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Microstructural evolution mechanism of hydrogenated Ti–6Al–4V in the friction stir welding and post-weld dehydrogenation process

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The $\alpha + \beta$ titanium alloy, Ti–6Al–4V, was thermohydrogen processed and friction stir welded using a W–Re pin tool. Defect-free joints were obtained with proper welding parameters. Hydrogen was removed from the joints through a post-weld dehydrogenation process. The microstructures of the as-welded and dehydrogenated joints were examined, and the microstructural evolution mechanism was revealed in the friction stir welding and post-weld dehydrogenation process.

Keywords: Friction stir welding; Titanium alloy; Thermohydrogen processing; Microstructural evolution

Friction stir welding (FSW) is a novel solid-state joining technique invented by The Welding Institute (TWI) [1]. In typical FSW, a non-consumable rotating tool with a specially designed pin is inserted into the abutting edges of the sheets or plates to be joined and moves along the joint line. The material is softened and flows around the rotating pin under a combined effect of frictional and adiabatic heating, thus forming a solid-state bonding between the two work-pieces.

Due to its many advantages, FSW attracts a great deal of attention in the industrial fields, and is successfully applied to weld various aluminum, magnesium and copper alloys. In recent years, FSW of high melting temperature materials such as steels and titanium alloys has become a research hotspot [2–4]. However, it is difficult to plasticize high melting temperature materials, especially titanium alloys, sufficiently, and this has restricted the development of FSW of titanium alloys [5–10].

Thermohydrogen processing (THP) is based on the modifying effects of hydrogen as an alloying element on phase compositions, the development of metastable phases and the kinetics of phase transformations in titanium alloys. In this approach, hydrogen is first added to the alloy by controlled diffusion from a hydrogen environment and then, after processing, is removed by a controlled vacuum annealing [11,12]. THP has proved to be a powerful tool for improving the hot workability of titanium alloys due to hydrogen-enhanced plasticity at high temperatures. In the present study, the $\alpha + \beta$ titanium alloy, Ti–6Al–4V, is thermohydrogen processed and friction stir welded using a W–Re pin tool. A subsequent dehydrogenation process is used to remove hydrogen from the joints. The microstructural evolution mechanism of hydrogenated Ti–6Al–4V in the friction stir welding and post-weld dehydrogenation process is discussed in detail.

The as-received material was a 2-mm-thick mill-annealed Ti–6Al–4V sheet. Hydrogenation was performed at 750 °C for 2 h in pure hydrogen atmosphere within a tubular furnace. A hydrogenated sheet with a hydrogen content of 5 at.% was obtained by controlling the hydrogen partial pressure. The hydrogenated sheet was friction stir welded using a W–Re pin tool installed in a special welding system designed by Harbin Institute of Technology, P.R. China. The chemical composition of the tool is 3 wt.% rhenium and tungsten balance. The tool shoulder diameter was 11 mm; the pin length was 1.8 mm and the pin was tapered from 6 mm at the shoulder to 4 mm at the pin tip. Welds were made at a tool rotation speed of 400 rpm and a welding speed of 100 mm min^{-1}, and the welding direction was perpendicular to the rolling direction of the sheet. During the FSW, a 2.5° tilt and a plunge depth of 2 mm were applied to the pin tool. Pure argon was used as a shielding gas to prevent the welding zone and pin tool from

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oxidizing. A subsequent dehydrogenation process was conducted at 750 °C for 2 h in vacuum to remove hydrogen from the as-welded joints. An ELTRA OH-900 oxygen/hydrogen analyzer was used to determine the hydrogen content in the as-welded and dehydrogenated joints.

Microstructural characteristics of the as-welded and dehydrogenated joints were examined by optical microscopy (OM; Olympus-PMG3) and transmission electron microscopy (TEM; Philips CM-12). The transverse cross-sections of joints were cut by electrical discharge machining and prepared by standard metallographic procedure. The polished cross-sections were chemically etched using Kroll's reagent and then observed on the optical microscope. OM observations for the weld zone (WZ) and the hydrogenated base material (BM) were performed at the weld center and a location well away from the WZ in the joint cross-section, respectively. Thin-foil disk specimens for TEM observation were cut from the BM parallel to the rolling direction and from the center of the WZ parallel to the welding direction, respectively, and finally observed on the transmission electron microscope, which was operated at 120 kV.

A typical cross-section of the as-welded joint is shown in Figure 1a. The central darker region corresponds to the WZ and the peripheral lighter zone is the BM. The WZ appears to be "basin-shaped", and no volumetric defects are observed in any of the joints. It should be noted that the thermomechanically affected zone is not apparent in this friction stir weld, and thus the WZ consists only of the stir zone. In Figure 1a, RD, ND and TD stand for rolling direction, normal direction and transverse direction of the sheet, respectively. WD corresponds to the welding direction and is parallel to the TD. RS and AS are the retreating and advancing side of the weld, respectively.

