MICROSTRUCTURE PROPERTIES OF TiNb ALLOYS PREPARED BY PLASMA METALLURGY

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Abstract

The TiNb-based alloys with nominal composition of Ti-22 at.% Nb and Ti-25 at.% Nb were prepared from Ti-45 wt.% Nb master alloy and Ti pieces (3N8) by means of plasma melting. The samples for metallographic observation were annealed for 12 hours at 1100°C in flowing Ar gas, water quenched and then electrolytic polished and etched. Metallographic observation together with microhardness measurement of both alloy compositions in plasma melted and annealed conditions confirmed the great influence of the heat treatment on the microstructure and mechanical properties. The results for the annealed and quenched samples have shown the significant increase in values for both alloy compositions.

Key words

shape memory alloys, TiNb alloys, plasma metallurgy, intermetallics, microhardness, microstructure

Introduction

Binary TiNb-based alloys with shape memory effect are investigated as Ni-free suitable substitution of TiNi-based alloys in biocompatible applications. It has been confirmed [1,2,3] that TiNb alloys exhibited shape memory effect and superelastic behavior at room temperature in wide range of Nb content (16.7 - 50 wt.% Nb) that is related to stress induced martensite. Mechanical and physical properties, as well as transformation characteristics are strongly influenced by concentration of Ti and Nb or eventually by other alloying elements (such as Mo, W, Fe, Si, B, Ta, Zr, Sn, V). It is well known that Nb is β stabilizer so that the TiNb alloys fall into β alloys, eventually into β rich α+ β alloys.

The aim of this work is the analysis of microstructure and phases formed after plasma melting and annealing of Ti-22 at.% Nb and Ti-25 at.% Nb alloys.

Experimental

The Ti-22Nb and Ti-25Nb (in at.%) were prepared from master alloy TiNb (50/45 wt.%) and elemental Ti (3N8) pieces. The ingots were melted and three times remelted using Ar-operated plasma torch in a water cooled copper crucible with feed rate of 1cm per minute. Fig. 1 shows the composition and melting temperature of master alloys as well as the ones of prepared alloys. The plasma melted samples were heat treated in high temperature furnace Linn HT 1800 at 1100°C for 12 hour in flowing Ar gas and water quenched from 700 °C.

Microstructure evaluation was performed on electrolytic polished and etched specimens (Kroll's solution: 8 HF: 15 HNO_3: 77 H_2O) by optical microscope OLYMPUS DP GX51. Microhardness measurement across the diameter of as-plasma melted and annealed specimens was carried out by means the LECO LM-100 instrument with load of 0.2 kg and indentation step of 1 mm.

The chemical composition of phases observed in the microstructure was determined by scanning electron microscope (SEM) JEOL JSM - 6490LV equipped with EDS INCA X - ACT probe.

