STRUCTURE AND MECHANICAL PROPERTIES OF TaNbHfZrTi HIGH ENTROPY ALLOY

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Abstract

High entropy alloys are a new material class with promising structural and mechanical properties in wide range of applications. In principle they consist of five or more elements in equimolar concentrations resulting basically in single phase microstructure. TaNbHfZrTi alloy was developed by Senkov for high temperature applications.

Since all of these elements are biocompatible the TaNbHfZrTi alloy can be investigated as a prospective bio implant material. The alloy was prepared by vacuum arc melting, no annealing treatment was applied. Tensile test and hardness test at room temperature were performed with very good results: yield strength 1155 MPa, rupture strength 1212 MPa, Vickers hardness 359 HV_{30}. Microstructure and fracture morphology was also investigated.

Keywords: High entropy alloys, microstructure, mechanical properties, tensile test

1. INTRODUCTION

First publication regarding high entropy alloys (HEAs) was published in [1]. Since then many research and review article has been published e.g. [2, 3]. High entropy alloys consist in principle of 5 or more principal elements in (near) equimolar proportion. Such alloy posses high configuration entropy which should stabilize solid solution structure of the alloy. Nevertheless many such systems contain intermetallic phases, but they are also called high entropy alloys [3].

Such a new group of materials became very attractive to scientists and researchers. Reason was their good properties and large space for experimental variations. Mechanical, microstructure, corrosion and other properties has been investigated in many alloy systems. Recently most investigated systems are based on Cr, Fr, Cu, Ni, Al. Other group of HEAs is based on elements with high melting temperature such as Ti, Nb, Ta, Zr, Hf, Mo, W, V [3, 4, 5]. These are investigated mainly for their promising high temperature properties [4]. Since some of these elements are considered biocompatible [6, 7], such alloy could be used for bio implant production. Therefore TaNbHfZrTi was chosen for trial casting. This alloy was investigated already by Senkov [5] and others. But to the authors knowledge published mechanical properties were measured only in pressure. Pressure tests are very common in case of HEAs. Therefore tensile test at room temperature was performed, together with microstructure and fractographic investigations.

2. EXPERIMENTAL

Experimental alloy was prepared by vacuum arc melting in water cooled copper crucible. Titanium, niobium and hafnium were in the form of bars with 10 mm in diameter. Tantalum and zirconium were in the form of 1 mm thick plates. Chemical purity of inserted elements was 99.9 %. Each element was added in equimolar proportion.

Final casting had approximately 100 mm in length, 30 mm in width, 10 mm in height and 400 g in weight. Casting was performed 6 times and flipped for each melt to mix the elements thoroughly and suppress chemical heterogenity.

Nor HIP, nor other heat treatment was applied to the casting. Tensile test body with 5 mm in diameter and 25 mm of measured length were stressed in deformation rate of $2\cdot10^{-4}$ s^{-1} using 1185 Instron test machine equipped with video-extensometer.

Metallographic sections perpendicular to the length of the casting were prepared. Vickers hardness HV_{30} was measured on that section using Zwick ZHU 250 Topline universal testing machine. Microstructure was examined by light (Nikon Epiphot 300) and scanning electron microscopy (JEOL JSM 7600F) on etched samples. Etching was performed in mixture of nitric acid (1.40) 30 ml, hydrofluoric acid (40 %) 10 ml and water 60 ml for approx. 10 seconds.

In order to determine the existing phase structures of the alloy, powder X-ray diffraction (PXRD) analysis was performed. D8 Discover diffractometer, CuKα radiation and 1D detector was used. Rietveld analysis was performed in TOPAS 4.2 with strain-related profiles' broadening modelled by Gaussian function and size-related by Lorentzian function.

Existing phase structure was also examined by EBSD (HKL NordlysS). EDS analysis of chemical composition was also performed by Oxford X-Max. The fractography analysis of broken specimen was performed on scanning electron microscope Jeol LV 5510.

