



Friction-stir welding of ultrafine grained austenitic 304L stainless steel produced by martensitic thermomechanical processing



S. Sabooni ^{a,*}, F. Karimzadeh ^a, M.H. Enayati ^a, A.H.W. Ngan ^b

^a Department of Materials Engineering, Isfahan University of Technology, 84156-83111 Isfahan, Iran

^b Department of Mechanical Engineering, The University of Hong Kong, Pokfulam Road, Hong Kong, China

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ABSTRACT

An ultrafine grained 304L austenitic stainless steel was produced by martensitic thermomechanical processing and joined by applying Friction Stir Welding (FSW). The thermomechanical processing comprised a cold roll procedure up to 80% reduction followed by annealing. After FSW, different grain structures in different regions of the weld nugget were observed due to the asymmetry in the heat generation during the welding process. Grain growth was found to be the most predominant phenomena in the region just ahead of the rotating tool during the thermal cycle of FSW. A banded structure was observed in the advancing side of the weld nugget. TEM observations revealed that nanometric sigma phase precipitates were present both in the grain boundaries and inside the grains of this region. Shear textures were clearly identified in the weld center. The lack of rotated cube texture shows that the discontinuous dynamic recrystallization (DDRX) is not active in the final microstructure. Increasing the welding speed can reduce the final grain size of the weld nugget leading to higher hardness. Hardness is found to increase in the weld and this is not just a grain refinement effect, but also due to the presence of sub-boundaries and a high density of dislocations.

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1. Introduction

Austenitic stainless steels are the most popular type of stainless steels with wide applications in different industries, from low-end to advanced applications like aerospace vehicles [1]. Although austenitic stainless steels possess high corrosion resistance, good formability and suitable welding properties, their relatively low hardness and yield strength have limited their wider applications [2–4]. Improving the mechanical properties of austenitic stainless steels have therefore become a critical concern, and advanced thermomechanical processing based on hot deformation or cold rolling–annealing is one of the most industrially applicable methods to produce nano- or ultrafine grained (UFG) austenitic stainless steels [5], which were found to exhibit high strength and ductility [2–4].

Most of the published research on UFG austenitic stainless steels to-date has focused on their production and mechanical property characterization. Weldability, as one of the most important factors of a material for being applicable, has not been systematically investigated. In the case of fusion welding of UFG materials, considerable grain growth in the heat affected zone,

and the coarse cast structure in the weld zone, are two significant phenomena that can jeopardize the mechanical properties of the welded material relative to the UFG base material [6]. Monte Carlo simulations have shown that the average grain size of the heat affected zone in a fusion weld of a 2 μm grain size steel can increase up to around 120–150 μm under high heat input conditions [7]. Some methods have been proposed to alleviate the weld quality problems associated with coarsening of microstructures, including heat input control in traditional welding techniques [7,8], the use of high-energy fusion welding processes such as laser welding [7], and the use of coolants such as liquid nitrogen behind the welding zone [9], but despite these, deterioration of mechanical properties in the weld zone is still common. Recently, solid-state welding processes like friction-stir welding have been successfully applied to join advanced materials [10,11]. Friction stir welding is a solid-state, hot-shear joining process in which a rotating tool penetrates in the material and the high heat caused by friction leads to material flow. FSW has been successfully applied to aluminum as well as magnesium alloys [12–14], but in comparison with these light alloys, limited research has been performed to study its applicability in high-temperature alloys such as steels, possibly due to the lack of suitable tools found for the FSW of such alloys [15]. According to the authors' knowledge, there has not been any published record on FSW of UFG

* Corresponding author.

E-mail address: s.sabooni@ma.iut.ac.ir (S. Sabooni).

stainless steels and their microstructural evaluation during the welding process. Therefore, the aim of this paper is to produce a UFG 304L stainless steel and to study its microstructural changes during FSW.

2. Materials and methods

A commercial AISI 304L stainless steel, in the form of a sheet with thickness 10 mm, was used as the initial material. Table 1 shows the chemical composition, and Fig. 1 shows the electron backscattered diffraction (EBSD) map, of the as-received material. The microstructure is equiaxed with an average austenite grain size of $\sim 30 \mu\text{m}$. The black¹, green and red lines in Fig. 1 represent high-angle grain boundaries (HAB) with misorientations larger than 15° , $\Sigma 3$ twin boundaries and low-angle boundaries (LAB) with misorientations between 2° and 15° , respectively, and it can be seen that the microstructure contains large fractions of HAB and $\Sigma 3$ twin boundaries, and just a small fraction of LAB. A small amount of elongated delta ferrite is also observed as indicated by blue color in the microstructure.

Specimens with dimensions $100 \text{ mm} \times 40 \text{ mm}$ were cut from the as-received material and cold rolled in a solution of ice and ethanol at -15°C up to 80% reduction, to enable the initial coarse-grained austenite to transform into fine structured martensite. The cold rolled samples were then annealed at 700°C for 300 min to obtain a UFG microstructure of austenite, through the reverse martensite-to-austenite transformation.

