Mechanical and microstructural analysis of Ti-6Al-4V material in a wide range of superplastic forming conditions

Alexander Sancho[1,a,*], Paranjayee Mandal[1,2,b], Craig Knowles[1,c], Diego Gonzalez[1,d]

[1]Advanced Forming Research Centre, University of Strathclyde, 85 Inchinnan Drive, Inchinnan, Renfrewshire PA4 9LJ, Scotland

[2]Now at BorgWarner Inc., Courteney Road, Gillingham ME8 0RU, England

[a]alexander.sancho@strath.ac.uk, [b]paranjayee.mandal@strath.ac.uk, [c]craig.knowles@strath.ac.uk, [d]diego.gonzalez@strath.ac.uk

Keywords: Superplasticity, Forming, Titanium, Ti-6Al-4V, Microstructure, Tensile, Strain-Rate Sensitivity

Abstract. In order to accurately define the superplastic forming (SPF) conditions of Ti-6Al-4V material, an understanding of the stress-strain behaviour, the initial microstructure, and their evolution during superplastic deformation are required. Ti-6Al-4V material with microstructure beneficial for SPF was superplastically tested according to the ASTM E2448 standard considering a wide range of forming conditions in terms of temperatures (750°C – 830°C) and strain-rates (seven strain-rates ranging from $5 \cdot 10^{-5}\ \mathrm{s}^{-1}$ to $5 \cdot 10^{-2}\ \mathrm{s}^{-1}$) – some of the tests of the 3x7 matrix are considered "extreme" conditions from an SPF point of view. The material showed improved superplastic behaviour, which was evident from the stress levels and strain-rate sensitivity values as estimated from the flow curves obtained for the different conditions. In comparison with other commercial alloys or results from similar analyses published in the last decades, low stress values and high strain-rate sensitivity (m) values were obtained despite the low temperatures and high strain-rates used in this analysis. The tests were interrupted when 0.5 true strain (65% engineering strain) was achieved followed by quenching, as this was the maximum local strain achieved when forming the component of interest. Samples did not show any sign of premature necking or failure, with the exception of the two most "extreme" cases. Particularly for the lower strain-rates (below $10^{-4}\ \mathrm{s}^{-1}$), some level of material hardening associated with a minimum grain growth was observed in the flow curves. In contrast, a noticeable material softening was observed for the higher strain-rate conditions (above $5 \cdot 10^{-3}\ \mathrm{s}^{-1}$), associated with the microstructural changes occurring due to dynamic recrystallization. These higher strain-rates led to formation of submicron-sized grains, which could have helped in the superplastic response of the material under these strain-rate conditions. Intermediate strain-rates ($5 \cdot 10^{-4}\ \mathrm{s}^{-1}$ and $10^{-3}\ \mathrm{s}^{-1}$) showed a different type of response in terms of microstructural behaviour (and flow curve outline) depending on the testing temperature. A negligible amount of cavitation was observed in the samples tested under extreme conditions.

Introduction

Ti-6Al-4V is a titanium alloy that has traditionally been used in the hot forming of aeronautic components. Examples of these hot manufacturing processes include hot creep forming and superplastic forming (SPF) [1, 2]. The industry is currently exploring and implementing methodologies for temperature and forming time reductions of the SPF process, as this has a real impact in production. Typical SPF temperatures in industry have been traditionally found in the range 850°C to 900°C. There are multiple advantages in that temperature reduction. Among others: energy savings (and the associated carbon footprint reduction), the possibility of using cheaper alloys for the tooling (generally very expensive Nickel alloys due to the Cr, Co, and Ni contents), increase in the tooling life (with less re-machining operations during its lifetime), the possibility to increase batch sizes before tooling needs to be replaced for cleaning (i.e., increase of productivity and reduction of maintenance) and cost reductions in terms of consumables (for example, the life of the cartridges increases at lower temperatures).

In order to achieve temperature and forming time reductions, a key parameter is the grain size of the material. Fine grain Ti-6Al-4V alloys present better superplastic behaviour (higher strain-rate sensitivities) than alloys with coarser grains, even at lower temperatures and faster strain-rates [3 – 6]. The refined grain structure tends to enhance grain boundary sliding allowing for larger plastic deformations [5, 7].

