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# Microstructural characterization of liquid nitrogen cooled Alloy 718 fusion zone

S.G.K. Manikandan[a,*], D. Sivakumar[a], K. Prasad Rao[b], M. Kamaraj[c]

[a] Indian Space Research Organization, Trivandrum, India
[b] University of Utah, Salt Lake City, United States
[c] Indian Institute of Technology Madras, Chennai, India

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ABSTRACT

The interdendritic Laves phase and the microsegregation have been investigated in Alloy 718 fusion zone cooled with liquid nitrogen during welding. Conventional GTA welding process was employed with modified waveform and two types of shielding gas and filler metal (solid solution and age hardenable). The weld cooling rate was enhanced using liquid nitrogen cooling during Gas Tungsten Arc welding process. The resultant fusion zone microstructures were characterized using the metallurgical tools. Dendrite remelting phenomenon was observed from the optical micrographs. It was found that the enhanced cooling rate with liquid nitrogen reduced the interdendritic phases which were confirmed in both the electron microscopic and the X-ray diffraction analysis. The elemental mapping in scanning electron microscope-energy dispersive spectral analysis also confirmed the reduced microsegregation. The dendrite arm spacing was reduced from the range of 15–54 μm (CCPHE–CCAR, conventional) to 3–17 μm (CCPHE–CCAR, liquid nitrogen cooled) for the employed process variables. The computed weld cooling rate was found to be enhanced from seven to fifteen times than the conventional welding process.

## 1. Introduction

Solute redistribution during weld pool solidification is an important phenomenon resulting in segregation that can significantly affect weldability, microstructure, and properties. David and Vitek (1989) studied the different solidification models to describe the solute redistribution during weld metal solidification and correlated the weld metal microstructures with solidification parameters such as temperature gradient, growth rate and the resultant interface undercooling (David and Vitek, 1989). Böttger et al. (2000) stated that such solute redistribution resulted in the formation of chemical inhomogenity and consequent interdendritic phases. Use of Nb as solute element and/or age hardener in Alloy 718 resulted in the formation of brittle Laves phase in the fusion zone. Radhakrishna et al. (1995) and Janaki Ram et al. (2004, 2005) found that the faster cooling rate in pulse current welding process reduced the level of interdendritic Nb segregation and the amount of Laves phase. Manikandan et al. (2012, 2013, 2014) studied the combined effect of shielding gas and the modified pulse wave form on Alloy 718 fusion zone. It was inferred that the cooling rate was enhanced with the steeper temperature gradient. High energy density welding processes like electron beam welding (EBW) are in use for enhancing the weld cooling rate due to the lower energy input rate. Madhusudhana Reddy et al. (2009) studied the behavior of Laves phase formation behavior in Alloy 718 fusion zone by adopting electron beam oscillation technique and inferred that the elliptical oscillation technique reduced the microsegregation. The complex and restricted structure of rocket systems had a limitation in adopting such processes. Ultimately the heat or energy input and heat dissipation dynamics during fusion welding process influenced the microsegregation and microstructures of fusion zone (FZ) and heat affected zone (HAZ). Hence a study on rapid heat dissipation using liquid nitrogen was initiated during manual GTA welding process for enhancing the weld cooling rate.

Jackson (1966) proposed the concept of time–temperature effects on the strength of aluminum welds and found that the strength characteristics improved with higher maximum temperature and lower time at temperature. It was reported by Koichi (1972) that the shorter time at temperature enhanced the weld

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*Abbreviations:* CCAR, constant current argon; CCPAR, compound current argon; CCHE, constant current helium; CCPHE, compound current helium; EBW, electron beam welding.

[*] Corresponding author. Tel.: +91 9442305959; fax: +91 44 22574752.
*E-mail addresses:* nehakutty06@gmail.com (S.G.K. Manikandan), d_sivakumar@vssc.gov.in (D. Sivakumar), jyothipr@gmail.com (K. Prasad Rao), kamaraj@iitm.ac.in (M. Kamaraj).

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**Table 1**
Chemical composition of base metal and filler metals.

| Elements | C | Mn | Si | Cr | Fe | Mo | Nb + Ta | Ti | Al | B | Cu | Ni |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Base metal | 0.05 | 0.11 | 0.11 | 18.15 | 19.77 | 2.89 | 5.02 | 1 | 0.62 | 0.002 | 0.03 | Bal. |
| FM1 | 0.05 | 2 | 0.5 | 16 | 5 | 16 | – | – | – | – | – | Bal. |
| FM2 | 0.08 | 0.55 | 0.35 | 21 | 16 | 3.3 | 5.5 | 1.15 | 0.8 | – | 0.3 | Bal. |

strength due to the reduction in microsegregation. The control of time–temperature effects was investigated using different methods by various researchers (Jackson, 1966; Koichi, 1972; Cole et al., 1966; Amuda and Mridha, 2012, 2013; Gabzdyl, 2002; Hamatani et al., 2006; Kala et al., 2014; Sharma et al., 2012). Those were briefed in the following paragraphs.

