu). The material has no propensity for the given boundary dispersion in welding due to the low concentration of silicon and carbon. This leads to the use of alloy in chemical processes with reducing and oxidizing media [1, 2]. Many engineering applications, such as pollution control, (FGD units of coal-fired power plants, incinerators, scrubbers, and wastewater treatment), pharmaceutical industries, chemical industries producing fluorinated and chlorinated chemicals, paper, agrichemicals, marine, oil and gas industries benefit from alloy 59 [3–7].

The absence of W and Cu in alloy 59 made it free of any localized or intergranular attack which makes it far calibre to alloy 22, alloy C-276, alloy 686 and C-2000 [3]. The lower iron (Fe) content in alloy 59 makes it highly corrosive resistant and it is beneficial in the welding of thick sections [4, 5]. The higher chromium content ‘C’
family alloys, such as alloy C-276 or alloy 59, were adaptable to oxidising species but lacked enough molybdenum to withstand acidic environments [6].

Helena Alves et al [7] reported that the critical areas of FGD units are fabricated by alloy 59 and also observed that the alloy can cope with aggressive conditions like localised corrosion compared to other Ni-base alloys such as C-276. Alloy 59 belongs to the BAM list (Bundesanstalt für Materialforschung und-prüfung (in German) — ‘Requirements for Tanks for the Transport of Dangerous Goods’ issued by the ‘Federal Institute for Material Research and Testing’ due to its high corrosion resistance. Shell materials for tank containers carrying corrosive dangerous goods are produced using alloy 59. The welded specimen of this alloy showed superior property compared with super austenitic steels of alloy 926 and alloy 31 [8].

The conventional fusion welding processes can be implied to weld alloy 59. Gas tungsten arc welding (GTAW) is more cost-effective and could yield better quality compared to other arc welding processes. The serious issue related to the welding of solid solution strengthened Ni–Cr–Mo alloys is the occurrence of intermetallic phases due to elemental segregation. This severely diminishes the service life of the weld joints. Ni–Cr–Mo alloys are also prone to premature failure in the weldments due to the presence of topologically closed packed phases (TCP) such as σ, μ and P at the fusion zone, which is formed during solidification. This is a result of the deliberate addition of alloying components to develop properties that exceed the eutectic reaction’s solubility limit. The molybdenum content is less than 15% of weight in Ni–Cr–Mo alloys which solidify as single-phase austenite with a minimal percentage of iron and tungsten causing resistance in the formation of brittle TCP phases [9, 10]. On the other hand, an increase in molybdenum and tungsten will promote the formation of P and μ phases (TCP) during solidification causing depletion in its property [11]. From the published pieces of literature, it is evident that no appropriate research was done in the welding of Alloy 59, which provides information on microstructure and mechanical characteristics. The following literature reports the thermal stability and behaviour of various Ni-based alloys whose chemical composition is appropriately similar to Alloy 59.

Cieslak et al [12] found that the occurrence of TCP phases causes hot cracking in Ni-based alloys (C–22, C–276, and C–4). They also noticed that alloy C–276 welds had a large proportion of TCP phases and it was more susceptible to hot cracking than other alloys. Arulmurugan et al [13] employed electron beam (EB) welding to examine the reaction of post-weld heat treatment (PWHT) on Ni-based alloy 686. According to the authors, the existence of secondary precipitates in the inter-dendritic region is induced by the microsegregation of alloying elements. The increased Cr content and higher weight percentage of Mo result in elemental segregation. The occurrence of secondary TCP phases (σ, μ and P) promotes premature failure and reduces the strength.

Srikanth and Manikandan [14] investigated the effect of Cr depletion in nickel-based superalloy (Alloy 600) by the conventional GTA welding method. The authors reported the welding technique to suppress the chromium carbide precipitates at weld joints using both continuous and pulse current GTA welding with dissimilar filler wires (ERNiCrMo-14, ERNiCr-3 and ERNiCrMo-3). In GTAW, the existence of carbides precipitates (M23C6) at inter-dendritic zones was exposed, but this was mitigated by PCGTAW welding.

In another study, Subramani and Manikandan [15] reported a welding method to reduce the elemental segregation (Cr23C6) in weldments of alloy 80A. Defect-free weldments were achieved in both the CCGTA and PCGTAW welding processes with fillers ERNiCrMo-3 and 263. Due to rapid cooling, PCGTAW welding produces a fine equiaxed structure at the weld centre (WC), whereas in CCGTA welding, coarser grain structures are formed. Moreover, at the interdendritic region of the weldments, secondary phases were noticed. The high content of molybdenum and chromium precipitates was noticed in CCGTA, but it was completely impoverished in PCGTAW mode.

The microsegregation at the fusion zone of weldments at dendritic and interdendritic regions of Ni–Cr–Mo alloys is due to the presence of Mo, Nb and Cr. This alleviates corrosion resistance property [10, 16, 17]. Sathishkumar and Manikandan [18] studied the welding method to deplete the formation of carbide in Hastelloy X with filler ERNiCrMo-1. The authors reported microsegregation of high chromium and molybdenum elements at the FZ of GTA weldments, but minimal segregation was observed in PCGTAW.

The influence of filler wire ERNiCrMo-4 and ERNiCrMo-17 to control the elemental segregation of alloy C-2000 with CCGTA & PCGTAW welding processes were studied by Arulmurugan et al [19]. The authors noticed the elemental segregation of molybdenum and chromium at the inter-dendritic region of all the weldments but it was low with PCGTAW-ERNiCrMo-4. Refined grain structure (8.9 to 9.77%) was observed while shifting from CCGTA to PCGTAW welding method.

