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TEM investigations on the effect of chromium content and of stress relief treatment on precipitation in Alloy 82

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HIGHLIGHTS

- Slight change of the Cr content does not affect the microstructure of the butt welds.
- Stress relief thermal treatment leads to the intergranular precipitation of $Cr_{23}C_6$.
- The $Cr_{23}C_6$ carbides are supposed to improve the SCC resistance of the butt welds.

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ABSTRACT

Nickel-base alloys are widely used in nuclear Pressurized Water Reactors (PWRs). Most of them have been found susceptible to Stress Corrosion Cracking (SCC) in nominal PWR primary water. The time to initiation depends on the material and is longer for weld metals than for Alloy 600. This study will focus on Alloy 82, which is used in Dissimilar Metal Welds (DMWs). In service, DMWs are either in the as-welded state or have undergone a stress relief treatment. Previous SCC studies showed that the heat treatment reduces significantly the SCC susceptibility of the weld. In this context, this study focuses on the microstructure characterization of the weld in the as-welded state and in the heat-treated state. As chromium content is also a key factor for the SCC susceptibility, welds with low chromium content and medium chromium content were studied. The lower SCC susceptibility of the heat-treated welds was attributed to intergranular $Cr_{23}C_6$ resulting from a combined effect of heat treatment and chromium and carbon contents. These intergranular carbides could explain the better behavior of Alloy 82, compared to other nickel-base alloys.

1. Introduction

Nickel-base alloys are used as construction materials in Pressurized Water Reactors (PWRs). Stress Corrosion Cracking (SCC) of wrought Alloy 600 and parent weld metals (Alloys 182/82) is a significant cause of failure in the primary circuit of PWRs [1]. Such cracking first occurred in Alloy 600 components such as steam generator tubes. After 100,000 h, the first cases appeared on components made of Alloy 182/82 such as Dissimilar Metal Welds (DMWs) between the low-alloy steel reactor vessel nozzle and stainless steel safe-end or pipe [2–6]. In France, most of DMWs are made of stainless steel, except for the last three N4 (1450 MWe) reactor vessel safe ends/to the main coolant piping DMWs that are made of Alloy 82. In France, unlike most other foreign DMWs, all Alloy 82 DMWs have undergone a stress relief treatment, due to a different manufacturing process. Only a few reliable crack growth rates (CGRs) data are available for Alloy 82 [7–11] and a few authors have studied the effect of a post-weld heat treatment on Alloy 82 CGR in a BWR environment [12] and in PWR environment [13]. This latter study concluded that heat-treated welds are less susceptible to propagation of stress corrosion cracks in PWR primary water than as-welded welds. In addition, it is generally accepted that SCC susceptibility of other nickel-base alloys depends on their chromium content [14]. The chromium content as specified by the RRC-M code (French code for nuclear component) must be in the range 18–22 wt%.

Therefore, this study will focus on Alloy 82 welds characterizations, in order to identify the influence of heat treatment and of chromium content on the microstructure. For this purpose, three welds were studied, differing by their chromium content and by their metallurgical state (heat-treated or as-welded). Characterizations were performed by Scanning Electron Microscopy (SEM), Electron BackScattered Diffraction (EBSD) and Transmission Electron Microscopy (TEM).

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2. Materials and experimental procedures

2.1. Materials

Three Alloy 82 butt welds were studied. Each butt weld was manufactured by Fouré – Lagadec. Alloy 82 was deposited between two AISI 304 L stainless steel plates (Fig. 1). The stainless steel plates were free to move during the welding process. Since the butt weld was not clamped, no mechanical stress was added. Two deposition processes were used. Butt weld 1 was manufactured by Flux-Cored Arc Welding (FCAW) with the following parameters: current (I = 200–210 A/V = 28–29 V), approximate number of beads: 90, wire diameter 1.2 mm. For butt welds 2 and 3, the filler metal was deposited by automatic Gas Tungsten Arc Welding (GTAW) with the following parameters: hot wire addition, pulsating current (I = 180 A/V = 13–14 V), approximate number of beads: 130, wire diameter: 1 mm, argon shielding.

Butt welds 1 and 2 were used in the as-welded conditions and after a laboratory heat treatment on extracted samples whereas the entire butt weld 3 was heat treated on site just after welding. The heat treatment reproduced the stress relieving procedure used during manufacturing for carbon steel and consisted of 7 h at 600 °C, with heating and cooling rates of 100 °C/h.