3. RESULTS AND DISCUSSION

3.1. Mechanical properties

Table 1 Mechanical properties of as-cast TaNbHfZrTi alloy

Specimen	$R_{p0.2}$ [MPa]	R_m [MPa]	A [%]	E [GPa]	HV_{30}
TaNbHfZrTi	1155	1212	12.3	59	359

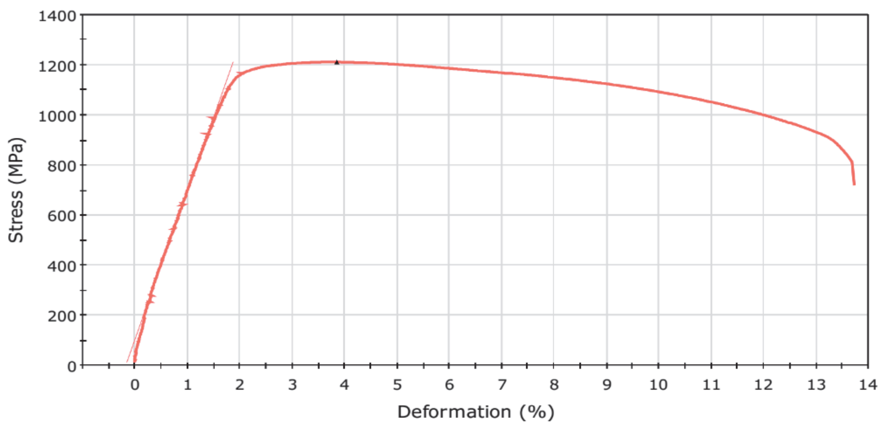

Fig. 1 Engineering stress strain diagram of as-cast TaNbHfZrTi alloy

Mechanical properties of as-cast TaNbHfZrTi alloy are given in **Table 1**. Diagram of engineering stress vs. engineering strain is depicted in **Fig. 1**. Necking starts rather early but is followed by large elongation. Measured yield strength 1155 MPa is larger than yield strength in compression 929 MPa measured previously on the same nominal composition alloy [5]. Hardness values are high, but not too much, therefore machining is possible.

