Short Communication

Low temperature superplasticity through grain refinement in Ti-6Al-4V by a novel route of quench-roll-recrystallise

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ARTICLE INFO

Article history:
Received 24 January 2015
Accepted 2 April 2015
Available online 13 May 2015

Keywords:
Low temperature superplasticity
Fine grain size
Water quenching
Annealing
Strain rate

ABSTRACT

A 'quench + roll + recrystallise' method was simulated through compression testing of initially ‘water quenched’ Ti-6Al-4V alloy at a temperature of 973 K and rolling strain-rate $10^3 \text{s}^{-1}$ in order to achieve superplasticity at lower temperature through grain refinement, with a view to increase die life. Subsequent annealing of wire-cut specimens of a rolled sheet at temperatures 1023, 1073, 1123, and 1173 K revealed that, the structures became finer and equi-axial in the range of 1-2 $\mu$m, when annealed at 1073 and 1123 K. In compliance to this behavior, a tensile sample from industrially 'quenched + rolled' sheet at 973 K could produce an elongation of 740% at a temperature of 1073 K under a strain-rate of $10^{-3} \text{s}^{-1}$. Significant elongation of 652% was obtained at further lower temperature of 1023 K under a strain-rate of $10^{-3} \text{s}^{-1}$. Quench-roll-recrystallise technique pushes down superplastic forming temperature to 1023 K.

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1. Introduction

Owing to its simplicity and high extendibility of difficult to manufacture materials, the superplastic forming (SPF) is increasingly used in aerospace industry to manufacture very complex geometries at much lower costs compared to conventional machining [1–3]. Materials such as titanium and aluminum alloys shall exhibit the phenomenon of superplasticity, when subjected to certain conditions of pressure, temperature and strain-rates [4,5]. The conditions are grain size $\leq 10 \mu$m, strain-rates $\leq 10^{-3} \text{s}^{-1}$ and temperatures $\geq 0.5 \Gamma_m$ where $\Gamma_m$ is the melting point of the material under investigation [6]. One of the pre-requisites for any material to be superplastic is that, it should be fine-grained and with equiaxed microstructure.

Ti-6Al-4V alloy is widely used as a structural material in the aerospace industry due to its low density, high specific strength, good corrosion resistance, excellent high temperature properties and high formability associated with
superplasticity [7] Because of these properties this alloy is preferred in aero engines, gas turbines, biomaterial industry, and other weight critical applications [8–11]. Deformation at high temperatures in different actual industrial processes such as hot forging or rolling is extensively used for both semi-finished and finished products of this alloy [12–14]. It is generally accepted that microstructure and variables such as grain-size morphology of secondary phase, strain-rate and temperature have great influence on the process [15,16]. The SPF of Ti-6Al-4V has been traditionally performed at 1198K. Though the SPF setup including dies is designed to withstand this high temperature, its life is limited and maintenance is high. A reduced working temperature in SPF by grain-refinement permits the use of low cost die materials such as die steel and stainless steel for forming. A low temperature SPF also reduces fuel consumption for heating the material and facilitates higher strength and toughness. Grain refinement is obtained by Severe Plastic Deformation (SPD). SPD can be used for the production of bulk UFG material with grain size less than 1 μm [17]. Grain refinement in Ti-6Al-4V alloy was achieved through High Pressure Torsion (HPT) by Misra et al. [18] and Sergueeva et al. [19], however, HPT has limited applications due to very small size of disk type samples, non-homogeneous and non-equilibrium microstructure. Grain refinement in Ti-6Al-4V was also achieved by Salishchev et al., by means of multi-step isothermal forging [20]. Equal-Channel Angular Pressing (ECAP) [21] has been suggested as an alternative SPD technique with scale-up potential. ECAP was studied on Ti-64Al-4V by Ko et al. [22], for low temperature superplastic forming, but the results show low percentage of elongation as the strain rate sensitivities have lower values. All the methods mentioned above need costly tooling to obtain ultra-fine grains. Therefore, the present investigation is aimed at developing an ultra-fine grained microstructure by an innovative method of ‘low temperature rolling of a water quenched structure’ thereby, examining the feasibility of lowering the conventional superplastic forming temperature. Recent works of Jun Liu et al., presents the forming technology of Ti-6Al-4V at low temperature 1073 K with short cycle time, which needs a die cavity with non-isothermal heating cartridge [23]. Superplasticity in an alloy can be evaluated generally by conducting tensile testing. However, superplasticity can be evaluated alternatively by localized tests like shear punch testing [24], indentation [25] and impression [26]. In the present study isothermal jump tests and total elongation tests are used to evaluate superplasticity in the material.

