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INFLUENCE OF HEAT TREATMENT ON PROPERTIES OF TI-NB ALLOYS

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Ti-Nb alloys are of a large potential for industrial, as well as biomedical utilisation. Oxygen content in the alloy is a parameter influencing its properties and cannot be neglected. The main focus of this paper is on observation of influence of annealing time on oxygen content in the structure of Ti – 20,7 (at.)% Nb alloy. Four groups of the samples were annealed at the temperature of 800 °C from 15 to 60 minutes with 15 min. step. As it was proven by the analysis, the oxygen content increased with increasing annealing time. Moreover, microhardness measurements showed increasing trend of HV microhardness value with increasing oxygen content.

Keywords: titanium, oxygen content, microhardness

INTRODUCTION

Oxidation behaviour of pure titanium is a complex issue and is influenced by several factors, such as temperature, oxygen partial pressure, water vapour content in the surrounding atmosphere etc. [1]. At increased temperatures (600 °C - 800 °C), titanium reacts with oxygen from the air and creates the TiO_2 oxide (rutile) and a coarse oxide layer on the sample surface develops [2].

Presence of oxygen in the structure influences mechanical properties of the alloy. In general, an alloy with higher oxygen content is more brittle but, at the same time, its hardness is increased [1]. Thickness of the layer, i.e. the depth from the surface of the sample in which the oxygen penetrated, can be determined with the help of microhardness measurements. Its thickness is dependent on annealing temperature and time.

Resistance of titanium to oxidation can be increased using alloying elements, such as Ta or Nb [1, 3]. These elements contribute to creation of nitride layer on the scale/surface interface and so that they contribute to decrease of oxygen solubility and further oxygen diffusion into the alloy. More resistant to oxidation than most of the other titanium alloys are titanium nitrides [4]. It is possible to enhance alloys with nitrogen by purpose at high temperatures and, therefore, to support creation of nitrides.

In some cases even oxygen can be added to an alloy by purpose. Oxygen content in specific Ti-Nb-O alloys is of an influence on damping characteristics of an alloy [5]; a shape memory effect was proven in Ti-Nb-O ternary alloys with oxygen content up to 1,5 % [6].

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Shape memory alloys are widely used in biomedicine as well. Between the most known shape memory alloys belong alloys based on Ti-Ni [7 - 9]. In biomedicine it is convenient to avoid using of nickel in the alloys from the reason of its negative influence to organisms [10]. Considering its high biocompatibility [10] and positive influence on mechanical properties, on decreasing of the young module above all [11], niobium is widely used as an alloying element to biomaterials. Above all, it is used as a replace for toxic vanadium [12].

The aim of this experiment is to observe the influence of annealing time on air to oxygen content in the structure of the Ti-22Nb alloy. The temperature of annealing was chosen to be 800 °C on the basis of the experiments previously made [1, 2], the annealing times were chosen as follows: 15 min, 30 min, 45 min and 60 min.

EXPERIMENT

For the purposes of this experiment the alloy Ti - 20,7 (at.) % Nb was used. The alloy was melted in plasma furnace from a pre-alloy and alloyed with pure Ti. The melted alloy was further swaged to a rod with diameter of 10 mm and then finally swaged to a wire with 3 mm diameter.

From those wires five groups of samples were created, the material was cut using electro-erosive cutting. Four groups of the samples were annealed on air at 800 °C and the annealing times were with a step of 15 minutes (15, 30, 45, 60 min), the last fifth group was left for comparison in the original condition. The groups of the samples and their annealing times are summarized in Table 1.

Each of the group contained six samples of which two were at a length of 6 mm for metallographic observation and the remaining four were at a length of 4 mm, those were supposed to be investigated for oxygen content.

Table 1 **Labels of the samples and annealing times**

Samples	A	B	C	D	E
Annealing time / min	-	15	30	45	60

At first, the samples were investigated using an EDX analysis on a QUANTA 450 FEG scanning electron microscope (SEM) to determine the real niobium content in the alloy.

