Hydrogen effect on compression mechanical properties of TiNb alloys at elevated temperatures

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Abstract. The study of this work was focused on the hydrogen effect on hot deformation behavior of hydrogen charged TiNb based alloys at three temperatures in comparison with non-charged specimens. The Ti24Nb and Ti26Nb (at.%) alloys were heat treated by three step regime in argon or hydrogen atmospheres. The hot compression tests were performed on a Gleeble 3800 machine at 800, 750 and 700°C with compression strain of $5x10^{-3}$ s^{-1} and deformation degree of 50 %. The microstructure resulting from heat treatment as well as from isothermal compression test was analyzed using optical and scanning electron microscopies. Measurement of microhardness revealed that higher microhardness values for Ti24Nb after thermo-hydrogen treatment corresponded to fine grained microstructure and strengthening by grain boundaries. Hydrogen contents determined using LECO RH600 analyzer showed that Ti26Nb contained higher amount of hydrogen due to the stabilization of beta phase by higher Nb concentration. Based on the hot compression test results, the plasticity at elevated temperatures was evaluated. The evolution of uniaxial compressive test curves showed the stabilization of flow stress due to the hydrogen for high deformations at lower temperatures. The differences in hot compression behavior for both niobium contents on the one hand and for hydrogen charged and non-charged specimens on the other hand were observed.

1 Introduction

The mechanical properties of most engineering metallic materials and especially of titanium alloys are generally deteriorated by hydrogen presence. The brittleness of alloys induced by the hydrogen is observed at room temperature as well as at elevated temperatures. Nevertheless, in some cases the hydrogen can act as beneficial element in improving particular properties. Titanium alloys show high strength-to-weight ratio, good corrosion resistance and biocompatibility, so they are of particular interest for use as biocompatible implants in orthopedics and traumatology [1].

Generally, workability of titanium alloys at room temperature is difficult and forming must be performed at elevated temperature. As it was discussed in [2-4], it is possible to improve hot workability of titanium and titanium alpha-beta alloys by using of hydrogen as a temporary alloying element in special heat treatment technology, so-called the thermo-hydrogen treatment (THT). The temporary hydrogen alloying allows hot working of alpha-beta titanium alloys at lowers temperatures with lower flow stresses. The microstructure refinement resulting from the THT technology allows improving fatigue and strength properties.

The study of this work was focused on the hydrogen effect on hot deformation behavior of hydrogen charged TiNb based alloys at three temperatures in comparison with non-charged specimens. The Ti24Nb and Ti26Nb (at.%) alloys that have predominantly beta phase structure were heat treated by three step regime in argon or hydrogen atmospheres.

2 Experimental

Experimental specimens of Ti-24Nb and Ti26Nb (at.%) alloys that nominal compositions are plotted in figure 1 were prepared of forged bars. Cylindrical samples of 10 mm in diameter and 12 mm in height were machined and heat treated in high temperature hydrogen charging furnace. Three steps thermo-hydrogen treatment according to [5, 6] and described in figure 2 was realized on four specimens in flowing hydrogen (5N).

Considering higher solubility of hydrogen [7] in predominantly beta phase TiNb, the time of hydrogenation steps was modified:

(1) heating to 600 °C and holding for 30 minutes,
(2) increasing temperature to 850 °C and holding for 18 minutes,
(3) cooling to 590 °C and holding for 60 minutes,
(4) hydrogen-cooling to 300 °C and then argon-cooling to room temperature.

In order to compare the high temperature deformation behavior with and without hydrogen in the alloy structure the same procedure of heat treatment was performed on four samples in flowing argon (5N).

The hydrogen charged and non-charged specimens were submitted to isothermal compression at constant strain rate of $5x10^{-3}$ s^{-1} on a Gleeble-3800 machine. The isothermal compression tests were performed at three selected temperatures: 800, 750 and 700 °C. Prior to compression reaching 50.0 % of maximum height reduction, the specimens were held for 3 min at the nominal deformation temperature and after the test, the samples were air-cooled to the room temperature. During isothermal compression, the flow stress was recorded as a function of strain.

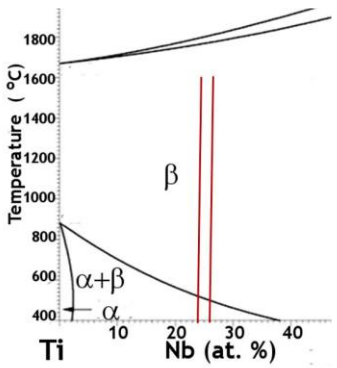

Figure 1. Binary Ti-Nb diagram for Ti rich alloys: ocurrence of alpha and beta phases with increasing Nb content .