The aim of this work was to test a fine-grain Ti-6Al-4V alloy in a wide range of strain-rates and temperatures in order to understand the new set of forming conditions for faster and colder SPF forming. The targeted temperatures are relatively low compared to traditional SPF temperatures for titanium (750°C to 830°C). This research work was funded by The Boeing Company.

Results

This section will show the obtained results in terms of mechanical testing and microstructural analysis of the Ti-6Al-4V material supplied by The Boeing Company as per AMS4911 and used in this work. This information will help to understand the material behaviour in terms of stress levels, strain-rate sensitivity and formability for each temperature and strain-rate condition, also the microstructure behaviour when the material is deformed at temperature, as well as the formability limits of the material. In addition, mechanical and microstructural results will be later used for the definition of SPF forming conditions.

- **Tensile Testing:**

Tensile testing at elevated temperature was conducted using a Zwick 250 Universal testing machine and a three zones clamshell furnace attached to the machine. The three zones furnace is controlled based on the temperature readings of three thermocouples attached to the sample. The gauge length of the samples was coated with glass lubricant (Prince Minerals, pink glass lubricant) in order to minimize as much as possible the appearance of alpha-case during the test. The initial thickness of test specimens was 2.03mm (0.080”). Table 1 collects the testing conditions in terms of temperature and strain-rate.

Table 1. Test matrix with three temperatures and seven strain-rates. Tests interrupted at 0.5 true strain.

Temperature (T)	**Strain-rate (s^{-1})**	**True strain (ε)**
Low temp. - T = 750°C Mid. temp. (T between 750°C - 830°C) High temp. - T = 830°C	$\dot{\varepsilon} = 5 \cdot 10^{-5}$	$\varepsilon = 0.5$
	$\dot{\varepsilon} = 10^{-4}$	
	$\dot{\varepsilon} = 5 \cdot 10^{-4}$	
	$\dot{\varepsilon} = 10^{-3}$	
	$\dot{\varepsilon} = 5 \cdot 10^{-3}$	
	$\dot{\varepsilon} = 10^{-2}$	
	$\dot{\varepsilon} = 5 \cdot 10^{-2}$	

Three different temperatures were used in the tests: 750°C (1382°F), 830°C (1526°F) and a middle temperature in the range between those two values. Seven strain-rate values have been defined for the material testing, covering a wide range of values, from $\dot{\varepsilon} = 5 \cdot 10^{-5} s^{-1}$ to $\dot{\varepsilon} = 5 \cdot 10^{-2} s^{-1}$. The idea for this wide range was to explore all the posibilities in terms of the strain-rate of the deformation undergone by the material (although the SPF forming cycle will be defined to run as fast as possible, there will be material points in the component that will see very low strain-rates at some point of the forming process).

The tests have been conducted as per the ASTM E2448 standard, increasing the strain-rate value of the test by 20% during specific ranges of strain. These so-called jump strain-rate tests allow to estimate the strain-rate sensitivity of the material for each test condition. Tests were interrupted at 0.5 true strain (65% engineering), as the maximum deformation achieved in the component of analysis was below this value.

Since microstructural analysis of specific tested samples was subsequently conducted, the samples were water quenched once the 0.5 true strain was achieved. This water quenching, which took no more than 6 seconds, “freezes” the existing microstructure at high temperature to be later analysed via SEM microscopy.

Figure 1 collects the tensile test results for 750°C. As the reader can see, there is material hardening for the slowest strain-rates associated to grain growth. For the highest strain-rates, material softening takes place due to grain refinement (linked to dynamic recrystallization phenomena). For the fastest strain-rate, $\dot{\varepsilon} = 5 \cdot 10^{-2}\mathrm{s}^{-1}$, fracture occured in one of the two tests.

