Effect of Ti content on the microstructure and mechanical behavior of
(Fe₃₆Ni₁₈Mn₃₃Al₁₃)_{100-x}Ti_x high entropy alloys

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Abstract

The microstructure and mechanical properties studies of a series of two-phase f.c.c./B2 (ordered b.c.c.) lamellar-structured, high entropy alloys (HEA) Fe₃₆Ni₁₈Mn₃₃Al₁₃Ti_x with x up to 6 at. % Ti have been investigated. X-ray microanalysis in a TEM showed that the Ti resided mostly in the B2 phase. The lamellar spacing decreased significantly with increasing Ti content from 1.56 μm for the undoped alloy to 155 nm with an addition of 4 at. % Ti, leading to a sharp increase in room-temperature yield strength, σ_y , from 270 MPa to 953 MPa, but with a concomitant decrease in ductility from 22% elongation to 2.3%. Annealing at 1173 K for 20 h greatly increased the lamellar spacing of Fe₃₆Ni₁₈Mn₃₃Al₁₃Ti₄ to 577 nm, producing a corresponding decrease in σ_y to 511 MPa. The yield strengths of all the doped alloys decreased significantly when tensile tested at 973 K with a concomitant increase in ductility due to softening of the B2 phase. The fracture mode changed from cleavage at room temperature to a ductile dimple-type rupture at 973 K. The results are discussed in terms of the Hall-Petch-type relationship.

Key words: High-entropy alloys; Microstructure; Mechanical properties; Hall-Petch-type relationship

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1. Introduction

High entropy alloys (HEAs), which are designed to contain several elements (≥ 5) to obtain a high mixing entropy, can exhibit good structural properties and, thus, show promise for engineering applications [1–5]. Initially, HEAs were originally designed based on the criterion that they contained at least five principal elements in equal or near equal atomic concentrations [6]. For example, an equiatomic CoCrFeMnNi HEA with the face-centered cubic (f.c.c.) crystal structure was produced [7,8] and its tensile properties in the temperature range 77-1073K was investigated by Otto et al. [7]. The alloy showed the highest strength and ductility at 77 K due to the formation of nanoscale deformation twins. Similarly, the $V_{20}Nb_{20}Mo_{20}Ta_{20}W_{20}$ refractory HEA with the body-centered cubic (b.c.c.) crystal structure, produced by Senkov et al. [9], exhibited a yield stress of up to 1246 MPa. Recently, Yao et al. [10] and Deng et al. [6] introduced the non-equiatomic $Fe_{40}Mn_{27}Ni_{26}Co_5Cr_2$ HEA and $Fe_{40}Mn_{40}Co_{10}Cr_{10}$ multicomponent alloys, respectively. Deformation twinning occurred at room temperature for the $Fe_{40}Mn_{40}Co_{10}Cr_{10}$ alloy, leading to excellent mechanical properties that were comparable to those of advanced TWIP steels [6].

Several studies have shown that the microstructure and mechanical properties of lamellar-structured two-phase FeNiMnAl alloys consisting of (Fe, Mn)-rich f.c.c. and (Ni, Al)-rich B2 (ordered b.c.c. structure) phases depend on the Al content [11–13]. The lamellar spacing increases with the decreasing Al content in the range of 15 to 13 at. %, leading to a decrease in strength but an increase in ductility. Further decreases in Al content (11-12 at. %) produce dendritic microstructures with lower strength, but higher ductility [13], while increases in Al content (20-21 at. %) produce a very fine (50 nm

phase width) aligned B2/f.c.c. microstructure that shows yield strengths up to 1180 MPa, but much poorer ductility [11].

The effect of alloying $\text{Fe}_{30}\text{Ni}_{20}\text{Mn}_{35}\text{Al}_{15}$ with up to 8 at. % Cr was investigated by Meng et al. [14,15] who found that the Cr alleviates the environmental embrittlement due to the formation of protective chromia-containing oxide scales on the surface. Cr additions up to 6 at. %, which produced little change in the lamellar structure, increased the room temperature elongation to failure significantly (from 8.5 % to 18 %) but slightly decreased the yield strength (from 820 MPa to 679 MPa). Later, Meng and Baker [16] performed nitriding treatment on the resulting HEA $\text{Fe}_{28.2}\text{Ni}_{18.8}\text{Mn}_{32.9}\text{Al}_{14.1}\text{Cr}_6$ to harden the surface through the formation of AlN precipitates.