Gallagher and Lippold identified a +ve exponential link with molybdenum and tungsten elemental levels in alloy C-22 using Scheil simulation analysis [20]. Natesh and Manikandan [21] studied the hot fissuring issues in Nickelvca 23 superalloy weldments. The authors noticed the Mo segregation in the interdendritic region which forms the Mo23C6 phases with CCGTAW. All the weld joints suffered from Si segregation. On employing ERNiCrMo-10 as a filler wire, elemental segregation was suppressed in PCGTAW. XRD results revealed the occurrence of various compounds like M23C6, M12C and MC which reduces the mechanical characteristics of
the weld joints. The fine grains and enhancement of strength were noticed during the transition from continuous to pulse current mode GTAW (in both cases with and without filler wire). PCGTAW showed the best results in comparison with other welding techniques \[22\text{–}24\]. Nickel-based alloys are efficiently welded by other GTA welding techniques, but on comparison, CCGTAW and PCGTAW are found to be cost-effective \[25\text{–}29\].

The elemental segregation in the Ni–Cr–Mo weldments lead to impoverishment in its properties and this could be alleviated by the proper choice of filler wire and welding parameters \[30\text{–}34\]. The welding issue related to solid solution strengthened Ni–Cr–Mo alloy can be reduced by rapid cooling while solidification, and this is achieved using pulse current mode arc-based welding. As it is evident from the above literature, the weldments of Ni–Cr–Mo alloys have been subjected to microsegregation and this has been overcome by proper selection of welding techniques.

Hitherto, limited studies have been reported in Alloy 59 welding and characterization. Since alloy 59 belongs to the family of Ni–Cr–Mo alloys, similar kinds of issues are expected in the weldments. The current study aims to investigate the microstructure, microsegregation and mechanical characteristics of alloy 59. A comparative analysis has been made between CCGTA and PCGTA welding. The base metal matching filler wire ERNiCrMo-13 of alloy 59 has been chosen based on the manufacturer’s recommendation \[1\]. The current research findings

### Table 1. Chemical composition of Alloy 59 and filler wire ERNiCrMo-13.

| Base metal / filler wire | Ni  | Cr  | Mo  | Fe  | Al  | Mn  | Si  | Cu  | S   | P   | C   |
|-------------------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Alloy 59 (Base Metal)   | Bal | 21.51 | 16.74 | 0.49 | 0.27 | 0.19 | 0.11 | 0.015 | 0.011 | 0.019 | 0.026 |
| ERNiCrMo-13 (Filler)    | Bal | 23  | 16  | 0.5  | 0.2  | 0.3  | 0.005 | —   | 0.003 | 0.01  | 0.005 |

Figure 1. Weld photographs of alloy 59 (a) autogenous-CCGTAW (b) autogenous-PCGTAW (c) ERNiCrMo13-CCGTAW (d) ERNiCrMo13-PCGTAW.
can be utilised by the industries as listed by BAM in the fabrication of tank containers for carrying dangerous goods, which would fall under the superior application of alloy 59 [8].

2. Experiment

2.1. Materials and welding procedure
Alloy 59 plate of 700 mm × 300 mm × 3 mm was procured in hot rolled and solution heat-treated condition. A small section of the received plate of size 15 mm × 10 mm × 3 mm was extracted using Wire-cut EDM (Electric

| Welding method          | No. of pass | Current Icc, lpc, lbc (A) | Voltage, V (V) | Welding speed S (mm s⁻¹) | Heat input, Hᵢᵣ (KJ mm⁻¹) | Total heat input Hᵢᵣ+Hᵢᵣ (KJ mm⁻¹) |
|-------------------------|-------------|---------------------------|----------------|--------------------------|----------------------------|-------------------------------------|
| CCGTAW- Autogenous Root | 1cc 160     | 10                        | 1.17           | 0.96                     |                            | 0.97                                |
| CCGTAW- ERNiCrMo-13 Root| 1cc 60      | 9.6                       | 1.03           | 0.39                     |                            | 0.77                                |
| First                   | 1cc 70      | 9.8                       | 1.25           | 0.38                     |                            | 0.54                                |
| PCGTAW- Autogenous Root | 1pc 120 Ibc 72 | 9.8                       | 1.17           | 0.54                     |                            | 0.54                                |
| PCGTAW- ERNiCrMo-13 Root| 1pc 75 Ibc 55 | 10                       | 1.11           | 0.38                     |                            | 0.64                                |
| First                   | 1pc 70 Ibc 35 | 9.8                       | 1.39           | 0.26                     |                            | 0.26                                |

Figure 2. Graphic layout of weld joints (a) detailed view, (b) tensile test sample as per ASTM standard E8/E-8M-13a and (c) impact test sample as per ASTM standard E23.
Discharge Machining) to divulge the alloying elements. The chemical composition was verified with Optical Emission Spectrophotometer and the values are listed in table 1. With the EDM machine, the plates were cut to the desired dimensions of 150 (L) x 55 (W) x 3 (T). Before using the plates, they were cleaned with acetone to remove any debris, grease, and impurities. The single ‘V’ groove of included angle 60° and a root gap of 1 mm was maintained to accommodate the molten metal. Impurities from the plates are removed by acid picking (concentrated HCL + distilled water). To avoid distortion, the plates were clamped above the copper plates. The weld was carried out into four categories: CCGTAW-Autogenous, PCGTAW-Autogenous, CCGTAW-ERNiCrMo-13, and PCGTAW-ERNiCrMo-13 using the KEMPPI DWE machine. Matching composition filler wire ERNiCrMo-13 of diameter 1.2 mm is employed in the present study and its chemical composition is listed in table 1. Shielding gas (Argon) is used to cover the weld pool from the surrounding air and prevent oxidation. This leads to concentration and transfer of heat during welding, as well as to create a stable arc. The shielding gas flow rate is maintained as 15 l min
−1. The welding is done in 3 passes. The oxide layer that has been formed at the interpass is scraped with a wire brush. Weld images of alloy 59 plates are depicted in figure 1. Bead on trial is carried out to determine the optimised weld process parameters. Table 2 lists the corresponding process parameters that are employed in the current study. The following equations are used to determine the total heat input to produce CCGTAW and PCGTAW.