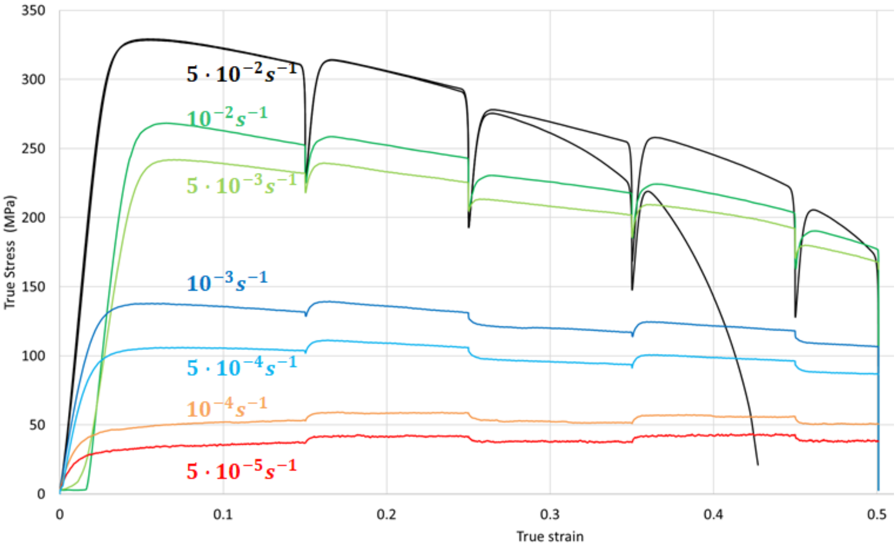

Figure 1. Flow curves obtained from the jump strain-rate tests at 750 °C.

Figure 2 collects the tensile test results for the temperature within the range between 750°C and 830°C. Similarly to the previous temperature, material hardening occurs for the slowest strain-rates whereas softening happens for the highest strain-rates. As the temperature has been increased, lower stress values than in the 750°C case have been achieved. No failure happened for any of the tests at this temperature (and samples did not show any sign of premature necking for the fastest strain-rates either).

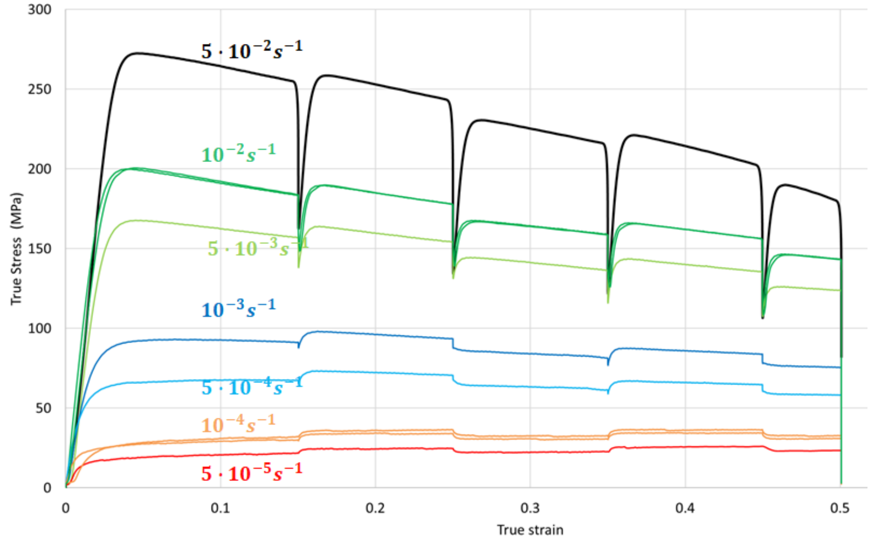

Figure 2. Flow curves obtained from the jump strain-rate tests at the temperature within the range between 750°C and 830°C.

Figure 3 collects the tensile test results for 830°C. As expected, lower stress values than in the previous temperatures were obtained.