In this paper, we present the **effect** of Ti additions on the microstructure of a similar alloy, i.e. $(\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13})_{100-x}\text{Ti}_x$ ($x = 0-6$ in at. %). Unlike Cr, we show that Ti increases the strength dramatically, largely through decreasing the lamellar spacing based on a Hall-Petch-type relationship. In addition, tensile tests were performed at 973 K to explore the higher temperature properties for the $(\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13})_{100-x}\text{Ti}_x$ (designated as $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ hereafter for simplicity) HEAs.

2. Experimental

Ingots of $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ with different amounts of Ti (0, 2, 4, 6 at. %) were arc melted from **high-pure metals (purity of 99.99 % Fe, 99.95 % Ni, 99.9 % Mn, 99.9 % Al and 99.9 % Ti)**. Since Mn tends to be lost by evaporation during melting, an additional 5 wt. % of Mn was added to each ingot to compensate. The pieces of metals were melted in a water-cooled copper crucible surrounded by argon. To ensure the homogeneous mixing

of the elements, the ingots were flipped over after each melting and were remelted three times in total. The as-cast alloys were annealed at either 823 K for 24 h, or 1173 K for 20 h, and cooled in air, depending on the composition.

Synchrotron X-ray diffraction (XRD) measurements were performed on the as-cast alloys at the Advanced Photon Source (APS) at the Argonne National Laboratory undulator beamline 2-ID-D with an X-ray photon energy of 18 keV (wavelength = 0.61992 nm). The total counting time for each diffraction pattern measurement was from 30-60 s. Accurate lattice constants were obtained by using the extrapolated function $f(\theta) = \frac{\cos^2\theta}{2} \left(\frac{1}{\sin\theta} + \frac{1}{\theta} \right)$ [17] for the lattice parameters calculated from individual diffraction peaks.

A scanning electron microscope (SEM) was used to study the microstructure of the alloys. Specimens were ground through increasingly fine grades of silicon carbide paper, polished using 0.3 μm alumina powder, followed by polishing using a suspension of colloidal silica with the particle size of 60 nm in a Vibromet polishing machine. The polished specimens were examined in an FEI XL30 field emission gun (FEG) SEM in the backscattered electron (BSE) mode, while the secondary electron (SE) mode was used for imaging the fracture surfaces of specimens after tensile tests. The f.c.c. phase fraction and the lamellar spacings of the two phases were obtained by analyzing the BSE images using Image J.

For examination in the transmission electron microscope (TEM), 3-mm diameter and 150- μm thickness disks were electro-polished by a Struers Tenupol 5 using an electrolyte containing 25% nitric acid in methanol at a temperature of -20 $^{\circ}\text{C}$, and an applied voltage of 10 V and current of 80 mA. The resulting thin foils were examined in a FEI Tecnai

F20 FEG TEM equipped with energy dispersive X-ray spectrometry (EDS) made by EDAX and operated at 200 kV. The library standards in the GENESIS software were applied to perform the quantification analysis based on EDS results.

The Vickers hardness of the specimens was measured with a Leitz MINIlload tester using a load of 1.96 N (200g). Flat dog-bone specimens with a gauge length of 10 mm, width of ~ 2.6 mm, and thickness of ~ 1.1 mm were used for tensile testing. The specimens were ground through 600 grit SiC paper and polished using 0.3 μm alumina powder. Tensile tests were performed at both room temperature and 973 K using an Instron at an initial strain rate of $5 \times 10^{-4} \text{ s}^{-1}$. The elongation to fracture was determined by measuring the gauge length before and after tensile testing using an optical microscope.

3. Result

SEM images of the $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ HEAs in both as-cast and annealed state are shown in Fig. 1. Fig. 1 (a) shows the lamellar microstructure of the Ti-free alloy. Based on previous observations of FeNiMnAl alloys [13], the phase with bright gray contrast is f.c.c. and the phase with dark gray contrast is B2. With Ti additions of up to 4 at. %, the alloys still exhibit lamellar microstructure (see Fig. 1 (b) and (d)), with the lamellar spacing decreasing significantly with increasing Ti. When the Ti addition was increased to 6 at. %, the lamellar structure disappears, see Fig. 1(f).

