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Achieving superior superplasticity from lamellar microstructure of a nugget in a friction-stir-welded Ti–6Al–4V joint

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A largest elongation of 728% was achieved in a nugget of a friction-stir-welded (FSW) Ti–6Al–4V alloy joint with a fully lamellar microstructure; this is much larger than the maximum elongation ever reported in fusion-welded nuggets (<400%). This superior superplasticity in FSW nugget vs. fusion-welded nugget was mainly attributed to easier globularization of the lamellae with smaller thickness (172 nm) and aspect ratio (8.1) in the FSW nugget during preheating and superplastic deformation.

Keywords: Friction stir welding; Titanium alloys; Superplasticity; Microstructure

Superplastic forming (SPF) has been used to produce complex Ti parts in the aerospace field because of its low cost and high efficiency [1]. However, because of the size limit of the rolled sheets for SPF, a process combining SPF and diffusion bonding (SPF/DB) has been developed to produce large-sized complex components [2]. Unfortunately, some problems are associated with the DB process such as low production efficiency, mechanical property reduction, etc. Therefore, there is a need to develop the SPF process combined with other welding techniques for Ti alloys.

Generally speaking, good superplasticity (SP) usually requires a fine-grained microstructure to promote the occurrence of grain boundary sliding. However, after fusion welding, a coarse cast lamellar microstructure is obtained in the nugget, which largely destroys the superplastic nature of the workpiece. For example, Kruglov et al. [3] reported that coarse-grained microstructure in a nugget produced by gas tungsten arc welding prevented SPF of the Ti alloy joints. Chen et al. [4] reported a SP of 397% in a nugget of Ti–6Al–4V alloy laser welds, the largest SP reported in fusion-welded nuggets, though still much lower than that achieved in the base material (BM) (∼900%). Therefore, fusion welds cannot be superplastically formed to produce large-sized Ti alloy components with excellent integrated properties. Friction stir welding (FSW), a solid-state welding technique, however, can retain a relatively fine-grained microstructure of the BM and thus its combination with SPF has great potential to produce large Ti alloy components.

Since its invention in 1991, FSW has been widely studied in low-melting-temperature alloys, such as Al and Mg alloys [5,6] and recently, in high-melting-temperature alloys such as Ti alloys [7–9]. It was reported that defect-free high-quality welds of Ti alloys could be obtained by FSW and the joint efficiency could reach 90% or more [10]. Meanwhile, some researchers investigated a combination of SPF and FSW of Ti alloys, and some preliminary results have been reported by Ramulu and his coworkers [11–16]. In their studies, the nugget was the main object of investigation because the nugget is the key zone influencing formation of superplasticity throughout the entire FSW joint.

Edwards and Ramulu [11] investigated the effect of process conditions on the SPF behavior in Ti–6Al–4V FSW joints. They suggested that the nugget temperature could be controlled to be above, below or near the β transus temperature by adjusting the welding parameters. When the nugget temperature was above or below the β transus temperature, a fully lamellar or an extremely fine equiaxed microstructure was obtained in the nugget, which exhibited less or much more SP than the BM, respectively. Only when the nugget temperature was near the β transus temperature did the nugget exhibit a SP similar to that of the BM.

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Sanders et al. [14] also found that it was possible for the nugget to exhibit SP equivalent to that of the BM by strictly controlling the nugget temperature with the help of cooling systems. In addition, they found that the strength of FSW and post FSW-SPF joints was close to that of the BM while the elongations decreased [12]. All these results suggest that the combination of SPF and FSW is a promising process to produce monolithic Ti alloy components with an excellent combination of properties.

However, it is very difficult to control the nugget temperature to be just near the β transus temperature because it is highly sensitive to the FSW parameters. Previous studies [9] showed that for near α or α + β Ti alloys, the nugget temperature was over the β transus temperature and a fully lamellar microstructure was obtained in the nugget under most FSW conditions. Therefore, it is of great significance to study the superplastic deformation behavior of a nugget with a lamellar microstructure. Considering that in the previous studies [11–16], all the FSW joints were superplastically formed under a single deforming condition, it is probable that the optimum SP was not obtained in the nugget. Moreover, the exact SP value in the nugget and its superplastic deformation mechanism remain unknown so far.