3.2. Chemical composition

Table 2 Chemical composition of as-cast TaNbHfZrTi alloy in at.%

Ti	Zr	Nb	Hf	Ta
18.16	20.55	20.96	20.65	19.68

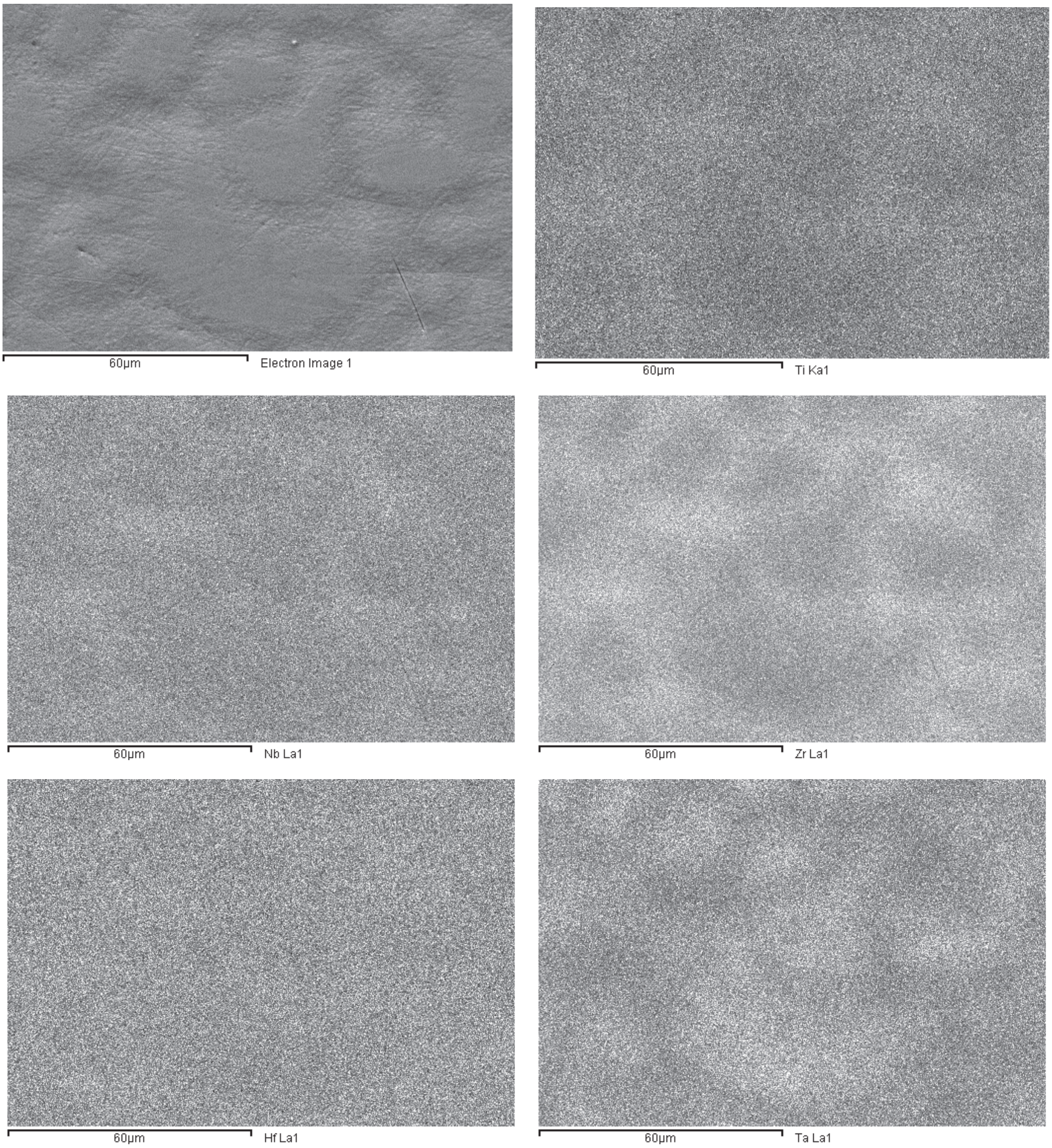

Fig. 2 SEM picture and EDS mapping of as-cast TaNbHfZrTi alloy

Chemical composition of the final casting was verified by EDS chemical analysis (see **Table 2**). No important difference from ideal equimolar composition has been found. Slightly smaller titanium content could be caused by evaporation during remelting since titanium has the lowest melting point.

Etching reveals dendritic microstructure of the alloy (see **Fig. 2**). EDS mapping analysis was also performed. Only minor chemical heterogeneity was found. It seems that interdendritic space is slightly rich on zirconium and slightly poor of tantalum. That is probably the cause of the origin of the etching relief.

3.3. Microstructure

TaNbHfZrTi alloy exhibits dendritic microstructure with grain size in the range of 100-200 µm (see **Fig. 3**). Grain size was estimated from EBSD analysis to 160 µm (see **Fig. 5**). The EBSD phase analysis identified only BCC titanium from the phase library of the analysis system.

