Microstructure and mechanical properties of an ultrafine Ti-Si-Nb alloy

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ABSTRACT

Nb-modified ultrafine Ti-Si eutectic alloy was made by cold crucible levitation melting, tested in compression at room temperature, and characterized by electron microscopy. Compression tests of $(\text{Ti}_{86.5}\text{Si}_{13.5})_{97}\text{Nb}_3$ specimens measured an ultimate compressive strength of 1180 MPa and a compressive plastic strain of 12%, both of which are higher than in eutectic $\text{Ti}_{86.5}\text{Si}_{13.5}$ alloy. Electron microscopy showed that the Ti-Si-Nb alloy had a bimodal microstructure with micrometer-scale primary α -Ti dendrites distributed in an ultrafine eutectic (α -Ti+ Ti_5Si_3) matrix. The enhanced ductility is attributed to the morphology of the phase constituents and to the larger lattice mismatches between α -Ti and Ti_5Si_3 phases caused by the Nb addition. The crystallographic orientation relationship of Ti_5Si_3 with α -Ti is $(1\bar{1}00)[\bar{1}\bar{1}26]\text{Ti}_5\text{Si}_3 \parallel (01\bar{1}1)[5\bar{1}\bar{4}\bar{3}]\alpha\text{-Ti}$.

Keywords: Alloys; Electron microscopy; Mechanical testing; Ductility;

Microstructure

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1. Introduction

Titanium alloys have relatively low density, high tensile strength and toughness, and good elevated-temperature oxidation resistance and strength. This combination of properties makes titanium alloys widely useful for aerospace, medical, structural, and chemical processing applications [1,2]. Ultrafine grained (ufg) and nanocrystalline (nc) Ti-based alloys would provide much greater strength than their coarse-grained counterparts, as indicated by the well-known Hall-Petch relation. Reducing the grain size will cause the material to become stronger [3]; however, ultrafine-grain (ufg) and nanocrystalline (nc) materials typically have low ductility and fracture toughness, making them vulnerable to catastrophic failure. This limits their engineering usefulness as structural materials [4]. The ductility limitations of ufg and nc materials have been attributed to artifacts from sample preparation, force instability in tension, and crack nucleation or propagation instability [5].

Improving the ambient temperature ductility of ufg and nc materials has been an important subject in the development of advanced structural materials. Various strategies have been employed to improve the ductility of nanostructured metals and alloys, including bimodal (or multi-modal) grain size distributions [3,6]. Wang et al. reported that an unusually large strain (~65%) and high yield strength (~400 MPa) were achieved by cold rolling Cu to create a bimodal grain size distribution with micrometer-sized grains embedded inside a matrix of nanocrystalline grains [7]. Other ductilizing approaches include materials with nano-twins, using phase transformation and twinning-induced ductility to achieve high strength, ductility and conductivity [3,6,8-10].

Silicon is an important alloying element in Ti-based alloys destined for elevated-temperature applications. It is used extensively as an effective strengthening agent in

solid solution. In addition, the precipitation of silicide compounds (e.g., Ti_5Si_3) was found to provide a useful contribution to dispersion strengthening and improved microstructural stability at elevated temperatures [11,12]. Si also improves creep resistance of TiAl-based alloys by forming Ti_5Si_3 silicide that pins dislocations [13]. Significant research efforts on transition-metal (TM) silicides with the 5:3 stoichiometry (TM_5Si_3) have been conducted to examine their synthesis [14,15], mechanical properties [16,17], deformation [18], thermal properties and oxidation behavior [19,20]. However, information about the microstructure and mechanical properties for eutectic Ti-Si alloys is still limited. Nb is a β -Ti stabilizer, and the Ti-Nb binary phase diagram reveals that Ti and Nb can form solid solutions at all compositions at elevated temperatures [21]. Thus, it is expected that Nb addition might improve the ductility of the eutectic Ti-Si alloys by changing the microstructural morphology.

