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Achieving superior low-temperature superplasticity for lamellar microstructure in nugget of a friction stir welded Ti-6Al-4V joint

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ABSTRACT

A low-temperature superplasticity of 442% was achieved at 650 °C for a fully lamellar microstructure in the nugget of friction stir welded Ti-6Al-4V joint. More importantly, the nugget showed a comparable superplasticity >395%) to the base material at 800 °C and $1 \times 10^{-4} \sim 1 \times 10^{-3}\ s^{-1}$; especially at $3 \times 10^{-4}\ s^{-1}$, both the elongation and flow stress of the nugget were similar to those of the base material, which provides the possibility of uniform superplastic forming of the entire weld. Good superplasticity in the lamella-structured nugget was mainly attributed to gradual globularization of the fine lamellae during static annealing and superplastic deformation.

Superplastic forming/diffusion bonding (SPF/DB) has been applied to produce complex Ti alloy components in the aerospace field [1,2]. As more integrated and larger components are highly required for the aerospace structures, the DB technique no longer fully meets the requirements because of low production efficiency, significant grain growth and some other limiting factors [2]. Therefore, increased efforts are made to produce integrated large-sized Ti alloy components by combining other welding techniques with SPF [3,4].

Generally speaking, a fine-grained microstructure is required to obtain a good superplasticity (SP), and rolled two phase Ti alloy sheets/plates with fine-grained microstructure show a good SP. When the whole weld is superplastically formed, the similar superplastic deformation ability (including SP and flow stress) in different zones of the weld is required to prevent local severe deformation or even being torn. However, for fusion welds, the SP nature of Ti alloy workpieces is largely destroyed by the coarse cast lamellar microstructure in the nugget [5], which hinders the combination of fusion welding and SPF.

Friction stir welding (FSW), a solid-state welding technique widely used to join Al alloys [6,7], has been recently studied for high-melting-temperature alloys such as Ti alloys [8–10]. Due to severe plastic deformation and dynamic recrystallization, a fine-grained microstructure can be obtained in the welds. Therefore, the combination of FSW with SPF has great potentials to produce large-sized Ti alloy components. However, due to the difficulty in FSW of Ti alloys, only some preliminary results on SPF of FSW Ti alloy joints have so far been reported [11–17]. Since the nugget is the key zone affecting the SP of the entire FSW joint [11] and there is a very narrow transition zone between it and the BM [10], the SP nature of the nugget becomes the main study object.

For FSW Ti-6Al-4V joints, Sanders et al. [17] reported that the nugget could show comparable SPF characteristics with the base material (BM) by optimizing process parameters. Edwards et al. [11] also suggested that the similar SPF ability for the nugget and BM could be obtained via strictly controlling the nugget temperature to approach the β transus temperature; however, the process window was extremely narrow. In other words, it is harsh and complex to control the nugget temperature to be just near the β transus by adjusting the FSW process. Actually, a fully lamellar microstructure was usually obtained in the nugget under most of FSW parameters [18–20]. Therefore, it is of great significance to study whether it is possible to obtain similar superplastic deformation ability for the lamella-structured nugget and the BM.

More recently, the present authors [21] reported the superplastic behavior of the lamella-structured nugget in a FSW Ti-6Al-4V joint at temperatures of 850–925 °C and strain rates of 3×10^{-4}–$3 \times 10^{-2}\ s^{-1}$, and a largest elongation of 728% was achieved at 925 °C and $3 \times 10^{-3}\ s^{-1}$. However, whether similar superplastic deformation ability could be achieved in the nugget and the BM is still unknown. Besides, although the optimum SP temperature for the nugget (925 °C) is within the conventional SPF temperature range (900–950 °C) of Ti alloys, a lower forming temperature is more attractive because it can reduce production cost, and maintain better post forming properties.

Ideal low-temperature SP (LTSP) should be obtained at half of the melting temperature of the matrix alloy, and the corresponding temperature for Ti alloys is below about 700 °C. However, the flow stress significantly increases below 700 °C, and it is difficult to obtain SP at too low temperature. For practical SPF, a low flow stress is required while the

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total elongation is less important but must be higher than 300% [22]. Therefore, the LTSP of Ti alloys were usually investigated at 650–800 °C considering both the flow stress and SP [15,22,23]. To our best knowledge, there is no report on LTSP for the lamella-structured nugget of FSW Ti alloy joints so far.

