AGEING STUDY OF TIMETAL LCB TITANIUM ALLOY

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Abstract
Phase transformations in TIMETAL LCB and their influence on mechanical properties were studied in this research. The ageing response of this material was studied by low temperature ageing at 400 °C, 450 °C and 500 °C. The evolution of volume fractions of α, β and ω phases during ageing was studied by X-ray diffraction (XRD). Mechanical properties were investigated using microhardness measurements and tensile tests. It has been proved that metastable ω phase is formed during annealing at 400 °C and 450 °C. ω particles further transform to very fine precipitates of α phase when exposed to annealing for longer time periods. These fine precipitates significantly contribute to increase of microhardness and achieving high value of yield stress.

Keywords:
TIMETAL LCB, metastable β titanium alloys, phase transformations

1. INTRODUCTION
Titanium and its alloys are of great interest due to their outstanding properties such as high specific strength, ductility and excellent corrosion resistance. This makes them ideal for use in automotive and aerospace industry as well as for manufacturing biomedical devices and components in chemical processing equipment [1]. The only drawback, which hinders wider use in all above-mentioned fields, is relatively high cost of titanium. In the last few decades, the importance of β titanium alloys has increased significantly. Metastable β titanium alloys retain body-centered cubic structure upon quenching to room temperature, since the solute content of β stabilizing elements in these alloys is high enough to suppress the martensitic transformation to low temperature α phase with hexagonal close-packed symmetry [2]. Moreover, hexagonal metastable ω phase is observed in metastable β titanium alloys in the form of uniformly dispersed submicron particles which are coherent with the parent β phase. The particles of ω phase have a significant influence on mechanical properties of titanium alloys, as they increase specific strength and hardness but they can also embrittle the material [3]. More importantly, ω phase serves as a nucleation site for α phase. The presence of ω particles has a great impact on β -> α phase transformation during ageing and on the resulting microstructure of the material [4, 5, 6].

This present work was conducted on one of the metastable β titanium alloys, TIMETAL LCB (Ti-6.8Mo-4.5Fe-1.5Al in wt.%). The material (10 mm diameter bar) was first solution treated for 30 min at 820°C. Then, the bar was directly quenched to 700 °C and aged for 10 min. This ageing temperature lies in the α + β phase field, which caused the α phase to precipitate on the grain boundaries of the β grains. The grain boundary α volume fraction was 1.5 and its average thickness was 0.26 μm [7]. This initial treatment was terminated by water quenching. To analyze the heating response of the material, the resulting specimens were aged in neutral salt baths at 400 °C, 450 °C and 500 °C for ten different times (0.5 h, 1 h, 2 h, 4 h, 8 h, 16 h, 32 h, 64 h, 128 h and 256 h). The low-temperature heat treatments were terminated by water quenching to room temperature.

In this work, phase transformations during low-temperature ageing in one of the metastable β titanium alloy, TIMETAL LCB, were studied. The relationship between phase composition and mechanical properties (microhardness and tensile properties) was observed and characterized.
2. RESULTS AND DISCUSSION

2.1 X-ray analysis

The measurement of X-ray diffraction (XRD) was done using a powder diffractometer Bruker D8 Advance with focusing Bragg-Brentano geometry. The diffractometer was equipped with a rotational sample holder and a laboratory Cu tube operated at 40 keV and 40 mA. The axial divergence of the incident beam was controlled by Soller slits. The diffracted beam was detected by Soller-X energy dispersive point detector. Following aged samples were selected for X-ray diffraction measurements: 0.5 h, 1 h, 2 h, 4 h, 16 h, 64 h and 256 h aged at 400 °C; 0.5 h, 1 h, 2 h, 4 h, 8 h and 16 h aged at 450 °C and finally 0.5 h, 1 h and 2 h aged at 500 °C. Only three samples from the ageing treatment at 500 °C were measured, since no ω phase was detected at this temperature. The samples were rotated during signal acquisition in order to minimize the influence of texture in the material and to reduce poor statistics due to large grains. The measurements were done in 2θ angle range of 32 - 112°. The resulting line profiles (plots of intensity versus 2θ angle) were analyzed using MStruct, a free computer program for microstructure analysis from powder diffraction data [8].

Analysis of the X-Ray line profiles showed that the ω phase was present in all measured samples aged at 400 °C (samples aged up to 256 h). At 450 °C, ω particles were detected up to 2 h of ageing, then they either dissolved or were too small or overlapped by secondary α phase for the system to pick up. The X-Ray diffraction showed no evidence of ω phase in samples aged at the highest ageing temperature, 500 °C. Volume fractions of individual phases were determined by analysis of the X-Ray diffraction data. The dependence of volume fractions on ageing time is shown in Fig. 1 and Fig. 2 for ageing temperatures of 400 °C and 450 °C, respectively.