2. Experimental procedure

Mill-processed Ti-6Al-4V plate of 15 mm thick was water quenched after soaking at a temperature of 1273 K for ½ h. The cylindrical test samples of 10 mm diameter and 15 mm height have been wire cut from the plate.

The test samples were then compressed from 15 mm to 4 mm at a simulated rolling temperature of 973 K and at two different strain-rates of 10⁻³ s⁻¹ and 10⁻⁶ s⁻¹. These hot compression tests were conducted on a floor type compression-testing machine (DARTEC). The compression rams were made from special heat resistant Ni base superalloys. Thermocouples were placed very near to the sample to monitor the temperature. Strain rates were kept constant by a programmable controlled change of velocity of the ram with deformation. For high velocity test, a high-pressure accumulator was used to generate maximum velocity up to 3 m/s. Subsequently, the compressed samples (compressed at strain rate of 10⁻⁶ s⁻¹, which is the matching strain rate of rolling) were annealed for ½ h at temperatures 1023, 1073, 1123 and 1173 K to refine the grain structure and assessing the grain size.

Another portion of ‘water quenched Ti-6Al-4V’ plate was rolled at 973 K up to a thickness of 2 mm in one heat. The finish-rolling temperature was recorded as 673 K. The rolling was done by two high hot & cold reversible rolling mill at Defence Metallurgical Research Laboratory, Hyderabad, India. Samples from the rolled plate were annealed at the selected temperatures 1073 and 1123 K and the microstructures were observed.

Isothermal strain-rate jump tests (compression) were conducted at 1023, 1073, 1123, 1173 and 1198 K for determining the strain-rate sensitivity ‘m’ values to know the suitability of material for SPF. Tensile tests were carried out at selective lower temperatures of 1023 and 1073 K and at a moderate strain rate of 10⁻³ s⁻¹. The test samples used for tensile tests are of gauge length 10 mm and cross sectional area of 12 mm².

Fig. 1 – Experimental plan.
as the thickness of the sample is 2 mm and width at the neck is 6 mm. The detailed experimental plan is shown in Fig. 1.

3. Results and discussion

3.1. Hot compression of WQ Ti-6Al-4V

Initial grain size of as-received α+β microstructure of Ti-6Al-4V alloy is approximately 16 μm. The micrograph of Ti-6Al-4V plate water quenched from 1273 K consists of fine acicular ‘α’ needles, revealed that, the structure is not conducive to superplastic deformation due to non-equiaxed nature of the structure. In view of this, hot compression tests have been designed to simulate and optimize the deformation parameters for rolling.

The as-quenched needles broke into fine particles when the sample was compressed by 73% at a temperature of 973 K. The deformed structure was slowly evolved to a fine grained equiaxed structure of approximate grain size 3 μm when compressed at a temperature of 973 K and at low strain rate of 10^{-3} s^{-1}. However, the microstructure was not re-crystallized when compressed at a temperature of 973 K and at high strain-rate of compression of 10^3 s^{-1}. This strain-rate nearly matches with the commonly applied rolling strain-rates. The annealing was carried out for 1/2 h at 1023, 1073, 1123 and 1173 K, assuming that, the structure obtained at a faster strain-rate could be suitable for accomplishing finer grains by annealing at a suitable temperature.

The microstructure remained without re-crystallization when annealed at lower temperature 973 K. In contrast, the microstructure became more equi-axial at higher temperature 1173 K, but the grains started growing. In order to develop ultrafine grains, annealing at 1073 and 1123 K is done which seems to generate fine and equi-axial microstructures. The most suitable structure seems to appear on annealing at 1073 K, where, the grain size is in the range of 1–2 μm. SEM images of water quenched test specimens annealed at different temperatures revealed the grain sizes as mentioned in Table 1.

3.2. Low temperature rolling of water quenched Ti-6Al-4V

In view of the results from hot compression tests as described under Section 3.2, ‘water quenched Ti-6Al-4V plate’ was rolled at a temperature of 973 K and subsequently annealed at the selected temperatures 1073 and 1123 K. A comparison of the two structures was made in Fig. 2(a) and (b) respectively. It is interesting to note that, the microstructure is more refined (~1 μm) due to annealing at 1073 K.

