Effect of silicon on creep properties of titanium 6Al-2Sn-4Zr-2Mo alloy

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Abstract

The alloy Ti-6Al-2Sn-4Zr-2Mo is a titanium alloy for elevated temperatures often used in aerospace applications. Minor additions of silicon have proven to improve the creep resistance of this alloy. In this work, three different amounts of silicon (0.015, 0.07 and 0.162 wt% Si) were added to cast Ti-6242 and creep tests were performed at different temperatures and loads. Creep resistance increased significantly with silicon addition by means of silicide precipitation hindering dislocations movement. Silicon rich nanoparticles in the microstructure were detected and their effect on creep resistance was investigated. The instruments used in this study were light optical microscope (LOM) and scanning electron microscopy (SEM).

Precipitates larger than 150 nm were found to be located heterogeneously in the microstructure, whereas smaller precipitates, ranging from 20-100 nm were homogeneously spread in the material. All silicides were predominantly situated next to the beta-phase in the alloy, either at the prior-beta grain boundaries or the beta-phase in between the alpha-colonies.

1. Introduction

Ti-6Al-2Sn-4Zr-2Mo (Ti-6242) is used in gas turbine aero-engines because of its good creep resistance at elevated temperatures, up to 540 °C [1,2]. It has been observed that an addition of silicon to titanium alloys can improve creep resistance significantly [3,4]. Additions of 0.04-0.13 wt% silicon to Ti-6242 showed a large increase in creep resistance up to 538 °C [5]. The improvement in creep resistance is believed to be caused by solid solution strengthening and pinning of the dislocations by silicide precipitates [6]. An increase in the maximum service temperature of Ti-6242 results in this material being able to be used in warmer parts of the engine, thus reducing weight.

Different types of silicides have been identified in silicon containing high temperature alloys. In Ti-6Al-5Zr-0.5Mo-0.25Si (IMI 685), so-called silicide precipitate S1 was identified to be (TiZr)₅Si₃ whereas S2 was experimentally confirmed as (TiZr)₆Si₃ in several alloys, such as IMI829 (Ti-6Al-2Sn-4Zr-1Nb-1Mo-0.25Si) and IMI 684 [6]. Popov et al. showed that precipitation of S1 silicides takes place during aging and that they can become enriched in zirconium atoms and transform into S2 and also S3, with (ZrTi)₂Si composition [7].

Silicides have been detected by using transmission electron microscope (TEM) in several studies. For Ti-6Al-2.7Sn-6Zr-0.4Mo-0.455Si precipitates were observed at the interfaces between the lamellar alpha and the beta-phase [8]. The size of the precipitates was approximately 650 nm large. Studies of silicides with SEM are however scarce. The aim of this study is to characterise silicides with SEM to get a better overview of their size and position in the alloy's microstructure.

2. Material and Experiments

The material used in this study was a cast Ti-6Al-2Sn-4Zr-2Mo material. The as-cast microstructure is shown in Figure 1. The large grains, expected in a cast material, show a fully lamellar Widmanstätten microstructure with grain boundary alpha in the prior beta grain boundaries. Some areas exhibit a basket weave microstructure. The creep testing was performed at Westmoreland Mechanical Testing & Research, Inc. in Youngstown, USA in tensile mode under strain control in air following ASTM E139-11 until fracture or a failure criterion of 1500 hours. The stress rupture creep tests were performed under 450 and 500 MPa at 500 °C and under 575 and 625 MPa at 500 °C. Samples with three different silicon

contents; 0.015, 0.07 and 0.162 wt% Si were tested, adding a total of 24 tests. Figure 2 shows a creep test sample, gage section dimensions are 6.35 mm diameter and 27.7 mm length.

Figure 1. As-received microstructure of cast Ti-6242.

Figure 2. Photograph of a specimen for creep tests.