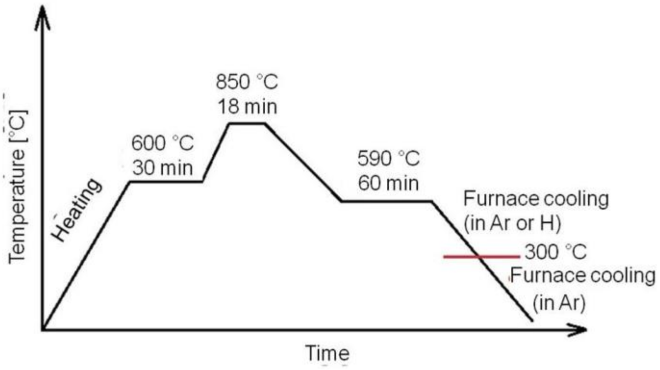

Figure 2. Three steps regime of thermo-hydrogen treatment (THT) before subsequent isothermal compression test.

The metallographic study was realized on the specimens ground and polished by conventional methods and etched using Kroll's reagent (2 HF : 4 HNO_3 : 80 H_2O). The amount of hydrogen in the specimens was determined before and after the compression test by hydrogen determinator LECO RH 600.

3 Results and discussion

The macroscopic view of specimens after the compression tests with an amount of reduction in height of 50 % is illustrated in figure 3. The surfaces of hydrogen charged specimens were cracked (red boxes in figure 3 b) while the non-charged specimens were cracked rather rarely on the surface.

The flow stresses recorded for both alloys are depending on the temperature as well as on the niobium content. It is clear that niobium is beta stabilizer, so for the Ti26Nb the flow stresses are lower then for Ti24Nb due to the higher content of more ductile beta phase. Comparing the deformation behavior at three different temperatures of hydrogen non-charged and charged specimens, it is clear that with increasing temperature the flow stress is decreasing and by reason of this the hot forming is improving for both composition and both heat treated stages.

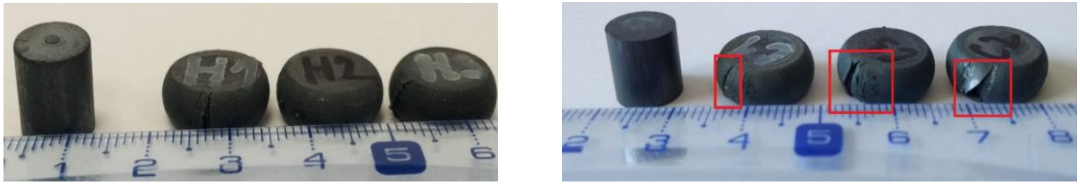

Figure 3. Specimens of TiNb alloys after the isothermal compression test: a) hydrogen non-charged, b) hydrogen charged (cracking in red boxes).

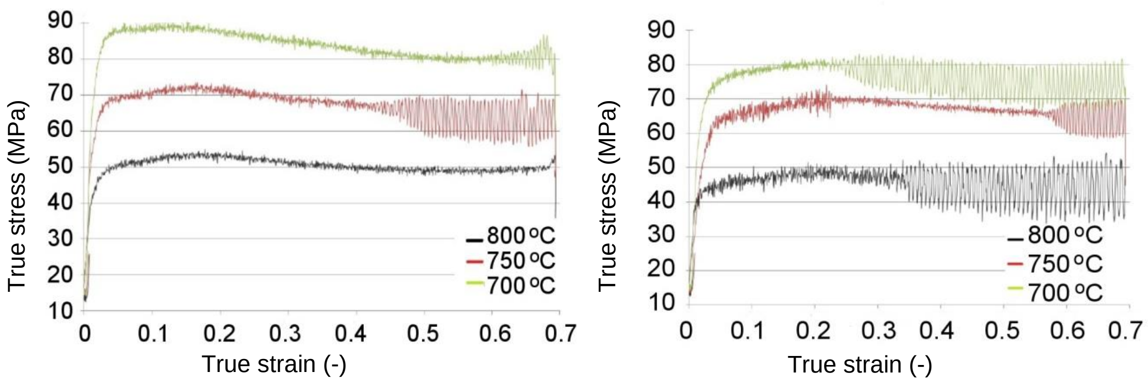

Figure 4. True stress - true strain curves for hydrogen non-charged: a) Ti24Nb; b) Ti26Nb.