![Fig. 1 Volume fractions of α, β and ω phases in samples aged at 400 °C](image1)

![Fig. 2 Volume fractions of α, β and ω phases in samples aged at 450 °C](image2)

2.2 Microhardness measurement

The microhardness measurements were done using Vickers indentation hardness tester Leco M-400-A. A load of 500 g and an indentation time of 10 s was applied. Microhardness measurement was done on each aged sample as well as on the two initial states. In order to obtain good statistics, ten indentations spaced 0.5 mm apart were made on each sample. The resulting values of microhardness were then calculated automatically for individual indentations. Mean and standard deviation of microhardness were then computed from the ten indentations. The variations in the microhardness with ageing time for each ageing temperature are plotted in Fig. 3.
At the lowest ageing temperature (400 °C), initial rapid increase in microhardness was observed in the first 30 min of ageing. The increase corresponds to the growth of particles of the ω phase, which form strong and efficient obstacles for dislocation movement due to their small proportions, homogeneous distribution and relatively high volume fraction. With continued ageing the microhardness steadily increases and reaches its maximum approximately after 128 h of ageing treatment. The increasing microhardness indicates the presence of ω particles and evolution of fine microstructure resulting in age hardening of the material. At 450 °C, the microhardness reached its maximum much earlier. The highest hardness values are observed between 4 - 16 h of ageing. With increasing ageing time, the microhardness decreases slightly and finally drops visibly between 128 h and 256 h. The slow decrease suggests the dissolution of ω phase followed by coarsening of secondary α phase which was formed by ω → α transformation. The rapid decrease of microhardness in samples aged for the longest time (256 h) at 450 °C corresponds to overageing of the alloy. At the beginning of ageing at the highest ageing temperature (500 °C), a steep increase in microhardness is observed as well. The maximum of microhardness is reached already after 2 h. However, the comparison of microhardness variations for all ageing temperatures in Fig. 3 indicates that the maximum at the highest temperature is much lower than the respective maxima at lower ageing temperatures 400 °C and 450 °C. Overageing and corresponding decrease of hardness was observed for the longest ageing times.

2.3 Tensile testing

For tensile tests, four heat treated conditions were selected, in particular the specimens aged for 8 and 16 h at 450 °C and 500 °C. These aged conditions were selected based on the results of microhardness and X-ray measurements. These samples exhibited high values of microhardness, while the analysis of X-ray spectra indicated that there is either none or very little ω phase left in the material. The tensile tests were performed at room temperature with the strain rate of $10^{-4}$ s$^{-1}$. The results are summarized in Table 1. The table lists the values of ultimate tensile strength (UTS), elongation, yield stress and plastic strain for each
tested sample. As all measured samples exhibited very low ductility, no necking and subsequent decrease in engineering stress-strain curve was observed. Therefore, the ultimate tensile strength corresponds to the fracture stress. The elongation was measured by a video extensometer during sample loading. The values of engineering yield stress (σ_{0.2}) and plastic strain (ε_p) in Table 1 were automatically evaluated by the tensile machine software. Some of the samples fractured already during the elastic deformation, thus they had no plastic strain and the yield stress could not be calculated, which is indicated by N/A in the σ_{0.2} cells of Table 1.

<table>
<thead>
<tr>
<th>Temperature [°C]</th>
<th>Time [h]</th>
<th>UTS [MPa]</th>
<th>Elongation [%]</th>
<th>σ_{0.2}</th>
<th>ε_p</th>
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</thead>
<tbody>
<tr>
<td>450</td>
<td>8</td>
<td>1403</td>
<td>1.4</td>
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<tr>
<td></td>
<td>16</td>
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<td>1.2</td>
<td>N/A</td>
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<tr>
<td>500</td>
<td>8</td>
<td>1527</td>
<td>1.9</td>
<td>1507</td>
<td>0.32</td>
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<td>16</td>
<td>1507</td>
<td>1.9</td>
<td>1482</td>
<td>0.41</td>
</tr>
</tbody>
</table>

Table 1  Tensile properties - ultimate tensile strength (UTS), elongation, yield stress (σ_{0.2}) and plastic strain (ε_p)

The results of the tensile tests show that the material exhibits very high strength in all aged conditions. At 450 °C overall lower values of ultimate tensile strength (UTS) are observed compared to the samples aged at 500 °C. Furthermore, the material aged at 450 °C exhibited brittle behaviour and fractured already in the elastic region of the stress-strain curve as evidenced by Table 1. This fact could be attributed to some ω phase still present in the samples aged at 450 °C. Since no ω phase was observed in these heat treated conditions by X-ray diffraction, this would mean the ω particles are either too small or have too low volume fraction to be detected by the X-ray diffraction.

3. CONCLUSIONS
This research focused on the characterization of the relationships between the evolution of phase composition during ageing and mechanical properties of TIMETAL LCB titanium alloy. The main results of the present work can be summarized as follows:

- The analysis of X-ray line profiles showed the presence of ω phase in all studied samples aged at the lowest temperature (400 °C) up to the longest ageing times. A quick dissolution of ω particles at 450 °C was observed. No ω phase was evidenced in the material aged at 500 °C. Dissolution of ω phase particles back to β matrix that accompanies the early stages of α precipitation has been proven.

- Microhardness measurements were done to characterize the evolution of mechanical properties accompanying the ongoing phase transformations during isothermal annealing. The highest values of microhardness were reached in samples aged for 400 °C and 450 °C after 256 h and 8 h, respectively. The shift of the maximum of microhardness towards shorter ageing times with increasing ageing temperature indicated that the onset of the phase transformations (ω phase formation and α phase precipitation) leading to higher hardness of the material occurred sooner at higher ageing temperatures.

- Tensile tests performed on selected conditions indicated that the aged material reaches high values of strength, however, the ductility is very low.
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REFERENCES