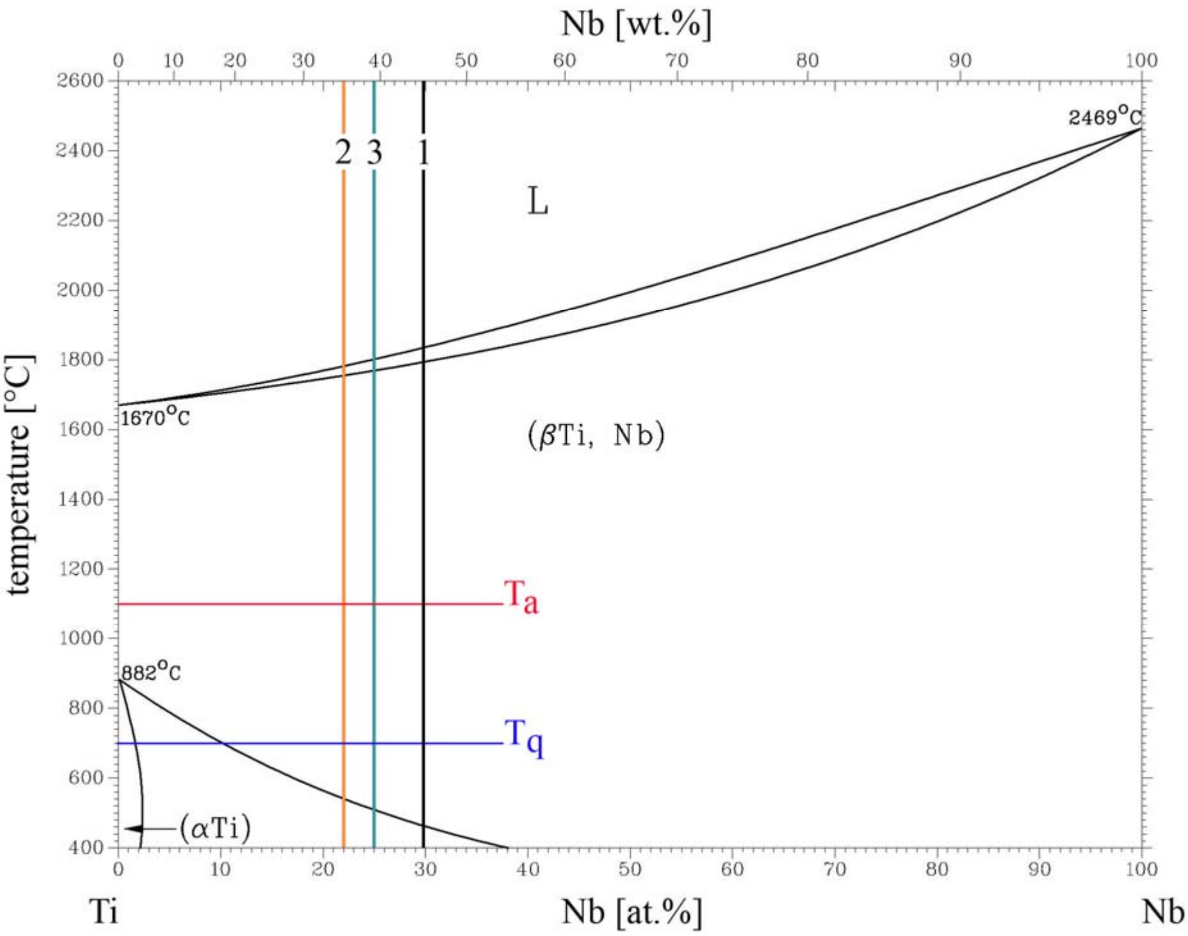

Fig. 1 *Binary diagram of the Ti – Nb system [1]: T_a-temperature of annealing, T_q-temperature of quenching. 1- master alloy TiNb, 2- Ti-22Nb alloy, 3- Ti-25Nb alloy*

Measurement and results

Fig.2 shows dendritic microstructure of large grains in as-plasma melted Ti-22Nb samples. Two phase lamellar structure in dendrites was observed at higher magnification (Fig. 4). The results of microanalysis summarised in Table 1 confirmed β phase in dendritic microstructure but fine laths formed during cooling of alloy could not be analysed because very little size.

The microstructure of the sample after heat treatment and detail at higher magnification are given in Fig. 3. The fully lamellar microstructure is formed of α and β phases that was confirmed by microanalysis (Table 2). The Nb contents of 5.55 at.% and 47.19 at.% correspond to α and β, respectively (Fig. 5). In accordance with binary diagram (Fig.1) water quenching of specimens was realised from lower temperature that was thought, so the microstructure below β transus was retained.

The microstructure of Ti-25Nb after plasma melting was also dendritic (Fig. 6, detailed in Fig.8). The results of microanalysis given in Table 3 confirmed the presence of β phase. After heat treatment, lamellar microstructure was formed. Fig. 7 shows coarse colonies of laths and grains with finer colonies that are detailed in Fig. 9. Therefore, the same effect of cooling below β transus is observed as the results of Nb content analysis confirmed (Table 4).

The microhardness results of Ti-22Nb and Ti-25Nb in as-plasma melted and annealed conditions are summarised and plotted in Figures 10 and 11, respectively. From the difference of mean values in plasma melted (253 HV and 212 HV, respectively) and annealed (725 HV and 619 HV, respectively) specimens, the great influence of the heat treatment on the microstructure and mechanical properties was found. Further detailed microstructure and phase analysis is needed for explanation of microhardness increase after annealing.