Therefore, the objective of this study is (i) to explore the exact superplastic properties of a nugget with a fully lamellar microstructure in a FSW Ti alloy joint; and (ii) to elucidate its superplastic deformation mechanism. In order to elucidate the reason for the excellent SP of FSW nugget compared to fusion-welded nugget, a comparison on the microstructural evolution was made between laser welding and FSW.

The as-received material was 2 mm thick mill-annealed Ti–6Al–4V sheets. The sheets were FSWed at a rotation rate of 500 rpm and a transverse speed of 150 mm min^{-1}. A W–5Re alloy welding tool was used, which consisted of a concave shoulder 11 mm in diameter and a pin tapered from 6.4 mm diameter at the root to 4.5 mm diameter at the tip, with a length of 1.6 mm. Argon shielding was employed to prevent the sheet surface from oxidizing.

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 about −25 °C. In order to statistically analyze the thickness and aspect ratio of the lamellar microstructure in the nugget, more than 400 lamellae were measured.

Dogbone-shaped tensile specimens with a gauge length of 2.5 mm, a width of 1.4 mm and a thickness of 1.0 mm were cut from the nugget of the FSW joint. Constant cross-head speed tensile tests were carried out on an Instron 5848 microtester. To reach thermal equilibrium, each specimen was held at the testing temperature for 30 min before tension. Each specimen stretched to failure was cooled rapidly in water to retain the microstructure information from immediately after failure. For brevity, we will refer to the nugget of the FSW joint as the "FSW-nugget" hereinafter.

The characteristic microstructure of the BM and the FSW-nugget is shown in Figure 1. The mill-annealed BM was characterized by elongated α, some equiaxed α, and a small volume fraction of transformed β (black contrast in Fig. 1a). After FSW, the nugget consisted of a fully fine lamellar microstructure (Fig. 1b). TEM observation revealed different orientated lamellar structures within prior β grains with a size of 8–20 μm and that the β grain boundaries were decorated by α phase with a thickness of ~500 nm (Fig. 1c). The fully lamellar microstructure suggested that the weld temperature during FSW was over the β transus temperature (~980 °C) in this study.

In addition, dislocations and sometimes {10–11} contraction twins could be observed in the lamellae (not shown), which suggested that the fine lamellar structure in this study was probably the α' martensite [4]. The average thickness and aspect ratio of the lamellae were 172 ± 97 nm and 8.1 ± 5.3, respectively (Fig. 1d and e). Both the thickness and aspect ratio of the lamellae for the FSW joints were much smaller than that for the fusion welds, such as laser welds (the average thickness ~1 μm and aspect ratio >>10) [4].

Figure 2a shows the variation of elongation with initial strain rate at 850–925 °C for the FSW-nugget. Elongations of >200% were obtained at all the temperatures and strain rates, which suggested that the lamellar microstructure exhibited SP at all the temperatures and strain rates. At 850–900 °C, the optimum strain rate was 1×10^{-3} s^{-1}, while it increased to 3×10^{-3} s^{-1} at 925 °C. A temperature increase from 850 to 925 °C resulted in an increase in the

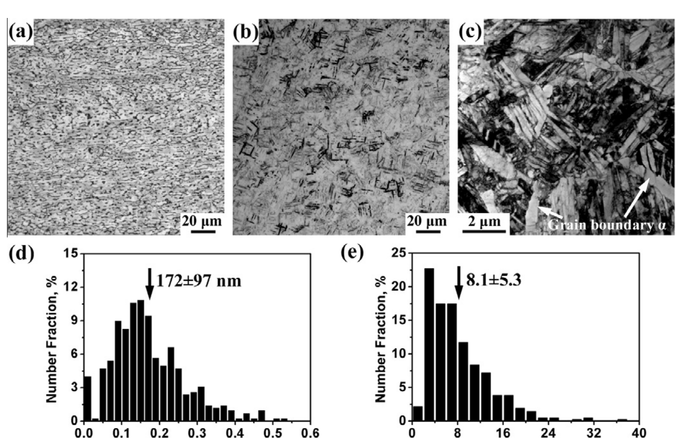

Figure 1. OM images of (a) base material and (b) nugget, (c) TEM image of nugget, (d) thickness and (e) aspect ratio distribution of lamellar microstructure in nugget for a FSW Ti–6Al–4V joint.