The alloys with 2 at. % and 4 at. % Ti were also subjected to various anneals to assess the thermal stability of the microstructures. Comparing Fig. 1 (b) with Fig. 1 (c), it is evident that the microstructure is largely unchanged by the anneal at 823 K for 24 h,

while obvious lamellar coarsening occurred after annealing at 1173 K for 20 h (compare Fig. 1 (d) and (e)). Table 1 summarizes measurements of the lamellar spacing and f.c.c. phase volume fraction. The alloys with smaller lamellar spacing had larger volume fraction of f.c.c. phase, which was consistent with our previous observation [13]. The lamellar spacing of the $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ HEAs is far more sensitive to the Ti content than the f.c.c. phase fraction, though both decreased with increasing Ti: the lamellar spacing of Ti- free alloy was 1560 nm, and this decreased to 155 nm, when 4 at. % Ti was added, although the volume fraction of the f.c.c. phase only varied from 85 % to 66 %.

The Synchrotron XRD patterns in Fig. 2 indicate that $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ alloys consist of only two phases: f.c.c. and B2. Fig. 3 shows the lattice constant for f.c.c. phases and B2 phases in $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ HEAs calculated from the XRD patterns. It should be noted that one peak at $\sim 10.5^\circ$ is unable to be indexed due to the limited information. The lattice constant of the f.c.c. phase is almost unchanged, while for the B2 phase the lattice constant almost increases linearly with increasing Ti content.

The bright field (BF) TEM images in Fig. 4 show the microstructure of the $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ HEAs. Again, the reduction in spacing with increasing Ti is evident. The X-ray spectra from as-cast $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_6$ in Fig. 4 e and f indicate that both phases contained all elements, but it is evident that the Ti strongly partitions to the B2 phase. Table 2 lists the compositions of phases in the as-cast $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ HEAs. Larger amounts of Fe and Mn were found in the f.c.c. phases, while the B2 phases was enriched in Ni and Al, showing similar results to a previous study [13]. The BF TEM image of $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_2$ HEAs annealed at 823 K for 24 h (Fig.4b) shows that no

precipitation occurred during annealing. The selected area diffraction (SAD) patterns in Fig. 4b and Fig. 4d confirm that the matrix phase with light gray contrast is f.c.c. and phase with dark gray contrast is B2, respectively.

Typical stress-strain curves for the $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ HEAs in both as-cast and annealed states are shown in Fig. 5. The room temperature yield strength and ultimate tensile strength (fracture strength) along with Vickers hardness, increases sharply with increasing Ti, i.e., the yield strength increased from 270 MPa for the Ti-free material up to 953 MPa for alloys with 4 at. % Ti. The elongation to the failure, exhibits the opposite trend, decreasing from 22.8 % when no Ti was present to 2.3 % when 4 at. % Ti was added. A slight increase in room temperature yield strength and hardness was observed after annealing $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_2$ and $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_4$ at 823 K, see Table 3. The yield strength and fracture strength, as well as the hardness values of $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_4$ decreased dramatically after annealing at 1173 K for 20 h, resulting from the coarsening of the phases; yet the anneal only led to a limited increase in the ductility. Table 3 summarizes the Vickers hardness, room-temperature strength and ductility of the $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ HEAs in both as-cast and annealed state.

Tensile tests were also performed at 973K at an initial strain rate of $5 \times 10^{-4} \text{ s}^{-1}$ for as-cast $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_2$, as-cast $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_4$, and as-cast $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_6$ (see Figure 5(b)), and the yield strength and elongation to failure are shown in Fig. 6. Both the yield strength and fracture strength of the $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ alloys were lower compared with their room-temperature strength, and the lack of work-hardening at elevated temperature resulted in only small differences between the yield strength and the fracture strength. The dependence of yield strength on Ti content disappears at 973 K,

while the elongation to failure continued to decrease with increasing Ti content; see Fig. 6.