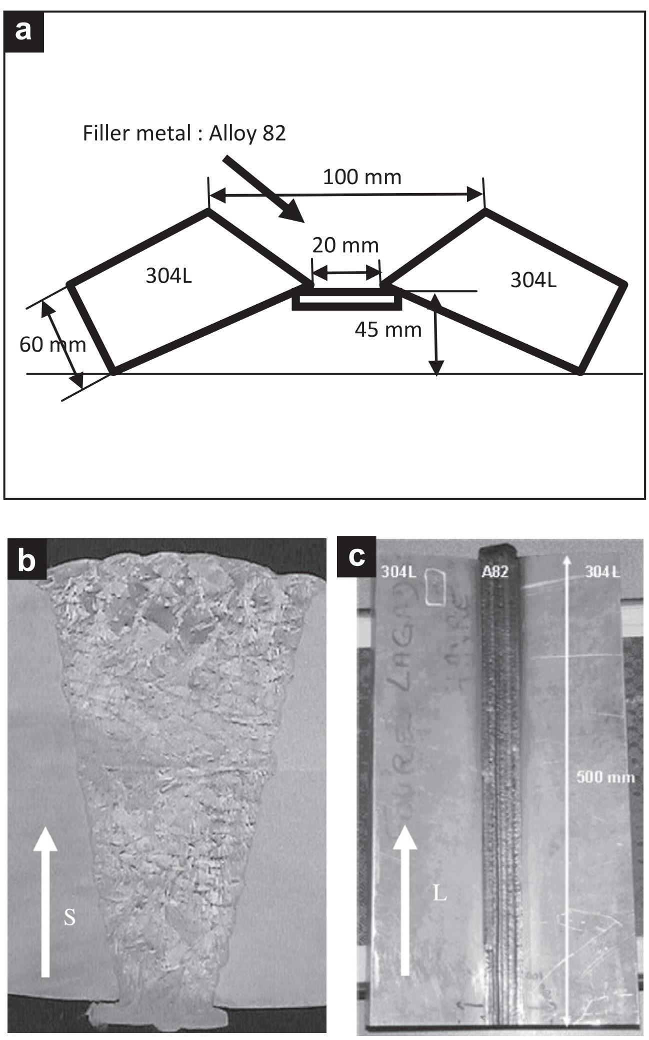

Fig. 1. (a) Schematic representation of butt weld manufacturing, (b) cross-section of the butt weld and (c) top view. The L direction is the welding direction and the S direction is the dendrite growth direction.

The chemical composition of the filler metal is presented in Table 1. It is compared to the RCC-M code composition and to the analyses performed in the middle of the welds after manufacturing. It can be noticed that the chromium content of the butt weld is lower than the chromium content in the wire.

The weld has a dendritic structure, each columnar grain being composed of slightly disoriented dendrites (Fig. 2).

2.2. SEM and EBSD

In this work, SEM and EBSD investigations were carried out on thin foils. SEM observations and EBSD investigations of TEM thin foils were carried out on a Zeiss DSM982 Gemini FEG microscope and a LEO 1450VP microscope equipped with high speed EDAX–TSL Hikari EBSD camera respectively. Working directly on TEM thin foils is astute and provides rapid and global information when dealing with grain boundary phenomena involving the boundary disorientation. Furthermore, it allows a direct correlation with TEM observation results.

2.3. TEM

TEM samples were extracted from the center of the weld. A 1 cm-thick slice was cut perpendicularly to the L direction. This slice was then cut in three equal parts perpendicularly to the S direction. At the center of the central part, a 3 mm diameter cylinder was machined parallel to the S direction. This direction was chosen in order to observe the LT plane where the number of grain boundaries is maximal. Then, 0.3 mm thickness disks were sliced from the two edges of the cylinder and mechanically polished to about 100 μm of thickness. Final thinning to electron transparency was achieved by twin-jet polishing at −10 °C using an electrolyte composed of 45% butoxyethanol, 45% acetic acid and 10% perchloric acid.

TEM observations and analyses were carried out on a 200 kV Tecnai F20 ST field emission gun microscope equipped with an Energy Dispersive X-ray (EDX) device, scanning transmission electron microscopy (STEM) capabilities, High-Angle Annular Dark-Field (HAADF) detector and Gatan Imaging Filter (GIF). STEM-HAADF technique consists in collecting high-angle diffused electrons. As the intensity of these electrons is proportional to the averaged atomic number Z, chemical contrast (Z-contrast) images can be acquired. Energy-filtered transmission electron microscopy (EFTEM) was also performed using the GIF to produce elemental maps. The technique consists in selecting, by the mean of a slit, only electrons of particular kinetic energies to form the image. Theses energies are characteristic of elements present in the studied material.

3. Results

Scanning electron microscopy on thin foils and transmission electron microscopy investigations were carried out to study the effect of Cr content and heat treatment on the microstructure of the weld and principally on grain boundary precipitation. The later is usually considered as a key parameter for the resistance to SCC of nickel base alloys [15].

3.1. SEM observations

SEM observations were carried out directly on TEM thin foils. They were then called samples 1, 2 and 3 in reference to the butt weld from which they were extracted. This method allows the investigation of the same regions from mesoscopic to nanometric scale. Thin foils were mounted on a dedicated specimen holder. Fig. 3 shows SEM images obtained on the three samples at different