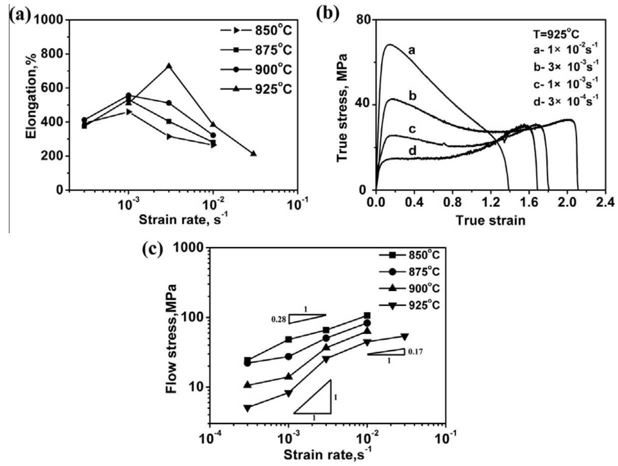

Figure 2. (a) Variation of elongation with initial strain rate at different temperatures, (b) true strain–stress curves at 925 °C, and (c) variation of flow stress with initial strain rate for FSW-nugget of Ti–6Al–4V alloy.

maximum elongation, and a maximum ductility of 728% was obtained at 925 °C and 3×10^{-3} s^{-1}. This result is surprising, because according to the conventional viewpoint, materials exhibiting a superior SP usually require an equiaxed microstructure, which is beneficial for grain boundary sliding (GBS)—the main mechanism for superplastic deformation.

Figure 2b shows typical true stress–true strain curves of the FSW-nugget at 925 °C for different strain rates. The optimum strain rate was determined to be 3×10^{-3} s^{-1}. At a high strain rate of 1×10^{-2} s^{-1}, a continuous flow stress softening took place after the initial strain hardening. This phenomenon is likely to be mainly related to the geometry factor resulting from necking. When the strain rate decreased, the trend of flow stress softening decreased. At the strain rate of 3×10^{-4} s^{-1}, a constant flow region before a true strain of ~0.7 was observed, a typical behavior when GBS dominates the deformation. At the last stage of superplastic deformation in the strain rate range from 3×10^{-4} to 3×10^{-3} s^{-1}, a continuous flow stress hardening took place after flow stress softening or constant flow regions, and the trend of hardening increased with decreasing strain rate. This phenomenon is mainly associated with grain growth during the later stage of the superplastic deformation, and the decrease in the strain rate means a longer time for grain growth, resulting in a larger hardening trend.

The variation of flow stress (at true strain of 0.1%) with the initial strain rate for the FSW-nugget is shown in Figure 2c. The strain-rate sensitivity m value varied from 0.17 to 1.0 at different strain rate ranges for different temperatures. The m values with the maximum elongations at various temperatures were ~0.55–1.0. GBS, as the main superplastic deformation mechanism, is usually related to an m value of ~0.5 or more [17]. It has been reported that α/β phase boundary sliding (PBS) occurred much more readily than GBS during superplastic deformation of Ti–6Al–4V [18]. Therefore, in this study, both PBS and GBS might dominate the superplastic deformation at various temperatures when the nugget exhibited the greatest elongation.

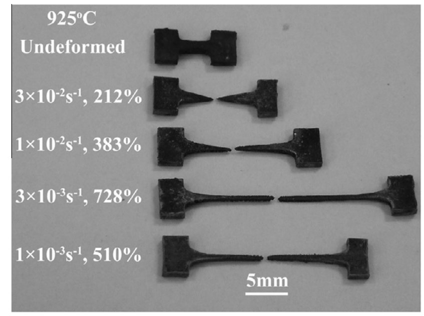

Figure 3. Tensile specimens pulled to failure at 925 °C at different strain rates.