Fig.7 shows the fracture surfaces of $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ HEAs after tensile testing at room temperature. The fracture surfaces of both as-cast and annealed Ti-doped HEAs appear to be largely transgranular cleavage (see Fig. 7 b and c), which is consistent with the elongation values shown in Table 3. The fracture surfaces of the alloys tensile tested at 973 K in Fig. 8 show numerous dimples, indicating ductile fracture occurred at elevated temperature tests.

4. Discussion

4.1 Microstructure evolution

The lamellar structures of two-phase FeNiMnAl alloys are extremely sensitive to the processing conditions and chemical composition, as shown in Fig.1. Liao and Baker [18] found that the lamellar spacing of the eutectic alloy $\text{Fe}_{30}\text{Ni}_{20}\text{Mn}_{35}\text{Al}_{15}$ alloys decreased with the increasing cooling rate after melting. Meng et al. [13] investigated the microstructure of lamellar-structured FeNiMnAl alloys with the varying Al content, and found that decreasing the Al content in the range of 15-13 at. % resulted in dramatic increases in the f.c.c. lamellar spacing. In this study, the Ti in $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ HEAs exhibited a similar effect on the microstructure (see Fig. 9), i.e., the greater the Ti content in the alloys, the smaller the lamellar spacing. But an increase in the Ti content to 6 at. % Ti caused a change in microstructure and the lamellar structure was no longer found. Likewise, an increase in the Al content in as-cast $\text{Fe}_{29}\text{Ni}_{19}\text{Mn}_{34}\text{Al}_{18}$ [19] and $\text{Fe}_{28}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{21}$ [11] produced aligned microstructure rather than lamellae.

Previous studies on the effects of Cr on $\text{Fe}_{30}\text{Ni}_{20}\text{Mn}_{35}\text{Al}_{15}$ alloys indicated that most of Cr residing in the f.c.c. phase [15]. Chromium additions slightly lower the yield strength but increase the ductility with little change in the microstructure. Yet the Ti concentrates into B2 phase in this study (see EDS results in Table 2), resulting in the decrease of lamellar spacing and the increase of strength.

Meng and Baker [20] showed that in the $\text{Fe}_{28}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{21}$ alloy, β -Mn-structured precipitates are produced by annealing at temperatures $\leq 1073\text{K}$, while dramatic coarsening of the two-phase B2/f.c.c. microstructure occurred upon annealing at temperatures $\geq 1073\text{K}$ without the formation of β -Mn-structured precipitates. Hence, in this study, the $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ alloys were annealed at 823 K and 1173 K to investigate their thermal stability. The microstructures of the alloys show little change after annealing at 823 K (see Fig. 1 b and c) with no β -Mn precipitation. In contrast, the lamellar spacing of $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_4$ increased from 155 nm to 577 nm after the 20 h anneal at 1173 K (comparison of Fig. 1 d and e); indicating that phase coarsening occurs at temperatures $\geq 1173\text{ K}$.

The lattice constant of the B2 phases was found to increase almost linearly with increasing Ti content in the $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ HEAs, see Fig. 3. EDS results in Table 2 also show that the Ti content in the B2 phase (from 0, 3.9, 8.2 to 12.4 at. %) also increases almost linearly with increasing Ti in $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ alloys (from 0, 2, 4 to 6 at. %). Thus, the influence of Ti content on the lattice constant for B2 phases can be understood by Vegard's law [21]. The lattice parameters (a) of B2 phase can be expressed as a function of Ti content (x) by considering two parts of B2 phase-with Ti and Ti-free: $a = (1 - x)a_{\text{B2 without Ti}} + xa_{\text{B2 with Ti}}$, which is $a = 2.9053 + 0.0018x$ based