In the present research, we report on the relation between microstructure and mechanical behavior of Ti-Si eutectic alloys modified by Nb addition. The as-cast Nb-modified Ti-Si eutectic alloy having bimodal structure shows a plastic strain of 12% while preserving an ultimate strength of 1180 MPa. We pay special attention to the correlation between the microstructures and mechanical properties. It is hoped that this study will improve understanding of ufg and nc materials by providing a better understanding of methods to produce high-performance ufg and nc structural materials via microstructural manipulation.

2. Experimental procedure

Ingots with a nominal composition (at.%) of $\text{Ti}_{86.5}\text{Si}_{13.5}$ and $(\text{Ti}_{86.5}\text{Si}_{13.5})_{97}\text{Nb}_3$ were fabricated from high-purity (99.99%) Ti, Si, and Nb metals by cold crucible levitation melting under argon atmosphere on a water-cooled Cu crucible. The melted

ingots were flipped and re-melted four times in order to minimize composition variation within the specimens. The phase constituents of the alloys were characterized by a D/MAX-3C X-ray diffractometer using Cu $K_{\alpha 1}$ radiation ($\lambda = 0.1541$ nm). Compression specimens 3 mm in diameter and 6 mm long were cut by electrical-discharge machining (EDM). The compression tests were carried out at a constant strain rate of $1 \times 10^{-3} \text{ s}^{-1}$ in a CMT5305 electronic universal testing machine at room temperature. Metallographic structure of the alloys and the fracture surfaces of compression specimens were examined using a scanning electron microscope (SEM, JSM-7001) equipped with energy-dispersive X-ray spectroscopy (EDXS). Transmission electron microscope (TEM) specimens were cut by EDM from the as-cast ingots, ground and thinned by electropolishing in a solution of 65 vol.% methanol, 30 vol.% butanol, and 5 vol.% perchloric acid at a voltage of 30 V and temperatures between -20° and -30°C . TEM bright field (BF) imaging and selected area electron diffraction (SAED) were performed in a Philips CM 200 ST microscope with a field emission gun operating at an acceleration voltage of 200 kV.

3. Results and discussion

Fig. 1 shows the X-ray diffractograms of the $\text{Ti}_{86.5}\text{Si}_{13.5}$ and $(\text{Ti}_{86.5}\text{Si}_{13.5})_{97}\text{Nb}_3$ alloys. X-ray diffraction (XRD) patterns reveal that both alloys were found to consist of the hexagonal α -Ti (space group: $P6_3/mmc$, A3 structure) solid solution and the ordered hexagonal Ti_5Si_3 (space group: $P6_3/mcm$, $D8_8$ structure) intermetallic compound. Addition of 3 at.% Nb does not change the phase constitution of $(\text{Ti}_{86.5}\text{Si}_{13.5})_{97}\text{Nb}_3$ alloy. The lattice parameters of the α -Ti solid solution of the $\text{Ti}_{86.5}\text{Si}_{13.5}$ and $(\text{Ti}_{86.5}\text{Si}_{13.5})_{97}\text{Nb}_3$ alloys are $a = 0.2965$ nm, $c = 0.4705$ nm and $a = 0.2968$ nm, $c = 0.4716$ nm, respectively, which is larger than that of pure α -Ti ($a = 0.2944$ nm, $c = 0.4678$ nm) due to dissolution with Si and Nb [22]. The chemical

Fig. 1. XRD diffractograms of (a) $\text{Ti}_{86.5}\text{Si}_{13.5}$ and (b) $(\text{Ti}_{86.5}\text{Si}_{13.5})_{97}\text{Nb}_3$ alloys.

composition of the α -Ti and Ti_5Si_3 phases in $\text{Ti}_{86.5}\text{Si}_{13.5}$ and $(\text{Ti}_{86.5}\text{Si}_{13.5})_{97}\text{Nb}_3$ alloys measured by EDXS is shown in Table 1. The lattice parameters for Ti_5Si_3 in $\text{Ti}_{86.5}\text{Si}_{13.5}$ alloy are determined to be $a = 0.7492$ nm, and $c = 0.5136$ nm. Nb addition results in lattice expansion for both the a - and c -directions compared to the $\text{Ti}_{86.5}\text{Si}_{13.5}$ alloy, the lattice parameters for Ti_5Si_3 in $(\text{Ti}_{86.5}\text{Si}_{13.5})_{97}\text{Nb}_3$ alloy are $a = 0.7498$ nm and $c = 0.5141$ nm. From the detailed analysis of these data, it is observed that Nb addition to $\text{Ti}_{86.5}\text{Si}_{13.5}$ alloy causes larger lattice mismatch between the A3 α -Ti and D8₈ Ti_5Si_3 phases.

