A Novel Series of Refractory High-Entropy Alloys $\text{Ti}_2\text{ZrHf}_{0.5}\text{VNb}_x$ with High Specific Yield Strength and Good Ductility

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Abstract

A series of $\text{Ti}_2\text{ZrHf}_{0.5}\text{VNb}_x$ ($x=0, 0.25, 0.5, 0.75$ and 1.0) refractory high-entropy alloys were prepared to investigate the alloying effect of Nb on the microstructures and mechanical properties. All the alloys displayed a simple BCC structure. The microstructures of the alloys changed from the initial single-phase columnar structure ($x=0$) to dendrite microstructure ($x>0$). At room temperature, all the alloys exhibited high ductility (with the compressive strains of more than 50%). With the increase in Nb content, the yield strength slightly decreased from 1160 to 980 MPa and the hardness dropped from 338 to 310 HV. Moreover, the alloys exhibited low density from 6.47 to 6.84 g/cm³ and high specific yield strength (SYS) from 143 to 179 kPa m³/kg. The comprehensive performance of ductility and SYS was superior to most of the reported high-entropy alloys. The yield strength of the alloys increased from 405 to 859 MPa and from 85 to 195 MPa with the addition of Nb element at 873 K and 1073 K, respectively.

Keywords Specific yield strength · Refractory metals · Structure · Mechanical properties

1 Introduction

Recently, a new alloy concept of high-entropy alloys (HEAs) or multicomponent alloys was proposed by Yeh et al. [1] and Cantor et al. [2]. HEAs generally contain principal elements with the concentration between 5 and 35 at% [3–11] and exhibit many excellent physical, chemical and mechanical properties, such as high strength and ductility [3, 4, 12, 13], high fracture toughness [5, 6], excellent corrosion resistance [7, 8] and good irradiation resistance [9, 10]. Thereinto, refractory high-entropy alloys (RHEAs) were designed using high-melting-point metals such as V, Nb, Ta, Mo, W, Ti, Zr and Hf, which possess high strength and excellent high-temperature softening resistance [11, 14, 15]. Hence, they

became potential candidates applying to the field of aerospace and aviation. For example, Senkov et al. first reported the WMoNbTa(V) RHEAs with a single BCC phase that could retain the yield strength of more than 500 MPa at 1600 °C, which were superior to conventional superalloys [16]. Since then, more RHEAs were designed, such as TiZrHfNbTa, TiZrHfVMo, TiZrHfMoNb, Ti(V)WMoNbTa, TiVNbTa and so on [17–23], which exhibited excellent strength at room temperature (RT) and high temperature. However, most of them generally had two disadvantages: poor ductility (compressive strain $\epsilon < 50\%$) or high density ($\rho > 7.5$ g/cm³), which were not conducive to industrial applications. Therefore, some light-weight HEAs containing Al and Cr elements, such as CrNbTiVZr, TiVNb(Ta)Al_x, TiVNbAlZr_x and AlMo_{0.5}NbTa_{0.5}TiZr etc. were designed to reduce the density [13, 24–29]. These alloys possessed high strength with low density (~ 6.5 g/cm³) but exhibited poor plasticity. Therefore, it was difficult for most reported RHEAs to have high strength, good ductility and low density at the same time.

To address the above mentioned problems in the real application of RHEAs, this study proposed to use light elements, such as Ti, Zr, Hf, V and Nb, to design low-weight RHEAs, aiming at high strength and good plasticity to resolve the strength-ductility trade-off. According to the

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valence electron concentration (VEC) model [30], the alloy is single body-centered cubic (BCC) phase when the VEC is less than 6.87. Therefore, the content ratio of Ti was raised to 2 and Hf element was reduced to 0.5 for further obtain a lower VEC value in order to stabilize the BCC phase, and reduce the density. Then, a new series of light BCC-structured $\text{Ti}_2\text{ZrHf}_{0.5}\text{VNb}_x$ ($x=0, 0.25, 0.5, 0.75, 1$) alloys with low VEC value were designed, and their microstructures and properties were investigated.