on a linear fit of the data in this study. Similar phenomena has been observed by He et al. [22] in $(\text{FeCoNiCrMn})_{100-x}\text{Al}_x$ HEA, of which the lattice constant shows a linear increase with increasing of Al in the single f.c.c. solid solution phase region. On the other hand, Fig. 3 shows that the lattice constant of f.c.c. phases remains unchanged with the different additions of Ti in $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ HEAs. Two reasons account for the latter phenomenon. First, only a very small amount of Ti is present in the f.c.c. phases based on the EDS results, i.e., only 2 at. % Ti was found in f.c.c. phase when 6 at. % Ti was added. Secondly, Fe and Mn with larger atomic radius are enriched in the f.c.c. phase, both of which have a similar atomic radius of 140 pm to Ti (see Table 4). The replacement of Fe and Mn by Ti leads to almost no change in lattice parameters. Tung et al. [23] also found that in AlCoCrCuFeNi HEA, the lattice parameters of the individual phase remain the same when varying the content of elements since the variation of elements has little influence on the lattice parameters of phase with a substantial fraction of large atoms.

4.2 Mechanical behavior at room temperature

Liao and Baker [12] showed that B2 phase is much harder than the f.c.c. phase at room temperature in lamellar-structured two-phase FeNiMnAl alloys, and thus, dislocation motion was confined to the f.c.c. phase during *initial* plastic deformation. Therefore, a Hall-Petch-type relationship between strength and f.c.c. lamellar spacing, λ , was exhibited in FeNiMnAl alloys [11,13]. For the $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ HEAs the room-temperature yield strength and elongation are plotted as a function of $\lambda^{-1/2}$ and λ^{-1} in Figure 10.

Hall [24] and Petch [25] suggested that the yield stress, σ_y , related to the grain size, d , through:

$$\sigma_y = \sigma_0 + k\lambda^{-1/2} \quad (1)$$

Where σ_0 is the lattice friction stress, and k is a constant. Following this approach, a plot of the yield stress versus $\lambda^{-1/2}$ with a correlation coefficient R^2 of 0.99 is shown in Fig. 10a, suggesting an almost perfect linear fitting here.

However, for the $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ HEAs, a negative, nonphysical value of σ_0 was produced in the above plot of σ_y versus $\lambda^{-1/2}$. A similar phenomenon occurred in the eutectic FeNiMnAl alloys over a range of Al content [13] and some pearlitic steels [26–28], when the Hall-Petch relationship was applied. Dollar et al. [26] found that in these pearlitic steels the stress at the tip of pile-ups is relatively low, which is unable to initiate the yielding. Instead, the pearlite yielding is proposed to be controlled by the onset of deformation in the ferrite matrix. The movement of dislocations is restricted in the ferrite between two cementite walls. The critical stress ($\Delta\sigma$) to maintain such movement is related to the ferrite shear modulus (G), lattice dislocation Burgers vector (b) and lamellar spacing (λ), and is given by: $\Delta\sigma \sim 3 \cdot Gb\lambda^{-1}$. Thus, the pile-up model is no longer suitable for the yield strength of pearlite and the alternative relationship

$$\sigma_y = \sigma_0' + k'\lambda^{-1} \quad (2)$$

was used to plot the data (see Fig. 10c). According to this modified relationship, the equation becomes: $\sigma_y = 301 + 0.1\lambda^{-1}$, where the value of k' at 0.1 N/mm here is close to that in pearlitic steels (0.06 N/mm) [26].

It is interesting to note that, based on this modified Hall-Petch relationship, the lattice friction stress σ_0' of the f.c.c. phase for $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ HEAs is ~ 50 MPa higher than that in FeNiMnAl alloys system [13]. EDS results in Table 2 show 1-2 at. % Ti was dissolved in the f.c.c. phase, indicating that this produced substitutional solid solution

strengthening. Zhou et al. [29] showed an excellent solid solution strengthening effect of Ti in AlCoCrFeNiTi_x HEAs, and found that the mechanical properties of $\text{AlCoCrFeNiTi}_{0.5}$ are even superior to most of the high-strength alloys such as bulk metallic glasses. Traditionally, solid-solution-hardening was obtained when the dislocation mobility in crystalline materials is hindered by the introduction of solute atoms [30]. Various types of dislocation-solution interactions, i.e., elastic interaction and a modulus interaction, that occur between a moving dislocation and solute atoms. Elastic interaction, for example, results from the mutual interactions between elastic stress fields around misfitting solute atoms and the core of edge dislocations, of which strengthening is directly related to the misfit of the solute. In our $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ alloys, the interaction energy increases when the solvent atom, i.e., Fe, Ni, Mn or Al, in f.c.c. phase is removed and replaced by Ti atom, leading to the increase in the lattice friction stress. He et al. [22] approximated the alloying effect of Al on the tensile properties of FeCoNiCrMn HEA via a standard model for substitutional solid strengthening [31] i.e. $\Delta\sigma_{ss} = Bc^{1/2}$, where c is the mole ratio of Al and B is a constant determined by the shear modulus of the alloy and the interaction parameter. A good approximation was obtained though the calculated value underestimated the experimental result [22].

