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# Materials Science & Engineering A





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# The role of Ti carbonitride precipitates on fusion zone strength-toughness in submerged arc welded linepipe joints

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## article info

Article history: Received 30 July 2014 Received in revised form 20 October 2014 Accepted 21 October 2014 Available online 13 November 2014

Keywords: Steel Submerged arc welding Mechanical characterization Precipitation Carbonitrides

# **ABSTRACT**

The role of micro-alloying in the submerged arc welding (SAW) of high strength low alloy steel linepipe is paramount in facilitating the high strength properties of the linepipe. In this study, transmission electron microscopy analysis revealed the presence of large (0.85  $\mu$ m) Ti (C,N) precipitates within the predominantly acicular ferrite SAW joint. Cross-weld Vickers hardness and Charpy impact tests revealed that the fusion zone has high hardness and low toughness properties in relation to the base metal and heat affected zone. Fractography observations made on the ductile fracture surface of the fusion zone revealed a high number of the large Ti (C,N) precipitates to be located within the microvoids – suggesting their role in nucleating microvoids. Finally, fracture micro-mechanics are used to evaluate the relationship between the coarse precipitates and reduced strength-toughness properties in the SAW weld of the API-5L grade X65 linepipe steel.

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## 1. Introduction

The increasing global demand for energy resources has led to the development of modern high strength low alloy (HSLA) linepipe steels which exhibit excellent strength and toughness properties. In the production of the tough HSLA steels, enhancement of strength levels can be achieved by microalloying; that is, the addition of elements such as niobium, vanadium and titanium at a level of only a few hundredths of a weight percent to the alloy. The micro-alloying elements have a strong affinity with the interstitial elements, such as carbon and nitrogen, and stimulate precipitation strengthening [\[1\]](#page--1-0). Precipitation strengthening, however, normally reduces impact toughness [\[2\]](#page--1-0) and so an accompanying refinement of the microstructure is required [\[3\]](#page--1-0). Most commonly, the necessary refinement is achieved by fine dispersions of stable TiN particles pinning prior austenite grains during a thermomechanically controlled rolling (TMCR) process [4–[6\].](#page--1-0) The strong reduction in the average grain size of the ferrite as a result of these pinning characteristics facilitates the high strength of the steel, along with improved toughness properties, as described by the Hall–Petch relationship [\[7\].](#page--1-0) In contrast, the welded joint of deep sea oil and gas linepipes, produced by localized heating and continuous cooling, is subjected to no such refinement. Consequently, the strengthening role of the TiN within the weld joint of

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oil and gas transportation linepipes is somewhat redundant in a grain refining sense.

Furthermore, studies [\[8,9\]](#page--1-0) have shown that TiN particles that form in the molten or semisolid matrix can grow large ( $> 0.5 \mu$ m) in size. As such, Ti and N levels, as well as the process parameters such as solidification rate, need to be carefully controlled in order to achieve optimum toughness levels. Yan et al. [\[10\]](#page--1-0) demonstrated that large TiN inclusions were found to affect the toughness of micro-alloyed steel plates. It was shown, that when the product of the Ti and N content is larger than the solubility product of TiN at the solidus temperature, TiN manifests in the liquid as large inclusions. On the other hand, if the contents are low so that TiN does not form until the solid state, then fine precipitates can be formed. Furthermore, the inherently brittle non-metallic inclusions have often been reported to initiate the cleavage  $\left[11-13\right]$ . It has also been reported in the literature that at low temperatures, the specimens could also remain in almost completely elastic state during the Charpy V-notch (CVN) tests, and it was found that TiN cleavage initiation occurs at a characteristic distance away from the notch tip [\[14](#page--1-0)–16]. Noticeably, in the case of room temperature, where considerable plastic fracture is present on the fracture surface, less attention has been paid to the function of the TiN particles on fracture initiation.

It is evident that the role of TiN in contributing towards cleavage in base metal of HSLA steel is well understood. Recent studies by the authors [\[17\]](#page--1-0) have highlighted that TiN within the fusion zone may be acting as nucleation sites for ductile fracture and as such reducing toughness properties. The study showed that there was no evidence for the particles contributing to reduced toughness in the base metal

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and HAZ. Yet, the role of TiN particles on the strength and toughness of the fusion zone in welded HSLA linepipe steel is still severely lacking and as such will be the focus of this research.

## 2. Materials and experimental methods

# 2.1. Materials and pipe manufacturing procedure

As one of the several grades controlled by American Petroleum Industry (API) standard [\[18\],](#page--1-0) API-5L grade X65 steel is widely used as the main linepipe material for transporting oil and gas in harsh and corrosive deep sea environments. The HSLA steel linepipes are normally produced by U-forming, O-forming, and Expansion (UOE) processes. UOE process involves forming steel plate into pipe through edge crimping, U-forming and O-forming before doublesided submerged arc welding (SAW) along the longitudinal seam [\[19\]](#page--1-0); after the SAW, the pipe is mechanically expanded to the nominal diameter to complete the UOE process.