In this study, a nugget with a lamellar microstructure was obtained in Ti-6Al-4V alloy by FSW and was subjected to superplastic investigation at 650–800 °C, and the objective is to explore (a) if similar low-temperature superplastic deformation ability could be obtained for the lamella-structured nugget and the BM and (b) what is the main factor contributing to the good SP for the lamellar microstructure.

The as-received material was 2-mm-thick mill-annealed Ti–6Al–4V sheet. The sheets were friction stir welded at a rotation rate of 500 rpm and a transverse speed of 150 mm/min. A W-5Re alloy welding tool was used, and the details on the tool can refer to our previous paper [21]. Microstructural characterization was carried out by optical microscopy (OM) and transmission electron microscopy (TEM). Specimens for TEM were prepared by twin-jet electropolishing with a solution of 6 vol.% $HClO_4$ + 34 vol.% CH_3OH + 60 vol.% C_4H_9OH at 11V and at about −25 °C.

Dogbone-shaped tensile specimens with a gauge length of 2.5 mm, a width of 1.4 mm and a thickness of 1 mm were cut from the nugget transverse to the welding direction. Constant crosshead speed tensile tests were carried out on an Instron 5848 micro-tester. To reach thermal equilibrium, each specimen was held at the testing temperature for 5 min before tension.

Each specimen stretched to failure was cooled rapidly in water to keep the microstructure information just after failure. To obtain the statistical data of thicknesses (or grain sizes) and aspect ratios of lamellae (or grains) in the nugget after annealing and superplastic deformation to different elongations, more than 300 lamellae (or grains) were measured under all the annealing and tension conditions.

The characteristic microstructures of the BM and the nugget are shown in Fig. 1. The BM consisted of a typical mill-annealed microstructure (Fig. 1a). After FSW, the nugget consisted of a fine fully lamellar microstructure (Fig. 1b), which suggested that the FSW process temperature was over β transus temperature (~980 °C). The detailed microstructure can refer to our previous paper [21], and the thickness and the aspect ratio of α lamellae were determined to be 172 nm and 8.1, respectively [21].

Fig. 2a shows the variation of elongation with initial strain rate at 650–800 °C for the nugget. At 650 °C, a largest elongation of 442% was obtained at $3 \times 10^{-5}\ s^{-1}$, which suggested that the lamella-structured nugget showed a good LTSP. This is the first report on LTSP of the lamellar microstructure in Ti alloys so far, and it is also the first time to report LTSP of welds. As the temperature increased, the optimum strain rate showed an increasing trend, and a maximum ductility of 500% was obtained at 800 °C and $1 \times 10^{-3}\ s^{-1}$. Fig. 2b shows the elongations of the BM at 650–800 °C. Compared the nugget with the BM, they showed comparable SPs (more than 395%) at 800 °C for the strain rate range of 1×10^{-4}–$1 \times 10^{-3}\ s^{-1}$ (marked by blue ellipses in Fig. 2a and b), and the maximum discrepancy was just 7.4%. The comparable SPs in the nugget and BM are very important because it means that the SP extreme of the workpiece is likely to be achieved without the limitation of low SP in some local zone. However, whether uniform forming of a weld could be achieved is also affected by flow stress in different zones of the weld during practical SPF.

Fig. 2c shows typical true stress-true strain curves of the nugget and the BM at 800 °C and 1×10^{-4}–$1 \times 10^{-3}\ s^{-1}$. For both the nugget and BM, an initial strain hardening took place, followed by a flow softening at each strain rate (except for the BM at $1 \times 10^{-4}\ s^{-1}$). It is noted that, at $3 \times 10^{-4}\ s^{-1}$, both the nugget and the BM exhibited similar true stresses (red curves B and b): the flow stress (at true strain of 0.1%) of the nugget (30.2 MPa) was almost equal to that of the BM (29.7 MPa), and the maximum discrepancy from the peak flow stress was just 9.1%.