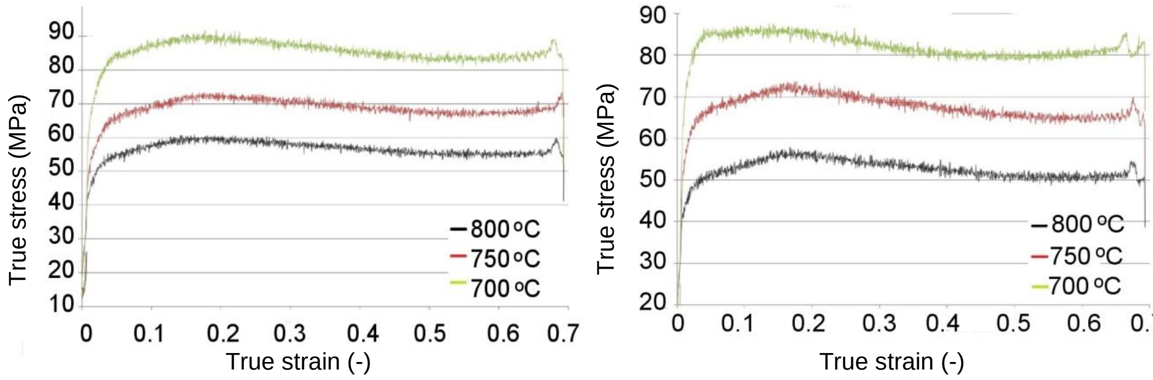

Figure 5. True stress - true strain curves for hydrogen charged: a) Ti24Nb; b) Ti26Nb.

In the case of the hydrogen non-charged specimens unusual behavior was registred during compression tests (figure 4). Large instabilities of flow stress are more evident for the specimens with higher niobium content and could be related with lower rate of dynamic recrystallization. The hydrogen effect on development of compression test was inconsistent for both alloy compositions.

Unlike the Ti24Nb alloy the hydrogen content in the Ti26Nb specimens increased the flow stress, but on the other side the flow stress instabilities were suppressed for both alloys. Indeed, the evolution of uniaxial compressive test curves (figure 5) showed the stabilization of flow stress due to the hydrogen for high deformations at all three temperatures and for both compositions of TiNb alloy. The differences in hot compression behavior for both niobium contents on the one hand and for hydrogen charged and non-charged stages on the other hand are related with increasing niobium and hydrogen amounts effecting strengthening by solid solution [8].
The hydrogen amounts measured after the THT and after the isothermal compression test (CT) are represented in figure 6. The highest amount (2572 wt. ppm) was determined in Ti26Nb after the THT that corresponds with higher hydrogen solubility in beta phase. Nevertheless, the decrease of hydrogen content after the CT at 800 °C was more important just for the alloys with more niobium. Unfortunately, the hydrogen promoted cracking on the surfaces of compression tested specimens.

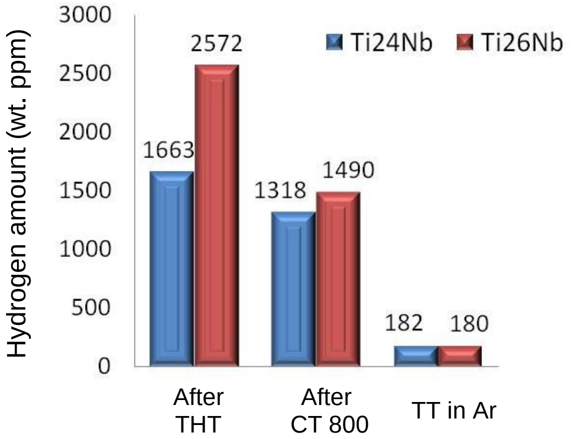

Figure 6. Amount of hydrogen in tested specimens .

