



304 stainless steel brazing incorporating tungsten nanoparticles



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ABSTRACT

Tungsten nanoparticles of 80 nm average particle size were utilized in the brazing of 304 stainless-steels. The nanoparticles were characterized by DTA and TEM, and the brazed samples were evaluated by SEM and Vickers microhardness tests. The interaction of the tungsten nanoparticles with the metallic filler in the melting zone modified the size and morphology of the formed phases into a finer and uniformly distributed microstructure. In samples treated at 1200 °C for 60 min, the microhardness decreased from 310 to 170 HV, being the latter value, close to that of the base metal. The nanoparticles and the microcracks develop a synergistic effect when they are in contact with the liquid phase in such a way that a rise in the threshold capillary-pressure leads to the filling of the interstices. The capillary-like system resulting from the wettability of the nanoparticles and microcrack surfaces by the liquid phase, leads to a solidified microstructure with fine and uniform phase distribution.

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1. Introduction

Owing to their excellent properties such as corrosion resistance and optimum hardness at room temperature, stainless steels are essential for medical, chemical, cryogenic containers and biotechnological applications. However, under certain conditions, the brazed stainless steels are susceptible to corrosion, which reduces their mechanical properties and gives place to fracture initiation. Those adverse effects are mainly present near the areas joined by brazing. In the case of micro-cracking with no soldering, that effect is ascribed to an inadequate penetration of the flux used during the brazing process, given the complex path of the microcracks. The foregoing situation can be solved using the transient liquid phase (TLP) bonding, considered as a preferred repairing/joining process for nickel base superalloys due to its ability to produce near-ideal joints, a weld bead with at higher melting point than base metal and free of intermetallics or eutectic phases, as shown by Pouranvari et al. (2009). The main advantage of TLP is the formation of a melt in the interlayer and dissolution of the substrate material in the bond region, as established by Cook and Sorensen (2011). The foremost

difference between the brazing and the TLP processes, is that the latter involves four well-defined steps whilst technically, brazing is simply the joining of two base materials with a filler metal, as defined by the AWS (American Welding Society). The four steps of TLP process are as follows: (1) setting up the bond; (2) heating to the specified bonding temperature to produce a liquid in the bond region; (3) holding the assembly at the bonding temperature until the liquid has isothermally solidified due to diffusion and (4) homogenizing the bond at a suitable heat-treating temperature. Sometimes in the TLP process, the dissolution of the substrate is not activated, thus avoiding the isothermal solidification (Cook and Sorensen, 2011). Studies such as that by Philips et al. (2008) have suggested that the microstructural evolution in the weld bead of austenitic stainless steels depends on thermochemical interactions with the parent alloy through dissolution, re-precipitation reactions, and solid-state diffusion during the TLP, but that it can be restricted for wide joints because the dissolution is not sufficient to eliminate the intermetallics. Analytical modeling conducted by Zhou (2001) established that liquid phase is at its maximum width when the isothermal solidification stage starts. Also, Jalilian et al. (2006) showed that at the bonding temperature, the inter-layer melts, filling the surface gaps with a thin liquid layer. An advantage can be taken of this liquid phase to fill the micro-cracks and micro-pores by capillary processes between the TLP and the melting filler metal. Byong-Ho and Dae-Up (2002) and Abdelfath

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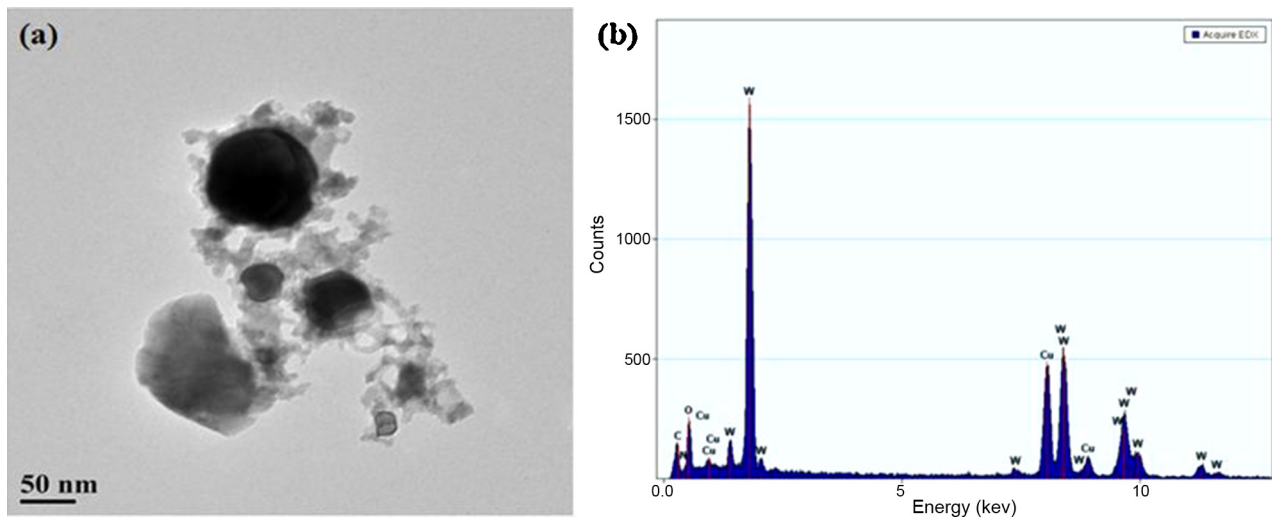