2 Experimental

The $\text{Ti}_2\text{ZrHf}_{0.5}\text{VNb}_x$ (when $x=0, 0.25, 0.5, 0.75$ and 1 , denoted as $\text{Nb}_0, \text{Nb}_{0.25}, \text{Nb}_{0.5}, \text{Nb}_{0.75}$ and Nb_1 , respectively) alloys were prepared by vacuum arc melting in a Ti-gettered argon atmosphere using pure elements of Ti, Zr, V, Nb and Hf (99.95 wt%). Approximately 35 g of alloy ingots was remelted at most seven times for improving the homogeneity of alloys. The samples were ground using SiC papers up to 2000 grit, and then the crystalline structure of the samples was characterized by the X-ray diffractometer (XRD, EMPYREAN with CuK_α radiation target) with the angle of 2θ scanning from 20° to 100° at a scanning rate of $0.106103^\circ/\text{s}$. After polishing and corrosion, the microstructure and compositions of the samples were analyzed using the scanning electron microscope (SEM/Zeiss Supra 55). The RT compressive tests were conducted using a Wippermann materials testing machine according to the standard of ASTM E9-09. The compressive properties at 873 K and 1073 K were tested by a Gleeble-3500 thermal simulation machine, and they were heated to the test temperature at a heating rate of 5 K/s and held for 5 min. The test specimens were cylindrical samples of $\Phi 5 \times 10$ mm and $\Phi 6 \times 8$ mm at room temperature and elevated temperature, respectively. The compression rates were all 10^{-3} s^{-1} . Hardness

measurements of the alloys (test standards used ASTM E384-11e1) were conducted using a Vickers hardness tester (MH-50) under a load of 1000 g for 15 s. Seven measurements were made on each sample to obtain the averaged experimental data. The density of the alloys was measured by the Archimedes drainage method (ME204E balance).

3 Results and Discussion

3.1 XRD Analysis and Phase Identification

Figure 1a shows the XRD patterns of the $\text{Ti}_2\text{ZrHf}_{0.5}\text{VNb}_x$ alloys with different Nb contents. A single disordered BCC phase was found in the $\text{Ti}_2\text{ZrHf}_{0.5}\text{VNb}_x$ alloys. No other phase diffraction peaks appeared with the increase in Nb content, indicating that the addition of Nb element has no obvious influence on the phase formation for this series of alloys. Figure 1b shows the detailed scans for the peak of (110) of BCC solid solution phase. It can be found that the peak of the BCC phase integrally shifted toward the higher 2θ angle with the addition of Nb element, indicating that the lattice constants decrease slightly on the whole, as shown in Table 1. It is because of the radius of Nb element is $1.429 \text{ \AA}$ which is a little bit less than the average value ($1.437 \text{ \AA}$) of $\text{Ti}_2\text{ZrHf}_{0.5}\text{V}$ matrix.

At present, several parameters have been summarized to predict the phase formation of HEAs. Guo et al. [30] concluded that the HEAs generally form the BCC phase at $\text{VEC} < 6.87$ and face-centered cubic (FCC) phase at $\text{VEC} > 8$, and Zhang et al. [31] summarized that $\Omega > 1.1$ and $\delta < 6.6\%$ are beneficial to the formation of solid solutions in many HEAs. In this study, the VEC of the alloys changes from 4.22 to 4.37 with the increase in Nb content which manifests the alloys tend to form BCC phase; Besides the maximum δ is 6.11 (< 6.6) and extreme large Ω (> 12.41)

Fig. 1 a XRD patterns of $\text{Ti}_2\text{ZrHf}_{0.5}\text{VNb}_x$ alloys, b detailed scans for peak of (110) of BCC solid solution phase

Table 1 Parameters of VEC, δ (atomic size difference), ΔH_{mix} (enthalpy of mixing), ΔS_{mix} (configurational entropy), Ω (solid solution formability) and a_{exp} (lattice constant) for Ti₂ZrHf_{0.5}VNb_x alloys

