Superplastic Behaviour of Ti54M and Ti64

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Abstract

Even though TIMETAL-54M (Ti-5Al-4V-0.6Mo-0.4Fe or Ti54M) has been commercially available for over 10 years, further study of its superplastic properties is still required in order to assess its applicability within the aerospace industry as a potential replacement for other commercial titanium alloys such as Ti-6Al-4V (Ti64). Ti54M is expected to obtain superplastic characteristics at a lower temperature than Ti64 due to its lower beta-transus temperature. The superplastic forming (SPF) capability of alloys that can be formed at lower temperatures has always attracted the interest of industry as it reduces the grain growth and alpha-case formation, leading to longer life for costly high temperature resistant forming tools.

In this work, the SPF characteristics of both Ti54M and Ti64 have been examined by conducting tensile tests according to the ASTM E2448 standard within a range of temperatures and strain values at a fixed strain rate of $1 \times 10^{-4}$/s. A high strain rate sensitivity and uniform deformation at high strains are key indicators in selecting the optimum superplastic temperature. This was observed at 815$^\circ$C and 925$^\circ$C for Ti54M and Ti64 respectively. The tensile samples were water quenched to freeze their respective microstructure evolution following superplastic deformation and SEM images were captured for grain size and volume fraction of alpha-phase analyses. A slightly higher alpha-grain growth rate was observed during superplastic deformation of Ti64. The initial fine-grain microstructure of Ti54M (~1.6 micron) resulted in a final microstructure with an average grain size of ~3.4 micron and optimum the alpha/beta ratio. Both the fine-grained microstructure and increased amount of beta-volume fraction promotes the superplastic behaviour of Ti54M by grain boundary sliding (GBS). Thus superplastic properties were observed for Ti54M at a lower temperature (~100$^\circ$C) than for Ti64.

1. INTRODUCTION

The ability of a polycrystalline material to exhibit very high tensile elongations prior to failure is referred as superplasticity. Normally the superplasticity of a material is defined by tensile elongations of more than 200 – 300% without localised necking and a high degree of strain rate sensitivity (m) in the range of 0.5 – 0.8. The basic requirements for achieving superplasticity are – (i) fine grain microstructure, usually with two phases, (ii) superplastic deformation temperature in the range of 0.5 to 0.8 of the absolute melting point temperature of the alloy, (iii) metallurgically stable microstructure at the superplastic temperature with restricted grain growth, and (iv) controlled strain rate, usually in the range of 0.01/s to 0.0001/s for maximum superplasticity. To some extent, these requirements are contradictory since the higher temperatures and the slow strain rates tend to facilitate detrimental grain growth. Therefore, other parameters such as grain growth kinetics, grain aspect ratio, grain size distribution, volume fraction and the texture of phases should be considered [1].

The most common titanium alloy within the aerospace industry is Ti-6Al-4V (Ti64), which provides a combination of corrosion resistance, satisfactory mechanical properties, high weldability, good machinability and a wide range of in-service temperatures (good for applications up to the range of about 315 $^\circ$C – 400$^\circ$C) [2]. The superplastic formability of Ti64 is maximized at temperature typically higher than 875$^\circ$C, with slow strain rates. At elevated temperatures the $\beta$ phase is considerably softer than the $\alpha$ phase, showing higher diffusivity and the presence of more available slip systems. Therefore the volume fraction of $\beta$ phase plays a critical role in determining the prevailing deformation mechanism. In this respect, the dominant deformation mechanism typically operating during superplastic deformation is Rachinger grain boundary sliding (GBS). Rachinger GBS is accommodated by several different mechanisms, such as (i) diffusion, (ii) dislocation movement or, (iii) a combination of both [3, 4]. Two other deformation processes, such as cooperative GBS and cavity formation, are also considered for superplastic behaviour [5, 6].