Fig. 1. (a) Tungsten nanoparticles of 80 nm and (b) EDX spectrum of the W NPs.

and Ojo (2009) suggest that the formation of a transient liquid phase (TLP) can lead to an enhanced homogeneous microstructure. This is possible by the insertion of an element to diffuse into the substrate material, which ultimately gives place to an isothermal solidification. The same beneficial effect is obtained by incorporating foils which contain nanoscale layers of Al and Ni that react exothermically as local heat sources for melting AuSn solder layers, as used by Wang et al. (2004). But, if the isothermal solidification is not complete, the liquid in the intermediate layer is solidified through eutectic phases during cooling, causing a decrease in the mechanical properties and corrosion strength of the weld bead, as shown by Yuan et al. (2012). Jang and Shih (2003) suggest that the metallic fillers containing silicon, boron or melting-point depressant solute with high solubility in the metal base, reduces the eutectic structures and diminishes the anomalous behavior at high-temperature. On the other hand, McGuire et al. (2009) established that tungsten reduces the size of borides and modifies the intermetallics; likewise, the effect of phase growth kinetics in stainless steels, by tungsten, has been confirmed by other authors (Park et al., 2006; Michalska and Sozańska, 2006; Kim et al., 1998).

In this context, this research work is aimed at modifying the brittle eutectic phase in the joining area of 304 stainless-steels, through a systematic study of incorporating tungsten nanoparticles (NPs) into microcracks, by the Brazing process.

2. Experimental procedure

2.1. Characterization and impregnation of tungsten NPs on the fractures of stainless steels

In order to study the brazing process and the effects of tungsten NPs (80 nm, Nanospyring), fractures were induced in 304 stainless steel rods of 10×60 mm by bending them with a mechanical testing machine; these fractures were inspected by scanning electron microscopy (SEM). Before conducting the brazing process, the tungsten NPs were characterized by transmission electron microscopy (TEM) and differential thermal analysis (DTA), at the rate of $10^\circ\text{C}/\text{min}$. Characterization of tungsten NPs by DTA was carried out to study the phase transformations or thermal events as a function of brazing temperature without mixing with paste filler metal. The tungsten NPs were successively dispersed five times in ethanol, using a mixture of 0.5 g of tungsten NPs in 100 ml of ethanol, sonicated for 1 h. Separately, the steel fracture samples

were ultrasonically cleaned with ethanol for 15 min to remove impurities of the microcracks and micropores and trapped air. Subsequently, the cleaned fracture samples were placed into the dispersed tungsten NPs and sonicated for 30 min. The sonication process promotes the incorporation of tungsten NPs into the microcrack fractures.

2.2. Brazing of fractured 304 stainless steel

In order to evaluate the effect of tungsten NPs in the brazing process of the stainless steel, the BNi-9 filler metal was characterized by the atomic absorption spectroscopy (AAS) method, and used in the fractured rods with and without tungsten NPs impregnation. The brazing process was conducted in a sealed tube furnace under an Ar gas flow of 0.1 L/min, the brazing temperature used was of 1200°C for 10, 20, 30 and 60 min; the heating and cooling rates were fixed at $10^\circ\text{C}/\text{min}$. The paste filler metal was spread on both types of fracture specimens, namely, those without and those with the dispersed tungsten NPs. This operation was followed by the manual joining of the fracture surface counterparts in such a way that the gap distance between the joined surfaces was close to 1 mm, which was measured with a Vernier and confirmed by optical microscopy. During the brazing tests, a soldering flux was not used because after fracturing, the samples were immediately cleaned and brazed, as described in this section. In order to determine the reactivity of the tungsten NPs on the stainless steel fractures, in another experiment, the impregnated fractures were exposed – without the paste filler metal – to the same brazing conditions (1200°C for 1 h). The resulting samples were characterized by SEM and optical microscopy.

3. Results and discussion

3.1. Characterization of tungsten NPs and the effect in the brazing process

Fig. 1a is a TEM micrograph showing that the size of the tungsten NPs used to impregnate the stainless-steel fracture surfaces is less than 80 nm. Fig. 1b) is an EDX spectrum, revealing the W, Cu, O and C peaks of the elements constituting the NPs. The chemical composition of the filler metal is presented in Table 1.

Differential thermal analysis (DTA) conducted on the tungsten NPs in the temperature range between 1100 and 1200°C

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