Total heat input to produce CCGTAW, equation (1)

\[ H_{\text{in}} = \frac{I \times V}{S} \times \eta \text{ in } \left( \frac{kJ}{mm} \right) \]  

Total heat input to produce PCGTAW, equations (2) and (3),

Mean Current \( I_{\text{in}} = \frac{(I_p \times t_p) + (I_b \times t_b)}{(t_p + t_b)} \text{ in Amphere} \)  

\[ H_{\text{in}} = \frac{I_{\text{in}} \times V}{S} \times \eta \text{ in } \left( \frac{kJ}{mm} \right) \]  

where, \( S \)—welding speed in mm min
−1; \( V \)—voltage; \( I_{\text{in}} \)—mean current in Amps; \( I_p \)—pulse current in Amps; \( I_b \)—background current in Amps; \( t_p \)—pulse current duration in ms; \( t_b \)—background current duration in ms; \( \eta \)—efficiency of the welding process 70% for CCGTAW and PCGTAW process [18, 19]. Table 2 lists the calculated heat inputs of four different welding modes employed in this study.

2.2. Metallurgical and mechanical evaluation

The test coupons for evaluation are shown in figure 2(a). The EDM was employed to slice the coupons perpendicular to the weld direction. The cross-section includes the base metal (BM), heat affected zone (HAZ)
and fusion zone (FZ). Bakelite powder was used to mount the metallographic samples using hot press mounting equipment. The polishing was done using 220–2000 grit Silicon Carbide (SiC) emery sheets, followed by alumina powder (Al₂O₃) of 0.5 μm. Finally, water was used to polish the samples to a mirror finish. To disclose the microstructure, the samples were electrolytically etched with 10% oxalic acid in distilled water at 12 V (D.C) for 30–40 s. Macro analysis was performed to identify weld defects/cracks in the welded samples (figure 3). Microscopic images of the samples (BM, HAZ, FZ) were captured using ZEISS optical microscope (figures 4–8). The microscopic analysis divulges the microstructure changes in the FZ in contrast to the BM. SEM analysis was employed to determine the presence of secondary precipitates at the weld joints. The elemental segregation in the interdendritic and dendritic regions of the weld centre and weld interface zones was studied using EDS.
analysis. XRD evaluation was made to find the crystalline grain size (d) with the help of the Scherrer equation. E8/E-8M 13a ASTM standard was employed to prepare tensile test samples (2(b)). The universal Testing Machine (make: Aimil) evaluated three tensile samples in each weld type at a strain rate of 2 mm min \(^{-1}\). Impact test samples were sliced as per ASTM E23–12C standard (figure 2(c)). To propagate the fracture at the weld centre (Cap region) a ‘V’ notch of depth 2 mm at an angle of 45° was made. SEM fractography examination was performed to determine the type of failure in tensile and impact. The hardness of the various weld joints was determined using a Vickers microhardness test. This test was performed by applying the load of 500 gf for a dwell period of 10 s.

3. Results and discussion

3.1. Macrostructure of Alloy 59

The macro images of alloy 59 weldments are illustrated in figures 3(a)–(d). It depicts the cross-sectional macro structures of continuous and pulse current mode GTA weld joints with autogenous and ERNiCrMo-13 filler. To examine the defects in the FZ, macro samples were taken in the transverse region. It is observed that there were no cracks or any other defects in the weldments. The weld pool morphology is found to be good and a full weld penetration is attained in all the weldments. Since all driving forces of the molten metal are well balanced, the steady flow of fluid is attained at the weld pool, which influences the shape and size of the weld bead. Many researchers stated that inappropriate selection of welding techniques and filler wire would cause defective weld joints, but in this case, the results indicate that the selected welding techniques and filler wire are appropriate for the base metal (BM). PCGTA weldments have a narrow weld bead width of 5.09 mm (autogenous) and 7.58 mm (ERNiCrMo-13). Whereas, in CCGTA weldments, a wider weld-bead width of 6.03 mm (autogenous) and 8.42 mm (ERNiCrMo-13) is reported. The total heat input supplied during pulse mode (Autogenous = 0.5365 KJ mm \(^{-1}\), Filler ERNiCrMo-13 = 0.6573 KJ mm \(^{-1}\)) is comparatively lesser than the total heat input.
supplied during continuous mode (Autogenous $= 0.9605 \text{ KJ mm}^{-1}$, Filler ERNiCrMo-13 $= 0.7741 \text{ KJ mm}^{-1}$). A narrower heat-affected zone can be achieved by using pulsed GTA welding rather than CCGTAW. This is because the heat energy is supplied at the peak current period ($I_p$) and allows it to be observed into the BM during the background current period ($I_b$). In comparison to CCGTAW, PCGTAW has a lower heat input, which results in a lower heat at the centre of the weld pool, resulting in lower shear stress from Marangoni convection and plasma jet. In addition, the frequent oscillations in peak and base current during PCGTAW welding allow a larger thermal gradient at the weld interface. Whereas, a lower temperature gradient would in turn result in a lower heat at the centre of the weld pool, resulting in lower shear stress from Marangoni convection and plasma jet. In addition, the frequent oscillations in peak and base current during PCGTAW welding allow space for solidification, thereby reducing heat supply. The governing forces (Buoyancy and Lorentz) of the weld bead profile are balanced [18]. This causes a decrease in weld bead width and achieves a full depth of penetration in PCGTAW welding. According to Saedi et al [35], the narrow weld beads are formed due to high electromagnetic forces that spin at the FZ during pulsing. The process parameters (table 2) determined in this study are optimal for welding a 3 mm thick Alloy 59 plate employing both autogenous and ERNiCrMo-13 filler wire.