Alloy	VEC	δ	ΔH_{mix} (kJ/mol)	ΔS_{mix} (J/(K mol))	Ω	a_{exp} (pm)
Nb ₀	4.22	6.11	− 1.78	10.58	12.41	335.42
Nb _{0.25}	4.26	5.95	− 1.2	11.74	20.78	335.83
Nb _{0.5}	4.3	5.81	− 0.72	12.23	36.61	336.05
Nb _{0.75}	4.33	5.67	− 0.33	12.48	82.59	336.15
Nb ₁	4.37	5.54	0	12.61	∞	336.58

also predict the series alloys would form single BCC solid solution phase without other intermetallics in accordance with the experimental results [32]. In addition, Zhang et al. also proposed that the HEAs would form the solid solution if the ΔH_{mix} is in the range − 15 to 5 kJ/mol. The absolute values of ΔH_{mix} of Ti₂ZrHf_{0.5}VNb_x series of alloys are very small, almost close to zero, indicating that Nb has a large solid solubility with other four elements to form single disordered BCC phase, and does not tend to form intermetallics. The parameters of the Ti₂ZrHf_{0.5}VNb_x alloys are displayed in Table 1.

3.2 Microstructure

The SEM images of Ti₂ZrHf_{0.5}VNb_x alloys are displayed in Fig. 2. It can be seen that the Ti₂ZrHf_{0.5}VNb₀ alloy shows a coarse columnar crystal structure, confirming the Nb₀ alloy has a single-phase structure. Combined with the XRD results, a conclusion can be drawn that the single-phase structure is the BCC solid solution phase. With the addition of Nb element, dendrite structures are observed in Nb_{0.25}, Nb_{0.5}, Nb_{0.75} and Nb₁ alloys. As the Ti₂ZrHf_{0.5}VNb_x alloys are a single-phase structure, the Nb_{0.5} alloy is chosen to carry out the transmission electron microscopy (TEM) test, as shown in Fig. 2f. It can be seen that the microstructure is uniform, and only one set of diffraction spots of BCC phase was found. In addition, the elemental mapping of Nb₁ alloy was investigated as a typical Ti₂ZrHf_{0.5}VNb_x alloys, and it can be seen that the all elements are evenly distributed as shown in Fig. 3. The above results prove that the series of alloys exhibit single BCC structure with homogeneous composition distribution.

3.3 Mechanical Properties

Figure 4a shows the compressive engineering stress–strain curves of the Ti₂ZrHf_{0.5}VNb_x ($x=0, 0.25, 0.5, 0.75$ and 1.0) alloys at RT. The values of the yield strength, fracture strain and Vickers hardness are listed in Table 2. It is noted that all the Ti₂ZrHf_{0.5}VNb_x alloys possess excellent plasticity, which can be compressed more than 50% without fracture; it means that the addition of Nb element does not cause significant plasticity reduction. The yield strength and Vickers hardness curves of Ti₂ZrHf_{0.5}VNb_x alloys (Fig. 4b) slightly decrease

from 1160 to 980 MPa and 338 to 310 HV with the increase in Nb content, respectively. Interestingly, both of them present an approximate linear decline. The linear line can be expressed as $Y_{\sigma} = -192x + 1166$ and $Y_{\text{HV}} = -27.3x + 338$, where Y_{σ} is the yield strength, Y_{HV} is the Vickers hardness, and x is Nb content in Ti₂ZrHf_{0.5}VNb_x. We calculated the atomic radius difference of the Ti₂ZrHf_{0.5}VNb_x RHEAs with the addition of Nb element by the following formula [31]:

$$\delta = \sqrt{\sum_{i=1}^n c_i (1 - r_i/\bar{r})^2}, \quad (1)$$

$$\bar{r} = \sum_{i=1}^n c_i r_i, \quad (2)$$

where c_i and r_i are the atomic fraction and the Goldschmidt atomic radius [33] of the i th component, respectively. $\bar{r}$ is the arithmetical mean value of atomic radius. It can be found that the values of δ decrease from 6.11 to 5.54 as shown in Table 1, which means the lattice distortion of the Ti₂ZrHf_{0.5}VNb_x RHEAs decreases with the increase in Nb content. The strength of the alloys is proportional to the degree of the lattice distortion. Therefore, the addition of Nb element reduces the yield strength and hardness of the Ti₂ZrHf_{0.5}VNb_x RHEAs at room temperature.