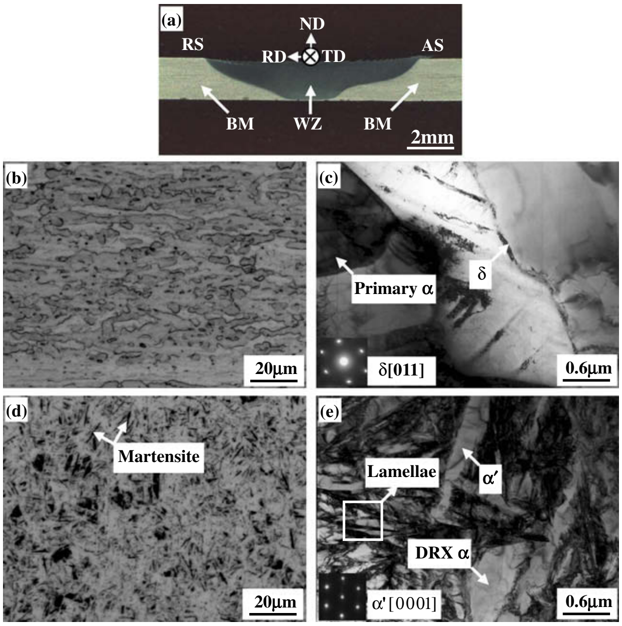

Figure 1. Microstructural features of the as-welded joint: (a) cross-section, (b) OM image for BM, (c) TEM image for BM, (d) OM image for WZ and (e) TEM image for WZ.

An optical micrograph of the BM is shown in Figure 1b. The BM maintains its typical microstructure in mill-annealed Ti–6Al–4V, i.e., elongated primary α + transformed β. The grey and black regions in the OM image represent primary α and transformed β, respectively. Titanium and titanium alloys have a high affinity for hydrogen and are capable of absorbing up to 50 at.% hydrogen at 600 °C, and face-centered cubic (fcc) δ hydride can be precipitated with increasing hydrogen content [13]. For Ti–6Al–4V, Kerr et al. [14] reported that the minimum hydrogen content needed to form δ hydride is 15 at.%. In the present study, however, δ hydride is found in the high diffusion path for hydrogen, i.e., the α/β interface, in hydrogenated Ti–6Al–4V with a hydrogen content of 5 at.% (see Fig. 1c), and this is consistent with the result reported by Mahajan et al. [15].

It has ben reported that the WZ temperature can be above or below the β-transus temperature of $\alpha + \beta$ titanium alloy, and thus a full lamellar microstructure [7,8] or a bimodal microstructure [9,10] can be formed in the WZ. In the present study, the WZ consists of fine-equiaxed primary α and transformed β (Fig. 1d), indicating that the temperature in the WZ is below the β-transus temperature. It is interesting to note that acicular martensite is seen in the WZ of as-welded joints, while such a martensite does not exist in the WZ of conventional Ti–6Al–4V alloy, as reported by Pilchak et al. [9] and Zhou et al. [10]. Therefore, the formation of acicular martensite is related to the reduction in the critical cooling rate to form martensite in Ti–6Al–4V due to the addition of hydrogen [16,17].

The detailed microstructural evolution of the WZ in the as-welded joint is further revealed by TEM. The WZ is characterized by dislocation-free equiaxed primary α and transformed β composed of acicular martensite and lamellar $\alpha + \beta$, as shown in Figure 1e. The type of martensite formed in the hydrogenated titanium alloy is dependent on the hydrogen level and process history [16,17]. In the current case, hexagonal close-packed α' martensite is formed in the WZ. The formation of acicular α' martensite and lamellar $\alpha + \beta$ indicates that the WZ temperature is in the upper two-phase region of the phase diagram (above the starting temperature for martensite transformation) and the cooling rate is larger than the critical cooling rate for martensite transformation. Face-centered cubic δ hydride is also observed at the α/β interface in the WZ.

The material in the WZ reaches a high temperature and experiences a large strain during FSW, and thus dynamic recrystallization (DRX) can be induced in the WZ. Similar to conventional titanium alloys, the hydrogenated Ti–6Al–4V may experience recrystallization as well as phase transformation when processed in the two-phase region. The existence of dislocation-free equiaxed α confirms the DRX process in the WZ. In addition to DRX, $\alpha \rightarrow \beta$ transformation can also occur due to the high temperature of the heating stage of the FSW. However, the $\alpha \rightarrow \beta$ transformation is not fully developed because the temperature is below the β-transus temperature and the high temperature dwell time is relatively short. Due to the addition of hydrogen, $\beta \rightarrow \alpha + \beta$ as well as $\beta \rightarrow \alpha'$ occurs during the cooling stage of the FSW, resulting in the formation of alternate