3.2. Microstructural investigation of Alloy 59

3.2.1. Alloy 59 microstructure

The microstructure analysis of Alloy 59 (Base Metal) is depicted in figure 4. It holds several twin annealed structures at the grain boundaries. These structures are formed due to the solution treatment of alloy [14]. The twin annealed structures possess’ coherent interface at the boundaries and these boundaries can impede dislocation during deformation, enhancing high strength in alloy 59 [33].

3.2.2. Microstructure of CCGTAW and PCGTAW with Autogenous

The microstructure of the weldments (autogenous-CCGTAW) at the fusion zone shows columnar and cellular dendrites (figure 5(a)). The HAZ which is near to the weld interface (WI) is exposed with a coarser grains structure (figure 5(b)) whereas in PCGTAW weldments fine equiaxed and columnar dendritic structures are noticed at both FZ and WI (figures 6(a) and (b)). The reduction in the width of HAZ is found in pulse mode (105 ± 5) when compared to the continuous mode (143 ± 5) of heat supply (figures 5(b) and 6(b)). The coarse grain structure in CCGTAW samples is due to high heat input (0.9605 KJ min$^{-1}$) with a low cooling rate on solidification. When the thermal gradient changes, it affects disintegration at the planar solid-liquid interface during solidification, resulting in columnar and cellular dendritic formations. Grain growth could be slowed by a larger thermal gradient at the weld interface. Whereas, a lower temperature gradient would influence higher grain growth at the weld centre. At the weld interface, grain growth in the crystallographic plane direction coincides with heat dissipated, which is diagonal to the weld pool, causing a columnar structure in both autogenous CCGTAW and PCGTAW samples [23].

In autogenous PCGTAW, fine equiaxed structures are formed at the WC. In general, an equiaxed grain structure is the preferred weld structure because it provides optimum mechanical performance. In comparison to the CCGTAW, the PCGTAW produces equiaxed structures due to its low heat input (0.5365 KJ min$^{-1}$) and faster cooling rate during crystallisation. Weld solidification is intended to occur during the base current period. During this, the arc is at its most stable condition and the least amount of heat is transferred to the weld. Peak current, on the other hand, refers to the maximum current delivered to the welding arc by the power source. During welding, this current is employed to provide the required heat to melt the surface of the base metal.

In comparison to CCGTA welding, the weld metal cools more quickly because of the alternate heating and cooling cycles, resulting in a finer grain structure. The rapid cooling of the weld pool surface contributes to the formation of new nucleants during the base current. These nucleants tend to settle down with time, resulting in a homogeneous distribution across the molten weld pool. Surface nucleation leads to the formation of a finer grain structure in the weld.

Due to pulse mode at various intervals of time refined grain structures are obtained. The temperature gradient during melting (peak current) and solidification (base current) results in the formation of new grains in the weldments. The occurrence of newer grains stops and remelting of growing grains occurs due to an increase in temperature in the subsequent cycles. This causes the refinement in the grain structure in PCGTAW. Many researchers have reported a similar type of observation [18, 19, 21].

3.2.3. Microstructure of CCGTAW and PCGTAW with ERNiCrMo-13 Filler

Figures 7 and 8 depict the microscopic images of the weldments made with CCGTAW and PCGTAW with filler ERNiCrMo-13. CCGTAW (figure 7(a)) indicates the presence of columnar and dendrite structures at the FZ whereas the WI (figure 7(b)) confirms the transition of planar to cellular structures. The HAZ of CCGTAW is wider (146.3 ± 5) than its previous case, which is due to higher heat input (0.7741 KJ mm$^{-1}$) during welding. In CCGTAW weldments, a coarser grain structure is observed at both the weld centre and weld interface due to a lower cooling/solidification rate. The continuous heat supply in CCGTAW mode results in a lower cooling/solidification rate in the weldments. The microstructure of PCGTAW weldments using ERNiCrMo-13 filler is...
shown in figures 8(a) and (b). Equiaxed dendritic and a few columnar dendrites are observed at the weld centre, while the weld interface shows columnar dendrites. The transition of columnar to equiaxed is confirmed. Equiaxed microstructures showed a better resistance to crack formation and propagation. It reduces the crack susceptibility of the weld since it forces the crack to follow a more complicated path than in a columnar structure. The lower heat supply (0.6373 KJ mm$^{-1}$) and higher cooling rate in PCGTAW result in grain refinement producing equiaxed structures. The HAZ size of PCGTAW (94.7 μm) is significantly less compared to CCGTAW with filler ERNiCrMo-13. In PCGTAW, the heat generated by the pulse current melts the base metal, and the background current dissipates the heat, resulting in a smaller heat-affected zone compared to CCGTAW [19].