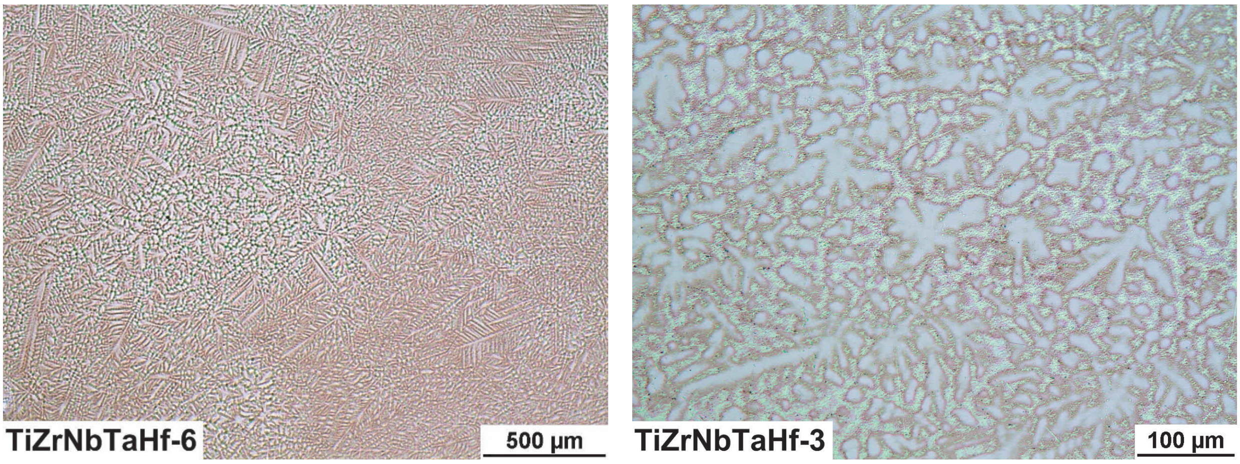

Fig. 3 Dendritic microstructure of as-cast TaNbHfZrTi alloy - light microscopy

In the obtained PXRD pattern (see **Fig. 4**), reflections of BCC phase are observed. According to the Rietveld refinement, the lattice parameter of this phase was 0.34089 nm. This value confirms values measured before 340.4 pm [5]. Taking advantage of whole powder pattern modeling (WPPM) [8], the average size of coherently scattering domains lies in the vicinity 300 nm, which means that the so-called size broadening effect is negligible in comparison with the micro-strain broadening which was computed to be $\varepsilon^{micro} = (0.881 \pm 0.009)\cdot 10^{-3}$.

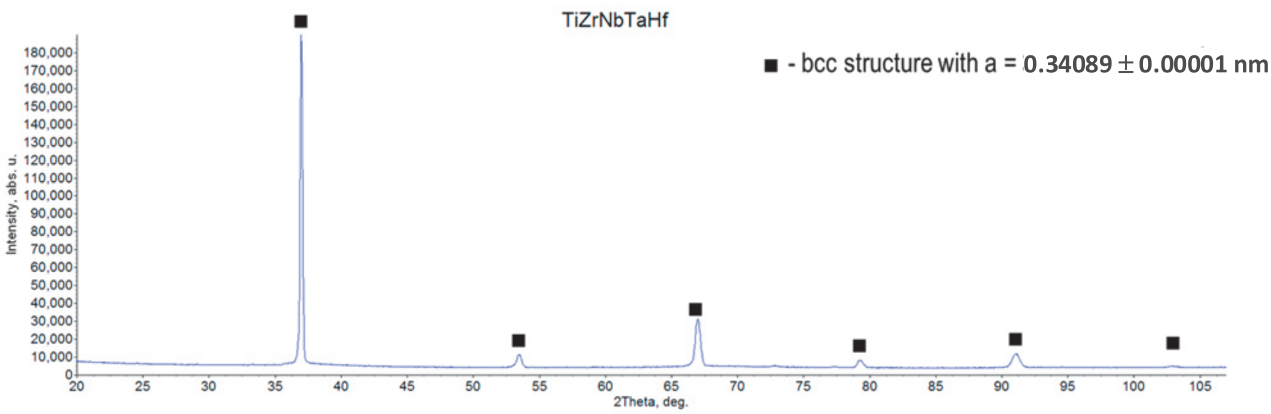

Fig. 4 PXRD pattern obtained on the surface of the as-cast TaNbHfZrTi alloy

3.4. Fractography

Fracture surfaces of the broken test specimen were analyzed. Fracture surface is inclined to tensile direction (see **Fig. 6**). Two different areas were found on the fracture surface. Large ductile shallow dimples in the middle of the fracture surface and small shallow ductile dimples in the surrounding (see **Fig. 7**). No signs of brittle fracture were found.