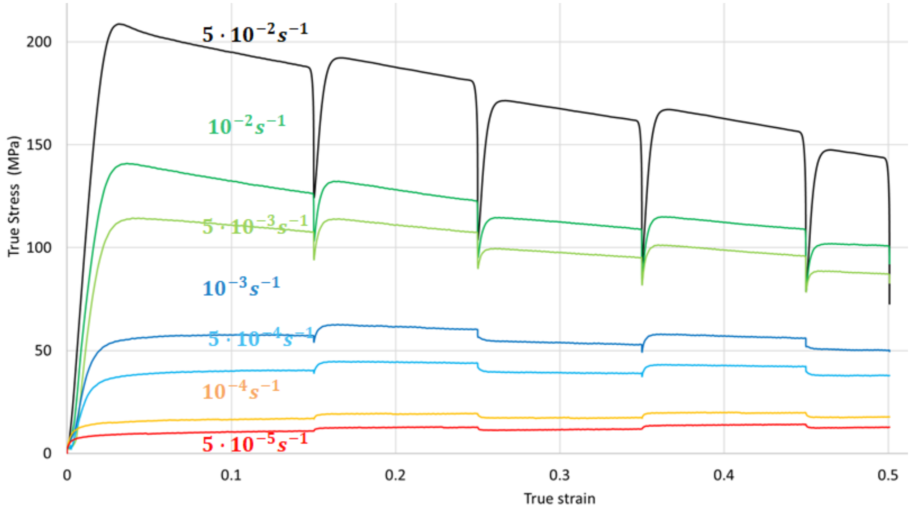

Figure 3. Flow curves obtained from the jump strain-rate tests at 830 °C.

From the jump strain-rate curves, strain-rate sensitivity values have been extracted for each strain-rate and for the three temperatures. Figure 4 collects the strain-rate sensitivity versus strain-rate (in logarithmic scale). As the reader can see, the strain-rate sensitivity increases as the strain-rate decreases (with the exception of the case with the lowest temperature and the lowest strain-rate). It is also evident that for the temperatures used in the present analysis, the strain-rate sensitivity improves as the temperature increases (reaching values close to 0.7 at 830°C for the slowest strain-rate).

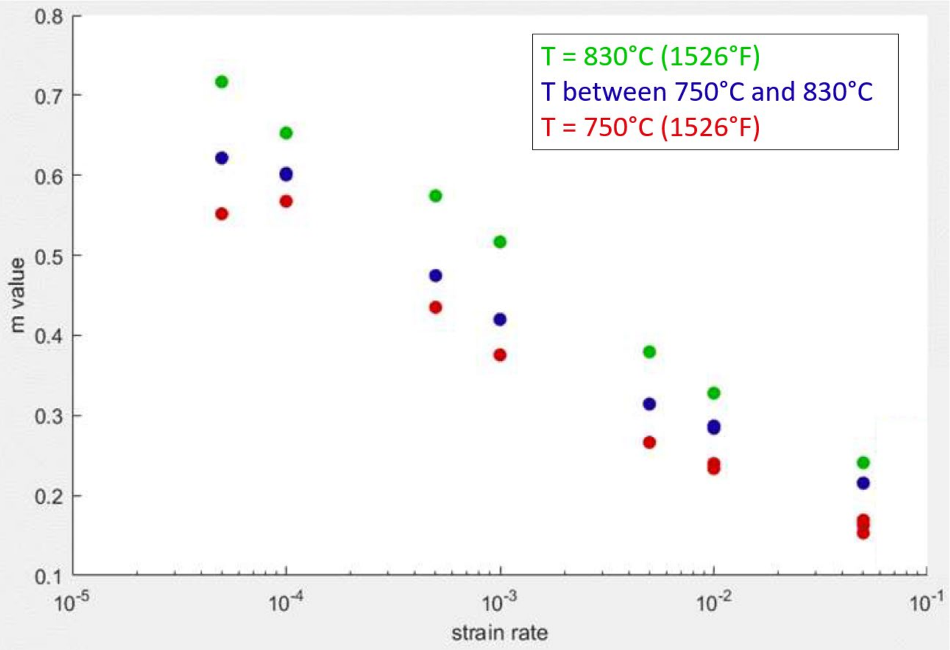

Figure 4. Comparison of strain-rate sensitivity values obtained for the three temperatures.