For optical metallography analysis the samples were hot-pressed into conductive bakelite, then grinded on SiC abrasive papers of coarseness up to 1 500 and electrolytically polished. Finally , they were etched in the $HF:HNO_3:C_3H_6O_3$ (2:1:2) etchant for 5 s.

Microhardness of the samples was observed using a Tuture-Tech FM100 microhardness tester; Vickers microhardness was measured with 100 g load and 7 s load hold time. Oxygen content in the samples was measured using an Eltra ONH 2 000 device.

RESULTS

EDX analysis

The SEM-EDX analysis proved that the fi nal content of niobium in the alloy was 20,7 (at)%.

Optical metallography

Optical metallography was focused on observation of microstructure and the phases. The microstructure of sample A (in the original condition after swaging) consisted of β-phase, which corresponded with the preposition of dynamically recrystallized structure that occurred during swaging (Figure 1).

Heat treatment usually leads to structure softening and grain growth and the recrystallized structure disappears. In the microstructure of sample B (Figure 2) there is still the recrystallized structure and, moreover , fine precipitates start to develop. The β-phase is present in the grains; the precipitates are created by some kind of α (α', α") phase.

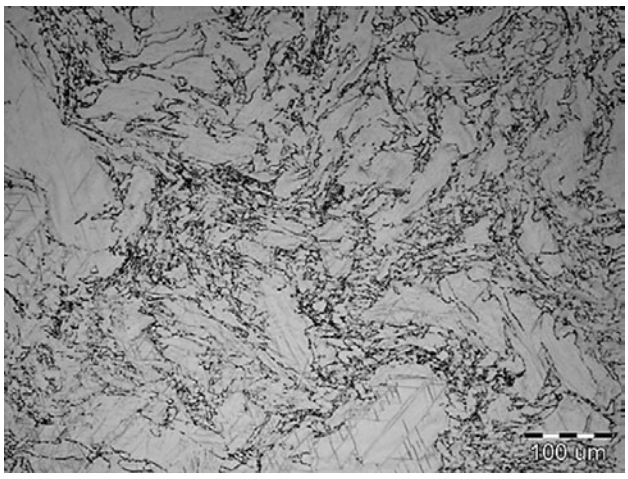

Figure 1 Sample A, microstructure after swaging

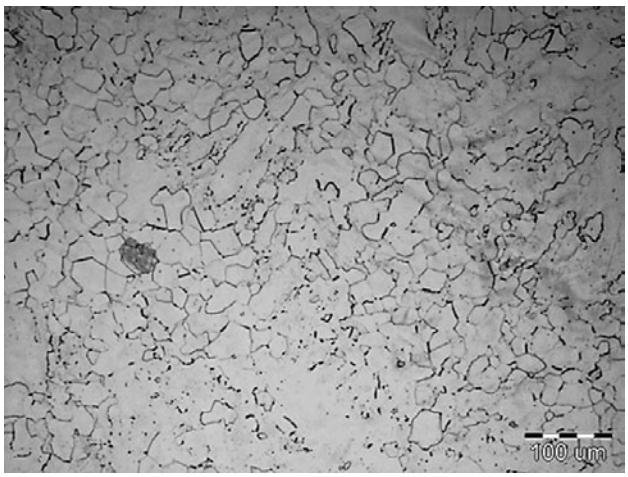

Figure 2 Sample B, microstructure after annealing 800 °C, 15 min

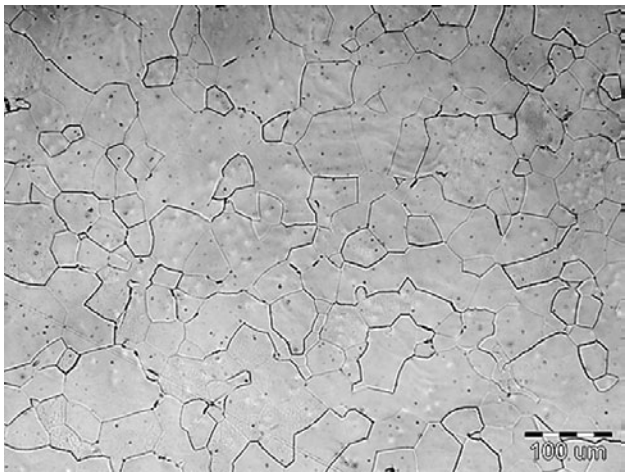

Figure 3 Sample E, microstructure after annealing 800 °C, 60 min

In Figure 3 there is the microstructure of sample E after annealing for 60 minutes. As it is evident from the figures, with increasing annealing time the grains grow and the precipitates become coarser.