Table 1

EDXS of the chemical composition (at.%) of the α -Ti and Ti_5Si_3 phases.

Sample	Primary α -Ti dendrite			α -Ti in eutectic matrix			Ti_5Si_3 in eutectic matrix		
	Ti	Si	Nb	Ti	Si	Nb	Ti	Si	Nb
$\text{Ti}_{86.5}\text{Si}_{13.5}$	—	—	—	84-89	11-16	—	65-78	22-35	—
$(\text{Ti}_{86.5}\text{Si}_{13.5})_{97}\text{Nb}_3$	91-94	2-4	4-5	78-87	11-18	2-4	65-69	27-33	2-4

Fig. 2a and b (higher magnification of (a)) present SEM backscattered electron (BSE) images of the eutectic $\text{Ti}_{86.5}\text{Si}_{13.5}$ alloy, displaying a typical rod-like eutectic structure with a rod spacing of 150-600 nm with the Ti_5Si_3 rods being 150-500 nm in diameter. EDXS analysis indicated that the brighter rod-like phase is Ti_5Si_3 , and the dark contrast area is α -Ti. A SEM BSE micrograph shown in Fig. 2c reveals a bimodal phase size distribution with primary α -Ti dendrites embedded in an ultrafine

matrix for the $(\text{Ti}_{86.5}\text{Si}_{13.5})_{97}\text{Nb}_3$ alloy. With higher magnification, Fig. 2d, it is seen that the diameter of the rod-like Ti_5Si_3 phase is about 150-700 nm. The rod spacing of the eutectic structure in the $(\text{Ti}_{86.5}\text{Si}_{13.5})_{97}\text{Nb}_3$ alloy (100-500 nm) is finer than that in the $\text{Ti}_{86.5}\text{Si}_{13.5}$ alloy. EDXS measurements shown in Table 1 reveal that the solubility of Si in α -Ti in the Ti-Si eutectic alloy is 11-16 at.%, indicating that the α -Ti is a solid solution. Si content in the Ti_5Si_3 phase in the Ti-Si eutectic alloy is 22-35 at.%, which is lower than the range shown in the binary Ti-Si phase diagram [21]. The solubility of Nb in primary α -Ti dendrites, eutectic α -Ti, and Ti_5Si_3 phases is 2-5 at.%. The Si content in eutectic α -Ti and Ti_5Si_3 phases in $(\text{Ti}_{86.5}\text{Si}_{13.5})_{97}\text{Nb}_3$ shows a similar value in comparison with the $\text{Ti}_{86.5}\text{Si}_{13.5}$ alloy. Therefore, it is clear that Nb acts as a substitutional impurity on Ti sites in the α -Ti solid solution and Ti_5Si_3 phase.

Fig. 2. SEM BSE micrographs of (a) $\text{Ti}_{86.5}\text{Si}_{13.5}$, (c) $(\text{Ti}_{86.5}\text{Si}_{13.5})_{97}\text{Nb}_3$ alloys, (b) and (d) high magnification of (a) and (c).