### 3.3. SEM/EDAX

The microsegregation in the weldments is examined using SEM/EDS. The EDS results provide the local chemical composition in the weldment at different locations. EDS analysis (30 shots) was done in the welded region. The mean value and its standard deviation are shown in tables 3–6.

#### 3.3.1. CCGTAW and PCGTAW with autogenous

SEM/EDS elemental analysis of the weldments fabricated by continuous and pulse current GTAW with the autogenous mode is shown in figures 9 and 10. Figure 9(a) represents the SEM image of the weld center (WC) and figure 9(b) shows the SEM image of the weld interface (WI). It is witnessed from the images that the secondary phases are distributed in the entire microstructure in the form of white colour particles. The images show the dendritic-core and inter-dendritic region of the WC are presented in figure 9(a) (i and ii). Table 3 shows the weight percentage consolidation of the key elemental levels at different places of the Autogenous CCGTA weld joint. As shown in table 3, the interdendritic region has more Mo and less Ni than the dendritic core. Figure 9(b) (iii and iv) illustrates an EDS examination of the weld interface, which yields results that are consistent with the weld centre. In CCGTAW, the high-temperature gradient and slow cooling rate

### Table 3. EDS results in weight % of Autogenous-CCGTAW.

| Weld type | Zone                | Ni       | Cr        | Mo       | Fe       |
|-----------|---------------------|----------|-----------|----------|----------|
| CCGTAW- Autogenous | WC dendritic Core | 65.81 ± 4.6 | 22.05 ± 1.3 | 15.57 ± 0.8 | 0.55 ± 1.2 |
|           | WC inter-dendritic core | 48.42 ± 6.8 | 23.07 ± 2.9 | 27.2 ± 1.6 | 0.56 ± 0.9 |
|           | WI dendritic core    | 61.63 ± 3.1 | 22.29 ± 1.7 | 15.07 ± 0.5 | 0.62 ± 0.6 |
|           | WI inter-dendritic core | 45.72 ± 7.4 | 22.62 ± 3.2 | 26.91 ± 1.4 | 0.54 ± 0.7 |

### Table 4. EDS results in weight % of Autogenous-PCGTAW.

| Weld type | Zone                | Ni       | Cr        | Mo       | Fe       |
|-----------|---------------------|----------|-----------|----------|----------|
| PCGTAW- Autogenous | WC dendritic Core | 61.79 ± 4.5 | 22.20 ± 5.1 | 16.23 ± 1.6 | 0.52 ± 0.5 |
|           | WC inter-dendritic core | 59.16 ± 6.1 | 21.77 ± 4.3 | 18.29 ± 1.1 | 0.63 ± 0.9 |
|           | WI dendritic core    | 57.79 ± 3.2 | 22.02 ± 2.4 | 16.14 ± 1.3 | 0.57 ± 1.1 |
|           | WI inter-dendritic core | 58.52 ± 2.7 | 22.81 ± 1.9 | 17.39 ± 1.7 | 0.55 ± 1.4 |

### Table 5. EDS results in weight % of ERNiCrMo13-CCGTAW.

| Weld type | Zone                | Ni       | Cr        | Mo       | Fe       |
|-----------|---------------------|----------|-----------|----------|----------|
| CCGTAW- ERNiCrMo-13 | WC dendritic Core | 62.32 ± 3.5 | 22.01 ± 2.1 | 15.78 ± 0.6 | 0.62 ± 0.9 |
|           | WC inter-dendritic core | 50.56 ± 4.7 | 23.26 ± 1.9 | 26.64 ± 1.3 | 0.57 ± 0.4 |
|           | WI dendritic core    | 63.63 ± 2.6 | 22.84 ± 1.4 | 12.65 ± 0.8 | 0.64 ± 1.2 |
|           | WI inter-dendritic core | 51.62 ± 2.1 | 23.08 ± 3.7 | 24.45 ± 0.9 | 0.53 ± 0.5 |

### Table 6. EDS results in weight % of ERNiCrMo13-PCGTAW.

| Weld type | Zone                | Ni       | Cr        | Mo       | Fe       |
|-----------|---------------------|----------|-----------|----------|----------|
| PCGTAW- ERNiCrMo-13 | WC dendritic Core | 64.98 ± 3.5 | 22.65 ± 2.3 | 16.59 ± 1.4 | 0.59 ± 0.8 |
|           | WC inter-dendritic core | 58.42 ± 4.4 | 23.25 ± 1.3 | 17.83 ± 0.9 | 0.63 ± 0.5 |
|           | WI dendritic core    | 63.95 ± 3.2 | 22.00 ± 1.1 | 13.37 ± 1.7 | 0.61 ± 0.7 |
|           | WI inter-dendritic core | 59.99 ± 2.6 | 23.09 ± 1.8 | 16.34 ± 0.8 | 0.56 ± 1.1 |
provide adequate time for the alloying elements to diffuse in the interdendritic region, allowing for microsegregation of Mo components. Higher magnification secondary precipitate in the WC is shown in figure 9(c). Its elemental mapping is shown in figure 9(d) and it clearly shows that Mo enriched and Ni impoverished in the secondary phases composition.

