



Investigation on the microstructure and toughness properties of austenitic and duplex stainless steels weldments under cryogenic conditions



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ABSTRACT

This study presents deep-resolved metallurgy and fractography of the weldments beyond the routine examination of the welded constructions working under cryogenic conditions. Duplex-austenitic and austenitic-austenitic stainless steel plates were welded by means of a shielded metal arc welding. The impact toughness of the weldments was assessed at both subzero and ambient temperatures. The weld microstructure was composed of Widmanstätten austenite and a ferrite matrix at the duplex-austenitic weldment. The microhardness values varied from a maximum of 330 HV0.1 at the duplex parent metal to 200 HV0.1 at the austenitic parent metal due to the phase transitions from an f.c.c. plus b.c.c. to a fully f.c.c. crystalline structure. Under cryogenic conditions, greater impact toughness for the weld metal was determined at the duplex-austenitic weldment relative to the austenitic-austenitic weldment. The weldments exhibited ductile fracture failures down to -80°C . In accordance with the decrease in the sub-zero test temperatures, the standard deviation in the impact energy values decreased, and the fracture was still ductile and stable for the weldments. At -80°C and -176°C , cleavage surfaces were observed in the duplex-austenitic welded impact bar samples and cleavage fracture data were more reproducible with respect to ductile fracture data.

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1. Introduction

In the steel selection process for making special pressure vessels such as chemical reaction, vacuum and cryogenic vessels, vessel designers take advantage of the superior features of austenitic stainless steels, including greater toughness, increased uniaxial tensile plastic strain (with respect to carbon steels [1]), corrosion resistance and hygiene. Because of their face-centered cubic (f.c.c.) crystalline structure, austenitic stainless steels exhibit higher strength coefficients than body-centered cubic (b.c.c.) crystalline steels during elasto-plastic deformation under cold-worked conditions [2]. In the case of monotonic loading over a yield stress of austenitic stainless steel and subsequent uniaxial tensile unloading cycle, the value of the yield stress increases at the next loading. Thus, a greater increase in the yield strength of austenitic stainless steels occurs after several loading/unloading cycles render the strain hardening [3]. The subzero temperatures slightly affect their toughness [1] and elongation at uniaxial tensile loading [4].

Duplex stainless steel (DS) has dual phases, including solid solutions of b.c.c. and f.c.c. iron, ferrite and austenite, respectively (they are also called “ternary ferrite” and “ternary austenite” [5] because of the substituted Cr and Ni together with interstitial C atoms in the cubic crystalline iron cells for the Fe–Cr–Ni steels). In general, DS includes almost equal volume fractions of the ferrite and austenite phases [6]. It is also known that the micro-hardness values of individual ferrite grains are greater than those of austenite grains in the microstructures of Fe–Cr–Ni alloys [7]. The differences in these phases can result in residual stress in the metals during or after deformation processes [2]. Thus, DS is prone to the residual stress states that contribute to micro-hardness. Because DS includes ferrite grains along with austenite grains, unlike the fully austenitic microstructures of austenitic stainless steels, DS has greater bulk hardness and finer grains. Its strong metallurgical constitution and greater strength can be maintained when exposed to reasonable plastic strains in applications such as pipes or vessels. The vessels that will be utilized under cryogenic conditions are constructed of austenitic and/or duplex stainless steel plates. In forms of pipe or vessel, austenitic and duplex stainless steel plates cannot be straightened with post-heat treatments as addressed in the deformation ageing [8] and strain ageing [9].

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However, straightening can be accomplished through cold-stretching with a reasonable internal pressure satisfying yield criteria in the stainless steels.

Assuming that vessels can be designed and made completely from austenitic or duplex stainless steels is not realistic because of the numerous weld beads required to join the pre-existing standard parts of a vessel wall that is composed of previously deformed sheets. In manufacturing facilities, a thin-walled cryogenic vessel body previously prepared from duplex stainless steel sheets is closed and welded at the elliptical steel heads and the similar steel filler, respectively. The standard pre-existing products made of commonly austenitic ASTM 304 pre-cast or rolled grades, i.e., pipes, fittings, elbows, flanges, manhole, or feed-through ports, are welded on the circumferential vessel wall by a consistent stainless steel fillet. The cold-stretching is performed after completion of the welding process at a reasonable diametric enlargement at a hydrostatic high internal pressure, sending and releasing pressurized water to and from the vessel several times. This treatment provides remarkable benefits resulting from the aforementioned improved mechanical properties compared to those attained in versions that are thicker-walled but are made of low-carbon steels with b.c.c. crystalline structures. The as-welded cryogenic vessels are then evacuated, with tests proceeding at low internal pressures (approx. 10^{-2} mbar) in vacuum to facilitate the detection of leakage. Liquefied carbon dioxide, LNG and CNG storage/transportation vessels (cargo tanks) are good examples of duplex stainless steel-welded constructions (2205 grade), and liquefied nitrogen storage vessel walls are commonly made from as-rolled 304 stainless steels sheets. The service safety of highly loaded welded constructions is predominantly dependent on the integrity and fracture resistance of their welded joints [10]. For these reasons, the weldments (butt-joints, lap joints and T-joints) of the austenitic (grade 304) and duplex (grade 2205) stainless steels play a significant role in the design and safety of cryogenic vessels. However, the advanced metallurgical findings associated with impact toughness data for thick-walled weldments have not been shared with recruiters, apart from those that are available in some relevant designations for failure analyses of the vessel constructions [11–15]. The literature survey indicates that although other mechanical properties of austenitic and duplex stainless steels have been examined, there is relatively less information about the toughness and impact behavior of these welded joints. Although the metallurgy and fracture toughness of the weldments have been investigated and reviewed at room temperature in several works using different metallic materials and processing variables [16–19], works related to evaluating subzero temperature behaviors of the weldments are limited [20–23]. Instead of readdressing the complex fracture toughness parameters in the previous studies [11,15,18,19], this study implements a fracture analysis on the weldments for the cryogenic vessels, supported with visual metallurgical findings.