The values of density and specific yield strength (SYS) of the Ti₂ZrHf_{0.5}VNb_x alloys are also presented in Table 2. Although the addition of high density Nb element increases the density of the alloys, they are still less than 7 g/cm³, and the lightest density can reach 6.47 g/cm³, which are still lower than that of most RHEAs. The SYS of the Ti₂ZrHf_{0.5}VNb_x alloys was calculated, and the maximum value can reach 179 kPa m³/kg. The SYS and ductility of some typical HEAs (including RHEAs) are summarized in Fig. 5 [11, 13, 17, 18, 20, 26–29, 34–42]. From Fig. 5, it can be seen that the CoCrFeNi-based HEAs located in the blue area rendered a left-sloping shape, they do not have both high SYS and plasticity. Most of RHEAs exhibit low SYS due to their characteristic high density, and some RHEAs with Al and Cr elements possess high SYS but poor ductility. The aforementioned findings suggest that the series of Ti₂ZrHf_{0.5}VNb_x alloys in this study (the red stars) display

Fig. 2 Microstructures of $\text{Ti}_2\text{ZrHf}_{0.5}\text{VNb}_x$ RHEAs: **a** $x=0$; **b** $x=0.25$; **c** $x=0.5$; **d** $x=0.75$; **e** $x=1$; **f** bright-field TEM image of $\text{Nb}_{0.5}$ alloy

much higher SYS and ductility than that of RHEAs. These results imply that the series of $\text{Ti}_2\text{ZrHf}_{0.5}\text{VNb}_x$ HEAs exhibit excellent comprehensive mechanical properties.

In order to further investigate the elevated-temperature mechanical properties of the series of $\text{Ti}_2\text{ZrHf}_{0.5}\text{VNb}_x$ alloys, the compressive curves of the Nb_0 , $\text{Nb}_{0.5}$ and Nb_1 alloys at 298 K, 873 K and 1073 K are shown in Fig. 6, and the values are listed in Table 3. With the increase in Nb content, the strengths of the alloys were improved from 405 to 859 MPa at 873 K and 85 to 195 MPa at 1073 K, respectively. The histogram of the temperature and yield strength of Nb_0 , $\text{Nb}_{0.5}$ and Nb_1 alloys is shown in Fig. 6c. It can be seen that the yield strength of the $\text{Ti}_2\text{ZrHf}_{0.5}\text{VNb}_x$ alloys decrease as the temperature rises, but the series of alloys can remain a

high strength at 873 K, especially for Nb_1 alloy. The yield strength of Nb_1 alloy decreased by only 120 MPa from RT (980 MPa) to high temperature 873 K (850 MPa). Then, the decline percentages of the yield strength are calculated from RT to 873 K, and the values are 65.1%, 32.6% and 12.3% for Nb_0 , $\text{Nb}_{0.5}$ and Nb_1 alloys, respectively. The addition of Nb element dramatically enhances the high-temperature strength and softens resistance. The results manifest that the Nb element is beneficial for improving the high-temperature mechanical performance of the $\text{Ti}_2\text{ZrHf}_{0.5}\text{VNb}_x$ alloys. Alloy melting point has a great influence on the high-temperature strength. The softening temperature of alloy is generally above $\sim 0.5 - 0.6 T_m$ (T_m melting temperature) [43, 44]. Therefore, the higher the melting point is, the stronger the

Fig. 3 SEM image (a), elemental mapping of Ti (b), Zr (c), Hf (d), V (e), Nb (f) for Nb_1 alloy, respectively

Fig. 4 **a** Compressive engineering stress–strain curves of $\text{Ti}_2\text{ZrHf}_{0.5}\text{VNb}_x$ alloys, **b** linear graphs of Vickers hardness and yield strength for $\text{Ti}_2\text{ZrHf}_{0.5}\text{VNb}_x$ alloys