Friction stir welding was performed on the resultant UFG 304L using a vertical milling machine. The welding tool was made of tungsten carbide with a shoulder diameter of 16 mm. A conical pin with upper and lower diameters of 5.5 and 5 mm, respectively, and length of 1.8 mm, was used (Fig. 2(a)). The sample plates were fixed onto a steel backing plate using a fixture to prevent any displacement during welding. For all tests, the tilt angle of the tool from normal direction was selected as 3° . Argon gas shielding was introduced around the tool at a flow rate of 10 l/min to prevent excessive oxidation during the welding process. Welding trials were performed at a constant rotational speed of 630 rpm and different welding speeds from 20 to 160 mm/min. Fig. 2(b) shows schematic illustration of different regions on the welded sample.

A ferritescope (Helmut Fischer GmbH, model MP30E) was used for the quantification of the ferromagnetic α' -martensite phase during the cold rolling process. Electrolytic etching was performed at 1–2 V for about 10 s in a solution mixture of 65 ml nitric acid and 35 ml distilled water to reveal the austenite grain boundaries. For cross sectional examinations, samples were cut using a slow-speed saw and mounted in epoxy using a hot-mount equipment. Subsequently the samples were mechanically ground and polished using papers down to 4000-grit followed by Al_2O_3 slurry. Vibration polishing was finally performed to obtain a surface quality suitable for EBSD. A Field Emission Scanning Electron Microscope (LEO 1530 FE-SEM) attached with an EBSD analyzer was used to characterize the grain structure of the samples. EBSD was performed using a step size of $0.05\text{--}2 \mu\text{m}$ based on the requirements. Transmission Electron Microscopy (TEM) analysis of the deformed and welded samples were performed using an FEI Tecnai G2 20 Scanning TEM. The TEM samples were produced by FIB using an FEI Quanta 200 3D system from related regions of the welds. Microhardness measurements were performed using a Buehler microhardness tester with a Vickers indenter at the load of 500 gf.

¹ For interpretation of color in Figs. 1, 6, 10, the reader is referred to the web version of this article.

3. Results and discussion

3.1. Production of ultrafine grained austenitic steel with bimodal grain-size distribution

Fig. 3 shows scanning electron micrographs of the α' /martensite morphology in different rolling reductions. As confirmed by ferritescope measurements in our previous report [16], 40% by volume of the structure would transform into martensite after 15% cold-rolled reduction. The morphology of martensite in this stage is lathy (Fig. 3(a)). Increasing the rolling reduction to 35% (Fig. 3(b)) caused breakdown of the lathy martensite into finer one, in addition to increasing the density of the martensite layers. After 55% cold-rolled reduction, almost all of the structure (98% by volume) has transformed into martensite, with only small regions still in austenite phase (Fig. 3(c)). Therefore, the rolling reduction of 55% can be described as a saturation state for martensite conversion.

The austenite stability index $M_{d30/50}$, defined as the temperature at which 50% of the austenite will transform into α' /martensite through cold deformation to a true strain of 0.3 [1], is a useful factor indicating the martensitic transformability during cold rolling. $M_{d30/50}$ effectively indicates the temperature that limits deformation-induced martensitic transformation since martensite becomes difficult to form above that temperature. The Nohara equation [17] relates the austenite stability index to the chemical composition and grain size (GS) of the alloy as:

$$M_{d30/50} (^{\circ}\text{C}) = 551 - [462(\%C + \%N) + 9.2\%Si + 8.1(\%Mn) + 13.7(\%Cr) + 29(\%Ni + \%Cu) + 18.5(\%Mo) + 68(\%Nb) + 1.42(\text{GS}-8)] \quad (1)$$

where elemental compositions are in wt.%, and GS is in terms of the ASTM grain size number. From eqn. (1), the $M_{d30/50}$ was calculated as 37°C for the present AISI 304L, meaning that there is sufficient driving force for martensitic transformation during rolling at -15°C . Further rolling reduction after the saturation state is necessary for obtaining ultrafine grains during the subsequent annealing, as many more possible nucleation sites are created which cause finer austenite grain sizes [18,19]. Therefore, rolling reduction was continued to 80% to ensure the formation of ultrafine grained austenite after the subsequent annealing.

Fig. 4 shows the SEM (Fig. 4(a)) and TEM (Fig. 4(b)) micrographs of the 80% cold rolled sample followed by annealing at 700°C for 300 min. The microstructure consists of mostly UFG austenite as the phase with lighter contrast in the SEM micrograph. Embedded in such a matrix are precipitates in dark contrast which are delta ferrite that was present in the as-received coarse grained 304L stainless steel and was not affected by the cold rolling and annealing. A bimodal distribution of the grain sizes can be easily identified in the UFG austenite matrix. The TEM micrograph in Fig. 4(b) also shows austenite grains with a high density of dislocations.

3.2. Microstructural observations during friction-stir welding

Friction-stir welding was performed with a constant rotational speed of 630 rpm and different welding speeds of 20–160 mm/min. The control of the heat input is very important during FSW of UFG stainless steels as it affects a number of processes such as grain growth, distortion and phase transformation. Therefore, knowledge about the generation and distribution of heat in the welded sample is necessary. Schmidt et al. [20] suggested that heat is generated by the friction between the rotating tool and material interface, and so the heat input dQ generated in an element of surface area dA during FSW is given by:

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