- **Microstructure Analysis:**

Analysis has been done on Ti-6Al-4V as-received material as well as on tested samples on both longitudinal (L) and transverse (T) directions of the samples via SEM microscopy. Several SEM micrographs were randomly obtained from different areas of each sample in order to have a representative analysis.

The analysis of the as-received material showed an average grain size of 2.4µm and a volumetric fraction of alpha and beta phases of 86% and 14%, respectively. The reader can see in Figure 5 an example of the as-received material where the globularity and the small grain size of the microstructure can be appreciated.

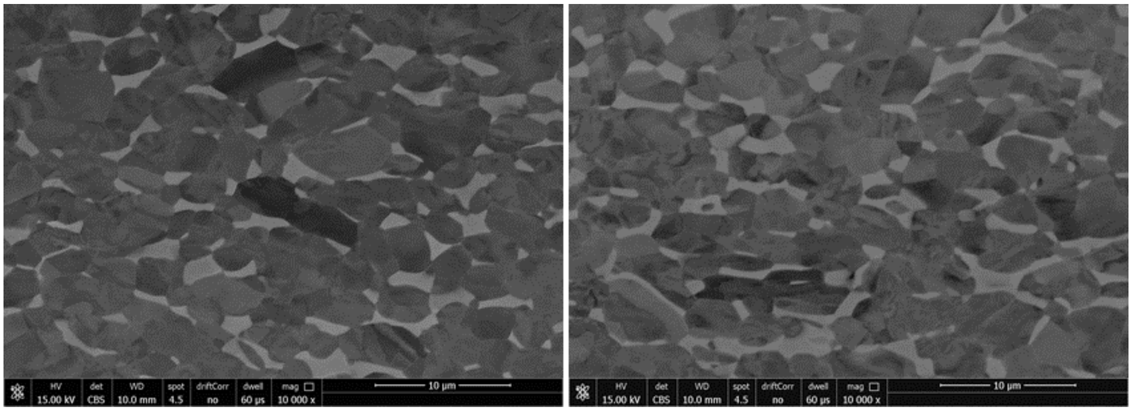

Figure 5. SEM micrographs of the Ti-6Al-4V as-received microstructure.

The SEM analysis of the tested samples confirmed the previously inferred concussions coming from the tensile tests. The average grain size increased for the slow strain-rates due the microstructure being at temperature during periods of time (the higher the temperature, the bigger the final grain size). On the contrary, for the fast strain-rates, dynamic recrystallization phenomena induced a decrease in the grain size. Figure 6 supports those conclusions for the strain-rates of $\dot{\varepsilon} = 5 \cdot 10^{-5}\mathrm{s}^{-1}$ and $\dot{\varepsilon} = 10^{-2}\mathrm{s}^{-1}$.

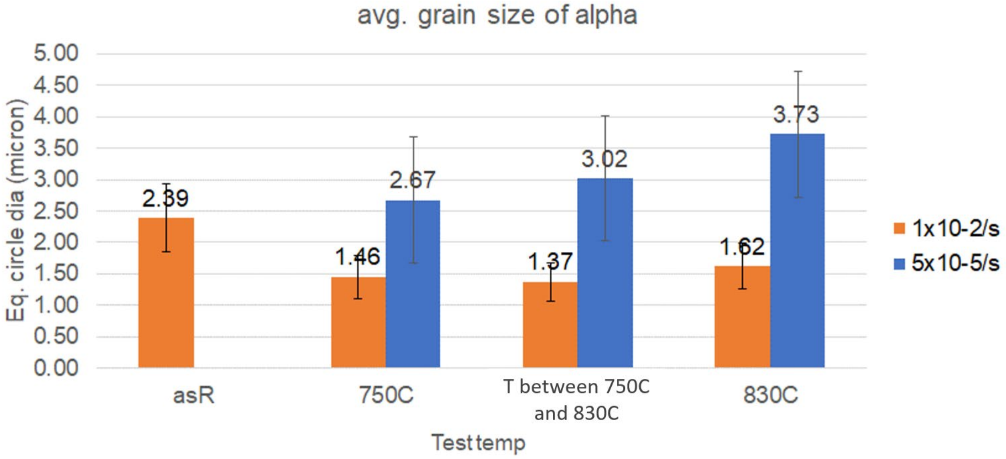

Figure 6. Average grain size of the tested samples at $5 \cdot 10^{-5} s^{-1}$ *and* $\dot{\varepsilon} = 10^{-2} s^{-1}$.