Tensile specimens pulled to failure at 925 °C at different strain rates are shown in Figure 3. At high strain rates of 1×10^{-2}–3×10^{-2} s^{-1}, failed specimens exhibited a necking characteristic. When the strain rate decreased to 3×10^{-3} s^{-1} or less, the failed specimens exhibited a relatively uniform elongation feature, which was usually the result of the occurrence of GBS/PBS. This phenomenon is consistent with the true stress–true strain curve in Figure 2b.

By observing the microstructure of the specimens pulled to failure at 925 °C, it was found that all the lamellar microstructures were fully globularized at all strain rates and typical microstructures after static annealing and superplastic deformation to failure at 925 °C and 1×10^{-2} s^{-1} are shown in Figure 4. After being preheated for 30 min before the superplastic tensile test, the lamellar microstructure obviously thickened and the aspect ratio decreased, with some even becoming equiaxed grains, as shown in Figure 4a (we defined grains with an aspect ratio of <2 as equiaxed in this study). After superplastic deformation, however, the lamellar microstructure was completely globularized and coarsened under dynamic loading (Fig. 4b). This indicates that it was very easy for the lamellae of the nugget to globularize due to the comprehensive effect of thermal and stress, which promoted the occurrence of GBS/PBS.

In this study, the nugget exhibited a maximum elongation of 728% at 925 °C and 3×10^{-3} s^{-1}. To the best of our knowledge, this is the first report of SP properties of

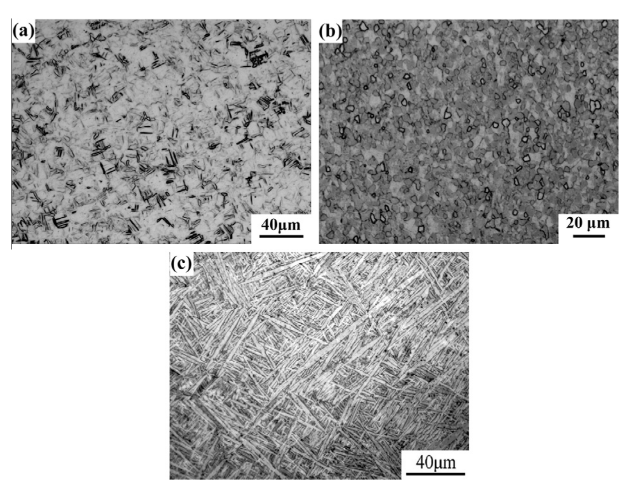

Figure 4. OM images showing microstructure of FSW-nugget of Ti–6Al–4V alloy after (a) static annealing for 30 min, and (b) being pulled to failure at 925 °C and 1×10^{-2} s^{-1}; (c) microstructure of laser-welded nugget after static annealing at 925 °C for 50 min [4].

FSW-nugget in Ti alloys. This result is encouraging because a typical lamellar microstructure produced by FSW under most conditions could achieve a high SP. By comparison, the maximum elongation in the nugget of fusion welds (achieved by laser welding) was <400% [4]. The higher SP achieved in the FSW-nugget was attributed to two reasons.

First, during static annealing prior to superplastic tension, it is easier to globularize the FSW-nugget than the laser-welded nugget. It has been reported that the termination migration mechanism is the main reason for the globularization of the lamellae [19] during heat treatment. It is very difficult to transform a fully lamellar microstructure into an equiaxed one by heat treatment alone because of the very slow kinetics of globularization, which is attributed to the low-energy interfaces of the lamellar microstructure resulting from the Burgers orientation relationship within two phases. Therefore, for the lamellae with a large aspect ratio in the laser weld, lamellae thickening predominated during static annealing (Fig. 4c) [4].

For the FSW-nugget, however, lamellae thickening resulted in a significant decrease in their aspect ratio during heat treatment because of the small initial thickness and length, and the lamellae with a small aspect ratio (e.g. <4) could be finally transformed into equiaxed grains with the help of termination migration (Fig. 4a). For example, the fraction of the lamellae with an aspect ratio of <4 was 24.9%, and when these lamellae coarsened, it is likely that they will globularize. That is to say, some of the lamellae were globularized during static annealing. In this case, these equiaxed α grains or lamellae with a small aspect ratio formed prior to the superplastic tension promoted the operation of GBS/PBS at the beginning of the superplastic deformation, thereby helping to enhance SP.

