INTERMETALLIC ALLOY BASED ON TiAl ALLOYED WITH 10 AND 15 at. % Nb

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Abstract
Subject of this work is an intermetallic alloy based on TiAl and doped with Nb with high creep resistance and good mechanical properties. The experimental TiAl-Nb material with two compositions of Ti45Al10Nb and Ti40Al15Nb was prepared from master alloy TiNb (55/45 wt. %), Ti and Al pieces by plasma melting, vacuum induction remelting and casting into the rod shape. The samples were homogenisation annealed at 1100°C for 12 hours in Ar atmosphere and water quenched from 700°C. Heat treatment at 700°C for 8 hours in Ar atmosphere as well as in hydrogen flowing gas was realised to prepare experimental material for hydrogen effect study. Microstructure of the specimens was studied by light and scanning electron microscopies, microhardness measurement and phase microanalysis were performed. The lamellar microstructure of the samples in as-cast and heat treated conditions formed of the γ (45 at.% Al) and α² (41 at.% Al) phases resulted from metallographic observation and EDS phase analysis. Microhardness values of the plasma melted samples decreased after annealing and quenching. Effect of hydrogen on the lamellar microstructure and microhardness values was studied in the hydrogen-charged and hydrogen-free specimens.

1. INTRODUCTION
Intermetallic alloys based on TiAl are suitable materials for high temperature applications due to the good oxidation resistance and high specific strength at higher temperatures than it is common for conventional Ti alloys [1]. The mechanical properties of TiAl alloys are strongly affected by microstructure morphology. The fully lamellar structure formed of the α²(Ti₃Al) and γ(TiAl) phases has been verified to have the best properties as compared to near lamellar, duplex, near gamma and gamma structures [2]. Nevertheless, creep properties and fracture toughness of fully lamellar alloys also depend on the microstructure parameters, such as lamellar spacing, orientation and their colony size. The interfaces of α₂/γ laths in the lamellar colonies act as obstacles to dislocation motion, but their orientation may promote creep anisotropy in duplex TiAl. A systematic research and alloy design have been realised to improve structural stability using microstructure manipulation, solid solution alloying and precipitation hardening. It was found that the fully lamellar microstructure possess the best mechanical properties if the lamellar orientation is aligned favourably according to the type of the load [3-5]. The objectives of the present study were to prepare two TiAl-based alloys alloyed with 10 and 15 at.% Nb, estimate the influence of Nb content on the microstructure features and on the microhardness values after plasma melting and heat treatment and evaluate hydrogen effect on microstructure and microhardness properties.

2. EXPERIMENTAL
The experimental Ti45Al10Nb and Ti40Al15Nb (at.%) alloys were melted from the master alloy TiNb (55/45 wt.%) and elemental Ti (3N8) and Al (5N) pieces by means of Ar-operated plasma torch. In
order to achieve the homogenised microstructure [6] the prepared alloys were vacuum induction remelted and cast to rods of 12 mm diameter. The alloys of both compositions were annealed for 12 hours at 1100 °C in flowing Ar gas and water quenched from 700 °C. Selected samples were heat treated at 700 °C for 8 hours in hydrogen gas and for comparison of microstructure changes the annealing of the same time and temperature conditions in flowing Ar was performed. High temperature annealing at 1300 °C for 4 hours in hydrogen and water quenching from 700 °C was realised to evaluate effect of hydrogen on microstructure features.

The samples prepared in this way were subjected to metallographic analysis and measurement of microhardness. The specimens of both compositions were polished, etched (Kroll’s reagent: 2 ml HNO$_3$:1 ml HF: 330 ml H$_2$O) and observed using metallographic microscope Olympus GX51 in as-cast as well as in heat treated conditions. Phase and microstructure analysis were carried out using scanning electron microscope JEOL JSM - 6490LV equipped with EDS INCA X - ACT probe. Microhardness values were measured by means of Future-Tech FM-100 instrument with load of 0.2 kg and step of 1 mm across the specimen diameter.

3. RESULTS AND DISCUSSION

Figure 1 shows the microstructure of plasma melted Ti45Al10Nb alloy in which large grains and (α$_2$+γ) laths were observed. After annealing and quenching the microstructure was formed of smaller grains and (α$_2$+γ) laths (Fig. 1b). The microstructures of plasma melted and heat treated Ti40Al15Nb alloy were also lamellar, as seen in Fig. 2a and 2b. In annealed and quenched condition of both compositions the finer laths of α$_2$ and γ phases were found.

Based on EDS microanalysis the average contents of elements were determined for Ti45Al10Nb: 46.5 at.% Ti, 42.6 at.% Al and 10.8 at.% Nb and for Ti40Al15Nb: 45.3 at.% Ti, 38.5 at.% Al and 16.2 at.% Nb. The concentrations of Al in dark and bright laths of lamellar (α$_2$+γ) microstructure in Fig. 3 correspond to 45 at.% Al in γ and 41 at.% Al in α$_2$. As compared of Fig. 1a and 2a it seems that higher content of Nb reduced lamellae size.