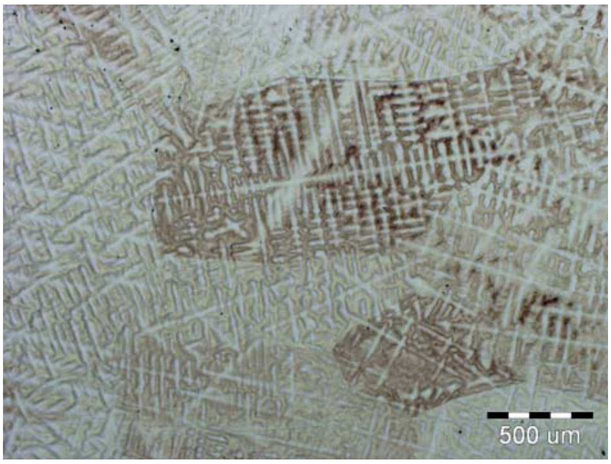

Fig. 2 *Dendritic microstructure of Ti-22Nb after plasma melting*

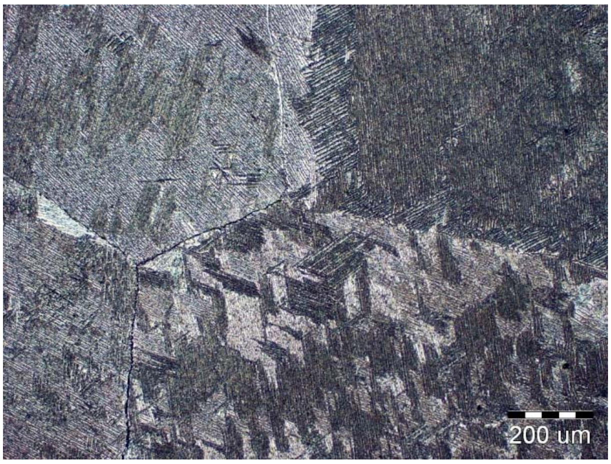

Fig. 3 *Lamellar (α+β) microstructure of Ti-22Nb after annealing*

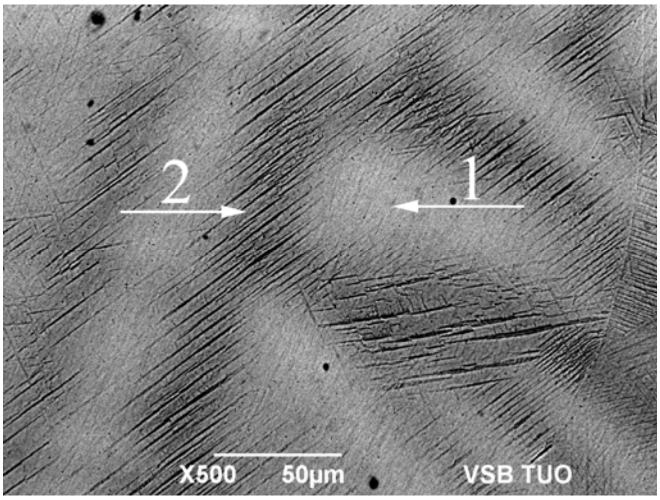

Fig. 4 *SEM micrograph of as-plasma melted Ti-22Nb alloy, detail of fig. 2*

MICROANALYSIS RESULTS OF Ti-22Nb AFTER PLASMA MELTING

Table 1

element **microstructure**	**Ti** [at.%]	**Nb** [at.%]
white dendrite (1)	78.81	21.19
dark interdendritic space (2)	76.46	23.54
general analysis of alloy	78.43	21.57