The aim of this study is to determine cryogenic effect on impact toughness of weld metal, heat affected zones, the 2205 and 304 parent metal. In this framework, the 2205–304 plates were welded through SMAW technique to produce the weldments. The study presents the experimentally observed differences in the metallurgy of austenitic and duplex stainless steel weldments with their change in impact toughness with respect to different cryogenic temperatures. The impact test results are also compared with those of the 304–304 weldment to benchmarking.

2. Experimental procedures

The 10 mm-thick sheet plates, EN 1.4301 (ASTM 304) and EN 1.4462 (ASTM 2205) grades, were provided by ISISAN ISI SAN.

A.Ş. Both steel plates have similar deformation ratio. They were previously analyzed by an OBLF spark spectrometer, and their chemical compositions are provided in Table 1. The ends of both sheets were prepared in an “X-joint” and butt-welded with shielded metal arc welding technique by using filler metals.¹ The weld bead was perpendicular to the rolling directions of both sheet plates to be welded. The welding parameters are shown in Table 2.

For the metallographic examination, after a grinding and subsequent polishing process, the transverse sections of the weldments were etched with a solution consisting of 30 ml HCl, 15 ml HNO₃ and 5 ml HF.² Microscopic hardness measurements (using the Vickers scale) were carried out on the transverse surfaces at the weld metal, heat affected zone (HAZ) and parent metal regions. To determine the microhardness distribution throughout the weld section, micro-Vickers indentations were performed by a Struers Duromin-5 microhardness tester under a loading of 100 gf at a step size of 100 µm. The microhardness values of the phases were measured under a loading of 50 gf.

X-ray diffraction measurements were carried out within a scan range of 5–90° two theta with a step size of 0.02° by an X-ray powder diffractometer (Brukers D8 Advance) with Cu K α monochromatic radiation at 40 kV, 40 mA and a fixed wavelength of 1.5418 Å. Crystallographic identifications such as d-spacing (interplanar space), lattice parameters and lattice planes were defined using Bragg's Law along with the X-ray measurements.

Impact test coupons were sectioned from three different regions of the weldments: the weld metal, the heat affected zone (HAZ) and the parent metals, as schematically shown in Fig. 1. The coupons were then machined to precision sample dimensions (7.5 mm × 10 mm × 55 mm, with 2 mm V notched at center). The notch angle was 45°. The overall notched-bar samples were tested according to ASTM E23-93a [24]. The samples were immersed into a liquid in a narrow and deep container and cooled for at least 20 min by keeping them balanced within ± 0.2 °C of the test temperatures and controlled with thermocouples prior to the impact testing. Charpy-notched impact bar tests³ were conducted at an initial potential energy of 300 J. Immediately after removal from the deep cooling container, the samples were fractured into max. 5 s. Impact toughness was determined at sub-zero temperatures⁴ of –10 °C, –20 °C, –40 °C, –80 °C and –176 °C. The impact behavior of the weldments was also demonstrated at the ambient condition of 25 °C. Every impact test was repeated three times and the collected impact data were tabulated and statistically analyzed.

Welding metallurgy and fracture morphologies were examined with an OM (Nikon SMZ 800) and SEM (Leo 440 with EDX attachment). EDX mapping was carried out at 15 kV and 70 mA by SEM (JEOL JSM-7001F) with the 80 mm² EDX attachment. The distributions of alloying elements on the transverse section of the weld were detected with EDX analyses, and the carbon equivalent was calculated for the weldments by the equation provided in Ref. 25. According to the position of the weld metal, a general

¹ A duplex stainless steel filler metal (electrode) was selected for the welding operations of duplex–austenitic weldment. An austenitic steel filler metal was used for the austenitic–austenitic weldment in this study.

² Hydrofluoric acid (HF) is a very effective etchant for exposing grains and phases boundaries of stainless steels and glazes. Because it is very hazardous to humans, HF must be added to the solution while wearing a respirator, protective glasses, long gloves and suitable clothing under special permission. For further precautions, refer to the CCMR and Laboratory Safety Instructions, Cornell University, NY, USA.

³ Izod tests are not appropriate for these impact tests under cryogenic conditions because of the excessive time and the rapid heat transfer from the sample to the metallic holders. Therefore, Charpy-notched bar impact tests were performed in this study.

⁴ The subzero test temperatures, –10 °C, –20 °C, –40 °C and –80 °C, were attained by gradually adding incremental quantities of solidified CO₂ into acetone (CH₃COCH₃) in a heat-insulated cooling container. The –176 °C was obtained by using exactly liquefied nitrogen in the cooling container.

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