The heat absorption and/or dissipation during welding were enhanced by adopting a forced cooling in the present work. This was achieved by impinging liquid nitrogen on the copper base plate in contact with the weldment. Cole et al. (1966) and Amuda and Mridha (2013) reported the usage of liquid nitrogen cooling during welding process for the improvement in strength of aluminum welds and grain refinement in ferritic stainless steel welds respectively. Solid $CO_2$ was employed in Laser welding for distortion control by Gabzdyl (2002). Hamatani et al. (2006) employed liquid nitrogen cooling during laser welding of the ultra fine grained steel to avoid the heat affected zone (HAZ) softening. Kala et al. (2014) used liquid nitrogen cooling and minimized the residual stresses in the fusion zone and HAZ of mild steel weldments. Sharma et al. (2012) employed in-process cooling using compressed air, liquid nitrogen and normal water during friction stir welding of AA7039 and found the mechanical properties of the weldment were improved. Amuda and Mridha (2012) demonstrated an improvement of weld ductility to the tune of 80% of the base metal by employing liquid nitrogen cooling during welding of AISI 430 ferritic stainless steels. In the present study, liquid nitrogen cooling was employed during welding and resultant microstructures were analyzed.

## 2. Experimental setup

Alloy 718 sheets (2 mm thick) in solution treated condition (980 °C) were joined by gas tungsten arc welding (GTAW) process using two filler metals (FM1 and FM2) of size Φ1.6 mm. The details of the chemical composition for base material and filler metal are given in Table 1. Square butt joints with tight fit-up were used. The weld parameters for an optimized heat input value of 0.75–0.77 kJ/mm were employed from the works of Manikandan et al. (2014) as shown in Table 2. The weld coupons were rigidly clamped on the welding fixture (box type cooper cooling system) as shown in Fig. 1.

The cooling system had a hole of 12 mm in diameter at the outlet end for avoiding pressure build up by nitrogen vapor during welding process. In addition, 6 numbers of equi-spaced holes of 1.5 mm diameter were drilled along the length of the copper cooling system as shown in Fig. 1. The nitrogen vapor escaped through these holes during welding had a cooling effect on the base material. This mechanism avoided the distortion of the welded sample due to the transverse temperature gradient. The entire assembly was kept inside the Helium filled welding hood and sealed to avoid ice formation in the weld coupon and copper cooling system under liquid nitrogen environment. Liquid nitrogen was supplied through the inlet of copper cooling system. The flow rate of liquid nitrogen was adjusted so those at the outlet of copper cooling system only liquid nitrogen were present (no gaseous nitrogen). Nitrogen vapor was formed during welding by extracting the heat. Thus a mixed flow ($LN_2$ and cold nitrogen vapor) could be achieved since the balancing of $LN_2$ inflow rate and boiling rate was found to be difficult during welding process.

**Table 2**
Welding process variables (Manikandan et al., 2014).

| Shielding gas | Process variable | Heat input (kJ/mm) |
|---|---|---|
| Argon | Constant current (CCAR) and compound current pulse (CCPAR) | 0.75–0.77 |
| Helium | Constant current (CCHE) and compound current pulse (CCPHE) | |

Welded samples were qualified for 100% X-ray radiography and dye penetrant test. The samples were etched using Kalling's reagent and the weld microstructures were observed under Leica optical microscope. Electron microscopy and X-ray elemental mapping were conducted on the weld metal using field emission gun scanning electron microscopy (FE-SEM) with a spatial resolution of 2–3 nm. Welded samples were subjected to the aging treatment as per AMS 5596G. Discs of 3 mm diameter were punched out from thin foils of 90 μm thickness in the as welded fusion zone. These discs were further thinned down by twin jet electro-polishing technique with an applied voltage of 18 ± 2 V. The electrolyte used was one part of perchloric acid and 4 parts of ethanol at a temperature of approximately 233 K. These foil type specimen were observed using Jeol make transmission electron microscope (TEM) equipment. XRD experiments were performed to identify the different phases in the fusion zone using Cu-Kα radiation.

## 3. Results and discussion

### 3.1. Microstructural characterization of fusion zone

Four levels of weld cooling rates were reported previously in the work of Manikandan et al. (2014) and the corresponding welding variables were employed in the present study. The rapid heat extraction using liquid nitrogen resulted with the enhanced weld cooling rate. Fig. 2 shows the base material micrograph. The micrographs of the conventionally GTA welded Alloy 718 fusion zone revealed thick and continuous Laves network associated with Nb enriched zones as shown in Fig. 3a. The microsegregation and Laves particles were seen in the heat affected zone as shown in Fig. 3b.

The fusion zone of liquid nitrogen cooled weld metal showed dendrites of different morphology as shown in Fig. 4a–e. The equiaxed growth of dendrite arms was disrupted in zone A (Fig. 4c) of the fusion zone (top side of weldment). The micrograph of zone

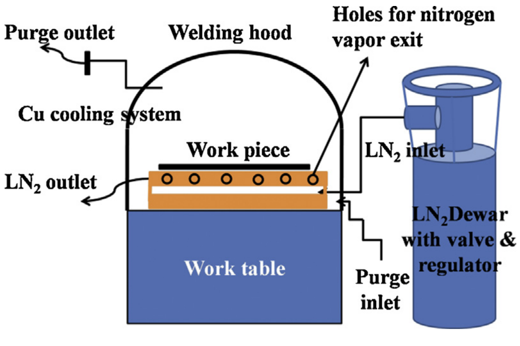

**Fig. 1.** Schematic of experimental set up.