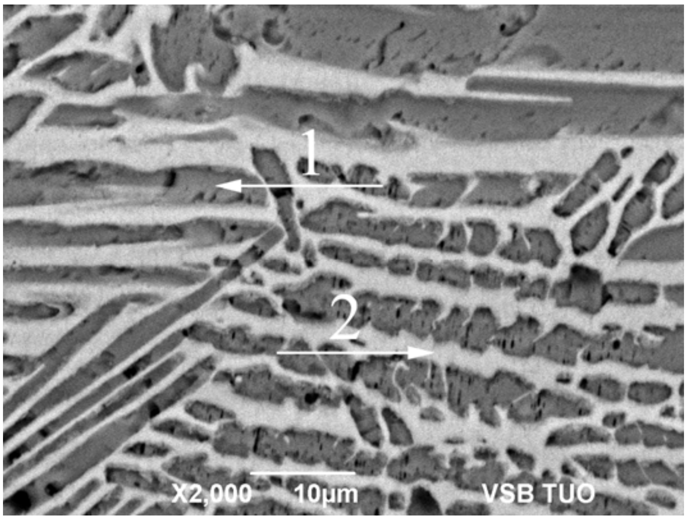

Fig. 5 *SEM micrograph of annealed Ti-22Nb, detail of fig. 3*

MICROANALYSIS RESULTS OF Ti-22Nb AFTER ANNEALING

Table 2

element **microstructure**	**Ti** [at.%]	**Nb** [at.%]
dark lath (1)	94.45	5.55
bright lath (2)	52.81	47.19

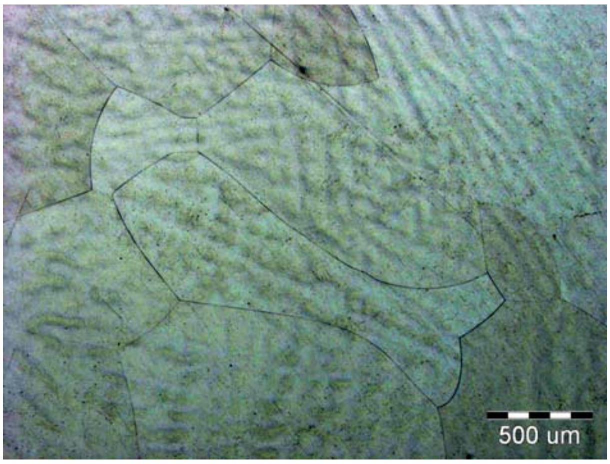

Fig. 6 *Dendritic microstructure of Ti-25Nb after plasma melting*

Fig. 7 *Lamellar ($\alpha+\beta$) microstructure of Ti-25Nb after annealing*

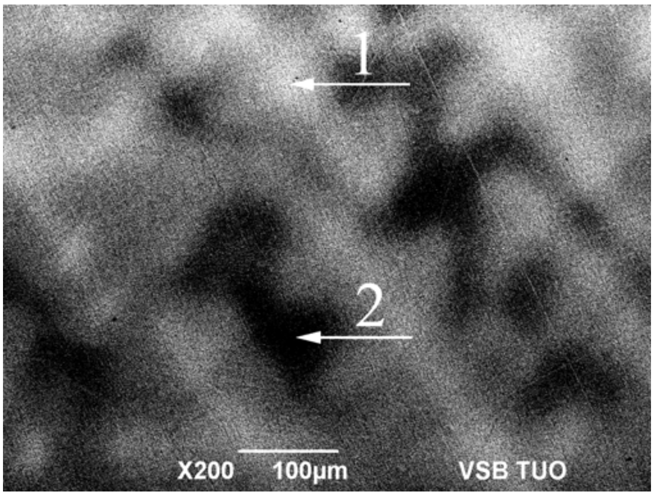

Fig. 8 *SEM micrograph of as-plasma melted Ti-25Nb alloy, detail of fig. 6*

MICROANALYSIS RESULTS OF Ti-25Nb PLASMA MELTED

Table 3

element microstructure	**Ti** [at.%]	**Nb** [at.%]
white dendrite (1)	71.04	28.96
dark interdendritic space (2)	75.21	24.79
general analysis of alloy	72.87	27.13

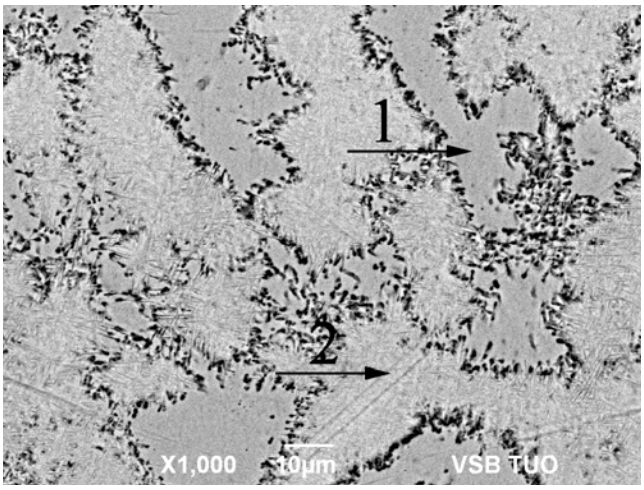

Fig. 9 *SEM micrograph of annealed Ti-25Nb alloy, detail of fig. 7*

MICROANALYSIS RESULTS OF Ti-25Nb AFTER ANNEALING

Table 4

element lamellar microstructure	**Ti** [at.%]	**Nb** [at.%]
fine grain (1)	73.23	26.77
large grain (2)	58.21	41.79