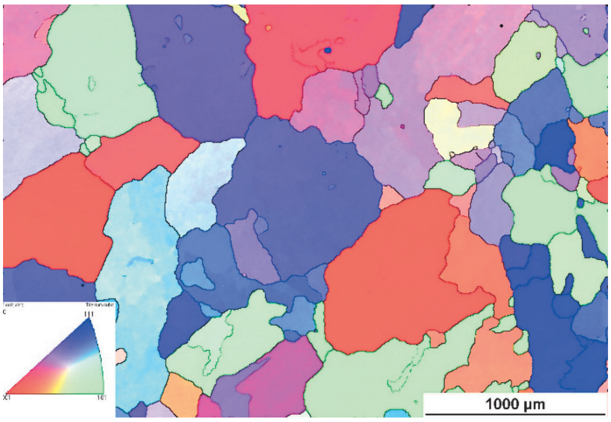

Fig. 5 EBSD microstructure analysis of as-cast TaNbHfZrTi alloy (left)

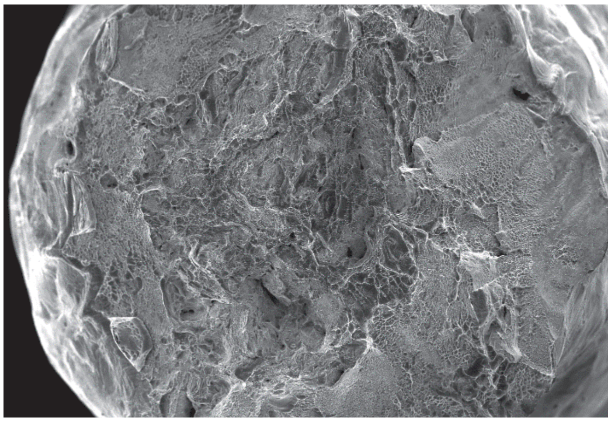

Fig. 6 Fracture surface of the broken tensile specimen of as-cast TaNbHfZrTi alloy (right)

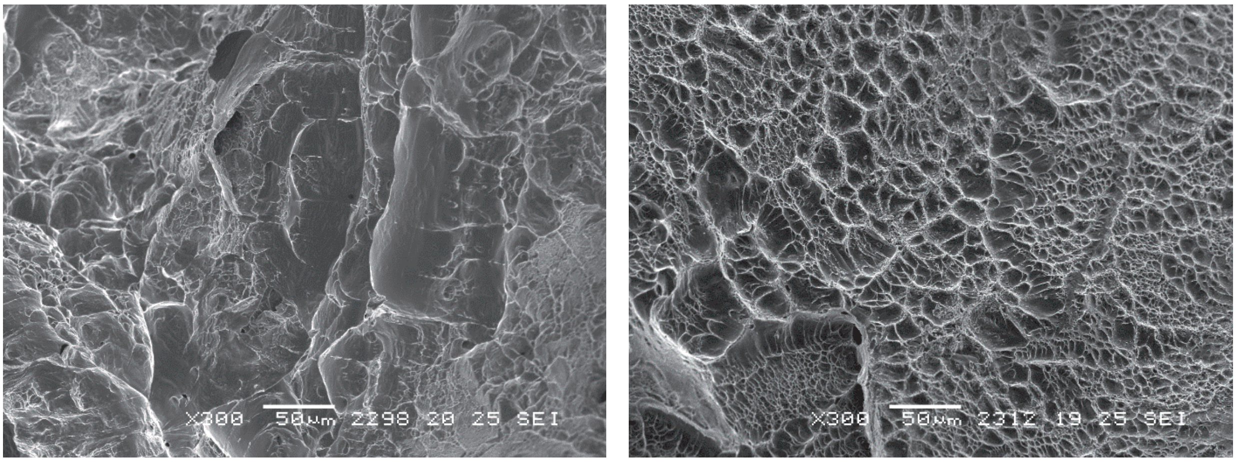

Fig. 7 Large ductile dimples in the centre (left) and small ductile dimples in the periphery (right of the fracture surface) of the broken tensile specimen of as-cast TaNbHfZrTi alloy

4. CONCLUSION

TaNbHfZrTi alloy was successfully prepared by vacuum arc melting from individual elements. Tensile tests at room temperature confirmed good mechanical (yield strength 1155 MPa, rupture strength 1212 MPa, Vickers hardness 359 HV_{30}) and deformation properties (A = 12.3 %). Microstructure of the alloy consists of dendritic grains with grain size 100-200 µm. EBSD and powder X-Ray Diffraction detected only BCC phase with lattice parameter of this phase 0.34089 nm. Fractography analysis revealed indications of ductile fracture only.

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