The results of microhardness measurement are summarised in Tables 1 and Tables 2. The higher values of microhardness for alloy with 15 at.% Nb measured for as-cast as well as for different heat
treatment of both alloys proved that higher content of Nb increased microhardness of samples independently on technology of melting and heat treatment. The microhardness of Ti45Al10Nb reached 520 HV after plasma melting and decreased to 406 HV after annealing. The similar results were obtained for Ti40Al15Nb, values of 526 HV and 503 HV corresponds to plasma melted and heat treated conditions, respectively. The higher values for plasma melted conditions than after annealing at 1100 °C and quenching from 700 °C of both compositions should be related to the phase presence after homogenisation treatment in compliance with phase diagram for Ti-Al-Nb ternary system [7]. The SEM and EDS phase analysis will be the subject of future work to determine the difference of the phase composition in both alloys after plasma melting and annealing.

![Fig. 2](image1.png)  ![Fig. 3](image2.png)

**Fig. 2** Optical micrographs of Ti40Al15Nb with (γ+α₂) lamellar microstructure
a) after plasma melting and b) after annealing at 1100 °C and quenching

**Fig. 3** SEM micrographs of plasma melted Ti45Al10Nb a) lamellar microstructure b) detail of (γ+α₂) laths

Table 1  Average values of Vickers microhardness (HV) measured for plasma melted TiAl-Nb samples in as-cast and annealed at 1100 °C conditions

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Alloy</th>
<th>Ti45Al10Nb</th>
<th>Ti40Al15Nb</th>
</tr>
</thead>
<tbody>
<tr>
<td>Plasma melted</td>
<td>Ti45Al10Nb</td>
<td>520</td>
<td>526</td>
</tr>
<tr>
<td></td>
<td>Ti40Al15Nb</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Annealed</td>
<td>Ti45Al10Nb</td>
<td>406</td>
<td>503</td>
</tr>
<tr>
<td></td>
<td>Ti40Al15Nb</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
The microstructure of induction melted alloys of both compositions was fully lamellar (Fig. 4). After homogenisation annealing 8h at 700°C in Ar the grains in both composition alloys were lamellar (Fig. 5a and 6a). Alloy with 10 at.% Nb contained little subgrains of size of about 20 µm that could be related to higher microhardness values (520 HV) in comparison of as-cast specimen (472 HV). The microstructure of annealed alloy with 15 at.% Nb remained fully lamellar, however the microhardness value also increased from 509 HV to 572 HV. The hydrogen heat treatment at 700°C has led only to coarsening of grains (Fig. 5b and 6b), indeed the hydrogen presence in the structure causes increasing of microhardness of both alloys to 572 HV and 588 HV in Ti45Al10Nb and Ti40Al15Nb, respectively.

Fig. 4 Microstructure after melting in induction furnace a) Ti40Al15Nb alloy and b) Ti45Al10Nb alloy

Fig. 5 Microstructure of Ti45Al10Nb alloy induction melted and annealed at 700°C a) in Ar and b) in H₂

Fig. 6 Microstructure of Ti40Al15Nb alloy induction melted and annealed at 700°C a) in Ar and b) in H₂
The microstructures of both alloys after hydrogenation at 1300°C and quenching from 700°C shows finer grains with fully lamellar feature (Fig. 7). Nevertheless, the results of microhardness decreased to values of 429 HV and 521 HV for Ti45Al10Nb and Ti40Al15Nb, respectively, compared to samples hydrogenated at 700°C. Further EDS phase microanalysis and X-ray analysis are needed to explain the role of hydrogen in microstructure changes related with reduced microhardness.

Table 2. Average values of Vickers microhardness (HV) measured for induction melted TiAl-Nb samples in as-cast, annealed at 700°C in Ar and annealed in hydrogen (700 and 1300°C) conditions.

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Alloy</th>
<th>Ti45Al10Nb</th>
<th>Ti40Al15Nb</th>
</tr>
</thead>
<tbody>
<tr>
<td>as-cast</td>
<td></td>
<td>472</td>
<td>509</td>
</tr>
<tr>
<td>annealed in Ar at 700°C</td>
<td></td>
<td>520</td>
<td>572</td>
</tr>
<tr>
<td>annealed in H₂ at 700°C</td>
<td></td>
<td>544</td>
<td>588</td>
</tr>
<tr>
<td>annealed in H₂ at 1300°C</td>
<td></td>
<td>429</td>
<td>521</td>
</tr>
</tbody>
</table>

4. CONCLUSIONS
Based on the experimental results the follows conclusions were drawn:
1. The microstructure optimisation was realised using plasma melting of master alloy and Ti and Al pieces followed by vacuum induction melting.
2. The microstructure of as-cast and annealed in Ar as well as in H₂ conditions was lamellar and consists of γ and α₂ laths.
3. The microhardness of Ti40Al15Nb alloy showed the higher values than for Ti45Al10Nb alloy in all heat treatment conditions which is in good accordance with higher Nb content.
4. The increasing microhardness after annealing at 700°C on the one side and decreasing microhardness after annealing at 1100°C on the other side could be related with phase changes in microstructure. Further phase analysis in accordance with ternary diagrams is needed to explain this phenomenon.
5. The effect of hydrogen treatment at 700°C manifested in increasing of microhardness due to presence of hydrogen in the alloy structure.
6. The drop of microhardness of both alloys after hydrogen treatment at 1300°C cannot be satisfactorily explained. What is role of the hydrogen in this event it could be determined with consideration of EDS microanalysis of phases in the microstructure.

ACKNOWLEDGEMENTS

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