A 0.5 m long section of SAW API-5L grade X65 linepipe was acquired for the study. Table 1 lists the nominal chemistry of the 25.4 mm gauge thickness, API-5L grade X65 steel source plate, and the welding wires used for SAW. The API-5L grade X65 source plate was manufactured through a thermo-mechanical process with accelerated cooling. The UOE pipe has a final outside diameter (OD) of 457.2 mm after expansion. Prior to expansion, the formed pipe was joined using a multi-wire SAW process in two main passes from inner diameter (ID) and outer diameter (OD). The ID welding was performed first using 4 wire welding heads with a travel speed of 1.65 m/min. Next, the OD welding was carried out using 5 wire welding heads with a travel speed of 1.9 m/min. The inter-pass temperature was kept at below 50 $\degree$ C in order to improve toughness performance.

# 2.2. Microstructure observation and mechanical testing

Samples were extracted from the SAW seam weld of the UOE linepipe section for specimen preparation and examination in the cross section transverse to the welding direction. All specimens were ground by silicon carbide abrasive paper, up to a final grade 1200. Specimens were then polished to a  $3 \mu m$  finish and lightly etched using nital solution (a mixture of 2% nitric acid and ethanol). The fusion zone microstructures were then examined at  $t/4$  ( $t$  is the thickness of the plate) from the OD using an Olympus BX51 optical microscope (OM) and an FEI Sirion 200 field emission gun in a

#### Table 1

Nominal and measured chemistries obtained through optical emission spectroscopy.

FEGSEM. Energy dispersive X-ray (EDX) analysis was performed on the SEM using Spirit software package. A FIB microscope (FEI FIB200) and a dual-column FIB (Carl Zeiss Nvision N40) were used to prepare TEM specimens. The TEM specimen preparation methodology for steel samples has been documented in [\[20,21\].](#page--1-0) Cross weld Vickers hardness tests were made at 1 mm intervals across the OD weld, transverse to the SAW joint, as depicted in [Fig. 1](#page--1-0) using a 10 kg load with a 10 s loading time. Cross-weld Charpy V-notch (CVN) samples (50 mm  $\times$  $10 \text{ mm} \times 10 \text{ mm}$ , with 2 mm notch depth and 0.25 mm root radius) were produced and tested at  $0^{\circ}C$  in accordance with API standard [\[18\]](#page--1-0), the details of which including the location and orientation of the CVN samples with respect to the weld joint have been described in a previous study [\[17\]](#page--1-0). Fracture toughness tests of the weld metal were also carried out at  $0^{\circ}$ C in accordance to ASTM E 1820 with samples of the same size, orientation and position as used for the Charpy specimens. A total of 6 fracture toughness tests were carried. Prior to the tests pre-cracks were generated for all the 6 specimens including a 3.5 mm notch depth located centrally and transverse to the weld, plus a targeted minimum of 1.3 mm fatigue crack growth.

# 3. Results

## 3.1. Chemistry

Table 1 summarises the measured chemistries of the as-welded SAW joint, obtained through optical emission spectroscopy (OES). Small additions of grain refining elements V (0.002 wt%), Nb (0.041 wt%), and Ti (0.013 wt%) were found within the grade X65 base metal. Similarly low levels of V (0.003 wt%) were found within the weld, however the weld exhibits higher levels of Ti (0.025 wt%) and lower Nb (0.025 wt%) concentrations in comparison to the base metal. As well as these intrinsic features within the respective weld regions, increased levels of Si and Mn in the fusion zone (weld) are seen in Table 1 as a result of the diffusion of small amounts of Si (0.1 wt%) and Mn (0.15 wt%) from the flux into the fusion zone during the SAW. The Ti and N contents play an important role in the formation of TiN particles. Higher contents of Ti and N can result in the formation of coarse TiN particles before solidification ends. Therefore, Ti and N contents should be controlled to be less than the solubility limits at solidus temperature to avoid the formation of coarse TiN particles. Also, the Ti/N ratio is significant to the growth of TiN particles during cooling. If Ti/N ratio is greater than the stoichiometric TiN (3.42), then TiN can



 $C_{eq}$  = Carbon equivalent value =  $C + \frac{Si}{30} + \frac{Mn + Cu + Cr}{20} + \frac{Ni}{60} + \frac{Mo}{15} + \frac{V}{10} + 5B$  wt% [\[18\].](#page--1-0)

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