In regard with the volumetric fraction of alpha and beta, as expected, part of the alpha phase transforms into beta. The higher the temperature and the time of exposure, the higher the increase of the volumetric fraction of beta phase (see Figure 7). As temperatures are still far away from the beta-transus temperature, this transformation does not equalise the amount of both phases (this generally happening at temperatures close to 900ºC).

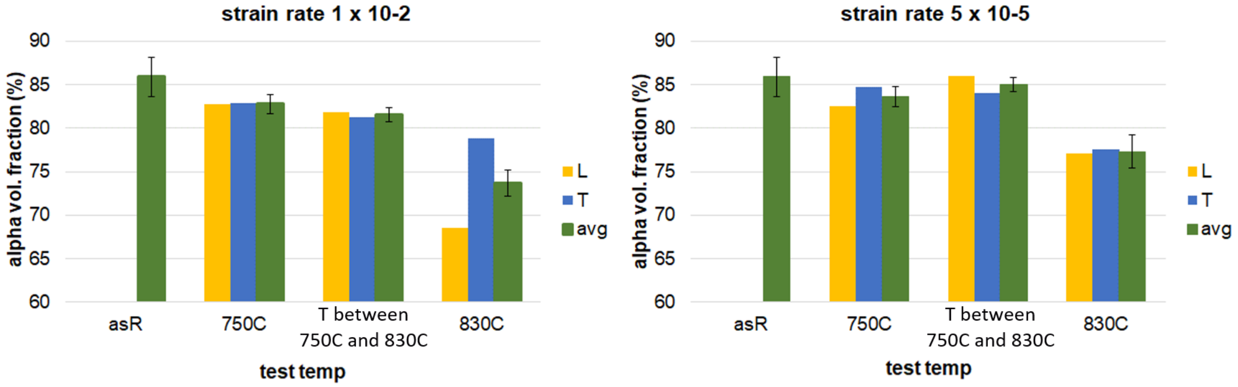

Figure 7. Alpha and beta volumetric fractions in the tested samples at $5 \cdot 10^{-5} s^{-1}$ *and* $\dot{\varepsilon} = 10^{-2} s^{-1}$.

Regarding cavitation, it can be stated that only extreme testing conditions (fast strain-rates above $\dot{\varepsilon} = 5 \cdot 10^{-3} \mathrm{s}^{-1}$) have developed some level of cavitation, always below the 0.3% volumetric fraction. Figure 8 shows two examples (at 750ºC and the temperature in the 750ºC - 830ºC range) of SEM micrographs with some level of cavitation for the fastest strain-rate $\dot{\varepsilon} = 5 \cdot 10^{-2} \mathrm{s}^{-1}$.