Stress-strain curves at room temperature for $\text{Ti}_{86.5}\text{Si}_{13.5}$ and $(\text{Ti}_{86.5}\text{Si}_{13.5})_{97}\text{Nb}_3$ alloys are shown in Fig. 3. The eutectic $\text{Ti}_{86.5}\text{Si}_{13.5}$ alloy has an ultimate strength of 1078 MPa and a plastic strain of 10% (fracture strain: 12%) in compression. The $(\text{Ti}_{86.5}\text{Si}_{13.5})_{97}\text{Nb}_3$ alloy displayed a substantially higher plastic strain of 12% (fracture strain: 14%) and an ultimate strength of 1180 MPa. Therefore, the Nb addition in $(\text{Ti}_{86.5}\text{Si}_{13.5})_{97}\text{Nb}_3$ alloy significantly improves compressive room temperature ductility and ultimate strength *vis-à-vis* $\text{Ti}_{86.5}\text{Si}_{13.5}$. Fig. 4 shows SEM secondary electron (SE) images of the fracture surface of the $\text{Ti}_{86.5}\text{Si}_{13.5}$ and $(\text{Ti}_{86.5}\text{Si}_{13.5})_{97}\text{Nb}_3$ alloys after compression at room temperature. The pull out of the Ti_5Si_3 phases and the relatively flat-faceted fracture surface (Fig. 4a and b (higher magnification of (a))) indicate little plastic flow before failure in eutectic $\text{Ti}_{86.5}\text{Si}_{13.5}$ alloy. The final fracture will take place by cleavage. The fracture surface (Fig. 4c and d (higher magnification of (c))) of $(\text{Ti}_{86.5}\text{Si}_{13.5})_{97}\text{Nb}_3$ alloy shows an apparent change in fractographic appearance. The dimple-like pattern in the fracture surface is evidence of ductile deformation.

Fig. 3. Stress-strain curves of (a) $\text{Ti}_{86.5}\text{Si}_{13.5}$ and (b) $(\text{Ti}_{86.5}\text{Si}_{13.5})_{97}\text{Nb}_3$ alloys.

Fig. 4. SEM SE images of the fracture surfaces of (a) $\text{Ti}_{86.5}\text{Si}_{13.5}$, (c) $(\text{Ti}_{86.5}\text{Si}_{13.5})_{97}\text{Nb}_3$ alloys, (b) and (d) high magnification of (a) and (c).

Fig. 5a is a TEM BF image of the eutectic $\text{Ti}_{86.5}\text{Si}_{13.5}$ alloy showing a typical rod-like structure. The rod spacing and the diameter of the Ti_5Si_3 rods are measured to be about 100-400 nm and 100-300 nm, respectively. Fig. 5b is the SAED pattern of the Ti_5Si_3 rod in Fig. 5a along the $[\bar{1}\bar{1}26]$ zone axis. Indexing showed that the Ti_5Si_3 phase has a D8_8 structure with lattice parameter $a = 0.749$ nm, and $c = 0.514$ nm. Fig. 5c is the SAED pattern of the α -Ti in Fig. 5a along the $[5\bar{1}\bar{4}\bar{3}]$ zone axis; from this we deduced that the hexagonal α -Ti structure has lattice parameters of $a = 0.297$ nm and $c = 0.472$ nm. Fig. 5d shows the SAED patterns of Ti_5Si_3 and α -Ti, yielding the orientation relationship $(1\bar{1}00)[\bar{1}\bar{1}26] \text{Ti}_5\text{Si}_3 \parallel (01\bar{1}1)[5\bar{1}\bar{4}\bar{3}] \alpha\text{-Ti}$. Electron diffraction reveals that the Ti_5Si_3 and α -Ti phases in the $(\text{Ti}_{86.5}\text{Si}_{13.5})_{97}\text{Nb}_3$ alloy have

the same SAED patterns as those of Fig. 5b and c. The crystallographic orientation relationship of the Ti_5Si_3 and $\alpha\text{-Ti}$ phases observed in the $(\text{Ti}_{86.5}\text{Si}_{13.5})_{97}\text{Nb}_3$ alloy is in good agreement with that obtained from the SAED patterns of Fig. 5d in the eutectic $\text{Ti}_{86.5}\text{Si}_{13.5}$ alloy.

Fig. 5. (a) TEM BF image of (a) $\text{Ti}_{86.5}\text{Si}_{13.5}$ alloy, SAED patterns of (b) $\text{D8}_8 \text{Ti}_5\text{Si}_3$ and (c) $\text{A3 } \alpha\text{-Ti}$ phases corresponding to $[\bar{1} \bar{1} 26] \text{Ti}_5\text{Si}_3$ and $[5 \bar{1} \bar{4} \bar{3}] \alpha\text{-Ti}$ zone axes, and (d) composite electron diffraction patterns of $\alpha\text{-Ti}$ and Ti_5Si_3 showing their orientation relationship.