Therefore, the lamella-structured nugget showed comparable SP and flow stress with the BM at 800 °C and $3 \times 10^{-4}\ s^{-1}$. To our best knowledge, it is the first time to report that the similar SP and flow stress were obtained in the nugget and BM. This implies that it is possible for the entire FSW Ti-6Al-4V joint to be superplastically formed without local tear. Furthermore, the superplastic temperate is 800 °C, ~100 °C lower than the traditional SPF temperature.

The variation of flow stress with the initial strain rate for the nugget is shown in Fig. 2d. The strain rate sensitivity m value varied from 0.12 to 0.76 at different strain rate ranges for different temperatures. At the optimum strain rate of each temperature, the m value exceeded 0.3, suggesting that grain boundary sliding (GBS) played an important role in the SP [21].

To check the main factor influencing the SP, microstructures after annealing and superplastic deformation at 800 °C are shown in Fig. 3. After holding 5 min at 800 °C, the nugget still consisted of a lamellar microstructure (Fig. 3a). A fully globularized grain is defined when its aspect ratio (simplified as AR) is <2. After tension to 50%, α lamellae largely coarsened and AR reduced, with part of the lamellae being fully globularized with β phase distributed at α grain boundaries (Fig. 3b). The lamellae were almost completely globularized after tension to more than 200% (Fig. 3c and d). Obviously, the lamellar microstructure in the nugget changed significantly during superplastic tension.

Fig. 4 shows the AR distributions of the lamellae at different elongations at 800 °C and $3 \times 10^{-4}\ s^{-1}$, and the variation of average AR and thickness with the elongation. After annealing, the fraction of globularized grains was 20.8% (Fig. 4a). As the superplastic elongation increased, the fraction of lamellae with large AR gradually decreased,

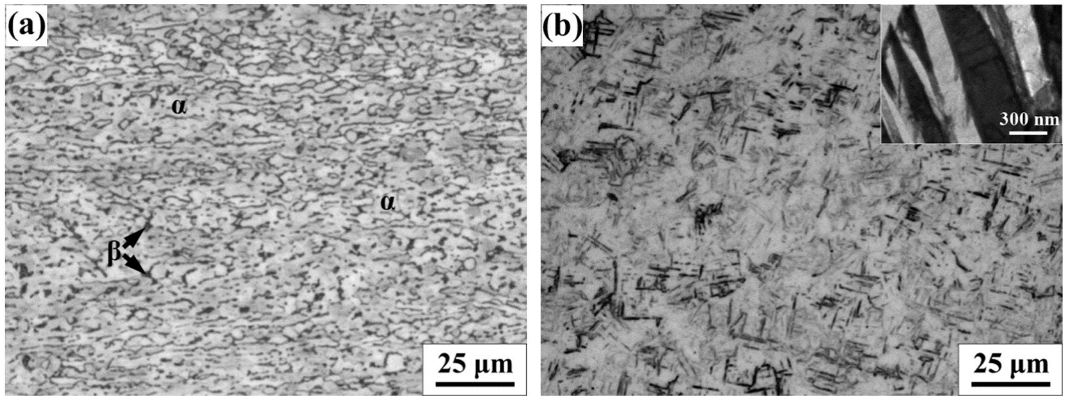

Fig. 1. OM images of (a) base material, and (b) nugget for FSW Ti-6Al-4V joint with an inserted TEM image.