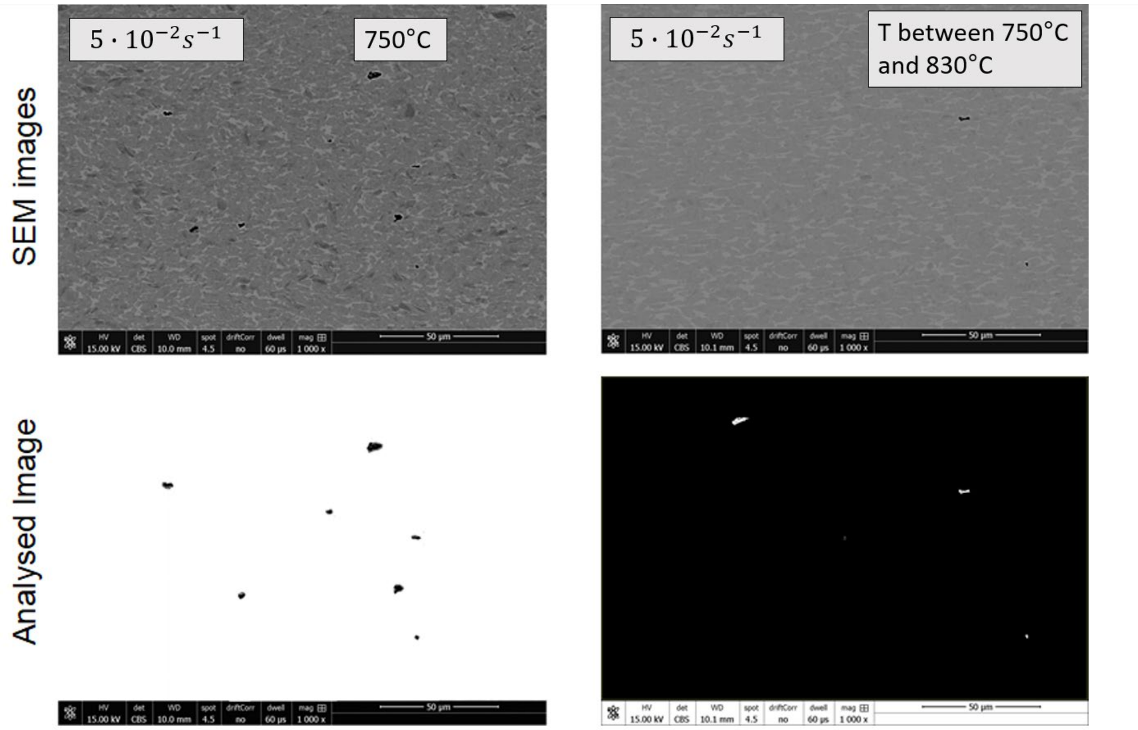

Figure 8. SEM micrographs showing negligible levels of cavitation at 750ºC and at the temperature in the 750ºC - 830ºC range (fastest strain-rate $5 \cdot 10^{-2} s^{-1}$*).*

A number of sub-micron grains are observed within the α colonies in the samples tested at fast strain-rates, regardless of the test temperature (see Figure 9). This phenomenon, which was not observed for the slow strain-rate tests, could have helped in the superplastic response of the material under the fast strain-rate conditions.

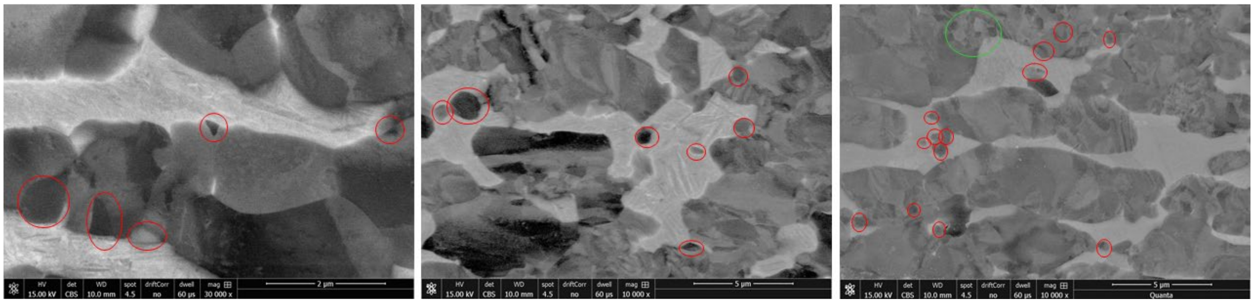

Figure 9. SEM micrographs showing sub-micron grains developed at $10^{-2} s^{-1}$ *for the three temperatures.*

- **Forming Parameters Definition:**

The tensile testing at temperature has allowed to define a material model for the Ti-6Al-4V material of analysis to be used in FE models of SPF. This modelling work will allow to define SPF forming conditions working at lower temperatures and shorter cycles. The limits in the temperature and time reduction would be established by the presence of cavitation in the material, which could compromise the final component in-service performance. Conditions for the presence of cavitation have been defined by the microstructural analysis of this work.