In titanium, higher forming temperatures tend to reduce die life, lead to the formation of excessive alpha case and reduce overall productivity due to longer heat up times and higher energy costs. Thus, there has been a focus on the development of alloys that can be formed at comparatively lower temperatures. Lower temperature SPF could be achieved by: (i) achieving finer grain size (~1 – 2 $\mu$m) (ii) increasing the diffusion rate for faster GBS, (iii) lowering the $\beta$-transus temperatures [7], and (iv) increasing the volume fraction of $\beta$ phase (~40% $\beta$ phase for optimum conditions).
superplasticity i.e. ~1200% elongation for Ti64 [1]) so that most of the deformation can be accommodated within the β phase due to its cubic crystal structure. Based on specific thermo-mechanical processing routes, finer globular primary α grains can be achieved. Incorporation of substitutional elements into the titanium alloy can also decrease the SPF temperature due to their ability to promote α grain refinement and to modify diffusion rate of β phase. Several titanium alloys have already been developed with this approach, and among them one notable commercial alloy is TIMETAL-54M (Ti-5Al-4V-0.6Mo-0.4Fe, or Ti54M) [8]. Ti54M is an α-β alloy, which provides superior machinability and strength comparable to similarly processed Ti64 [7, 9]. Compared to Ti64, Ti54M has lower Al content (α stabiliser) that reduces the material hardness leading to improved machinability [10]. The β-transus temperature of Ti54M (in the range of 938°C – 966°C depending on exact composition) is between 30°C – 50°C below that of Ti64, and hence the superplastic temperature range of Ti54M is considerably lower than Ti64.

In this work, a Ti54M sheet with 1.6 mm thickness and an average grain size of ~1.5 μm was thermo-mechanically processed and its microstructural evolution was investigated during superplastic forming and then compared to a Ti64 sheet with similar thickness, though with a larger initial grain size. Both the materials were supplied by Timet.

2. EXPERIMENTAL DETAILS

Interrupted tensile tests were carried out for both materials in order to understand the microstructural evolution during SPF. The interrupted tensile tests were done at 925°C and 815°C for Ti64 and Ti54M respectively [11] at three different true strain values (35%, 101% and 171%) and at a true strain rate of 1x10^-4/s according to ASTM E2448 standard. This strain rate was particularly chosen because the highest m value for Ti54M was reported at this true strain rate in the temperature range of 780°C – 850°C [7]. Further to that, this strain rate is widely applicable for commercial superplastic forming applications of Ti64 [12]. The tensile test samples were glass coated prior to testing to avoid alpha case formation. Following testing, the tensile samples were water quenched to freeze their respective microstructures. The as-received material and the tensile test samples were cut along the longitudinal direction for grain size and phase-volume analysis. Polishing was performed using a Buehler EcoMet 300 automatic grinder/polisher and the polishing steps included 600 and 1200 grit papers followed by micro cloths for 9 μm diamond paste and 0.02 μm colloidal silica until the surface became mirror-like. Backscattered electron (BSE) images were captured using FEI Quanta 250 FEG SEM and the accelerating voltage and working distance were 15kV and 10mm respectively. ImageJ software was used for grain size analysis according to ASTM standard E112 (200 – 300 α grains were considered for average grain size analysis). To calculate the volume fraction of α phase (primary α only) and the β parent grains, the primary α grains were manually highlighted. The ImageJ software was then used to measure the relative percentages of α and β grains.

3. RESULTS AND DISCUSSION

FIGURE 1 collates the normalized stress-strain curves of Ti54M at a strain rate of 1x10^-4/s at 815°C. These curves were typical superplastic stress-strain curves in which the majority of the flow curves were dominated by a broadly linear response with gradual strain hardening. The positive gradient on the curve was evidence of the work hardening flow behaviour of the material. This behaviour was also observed in previous studies of Ti54M although that the material investigated previously had a different average grain size and volume fraction of primary α phase [7].

FIGURE 2 shows the microstructures of the as-received and superplastically formed Ti54M samples. The thermo-mechanical processing of as-received Ti54M sheet material led to a microstructure containing equiaxed primary α grains within the β matrix (FIGURE 2a). The yellow circles indicate clusters made of very fine α grains having less than a micron diameter, whereas the red circles show the presence of large α grains having 3 – 6 μm diameter. Both the
fine and large α grains were included in the average grain size calculation, which led to an average grain size of ~1.56 ± 0.65 μm. It proved difficult to separate the individual β grains, thus the β grain size was not measured. Therefore, the average grain size represented the α phase only. The average aspect ratio of the α grains was found to be 1.59 and the percentage globularisation of the primary α grains was observed as 76.5% indicating the presence of mostly globular grains in the as-received Ti54M material. This specific microstructure of Ti54M was found to be favorable for SPF. FIGURE 2b - FIGURE 2d show the respective microstructures of the samples deformed at 35%, 101% and 171% true strain values. With increase in strain values, the microstructure remained equiaxed, however the grain growth was observed. The deformed samples showed a microstructure containing both equiaxed primary α grains (including fine α grains having ~2 μm diameter and large α grains having ~7 μm diameter) and the martensitic α prime grains within the β matrix – the latter forming during the water quenching. No clusters of very fine α grains, as observed in the as-received material, were observed after the tensile tests. The grain growth led to the formation of separate and comparatively large α grains. The β grains formed a matrix with no detectable grain boundaries between individual grains. Thus both martensitic α prime and β grains were excluded during the grain size analysis.