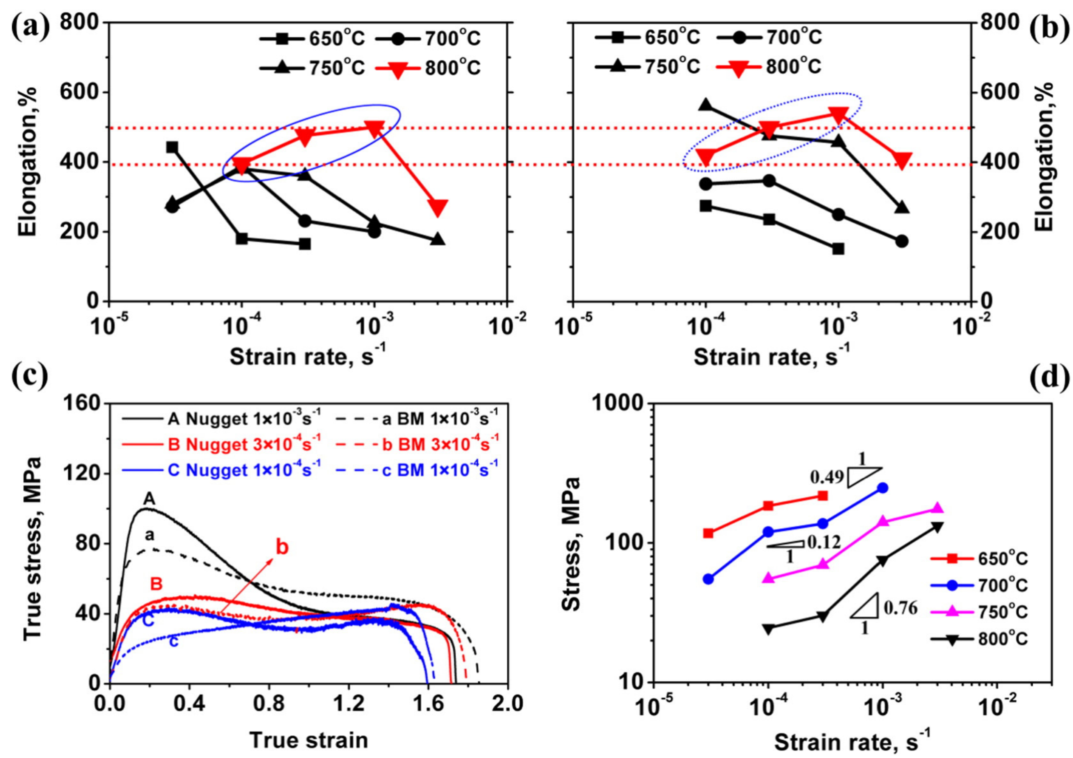

Fig. 2. Variation of elongation with initial strain rate at different temperatures for (a) nugget, and (b) base material, (c) true stress-strain curves at 800 °C and1×10^{-4}–1×10^{-3} s^{-1} for nugget and base material, and (d) variation of flow stress with initial strain rate for nugget of FSW Ti-6Al-4V alloy.