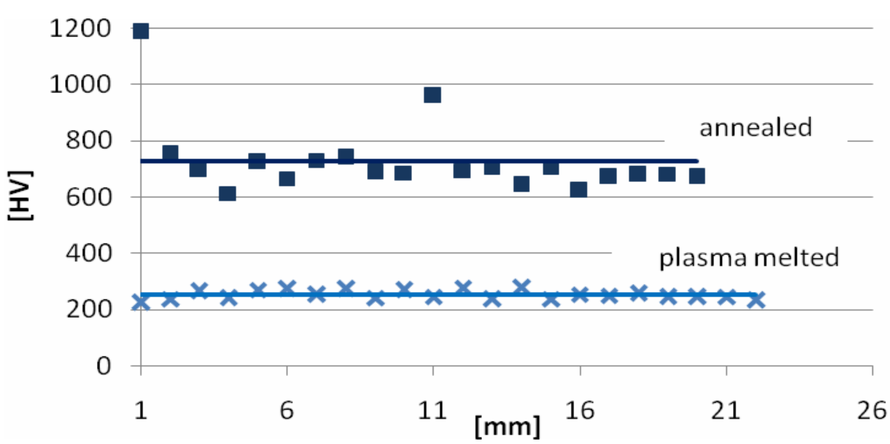

Fig. 10 *Comparison of microhardness values of Ti-22Nb in plasma melted and annealed conditions with mean values 253 HV and 725 HV, respectively*

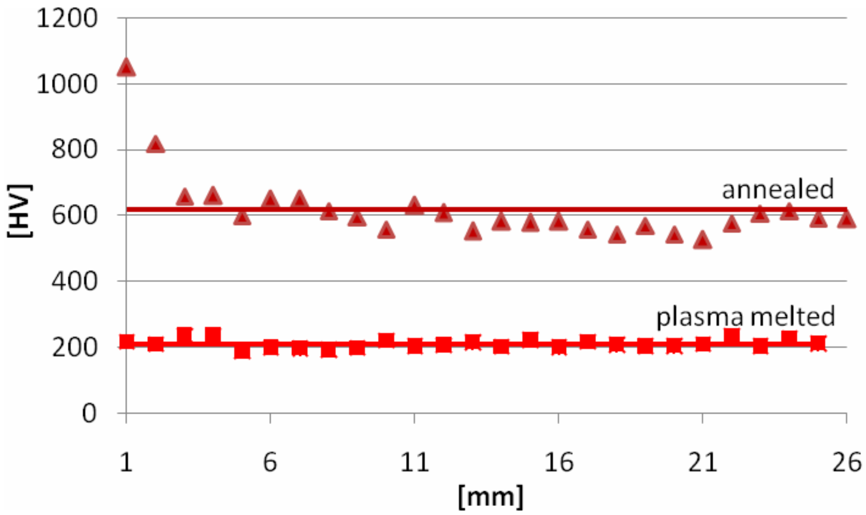

Fig. 11 *Comparison of microhardness values of Ti-25Nb in plasma melted and annealed conditions with mean values 619 HV and 212 HV, respectively*

Conclusions

The metallographic observation and microstructure analysis has led to the following conclusions:

1. The Ti-22Nb and Ti-25Nb alloys were prepared with 21.57 at.% and 27.13 at.% of Nb content, respectively.
2. The microstructure after plasma melting as well as after annealing was formed of β phase with coarse or fine laths of α phase.
3. The increase of the Nb content in Ti-25Nb alloys decreases the microhardness of both as-melted and annealed conditions by reason of microstructure features.
4. Microhardness measurement of both alloy compositions confirmed the significant effect of heat treatment on increase of the values.

The explanation of effect of Nb content and heat treatment need further phase analysis that will be the subject of future research work.

Acknowledgements

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