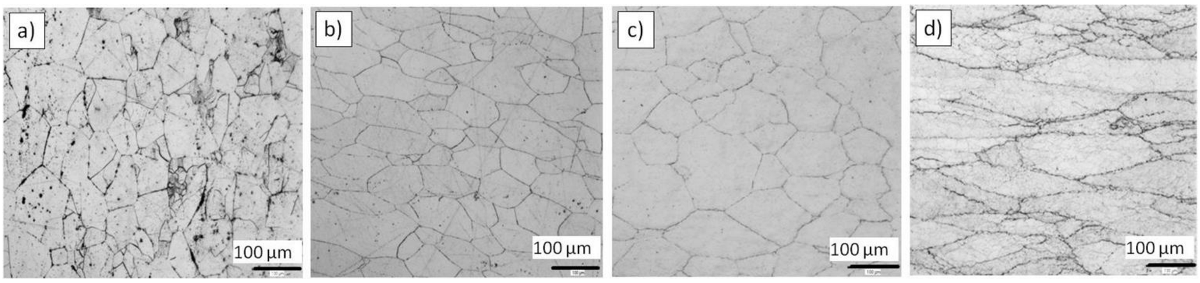

Figure 7. Optical micrograph of Ti24Nb alloy treated by three steps heat treatment in Ar (a) and deformed by compression test at 700 °C (b), 750 °C (c) and 800 °C (d).

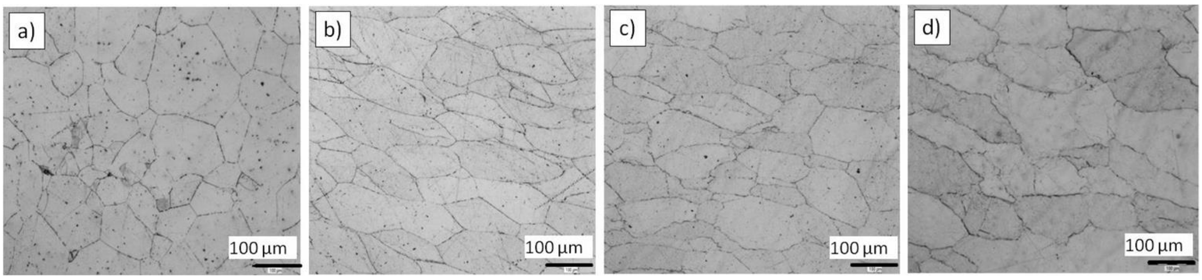

Figure 8. Optical micrograph of Ti26Nb alloy treated by three steps heat treatment in Ar (a) and deformed by compression test at 700 °C (b), 750 °C (c) and 800 °C (d).

The microstructure of hydrogen non-charged samples was formed of beta grains with fine alpha precipitates (figures 8 a and 9 a). After the hot compression tests, the microstructures of both alloys were dynamically recrystallized to large grains with very fine subgrains (figures 7 d and 8 d). The hydrogen charging led to the important refining of microstructure after the THT as well as after the CT, as seen in figures 9 and 10.

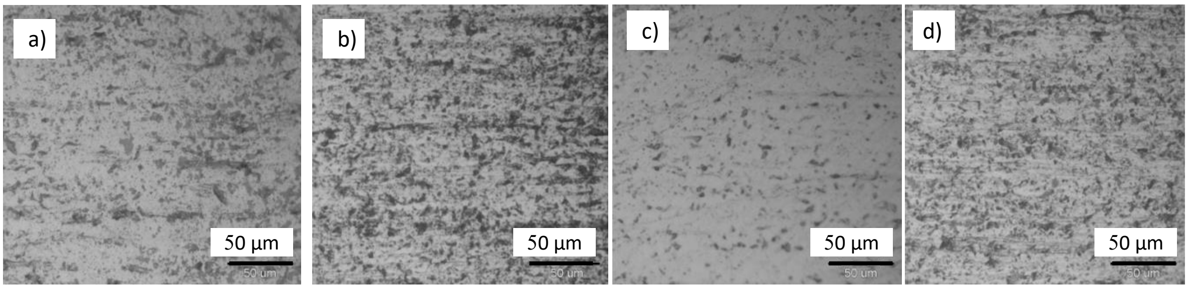

Figure 9. Optical micrograph of Ti24Nb alloy treated by three steps heat treatment in hydrogen (a) and deformed by compression test at 700 °C (b), 750 °C (c) and 800 °C (d).