![Fig. 2 – BSE images of ‘WQ + rolled’ (973 K) plate after annealing at (a) 1073 K and (b) 1123 K.](image)

<table>
<thead>
<tr>
<th>Annealed temperature, K</th>
<th>Grain-size (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1023</td>
<td>2</td>
</tr>
<tr>
<td>1073</td>
<td>1–2, equi-axial</td>
</tr>
<tr>
<td>1123</td>
<td>1–2, equi-axial with slight grain growth</td>
</tr>
<tr>
<td>1173</td>
<td>2–3, equi-axial with large grain growth</td>
</tr>
</tbody>
</table>

Table 1 – Grain size of the specimens at different temperatures.

![Fig. 3 – Stress Vs strain-rate and ‘m’ Vs strain-rate curves of ‘WQ + rolled’ Ti-6Al-4V Alloy at temperatures 1023, 1073, 1123, 1173 and 1198 K.](image)
Table 2 – Total % elongation at different temperatures and strain-rates.

<table>
<thead>
<tr>
<th>Temperature, K</th>
<th>Strain-rate (s⁻¹)</th>
<th>Elongation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1023</td>
<td>3 × 10⁻⁴</td>
<td>619</td>
</tr>
<tr>
<td>1023</td>
<td>10⁻³</td>
<td>652</td>
</tr>
<tr>
<td>1073</td>
<td>10⁻³</td>
<td>&gt;740</td>
</tr>
</tbody>
</table>

Fig. 4 – Total % elongation of ‘WQ+ rolled’ Ti-6Al-4V at temperatures 1023 (middle) and 1073 K (bottom) compared at a strain-rate of 10⁻³ s⁻¹.

3.3. Isothermal strain rate jump tests and total elongation tests

The strain-rate sensitivity ‘m’ was determined at temperatures of 1023, 1073, 1123, 1173 and 1198 K and plotted in Fig. 3. It was observed that, the maximum value of ‘m’ is attained at 1173 K. The humps are broad at temperatures 1023, 1073 and 1123 K, which is advantageous for superplastic deformation. Since the aim of the present investigation is to achieve superplasticity at low temperatures, the total elongation tests were confined to 1023 and 1073 K only. The grain growths are also expected to be low at these temperatures. The elongation at 1073 K under a moderate strain-rate (10⁻³ s⁻¹) was 740%, it reduced marginally (to 652%) at 1023 K as shown in Table 2 and Fig. 4.

4. Conclusions

This paper presents the possibility of reducing forming temperature of Ti-6Al-4V by grain refinement using a novel route of quench-roll-recrystallise. The conclusions drawn from this work are as follows:

1. As-received α+β microstructure of Ti-6Al-4V changes in to fine acicular ‘α’ needles after water quenching and these as-quenched needles broke into fine particles when the sample was compressed to 73% at 973 K.
2. The deformed structure after slower rate (10⁻³ s⁻¹) of hot compression gradually evolved to a fine grained equiaxed structure of approximate grain size 3 μm, when compressed to 73% at 973 K, deformed structure at faster strain-rate (10⁰ s⁻¹) of compression remained without being recrystallized.
3. Deformation at higher rate (10⁰ s⁻¹) of compression was observed to be suitable to accomplish further grain refinement by annealing at temperatures of 1073 and 1123 K.
4. Annealing of rolled plate at two different temperatures 1073 and 1123 K revealed that microstructure was more refined when annealed at 1073 K.
5. By quench-roll-recrystallise technique, an ultrafine equiaxed microstructure can be obtained in Ti-6Al-4V alloy.
6. Results of jump tests revealed that higher value of strain rate sensitivity was obtained at a temperature of 1123 K, whereas the humps were broad at lower temperatures, 1023, 1073 K, which could be advantageous for superplastic deformation.
7. Total elongation tests revealed that the elongation at 1073 K and at a strain-rate of 10⁻³ s⁻¹ was 740% and the significant elongation of 652% was observed at relatively lower temperature 1023 K and at a strain-rate of 10⁻³ s⁻¹, which showed the ultrafine grained microstructure developed by quench-roll-recrystallise technique pushed down the superplastic forming temperature to 1023 K.

Conflict of interest

The authors declare no conflicts of interest.

Acknowledgements

The authors are grateful to the Director, Defence Metallurgical Research Laboratory (DMRL), Hyderabad, for granting permission to use the facilities for conducting experiments. One of the authors Dr. J. Babu is thankful to Director, DMRL for allowing him to do Ph.D. work at DMRL.

REFERENCES