Table 2 Mechanical properties of $\text{Ti}_2\text{ZrHf}_{0.5}\text{VNb}_x$ alloys

Alloy	σ (MPa)	ε (%)	Hardness (HV)	ρ (g/cm ³)	SYS
Nb_0	1160	> 50	338.3	6.47	179.3
$\text{Nb}_{0.25}$	1115	> 50	332.4	6.57	169.7
$\text{Nb}_{0.5}$	1065	> 50	322.5	6.66	159.9
$\text{Nb}_{0.75}$	1025	> 50	318.9	6.77	151.4
Nb_1	980	> 50	310.4	6.84	143.3

high-temperature softening resistance of the alloy becomes. In this work, Nb element possesses the highest melting point compared with other four elements. In terms of the rule of mixture (ROM), the melting point of the $\text{Ti}_2\text{ZrHf}_{0.5}\text{VNb}_x$ alloys is calculated by the formula:

$$T_m = \sum_{i=1}^n c_i T_i, \quad (3)$$

where T_i are the melting point of the i th element. The melting points of pure elements are given in Table 4. It can be found that the addition of Nb element increases the melting points of the $\text{Ti}_2\text{ZrHf}_{0.5}\text{VNb}_x$ alloys drastically from 2089 to 2209 K as shown in Table 3, so the softening temperature of the alloys increases correspondingly. Then, high-temperature softening resistance of the series alloys is improved significantly with the addition of Nb element. Although Nb element decreases the lattice distortion of the $\text{Ti}_2\text{ZrHf}_{0.5}\text{VNb}_x$ alloys, the effect of the melting point of alloys is greater

Fig. 5 SYS and ductility of series of $\text{Ti}_2\text{ZrHf}_{0.5}\text{VNb}_x$ alloys at RT compared with other reported HEAs

Fig. 6 Compressive stress–strain curves of Nb_0 , $\text{Nb}_{0.5}$ and Nb_1 alloys at 873 K (a), 1073 K (b), linear graphs of temperature and strength (c)

Table 3 Values of compressive properties of $\text{Ti}_2\text{ZrHf}_{0.5}\text{VNb}_x$ alloys at RT, 873 K and 1073 K

Alloy	Temperature (K)	σ (MPa)	T_m (K)
Nb_0	298	1160	2089
	873	405	
	1073	85	
$\text{Nb}_{0.5}$	298	1065	2155
	873	718	
	1073	135	
Nb_1	298	980	2209
	873	859	
	1073	195	

Table 4 Characters of experimental elements (r atomic radius)

Element	VEC	r (Å)	ρ (g/cm ³)	T_m (K)
Ti	4	1.42	4.51	1933
Zr	4	1.55	6.51	2125
Hf	4	1.54	13.31	2500
V	5	1.31	5.96	2163
Nb	5	1.43	8.57	2750

4 Conclusions

- (1) The XRD and SEM results reveal that the $\text{Ti}_2\text{ZrHf}_{0.5}\text{VNb}_x$ alloys are single disordered BCC phase, and the microstructure changed from coarse columnar grain ($x=0$) to dendrite structure ($x>0$) with the increase in Nb content.

than that of the lattice distortion of the alloys at the high temperature.

- (2) At RT, all the $\text{Ti}_2\text{ZrHf}_{0.5}\text{VNb}_x$ alloys exhibit excellent compressive plasticity of over 50%, the yield strength has slight decrease from 1160 to 980 MPa with the increase in Nb content due to the reduction of lattice distortion. The $\text{Ti}_2\text{ZrHf}_{0.5}\text{VNb}_x$ alloys also possess low density less than 6.84 g/cm^3 and high specific yield strength ($179 \text{ kPa m}^3/\text{kg}$) which are greater than most of RHEAs, and even HEAs.
- (3) The addition of Nb element into the $\text{Ti}_2\text{ZrHf}_{0.5}\text{V}$ alloy significantly improves the high-temperature strength and soften resistance. Thereinto, the Nb_1 alloy can remain a high yield strength $\sim 850 \text{ MPa}$ at 873 K , and it only reduced by 12.3% compared with that at RT.

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