After the creep tests, 4 mm long samples were cut with a Discotom cutting machine and embedded in Bakelite by hot mounting. The cross-sections were polished with mechanical polishing up to grit 1200, followed by electropolishing in a LectroPol-5 equipment from Struers. A3 solution (methanol, 2-butoxyethanol and perchloric acid) was used, with a voltage of 20V, flow rate of 16 during 25 seconds. FEI Magellan 400 high resolution SEM was used to analyse the silicides. The instrument has five detectors; Everhart-Thornley detector (ETD), Through-lens detector (TLD), low voltage high contrast detector (vCD), SETM and EDS. The TLD detector was used for topography using secondary electrons. The vCD detector achieves strong contrast pictures using back-scattered electrons. The immersion mode was used for observation of silicides at high magnification.

3. Results and Discussion

Table 1 shows all the creep test results. The specimens with lowest amount of silicon (A-samples) broke at loading during testing at 450 °C for both loads (575 and 625 MPa). For the medium silicon amount samples (B-samples) the two samples tested at 450 °C with 625 MPa broke at loading. When decreasing the stress to 575 MPa the sample resisted until the maximum testing time (1500 hours) and the test was finished before rupture. The C-samples, which contain the highest amount of silicon (0.162 wt%), endured all testing conditions. The yield strength of the alloy Ti-6242 with 0.07-0.08%Si at 450 °C and at 500 °C is limited to 500 MPa and 520 MPa, respectively [9]. Therefore, the creep loads during testing were found to be too high for several of the samples, provoking a premature rupture upon loading. However, it is worth noticing that with a higher amount of silicon more samples were able to withstand the high stresses, meaning that the yield stress is increased for Si-rich Ti-6242 alloys.

Table 1. Creep test results for cast Ti-6242 with different Si-contents. A: 0.015 wt% Si, B: 0.07wt% Si and C: 0.162wt% Si.

Sample	Temp [°C]	Stress [MPa]	Total creep %	Rupture time [hrs]
1A	450	575		On loading
2A	450	575		On loading
3A	450	625		On loading
4A	450	625		On loading
5A	500	450	4.6125	806.2
6A	500	450	4.9325	795.2
7A	500	500	5.1319	232.9
8A	500	500	6.5639	116.2
9A	450	575		On loading
10A	450	625		On loading
11A	450	575		On loading
12A	450	625		On loading
1B	450	575	0.5916	1500
2B	450	575	0.7885	1500
3B	450	625		On loading
4B	450	625		On loading
5B	500	450	0.4179	625.2
6B	500	450	1.0635	715.0
7B	500	500	1.2002	313.1
8B	500	500	1.2841	396.4
1C	450	575	0.5650	1500
2C	450	575	5.5173	1500
3C	450	625	1.8724	672.1
4C	450	625	1.5013	487.1
5C	500	450	1.2313	1469.0
6C	500	450	0.7830	1500
7C	500	500	1.0240	225.9
8C	500	500	1.0428	471.5

The curves in Figure 3 show the creep curves (creep strain vs. time) for all samples that did not break on loading. At 500 °C the samples with lowest amount of silicon (A: 0.015 wt% Si) show a creep strain of around 5%.The creep rupture life is 100-200 hours for 500 MPa load and approximately 800 hours for the lower load (450 MPa). It is evident that the addition of silicon decreases the creep strain significantly, to below 1%. The longest creep rupture life is achieved for an addition of 0.162wt% Si, for the lower load (450 MPa) where the samples survived until 1500 hours, which was the failure criterion, or just before. For the higher load (500 MPa) the rupture time was between 400-500 hours. At 450 °C at 575 MPa load, all samples with an extra addition of silicon survived the whole testing time, 1500 hours and the creep strain is below 0.8 %. At the higher load, 625 MPa, only the samples with the highest amount of silicon addition (0.162 wt%) did not break on loading. The creep strain was between 1.5-2 % and the creep rupture life was 487 and 672 hours.

Figure 3. Creep curves for all samples that did not break upon loading. Specimens A: 0.015 wt% Si, B: 0.07 wt% Si and C: 0.162 wt% Si.

Figure 4 shows the creep strain in relation to silicon content divided into primary and secondary (or steady state) creep at the different loads and temperatures. It can be seen that the addition of small amounts of silicon dramatically reduces primary creep. The largest decrease is seen when adding 0.07 wt% Si and further silicon addition reduces gradually the primary creep. The effect of silicon addition on secondary creep is given with a logarithmic scale of the y-axis to separate the data points. Once a small amount of silicon is added, the steady state creep rate becomes more stable.