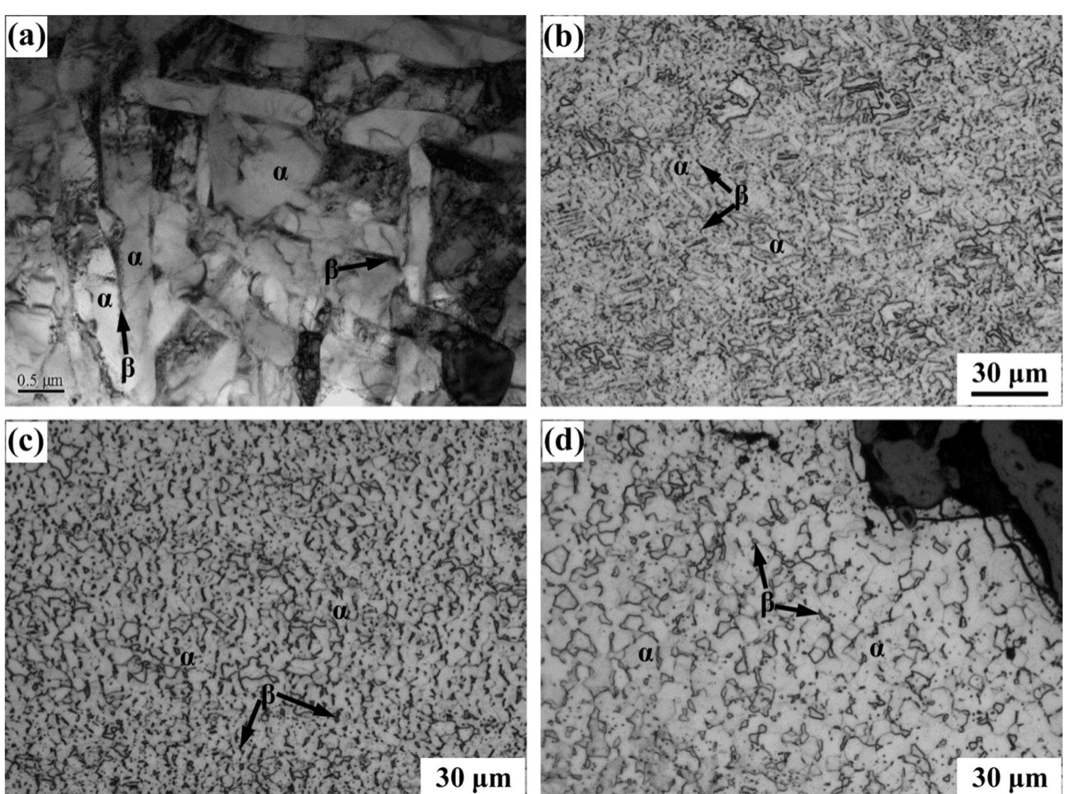

Fig. 3. (a) TEM images of nugget after holding for 5 min, and OM images of nugget in FSW Ti-6Al-4V joint after superplastic deformation at 800 °C and 3×10^{-4} s^{-1} to (b) 50%, (c) 200%, and (d) failure.

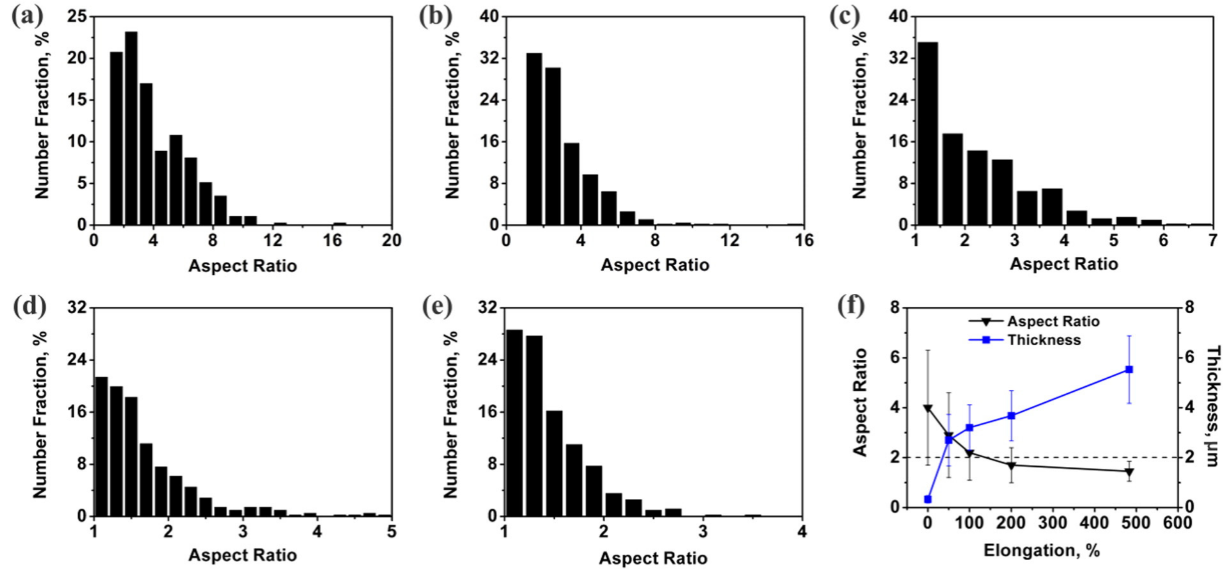

Fig. 4. Aspect ratio distribution of nugget after static annealing and superplastic deformation to different elongations at 800 °C and 3×10^{-4} s^{-1}: (a) annealing for 5 min, (b) 50%, (c) 100%, (d) 200%, (e) tension to failure, and (f) variation of average aspect ratio and thickness with elongation.

while that of globularized grains increased gradually from 20.8% in the annealing specimen to 91.3% in the failed specimen (Fig. 4a–e), indicating that the lamellae were gradually globularized due to the combination effects of thermal and stress. Fig. 4f more clearly reflected the trend of gradual globularization and coarsening of the lamellae with increasing the elongation.