Figure 9. SEM/EDAX images of autogenous-CGTA weldments (a) SEM images of WC (b) SEM images of WI (c) SEM-Secondary phase (d) elemental Line mapping and (i) weld center EDS analysis-dendritic core (ii) weld center EDS analysis-interdendritic core (iii) weld interface EDS analysis-dendritic core (iv) weld interface EDS analysis-interdendritic core.
SEM photographs of the WC and WI of pulse current GTA weld joints are depicted in figures 10(a) and (b). The elemental levels in several locations of the weldment are summarised in table 4. Figure 10(a) displays the micrographs of SEM analysis at the composite regions which possess an equiaxed structure. In the interdendritic zones, the micrograph indicates a very low distribution of secondary phases when compared to CCGTAW. The findings of an EDS study of the dendritic-core and inter-dendritic core of WC and WI are illustrated in figures 10. Figure 10(b) displays the micrographs of SEM/EDAX analysis at the composite regions which possess an equiaxed structure.
It is noticed from the EDS images that in the interdendritic region, the Mo concentration is slightly higher and the Ni content slightly declines. PCGTAW offers a fast-cooling rate and less active diffusion by nature, which does not provide enough time for the alloying components to be segregated to a larger extent. Secondary precipitate at the WC is shown in figure 10(c) and line mapping (10.d) also confirms Mo enrichment.

Figure 11. SEM/EDAX images of ERNiCrMo13-CCGTA weldments (a) SEM images of WC (b) SEM images of WI (c) SEM-secondary phase (d) elemental line mapping and (i) weld center EDS analysis-dendritic core (ii) weld center EDS analysis-interdendritic core (iii) weld interface EDS analysis-dendritic core (iv) weld interface EDS analysis-interdendritic core.
3.3.2. CCGTAW and PCGTAW with ERNiCrMo-13 filler

SEM micrographs of CCGTAW-ERNiCrMo-13 are displayed in figures 11(a) and (b). Columnar dendrites can be seen in both the fusion zone and weld interface. Secondary precipitates are noticed in the weld centre and weld interface region. Dendritic-core and inter-dendritic zones of CCGTAW with its elemental levels are seen in figure 11 (i) and (ii). As shown in table 5, the interdendritic region has a high Mo and low Ni content. The WI

![Image of SEM micrographs and EDAX analysis](attachment:image.png)

**Figure 12.** SEM/EDAX images of ERNiCrMo13-PCGTA weldments (a) SEM images of WC (b) SEM images of WI (c) SEM-secondary phase (d) elemental line mapping and (i) weld center EDS analysis-dendritic core (ii) weld center EDS analysis-interdendritic core (iii) weld interface EDS analysis-dendritic core (iv) weld interface EDS analysis-interdendritic core.
The SEM photograph of the PCGTA-ERNiCrMo-13 weld centre and interface zone is shown in figures 12(a) and (b). In this case, the major alloying elements (Ni, Cr, Mo, and Fe) from EDS analysis are listed in table 6. Figure 12(a) (i and ii) demonstrates the EDS analysis of the dendritic-core and inter-dendritic zones of the WC. The dendritic-core and interdendritic areas have very minimal variation in their elemental levels. Similar results were revealed in the WI regions. Line mapping analysis 12(c) and (d) also confirms the Mo augmentation.

It can also be seen in the SEM images that there are no micro-cracks at the composite region of the weldments. The results are consistent with the macrostructure study. As a result of the consolidation of EDS results, it has been revealed that the interdendritic zone of both CCGTAW weldments has higher segregation. Whereas, in PCGTAW, the secondary TCP phases are the higher cooling, lower diffusion rate, and lower G ratio. In this investigation, it was found that GTA weld joints had more detrimental secondary TCP phases than PCGTAW weld joints. Secondary phases are significantly reduced in pulsed current, as evidenced by current research, both in autogenous and ERNiCrMo-13 filler cases.

The microsegregation of the alloying element is calculated using the scheil equation. This technique is widely used in solid solution strengthened Ni–Cr–Mo alloys to enumerate the elemental segregation [36]. The Scheil equation is given below in equation (4).

\[
\text{Distribution coefficient } (k) = \frac{C_{\text{core}}}{C_{0}} \tag{4}
\]

Where \(C_{\text{core}}\) denotes the elemental level in the dendritic core and \(C_{0}\) signifies the elemental level in the alloy’s nominal composition. During solidification, the level of microsegregation in the alloying element can be measured by distribution coefficient \(k\). If \(k < 1\), there is a chance of more segregation in the interdendritic area, which leads to the generation of secondary phases. However, if \(k > 1\) then the segregation occurs in the dendritic core. Table 7 depicts the \(k\) value of alloy 59 weldments using CCGTAW and PCGTAW employed with and without filler wire. It has been proven that the Molybdenum \(k\) value is significantly less than one. This shows that the interdendritic region of both CCGTAW weldments has higher segregation. Whereas, in PCGTAW, the ‘\(k\)’ value of Mo is nearly one, indicating reduced microsegregation and suppression of secondary phase
formation. Many authors have reported the same issues during the welding of Ni–Cr–Mo alloys \[19, 30, 36, 37\]. Mo segregation in CCGTAW is caused by a large quantity of heat input during welding. This leads to slow cooling during solidification, and there is a reduction in the diffusion rate. But, in PCGTAW, a lower quantity of heat is supplied, providing a higher cooling rate during solidification. The diffusion of alloying elements is much faster, resulting in minimum segregation. Another cause of elemental segregation, notably in Molybdenum, is the difference in atomic-radii of nickel and molybdenum, where Ni is found to be a solvent and Mo to be a solute. Ni and Mo have a 9% difference in atomic radii. However, the size difference between Ni and other elements (Cr and Fe) is only 1% and did not influence microsegregation.