![Microstructures of the (a) as-received and superplastically formed Ti54M samples at (b) 35%, (c) 101% and (c) 171% true strain (scale shows length of 10 mm)](image)

FIGURE 2: Microstructures of the (a) as-received and superplastically formed Ti54M samples at (b) 35%, (c) 101% and (c) 171% true strain (scale shows length of 10 mm)

TABLE 1 summarises the microstructural properties of as-received and superplastically formed Ti54M samples at 815°C. The grain growth observed was in the range of ~57% – 116% from the as-received to deformed condition with increase in strain. No significant changes were observed in terms of grain shape (i.e. elongation as denoted by average aspect ratio) and percentage globularisation. Deformation at high temperature increases the volume fraction of β phase, which results in a noticeable decrease in equiaxed primary α phase from the as-received to deformed condition. It should be noted that a large volume fraction of β phase enhances superplastic behaviour, but at the same time it promotes grain growth, especially at higher temperature. Thus a substantial volume fraction of α phase is needed to provide the required microstructural stability of the material [4]. Therefore the optimum value of α/β ratio depends on the specific material and its superplastic temperature. In this study, the α/β ratio was observed in the range of 1.66 – 1.83 for Ti54M. As noted earlier, the dominant deformation mechanism typically observed is Rachinger GBS; this can enhance grain growth rates (as compared to static heating), which in turn influences the main characteristics of superplasticity – (i) strain rate sensitivity of flow stress and (ii) ductility limit.

TABLE 1: Summary of microstructural properties of as-received and superplastically formed Ti54M samples at 815°C

| Microstructural properties of equiaxed primary α grains | As-received material | 35% | 101% | 171% |
|--------------------------------------------------------|----------------------|-----|------|------|
| Average grain size (μm)                               | ~1.56 ± 0.65         | ~2.45 ± 1.09 | ~2.87 ± 1.27 | ~3.41 ± 1.53 |
| Average aspect ratio                                   | 1.59                 | 1.63 | 1.68 | 1.63 |
| Percentage globularisation                            | 76.51%               | 83.33% | 81.09% | 81.15% |
| Phase volume fraction                                  | 78.26%               | 62.4% | 64.03% | 64.67% |

TABLE 1: Summary of microstructural properties of as-received and superplastically formed Ti54M samples at 815°C
FIGURE 3 collates the normalized stress-strain curves of Ti64 at a strain rate of $1 \times 10^{-4}/s$ at 925°C. As with Ti54M, these curves were typical superplastic stress-strain curves in which the majority of the flow curves were dominated by a broadly linear response with gradual strain hardening. Compared to Ti54M, the higher gradient of the Ti64 curves was suggestive of a more rapid rate of grain growth, which would ultimately tend to reduce the superplastic response.

FIGURE 4 shows the microstructures of the as-received and superplastically formed Ti64 samples. The as-received Ti64 material contained equiaxed primary $\alpha$ phase (consisted of fine $\alpha$ grains having 1 – 5 $\mu$m diameter as well as large $\alpha$ grains having 6 – 11 $\mu$m diameter) within a $\beta$ matrix (FIGURE 4a). Similar to the method described above, both the fine and large $\alpha$ grains were considered during average grain size calculation, which produced an average grain size of 3.14 ± 1.45 $\mu$m. The average aspect ratio of the $\alpha$ grains was measured as 1.47 and the percentage globularisation of primary $\alpha$ grains was observed as 88.74% indicating the presence of mostly globular grains in the as-received Ti64 material. Overall, the as-received Ti64 material showed a higher average grain size and the presence of more globular $\alpha$ grains as compared to the as-received Ti54M. FIGURE 4b - FIGURE 4d show the respective microstructures of the Ti64 samples deformed at 35%, 101% and 171% true strain values. Measurements of the grain size revealed a considerable variation with the $\alpha$ grains in the diameter range of 2.5 – 14.5 $\mu$m. The $\beta$ grain growth was notable for the sample experiencing a true strain of 171% leading to an average grain size of ~7.09 ± 2.52 $\mu$m. The microstructure contained both equiaxed primary $\alpha$ grains and the martensitic $\alpha$ prime grains within the $\beta$ matrix. The average grain size was determined by averaging the size of equiaxed primary $\alpha$ grains only.