Although Ti-6Al-4V is two-phase Ti alloy, it only contains no more than 20% of equilibrium β phase at temperatures of <800 °C [24]. Therefore, α was the main phase dominating the deformation at temperatures of <800 °C [25–27]. It was reported that the smaller for the grain size, the larger contribution of GBS to the LTSP of Ti-6Al-4V, and for (ultra-)fine equiaxed microstructure, α/α GBS dominated the low-temperature superplastic deformation [27]. The dominating effect of GBS has been recently confirmed by in situ superplastic deformation at temperatures of >700 °C [26]. Furthermore, α/β phase boundary sliding (PBS) occurred even more readily than the GBS during superplastic deformation [28], and thus α/α GBS and α/β PBS concurrently dominated the superplastic deformation of equiaxed Ti-6Al-4V [26,28].

In this study, although the initial microstructure was lamellar, during static annealing before superplastic tension, part of lamellae were globularized into the ultrafine grains. For example, the fraction of globularized grains was 20.8% after annealing at 800 °C (Figs. 3a and 4a), which was much higher than that in the as-welded nugget (2.2%), while it was close to that of the original lamellae with AR < 4 (24.8%) [21]. It is well-known that it is difficult for those lamellae with large AR to be globularized only by annealing because of low energy α/β interfaces, while it is likely achieved via coarsening of lamellae with small AR [25]. Therefore, these globularized grains after annealing mainly resulted from the coarsening of the lamellae with AR < 4, which promoted the occurrence of GBS/PBS, thereby enhancing SP.

Moreover, it was reported that the thickness and AR of α lamellae were the two main effect factors on dynamic globularization, and the smaller for the thickness or AR, the easier to be globularized [25]. During superplastic deformation in this study, the lamellae were easily globularized due to their small thickness and aspect ratio, and the globularized grains were still very fine (Figs. 3 and 4f). As a result, these gradual globularized fine grains further promoted the occurrence of GBS/PBS, thereby further increasing the SP. Therefore, gradual globularization of the fine lamellae during static annealing and superplastic deformation was the main reason for achieving good SP in the nugget.

It is important to point out that although GBS/PBS was proposed as the main mechanism in the superplastic deformation for equiaxed microstructure, recent studies suggested that GBS/PBS also played an important role in the deformation even for non-equiaxed fine microstructure [29–32]. For example, it was reported that a good SP could be obtained in a fine lamellar TiAl alloys, attributing to the occurrence of GBS [29]. It was even reported that lath boundary sliding similar to GBS possibly occurred in TiAl alloys, though there was controversy on it [31,32].

For a given grain size, GBS/PBS possibly takes place more easily for grains with smaller AR, because of smaller sliding stress, resulting from the reduced sliding area and easier accommodation between the neighbor grains. In this study, after holding for 5 min at 800 °C before superplastic tension, lots of fine lamellae had a relative small AR (e.g. 60.9% for the lamellae with AR < 4), which might also contribute to the occurrence of GBS/PBS. However, whether the fine lamellae with small AR promoted the occurrence of GBS/PBS still needs in-depth investigations to confirm.

In summary, the fine lamellar microstructure in the nugget of FSW Ti-6Al-4V alloy joint exhibited a superior LTSP of 442% at 650 °C. A comparable SP was achieved for the nugget and BM at 800 °C and 1×10^{-4}–1×10^{-3} s^{-1}, and especially at 3×10^{-4} s^{-1}, both superplasticity and flow stress were comparable. Good SP in the nugget was mainly contributed to gradual globularization of the fine lamellae during static annealing and superplastic deformation.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at http://dx.doi.org/10.1016/j.scriptamat.2016.05.020.

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