### 3.4. XRD Analysis

To determine the crystalline size (d) of the fusion zone, XRD analysis was performed. XRD test details are given in table 8. Scherrer formula (v) and the Gaussian method are used to calculate this value.

\[
d = \frac{k \lambda}{\beta \cos \theta} \text{in nm}
\]

whereas 'λ' - x-ray wavelength in m (1.54178 × 10⁻² m), 'k'—dimensionless shape factor = 0.94, 'd'—average grain size (nm), 'β'—line broadening at full width half maximum intensity in radian and 'θ'—Bragg angle (in degree).

When using the CCGTAW-Autogenous mode, the grain size (d) is 540.53 nm; whereas in PCGTAW-Autogenous, the average grain size (d) is 450 nm. The CCGTAW-filler-13 case has a 'd' value of 460 nm, while PCGTAW-filler-13 has a 'd' value of 380.11 nm. There is a 16.7% improvement in grain refinement in the Autogenous and a 17.4% improvement in the filler-13 when shifting from CCGTAW to PCGTAW. It implies that when there is a transition from continuous to pulse mode of welding, there is significant refining of grain that occurs. It also adds to the advantage of enhancing the mechanical qualities.

### Table 9. Measuring of average grain size and dendritic arm spacing.

| Welding method          | Average grain size, nm | Average primary dendritic arm spacing, μm | Average secondary dendritic arm spacing, μm |
|-------------------------|------------------------|-----------------------------------------|-------------------------------------------|
| Autogenous-CCGTAW       | 540.53                 | 4.75                                    | 2.67                                      |
| Autogenous-PCGTAW       | 450                    | 4.02                                    | 2.27                                      |
| ERNiCrMo-13-CCGTAW      | 460.5                  | 4.97                                    | 2.51                                      |
| ERNiCrMo-13-PCGTAW      | 380.11                 | 3.78                                    | 1.90                                      |

### Figure 13. Photographs of fractured tensile test samples of alloy 59 employing (a) autogenous-CCGTAW (b) autogenous-PCGTAW (c) ERNiCrMo13-CCGTAW (d) ERNiCrMo13-PCGTAW.
ImageJ software is used to measure the primary dendritic arm spacing (PDAS) and secondary dendritic arm spacing from the microstructures of the weldments (figures 5–8) and the results are recorded in table 9. The PDAS and SDAS of the weld joints made with PCGTAW are found to be much lower. These favourable dendritic features are formed due to the effective cooling rate in PCGTA welding. PCGTAW- ERNiCrMo-13 exhibits the greatest dendritic characteristics.

3.5. Tensile test

Figures 13(a)–(d) depicts the fractured tensile test samples of alloy 59. Table 10 shows the average tensile ductility and toughness of the weldments. PCGTA welding (autogenous and ERNiCrMo-13) is found to be superior in strength and ductility compared to CCGTAW. In CCGTAW, the tensile fracture occurred at the FZ of the weldments. At the end of solidification, the thermal gradient is reduced, which causes grain growth in the FZ. Grain coarsening at the fusion zone are the reason for fracture [38]. In CCGTAW (autogenous and ERNiCrMo-13), the development of a Mo-rich secondary phase at interdendritic regions leads to a depletion in the strength of the weldments. The influence of secondary precipitates on the ductility and toughness of Ni-based alloys was examined by DuPont et al [10]. In PCGTAW, the weldments fractured at the base metal. This proves that PCGTAW samples have higher UTS than CCGTAW. The minimal heat input, high cooling rate, and

| Welding method       | Average tensile strength (MPa) | 0.2% Yield strength (MPa) | Elongation (%) | Joint efficiency (%) | Failure location |
|----------------------|--------------------------------|---------------------------|----------------|----------------------|------------------|
| Base metal           | 803                            | 704                       | 61             | —                    | —                |
| Autogenous-CCGTAW    | 793 ± 1.24                     | 518 ± 2.2                 | 29 ± 5.6       | 0.98                 | Weld Zone        |
| Autogenous-PCGTAW    | 805 ± 1.6                      | 539 ± 1.8                 | 33 ± 2.3       | 1.00                 | Base Metal        |
| ERNiCrMo13-CCGTAW    | 797 ± 5.5                      | 522 ± 3.7                 | 36 ± 7.5       | 0.99                 | Weld Zone        |
| ERNiCrMo13-PCGTAW    | 809 ± 3.2                      | 553 ± 4.3                 | 43 ± 1.2       | 1.01                 | Base Metal        |
quick solidification result in grain refinement and a reduction in Mo segregation in the fusion zone. This refinement of grain structure and lowering of elemental segregation enhances the strength and ductility of PCGTAW samples. As well as a greater number of equiaxed grain structures in PCGTA welding ensures that mechanical properties are equal in all three directions and helps to retain isotropic qualities in the weld microstructure. In addition, the spacing of the dendritic arms of an equiaxed dendrite is equal in all directions. Because of this, equiaxed grains are more ductile than columnar grains. As a result, they may deform more rapidly to sustain compression strains, resulting in superior tensile properties\[39\]. Tensile fracture of alloy 59 weldments was analysed using SEM and are shown in figures 14\(\text{(a)}\)–\(\text{(d)}\). Fractography images reveal the formation of micro-voids, ductile tear ridges, and cleavage facets. This proves the mode of failure is ductile. Due to grain refinement and the absence of grain coarsening at the weld region, the ductility of the PCGTAW sample is improved in comparison to CCGTAW.