Si and Nb are known as α - and β -Ti stabilizer elements, respectively.

Dobromyslov et al. [23] reported that the minimum concentration limit of the alloying metal Nb necessary for the complete stabilization of the β -Ti in binary Ti-Nb alloy is about 23 at.%. Therefore, there is only α -Ti(Si,Nb) phase formation with the addition of 3 at.% Nb in the $(\text{Ti}_{86.5}\text{Si}_{13.5})_{97}\text{Nb}_3$ alloy, which has been confirmed by XRD and SEM results shown in Fig. 1 and 2. The lattice parameters of the α -Ti in the eutectic $\text{Ti}_{86.5}\text{Si}_{13.5}$ and $(\text{Ti}_{86.5}\text{Si}_{13.5})_{97}\text{Nb}_3$ alloys are larger than those of pure α -Ti ($a = 0.2944$ nm, $c = 0.4678$ nm) due to dissolution of Si and Nb (Table 1). XRD analysis indicates that Nb addition leads to the expansion of the lattice parameters in both the a - and c -directions for the Ti_5Si_3 phase. The lattice parameters of the Ti_5Si_3 phase in the Nb-modified $(\text{Ti}_{86.5}\text{Si}_{13.5})_{97}\text{Nb}_3$ alloy were larger than those in the eutectic $\text{Ti}_{86.5}\text{Si}_{13.5}$ alloy due to the Nb substitution for part of the Ti in Ti_5Si_3 crystals, as shown in Table 1. The observed maximum amounts of Nb and Cr dissolved in Ti_5Si_3 phase reportedly can reach values as high as 21 and 12 at.% [16], respectively.

The precipitation behavior of Ti_5Si_3 during tempering of quenched Ti-2 wt.% (3.4 at.%) Si alloy showed that two forms of the silicide are observed [11]. Higher tempering temperatures ($T > 973\text{K}$) favor the formation of rods, while lower temperatures (773-873 K) favor formation of an hexagonal plate morphology. The orientation relationships of the rod-like and hexagonal plate Ti_5Si_3 with α -Ti are $(\bar{1}2\bar{1}1)[1\bar{2}16] \text{Ti}_5\text{Si}_3 \parallel (\bar{1}2\bar{1}0)[0001] \alpha\text{-Ti}$, and $(0001)[10\bar{1}0] \text{Ti}_5\text{Si}_3 \parallel (0001)[21\bar{3}0] \alpha\text{-Ti}$, respectively. However, a different orientation relationship of $\text{D8}_8 \text{Ti}_5\text{Si}_3$ with $\text{A3} \alpha\text{-Ti}$ in binary and Nb-modified Ti-Si alloys was observed in the present research, which is $(1\bar{1}00)[\bar{1}\bar{1}26] \text{Ti}_5\text{Si}_3 \parallel (01\bar{1}1)[5\bar{1}\bar{4}\bar{3}] \alpha\text{-Ti}$. The orientation relationship between Ti_5Si_3 and $\alpha\text{-Ti}$ determined above is analyzed in terms of the coincidence site lattice model [24], i.e., by fitting corresponding lattice planes on both sides of the

interface. The calculated lattice mismatches with room temperature lattice parameters in two different directions in the $\text{Ti}_{86.5}\text{Si}_{13.5}$ and $(\text{Ti}_{86.5}\text{Si}_{13.5})_{97}\text{Nb}_3$ alloys are 4.12 and 4.17 %, respectively, yielding low lattice strains. This suggests that there is a slight increase in the lattice mismatch due to the addition of Nb.