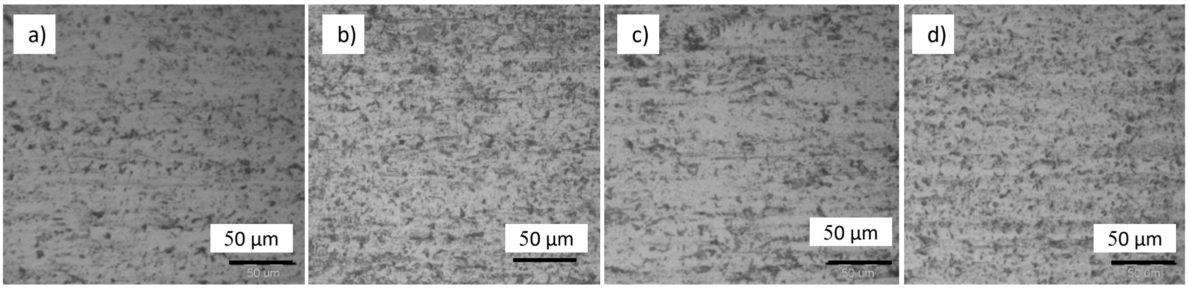

Figure 10. Optical micrograph of Ti26Nb alloy treated by three steps heat treatment in hydrogen (a) and deformed by compression test at 700 °C (b), 750 °C (c) and 800 °C (d).

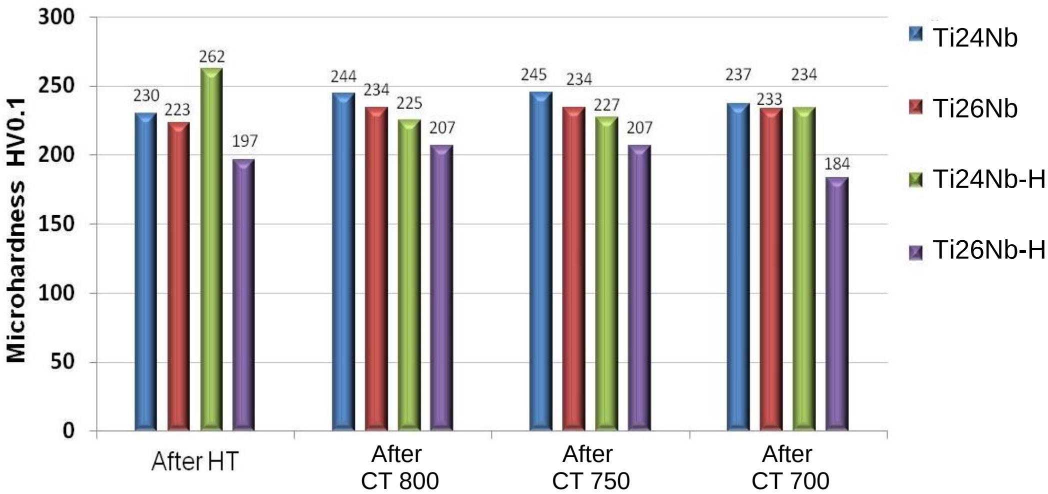

Figure 11. Microhardness of TiNb samples before and after compression test (CT) at 800, 750 a 700 °C.

The values of microhardness measured for hydrogen non-charged Ti24Nb alloy are higher in all treated stages (figure 11) and are related with precipitation of fine particles of alpha phase in beta grains and on grain boundaries. The finer microstructure of Ti24Nb after the THT caused the higher microhardness unlike the specimens with higher niobium content in which the hydrogen decreased the microhardness in all treated stages. As for the compression test curves, understanding the inconsistency of hydrogen influence on microstructure together with mechanical properties is presently limited and must be completed by other phase analysis. The hydrogen on the one side allows stabilizing more ductile beta phase but on the other side provides strengthening by solid solution. Nevertheless, it is clear that thermo-hydrogen treatment in the case of TiNb alloys is not as favorable as it is for Ti6AlV, at least for the niobium contents studied in this work.

4 Conclusions

The hydrogen effect on the microstructure and flow stress was studied using isothermal compression test at three different temperatures for two TiNb alloy compositions. Comparing the results obtained for hydrogen non-charged and hydrogen charged specimens it was found that the hydrogen content as high as 1663 and 2572 wt. ppm for Ti24Nb and Ti26Nb, respectively, has an obvious benefit effect on the supression of instabilities in true stress - true strain curves.

However, the higher amount of hydrogen induced increasing flow stress and resulted in cracking of specimens. The benefit effect of hydrogen on increasing formability of TiNb based alloys at lower temperatures was not evident as it was observed in the case of Ti6Al4V alloy.

Acknowledgement

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