### 3.6. Impact results

The toughness value of CCGTA and PCGTA weldments of alloy 59 are listed in table 11. Figures 15\(\text{(a)}\)–\(\text{(d)}\) show images of impact fractured weld samples. Higher toughness values are observed in PCGTA weldments both

| Type of welding          | Average impact toughness, J |
|--------------------------|-----------------------------|
| Base metal               | 80                          |
| Autogenous-CCGTAW        | 62.93 ± 4.1                 |
| Autogenous-PCGTAW        | 65.7 ± 1.6                  |
| ERNiCrMo-13-CCGTAW      | 64.03 ± 5.8                 |
| ERNiCrMo-13-PCGTAW      | 67.53 ± 3.7                 |

![Figure 15. Photographs of fractured impact test samples of alloy 59 employing (a) autogenous-CCGTAW (b) autogenous-PCGTAW (c) ERNiCrMo13-CCGTAW (d) ERNiCrMo13-PCGTAW.](image-url)
Table 12. Average hardness of Alloy 59.

| Welding process                  | Average hardness at weld zone (HV) | % increase with base metal |
|----------------------------------|-----------------------------------|---------------------------|
| Base metal                       | 220                               | —                         |
| Autogenous- CCGTAW               | 230 ± 11.8                        | 4.54%                     |
| Autogenous- PCGTAW               | 241 ± 8.3                         | 8.71%                     |
| CCGTAW- ERNiCrMo-13              | 234 ± 9.6                         | 5.98%                     |
| PCGTAW-ERNiCrMo-13               | 252 ± 9.1                         | 12.69%                    |
Autogenous and Filler wire-ERNiCrMo-13 when compared to CCGTA weldments and the values are found to be nearer to the base metal. It is believed that the decrease in toughness in CCGTAW samples is due to grain coarsening and the occurrence of Molybdenum rich secondary precipitates in the weld region. The reduced segregation and refinement of grains at the FZ of PCGTAW samples are considered to be responsible for the above results. The presence of a greater number of equiaxed fine grains is also the reason for the higher toughness of PCGTA weldments. It is relevant to note that many authors have reported the improvement of impact toughness of Ni–Cr–Mo alloys after switching from CCGTAW to PCGTAW [21–23]. Figures 15(a)–(d) indicates that the impact test samples did not break completely. This indicates that the type of failure is ductile. The SEM fractography analysis of fractured samples of Alloy 59 is shown in figure 16(a)–(d). The images expose the presence of ductile tear ridges, cleavage facet and dimples which indicates ductile fracture.

3.7. Microhardness examination

Figure 17 shows the hardness profile of alloy 59 weldments, which is taken across the transverse direction of the weld joint. The hardness profile demonstrates that the fusion zone (FZ) hardness is higher than the BM and HAZ in all the cases. It is also noticed that the large angle of undulations in CCGTAW weld joints is owing to the existence of high content of secondary precipitates. Table 12 show that PCGTAW welds have higher hardness rather than CCGTA welds. According to microstructural examinations, the lower hardness of the CCGTA weld joints is due to the occurrence of coarser grains in the FZ and HAZ (figures 5 and 7). The highest average hardness value is observed in the weld joint made by PCGTAW. PCGTAW welding resulted in 4.8%–7.7% greater average hardness when compared to CCGTA mode. The molten pool is agitated by high peak current values, which leads to grain refinement in the weld zone and consequently, enhances the hardness. More amount of fine equiaxed grains existing in PCGTAW (which have a higher number of atoms per volume) can withstand indentation better than coarser columnar grains in CCGTAW (lower number of atoms per volume). The Hall-Petch equation validates that a material’s yield strength increases when its grain size is reduced. The yield strength of a material is proportional to its hardness.

4. Conclusions

The following observations are made from this study

1. Defect-free weldments of Alloy 59 are produced by both CCGTAW and PCGTAW with optimised process parameters.

2. Microstructure of PCGTAW weldments possesses refined equiaxed microstructure in the fusion zone whereas coarser grain cellular/columnar structures are noticed in CCGTAW weldments. The rapid cooling rate, low heat input and high thermal gradient adapted in PCGTAW produce refinement in grain structures and small HAZ compared to GTA welding.

3. SEM/EDAX analysis revealed the occurrence of Mo rich secondary precipitates and severe elemental segregation in weld centre (Autogenous–CCGTAW) and weld interface (Filler ERNiCrMo-13–CCGTAW). But it was found significantly lesser in PCGTAW at weld centre and weld interface. The lower cooling rate and constant heat supply in the continuous mode resulted in severe elemental segregation of Mo which led to the formation of secondary precipitates.

4. The XRD results show that switching the welding technique from continuous to pulsed mode results in grain refinement. The grain refinement in pulse mode is 16.7% for autogenous and 17.4% for filler ERNiCrMo-13 compared to the continuous mode.

5. Mechanical testing reveals that the weld joints fabricated by PCGTAW offer higher tensile strength (~1.4 to 1.6%), higher toughness (~4.4 to 5.4%), and higher hardness (~4.8 to 7.7%) than CCGTAW weld joints. The refined microstructure and low segregation improve the mechanical characteristics of the weldments as seen in PCGTAW.

6. The detailed outcomes of the research demonstrate and recommend the use of PCGTAW with ERNiCrMo-13 filler wire to weld Alloy 59. This satisfies the superior application of Alloy 59 in FGD and in the fabrication of tanks for carrying dangerous goods as listed by BAM.

Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.
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