Oxygen content

Observation of oxygen content in the groups of the samples has proven that annealing time is of an indispensable influence on the oxygen content in the material. In Table 2 there are the results of measurements of oxygen content for the A-E groups of samples summarised. As it is evident, the lowest oxygen content, 0,207 %, is in the sample group A, which had been left in the original condition. With increasing annealing time the oxygen content increases up to the value of 1,127 % for the sample group E.

Table 2 **Oxygen content in the samples**

Samples	A	B	C	D	E
Oxygen content / WT %	0,207	0,790	0,922	0,924	1,27

Microhardness measurement

Microhardness measurements were performed to observe the influence of annealing time, i.e. the oxygen content, on the microhardness of the sample. In Figure

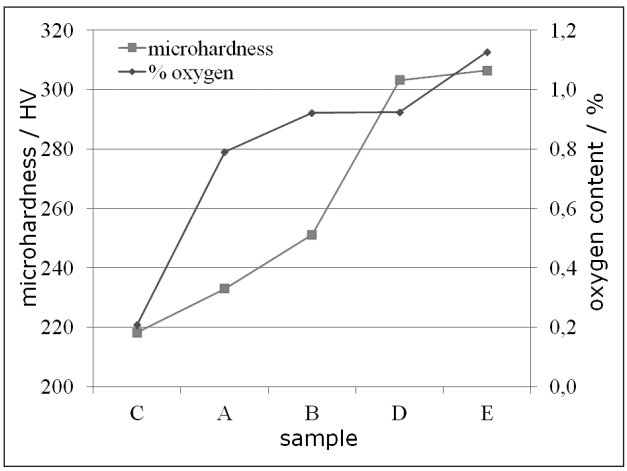

Figure 4 Graph of microhardness and oxygen content

4 there is a graph in which the results of microhardness measurements, as well as the results of measurements of oxygen content for the A-E groups of samples are summarised. In the figure it can be seen that microhardness of the samples increases with increasing oxygen content, therefore with increasing annealing time. Again, the microhardness is the lowest in the sample group A (218 HV), while it slowly increases with increasing annealing time up to the value of 306 HV for the sample group E.

DISCUSSION

Similar alloy had been observed before; the results of microhardness measurements of the sample group A are in accordance with the results obtained in a previous experiment [13]. After heat treatment increase in microhardness is evident; with increasing annealing time the microhardness and strength mechanical properties increase as well. The microhardness increase is caused by creation of precipitates in the alloy during annealing [14, 15]. To determine the type of the α-phase precipitates exactly X-ray analysis would be needed.There is a supposition of increase of mechanical properties (ultimate tensile strength) with microhardness increase according to experiments previously made [16], where UTS increase was proven by the results.Annealing time and temperature are of high influence on the properties of titanium-based shape memory alloys as well [17].

As it is evident from the results summarised inTable 2 and from the graph in Fig. 4, the oxygen content in the material increases quickly after annealing start and further oxygen dif fusion slows down. This is in accordance with results achieved in the paper [2], where the explanation of dif fusion deceleration is in creation of oxide layer on the sample surface.

CONCLUSION

Four groups of samples of a titanium alloy with alloying niobium content of 20,7 (at)% were annealed on air at the temperature of 800 °C for 15, 30, 45 and 60 minutes and after alloying they were subjected to oxygen content observation, microhardness measurements and OM.

The results proved that annealing time is of an indispensable influence on the oxygen content – it increased from 0,207 % in the initial condition after forming (sample group A) up to 1,127 % after annealing for 60 minutes (sample group E). Together with increasing oxygen content microhardness increased from 218 HV at the A sample to 306 HV at the E sample group.

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Note: The responsible for English language is the lecturer from TU Ostrava, Ostrava, Czech Republic