FIGURE 3: Normalized stress-strain curves showing superplastic behaviour of Ti64 at 925°C
FIGURE 4: Microstructures of the (a) as-received and superplastically formed Ti64 samples at (b) 35%, (c) 101% and (c) 171% true strain (scale shows length of 20 mm)

TABLE 2 summarises the microstructural properties of the as-received and superplastically formed Ti64 samples at 925°C. The grain growth was observed in the range from ~88% – 126% from the as-received to the deformed condition with increase in strain. However, no significant changes were observed in terms of grain shape and percentage globularisation. It should be noted that the rate of grain growth was observed to be higher in the Ti64, which might be influenced by not only the high temperature (almost 100°C higher temperature than Ti54M) but also by the material composition and α/β ratio. Rachinger GBS was reported as the dominant deformation mechanism for high temperature superplasticity of Ti64 (>850°C) [3]. At this high temperature, the β phase becomes soft and deformable and the volume fraction of β phase significantly increases with increase in temperature resulting in a α/β ratio in the range of 1.46 – 1.82 for Ti64.

TABLE 2: Summary of microstructural properties of as-received and superplastically formed Ti64 samples at 925°C

| Microstructural properties of equiaxed primary α grains | As-received material | 35% | 101% | 171% |
|---------------------------------------------------------|----------------------|-----|-----|-----|
| Average grain size (μm)                                | ~3.14 ± 1.45         | ~5.91 ± 1.69 | ~6.77 ± 2.38 | ~7.09 ± 2.52 |
| Average aspect ratio                                   | 1.47                 | 1.47 | 1.53 | 1.56 |
| Percentage globularisation                             | 88.74%               | 89.7% | 86.06% | 85.81% |
| Phase volume fraction                                  | 92.93%               | 62.87% | 59.27% | 64.48% |

4. CONCLUSIONS

In this work, the SPF characteristics of both Ti54M and Ti64 were examined within a range of temperatures and strain values at a fixed strain rate of $1 \times 10^{-4}$/s, and their respective microstructure evolution following superplastic deformation was analysed. The α-grain growth was observed to have slightly higher values for Ti64 (~88 – 126%) as compared to Ti54M (~57 – 116%) during superplastic deformation at all strain values. This correlated with the higher gradient of the flow curves for Ti64. The initial fine-grained microstructure of the Ti54M led to greater amount of grain boundary area per unit volume, which would facilitate grain boundary sliding (GBS). The finer grained initial structure combined with a lower β transus allowed the Ti54M alloy to exhibit superplasticity at ~100°C lower than Ti64. The lower forming temperature for Ti54M may allow the use of alternative tooling materials with lower alloying levels (and thus cost) and reduced lead times to source. In situations where larger numbers of parts are being manufactured a lower forming temperature will also mean less oxide build up on tooling (and may eliminate issues due to α case) and therefore provides an opportunity to manufacture increased numbers of parts before tools are required to be cooled down and removed for cleaning and fettling operations.

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6. REFERENCES

1. A. K. Ghosh and C. Hamilton, Defence Science Journal, 36-2 (1986) 153-177.
2. R. Boyer, Materials Science and Engineering, A213 (1996) 103-114.
3. E. Alabort, P. Kontis, D. Barba, K. Dragnevski and R. Reed, Acta Materialia, 105 (2016) 449-463.
4. N. Ridley, Superplastic Forming of Advanced Metallic Materials: Methods and Applications, Woodhead Publishing Limited, 2011 pp. 3 - 33.
5. K. Higashi, M. Mabuchi and T. G. Langdon, ISIJ International, 36-12 (1996) 1423-1438.
6. E. Alabort, D. Putman and R. Reed, Acta Materialia, 95 (2015) 428-442.
7. Y. Kosaka and P. Gudipati, Key Engineering Materials, 433 (2010) 311-317.
8. Y. Kosaka and P. Gudipati, Materials Science Forum, 838-839 (2016) 190-195.
9. N. Khanna and K. S. Sangwan, Proc IMechE Part B: Journal of Engineering Manufacture, 227-1 (2012) 96–101.
10. L.S. Croan, F.J. Rizzitano, W.L. Warner, Watertown arsenal laboratories, 1959.
11. A. Gomez-Gallegos, P. Mandal, D. Gonzalez, N. Zuelli and P. Blackwell, in Superplasticity in Advanced Materials (ICSAM 2018), 2018.
12. M. Kobayashi and M. Miyagawa, Transactions ISIJ, (1987) 685-695.