Figure 4. Primary and secondary (steady state) creep vs. amount of silicon addition at different loads and temperatures.

Regarding the silicides precipitation, these particles were found to belong to two populations. Larger silicide precipitates of sizes above 150 nm are inhomogeneously dispersed in the samples, localized in certain regions with high precipitate density. Apart from the large silicides, smaller silicides with sizes of 30-50 nm are evenly spread across the whole cross-section for all samples, see Figure 5 (left). Both types of silicides were found in the as cast material, after precipitation heat treatment. It is believed that the regions with larger precipitates originate from segregation during the casting process. The small silicides are positioned in the secondary alpha phase inside prior-beta grain boundaries (see Figure 5 (right)) and in the interfaces between alpha colonies. Silicon solubility is larger in the beta-phase than in the alpha-phase, and it is known that zirconium decreases the silicon solubility in titanium. During the transformation from beta to alpha, silicon will be rejected from the beta phase and precipitate in the form of silicides.

The precipitated large silicides show different morphologies: Squared, oval and rectangular, as shown in Figure 6. The small silicides are normally rod-like in shape.

Figure 5. As cast material with silicide precipitates (left, indicated with arrows) and a backscatter image of a rod-like silicide of 90 nm size inside secondary alpha in the beta phase (right).

Figure 6a) shows a large silicide that is square-shaped and b) shows an oval-shaped silicide with a thin oxide layer (appearing as “orange peel”) from the electropolishing process. It can also be noted that the silicide is harder to etch by the electropolishing, thereby sticking up in relation to the alloy surrounding it. The difference in morphology indicates that there are differences in the silicide composition. All large silicides are between 150 and 550 nm large and are usually oval. The corresponding EDS results verify that both particles are silicides, with the presence of zirconium. The silicides exact composition could not be determined, but it is suggested to be S2 ((Ti,Zr)₆Si₃) or S3 ((Zr,Ti)₂Si). As addition of even small amounts of beta stabilizing elements to titanium alloys prevents the formation of S1 silicide ((Ti,Zr)₅Si₃) [10], the alloy in this study is not believed to contain any S1-silicides given its Mo-content.

Figure 6. Different morphologies of large silicides, a) square (diagonal 200 nm) and b) oval-shaped (580 nm), with corresponding EDS data.

In terms of the location of the silicides, the large ones are located next to the beta-phase and have a clear preferential orientation with the alpha-phase. It has previously been observed that the long axis direction of the S2 silicide is oriented in 30° and 60° towards the direction of the alpha laths, see Figure 7. This is in accordance with previous research [8]. There was also a significant amount of silicides oriented in 40° to the alpha laths. This could be an indication of these silicides being of the S3 type instead of S2, needing further investigation. The smaller silicides were too small to observe their orientation through SEM imaging.

Figure 7. Orientation of the silicides in 30 and 60° towards the alpha laths.

4. Conclusions

Creep rupture time increases with silicon content, with the largest creep resistance improvement found during the secondary creep. Samples with no extra silicon addition had the highest primary creep strain, whereas there was no significant difference between medium and high silicon addition. No difference was observed between the different silicon additions for tertiary creep.

Silicon addition to alloy Ti-6242 leads to silicide precipitation. This work includes the observation of silicide precipitates in Ti-6242 through SEM investigations. This allows studying the distribution of these particles, their morphology and location in the microstructure.

Small (20-100 nm) silicides are evenly spread in the microstructure. Most of them are located in the secondary alpha-phase inside prior-beta grain boundaries and in the interfaces between alpha-colonies. The morphology of the small silicides is mainly rod-like.

Larger silicides (>150 nm) are inhomogeneously dispersed in the microstructure and are restrained to certain regions of the cross-sections. In these regions, there is a high-density of large silicides. These large particles are located next to the beta-phase and have a clear preferential orientation of 30° and 60° to the alpha-phase. The larger silicide morphology is mainly oval or squared.

Both the small and the large silicide exist in the as-cast material.

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