Second, it was much easier for the FSW-nugget to further globularize during superplastic deformation. During thermomechanical processing, the fragmentation of the lamellae via boundary splitting by the groove of the β phase was considered to be the main mechanism of globularization, and thus the finer the lamellae, more easily the lamellae globularized. Obviously the smaller the aspect ratio was, more easily the lamellae were transformed into equiaxed grains [19]. Therefore, compared to laser welding, lamellae with a smaller thickness and aspect ratio in the FSW-nugget were easier to further globularize via the combined effect of stress and heat during superplastic deformation (Fig. 4b). The globularized microstructure further promoted the occurrence of GBS/PBS and thus a higher SP was obtained.

Clearly, compared to laser welding, a higher SP in the FSW-nugget was associated with easier globularization of the lamellae during both static annealing and superplastic deformation due to their smaller thickness and aspect ratio, which resulted in GBS/PBS dominating during the superplastic deformation.

In summary, FSW is a promising way to produce a lamellar microstructure with small thickness and aspect ratio in Ti–6Al–4V alloy. The lamellar microstructure in the nugget of a FSW Ti–6Al–4V joint with a thickness of 172 nm and an aspect ratio of 8.1 exhibited a superior superplasticity of 728% at 925 °C and 3×10^{-3} s^{-1}.

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[1] G. Lütjering, J.C. Williams, Titanium, 2nd ed., Springer, New York, 2007.
[2] D.G. Sanders, M. Ramulu, J. Mater. Eng. Perform. 13 (2004) 744.
[3] A.A. Kruglov, F.U. Enikeev, R.Y. Lutfullin, Mater. Sci. Eng. A 323 (2002) 416.
[4] S.H. Chen, J.H. Huang, D.H. Cheng, H. Zhang, X.K. Zhao, Mater. Sci. Eng. A 541 (2012) 110.
[5] J. Yang, B.L. Xiao, D. Wang, Z.Y. Ma, Mater. Sci. Eng. A 527 (2010) 708.
[6] Z. Zhang, B.L. Xiao, Z.Y. Ma, J. Mater. Sci. 47 (2012) 4075.
[7] Y. Zhang, Y.S. Sato, H. Kokawa, S.H.C. Park, S. Hirano, Mater. Sci. Eng. A 485 (2008) 448.
[8] L.H. Wu, D. Wang, B.L. Xiao, Z.Y. Ma, Scr. Mater. 78–79 (2014) 17.
[9] L.H. Wu, D. Wang, B.L. Xiao, Z.Y. Ma, Mater. Chem. Phys. 146 (2014) 512.
[10] L. Zhou, H.J. Liu, Q.W. Liu, Mater. Des. 31 (2010) 2631.
[11] P. Edwards, M. Ramulu, Sci. Technol. Weld. Joining 14 (2009) 669.
[12] D.G. Sanders, M. Ramulu, E.J. Klock-McCook, P.D. Edwards, A.P. Reynolds, T. Trapp, J. Mater. Eng. Perform. 17 (2008) 187.
[13] M. Ramulu, P.D. Edwards, D.G. Sanders, A.P. Reynolds, T. Trapp, Mater. Des. 31 (2010) 3056.
[14] D.G. Sanders, M. Ramulu, P.D. Edwards, A. Cantrell, J. Mater. Eng. Perform. 19 (2010) 503.
[15] D. Sanders, M. Ramulu, P. Edwards, Materialwiss. Werkstofftech. 39 (2008) 353.
[16] P. Edwards, M. Ramulu, D. Sanders, Key Eng. Mater. 433 (2010) 169.
[17] F.C. Liu, Z.Y. Ma, Scr. Mater. 59 (2008) 882.
[18] J.S. Kim, J.H. Kim, Y.T. Lee, C.G. Park, C.S. Lee, Mater. Sci. Eng. A 263 (1999) 272.
[19] S. Zherebtsov, M. Murzinova, A. Salishchev, S.L. Sennatin, Acta Mater. 59 (2011) 4138.