Typical eutectic structures are rod-like, lamellar, globular, and acicular [25]. Fig. 2a and b indicate that the eutectic $\text{Ti}_{86.5}\text{Si}_{13.5}$ alloy shows a rod-like morphology. The rod-like structures present in the eutectic $\text{Ti}_{86.5}\text{Si}_{13.5}$ alloy could be modified by the addition of Nb to obtain a bimodal structure with micrometer-sized primary α -Ti dendrites embedded in the ultrafine matrix (α -Ti + Ti_5Si_3) as shown in Fig. 2c. The reduced eutectic rod spacing of α -Ti and Ti_5Si_3 shown in Fig. 2c and d in the $(\text{Ti}_{86.5}\text{Si}_{13.5})_{97}\text{Nb}_3$ alloy results from a decrease of interface energy caused by the addition of Nb. Park et al. reported a reduced lamellar spacing in binary Fe-Nb alloys with Al additions [26]. The precipitation of the primary α -Ti dendrites in the $(\text{Ti}_{86.5}\text{Si}_{13.5})_{97}\text{Nb}_3$ alloy compared with the Ti-Si eutectic results in both increased strength and ductility. The $(\text{Ti}_{86.5}\text{Si}_{13.5})_{97}\text{Nb}_3$ alloy exhibited a substantially higher plastic strain of 12% while preserving an ultimate strength of 1180 MPa.

The eutectic $\text{Ti}_{70.5}\text{Fe}_{29.5}$ alloy with the addition of 3 at.% Nb also shows a bimodal structure with micrometer-sized primary β -Ti dendrites embedded inside an ultrafine eutectic (β -Ti+TiFe). For the $(\text{Ti}_{70.5}\text{Fe}_{29.5})_{97}\text{Nb}_3$ alloy, the compression specimen fractured at a plastic strain of 15% and a strength of 2.53 GPa, while for the $\text{Ti}_{70.5}\text{Fe}_{29.5}$ alloy the plastic strain is 7% and the strength is 2.14 GPa in compression [27]. Similar results have been reported for a $\text{Ti}_{60}\text{Cu}_{14}\text{Ni}_{12}\text{Sn}_2\text{Nb}_{10}$ alloy with an *in-situ* formed β -Ti dendrite/nanostructured matrix microstructure. That alloy had a high compressive strength of 2.4 GPa and 14.5% plastic strain to failure at room temperature [4]. We believe that the formation of a bimodal structure and the good

compatibility between the α -Ti (A3) and Ti_5Si_3 (D8₈) structures are responsible for the improved ductility of the Nb-modified Ti-Si eutectic alloy. The larger lattice mismatch between the A3 and D8₈ phases in the $(\text{Ti}_{86.5}\text{Si}_{13.5})_{97}\text{Nb}_3$ alloy due to the addition of Nb may relieve the dislocation pile-ups by slip transfer across the A3/D8₈ interface [28,29], thereby improving the ductility of the $(\text{Ti}_{86.5}\text{Si}_{13.5})_{97}\text{Nb}_3$ alloy.

4. Conclusions

Nb-modified ultrafine Ti-Si eutectic alloys were fabricated by cold crucible levitation melting, and their mechanical properties were tested in compression at room temperature. Microstructural investigations indicated that the rod-like structure present in the eutectic $\text{Ti}_{86.5}\text{Si}_{13.5}$ alloy could be modified by the addition of Nb to obtain a bimodal structure with micrometer-sized primary α -Ti dendrites embedded in the refined ultrafine eutectic matrix. The enhanced plasticity of the Nb-modified Ti-Si eutectic alloy is attributed to the bimodal structure, reduced ultrafine rod spacing, and the compatibility between the α -Ti (A3) and Ti_5Si_3 (D8₈) structures. The $(\text{Ti}_{86.5}\text{Si}_{13.5})_{97}\text{Nb}_3$ alloy exhibited a substantially higher plastic strain (12%) while preserving high ultimate strength (1180 MPa). The orientation relationship of D8₈ Ti_5Si_3 with A3 α -Ti is $(1\bar{1}00)[\bar{1}\bar{1}26] \text{Ti}_5\text{Si}_3 \parallel (01\bar{1}1)[5\bar{1}\bar{4}\bar{3}] \alpha\text{-Ti}$.

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