Summary

Tensile tests have been performed on Ti-6Al-4V material with an as-received average grain size of 2.4 µm. The tests were run at three different temperatures (750°C, 830°C and a third temperature in between those two) and seven strain-rates (ranging from $\dot{\varepsilon} = 5 \cdot 10^{-5}\ \mathrm{s}^{-1}$ to $\dot{\varepsilon} = 5 \cdot 10^{-2}\ \mathrm{s}^{-1}$). The tests were interrupted and specimens quenched when 0.5 true strain was achieved in order to subsequently analyse the microstructure.

For the lowest temperature tested (750°C), strain-rate sensitivities between 0.16 for the quickest test ($\dot{\varepsilon} = 5 \cdot 10^{-2}\ \mathrm{s}^{-1}$) and 0.57 for the slowest ($\dot{\varepsilon} = 5 \cdot 10^{-5}\ \mathrm{s}^{-1}$) were obtained. Those results were only slightly lower than the ones obtained for the highest temperature tested (830°C), which showed sensitivities between 0.23 for $\dot{\varepsilon} = 5 \cdot 10^{-2}\ \mathrm{s}^{-1}$ and 0.71 for $\dot{\varepsilon} = 5 \cdot 10^{-5}\ \mathrm{s}^{-1}$. The values of strain-rate sensitivity obtained and the absence of significant necking in almost all the samples denote an outstanding SPF behaviour of the material at such low temperatures and fast strain-rates.

The microstructure assessment showed that apart from the overall grain distribution, during the deformation process clusters of sub-micron size grains appeared in some of the grain boundaries. This remarkable behaviour is thought to have contributed to the grain boundary sliding and the enhancement of superplastic properties. Additionally, presence of cavitation has been analysed in order to define the forming conditions to be avoided. This material has shown cavitation presence only under the extreme testing conditions, and its volumetric fraction can be considered as negligible.

Acknowledgements

We would like to thank The Boeing Company for funding this research as part of project AFRC-DIRF-1172. In particular, we thank Larry Hefti, David Milliken and Nicola Zuelli for their contribution. We also thank the laboratory technicians at the Advanced Forming Research Centre (AFRC) for their contribution running the experimental tests presented in this study.

References

[1] J. Beal, R. Boyer, and D. Sanders, Forming of Titanium and Titanium Alloys. ASM Handbook, Volume 14B: Metalworking: Sheet Forming, S.L. Semiatin, pp. 656-669. https://doi.org/10.31399/asm.hb.v14b.a0005146

[2] L.D. Hefti, Innovations in the Superplastic Forming and Diffusion Bonded Process, J. Mater. Eng. and Perf. 17 (2008) 178-182. https://doi.org/10.1007/s11665-007-9178-0

[3] Y.G. Ko et al., Low-Temperature Superplasticity of Ultra-Fine-Grained Ti-6Al-4V Processed by Equal-Channel Angular Pressing, Metall. and Mater. Trans. A. 37A (2006) 381-391. https://doi.org/10.1007/s11661-006-0008-z

[4] H. Matsumoto et al., Ti-6Al-4V alloy with an ultrafine-grained microstructure exhibiting low-temperature-high-strain-rate superplasticity, Mater. Letters 98 (2013) 209-212. https://doi.org/10.1016/j.matlet.2013.02.033

[5] E. Alabort et al., Alloys-by-design: Application to titanium alloys for optimal superplasticity, Acta Mater. 178 (2019) 275-287. https://doi.org/10.1016/j.actamat.2019.07.026

[6] L. Despax et al., Influence of strain rate and temperature on the deformation mechanisms of a fine-grained Ti-6Al-4V alloy, Mater. Sci. and Eng. A. 790 (2020) 139718. https://doi.org/10.1016/j.msea.2020.139718

[7] J.S. Kim et al., Microstructural analysis on boundary sliding and its accommodation mode during superplastic deformation of Ti-6Al-4V alloy, Mater. Sci. and Eng. A. 263 (1999) 272-280. https://doi.org/10.1016/S0921-5093(98)01157-5