The strengthening effects of Ti on the $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ HEAs can largely be ascribed to Hall-Petch-type relationship strengthening through a relationship of the form $\sigma_y = \sigma_0' + k'\lambda^{-1}$ (2). The increase of yield strength, largely resulting from the decrease of the lamellar spacing (λ) as the Ti content is increased. However, the Ti dissolved in the f.c.c. phase, leads to a 50MPa of lattice friction stress σ_0' due to the substitutional solid solution strengthening.

Plots of the elongation to failure versus $\lambda^{-1/2}$ (Fig. 10 b) and λ^{-1} (Fig. 10 d), where λ is the f.c.c. lamellar spacing both show good linear fits. It should be noted that the elongation of $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_4$ after annealing at 1173 K for 20 h was excluded in the plots. After annealing, the strength of $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_4$ alloys dropped dramatically from 953 MPa to 511 MPa due to the coarsening of lamellar spacing, yet surprisingly, the elongation only increased from 2.3 % to 5.6 %, which was far less than the elongation of $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_2$ alloys (13.9 %) with smaller lamellar spacing. Table 1 illustrates that annealing $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_4$ decreases the f.c.c. phase volume fraction to less than that of the $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_2$ alloy. However, the $\text{Fe}_{29}\text{Ni}_{19}\text{Mn}_{38}\text{Al}_{14}$ alloy, which has a similar f.c.c. phase volume fraction to the annealed $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_4$ alloy, exhibited much better ductility [13], i.e., the elongation of the $\text{Fe}_{29}\text{Ni}_{19}\text{Mn}_{38}\text{Al}_{14}$ alloy is 19.5 %, suggesting that the f.c.c. phase volume fraction did not play a dominant role in the ductility of $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ HEAs. As discussed before, the lattice distortion energy of f.c.c. phase increases significantly due to the introduction of Ti atoms, and the elastic interactions strongly restrict the moving of dislocations, leading to a sharp decrease in ductility of f.c.c. phase.

4.3 Mechanical behavior at elevated temperature

Both the yield stress and the fracture strength of the $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ HEAs were much lower at 973 K compared with their values at room temperature. Previous studies of the temperature dependence of $\text{Fe}_{30}\text{Ni}_{20}\text{Mn}_{35}\text{Al}_{15}$ [18] and $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloys [13] showed that the elevated temperature mechanical behavior is dominated by B2 phase. A rapid decrease in yield stress occurs once the temperature is above $0.45 T_m$, where T_m is the melting point, a common feature exhibited by many B2 intermetallic compounds [32].

The B2 phase is impenetrable at room temperature and impedes the dislocation motion, while at high temperature, plastic deformation appears in B2 phase and it gradually loses the dislocation impediment capability. TEM observations revealed that a number of dislocations exist in the B2 phase in $\text{Fe}_{30}\text{Ni}_{20}\text{Mn}_{35}\text{Al}_{15}$ alloys strained at 900 K [18]. Thus, the dramatically drop of the strength for $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ HEAs and the disappearance in the dependence of yield strength on Ti content are largely due to such soften behavior of B2 phase at elevated temperature.

As a typical behavior of B2 compounds [32], the ductility of $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ alloys increases significantly at high temperature, since the B2 phase undergoes plastic deformation and becomes ductile. The elongation of the alloys at 973 K decreases with increasing Ti content, which is the same with the trend at room temperature.

5. Summary

The microstructure and mechanical properties of $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ ($x = 0-6$ at. %) HEAs in both as-cast and annealed state have been investigated. The main conclusions are as follows:

1. For $x \leq 4$ at. %, an increase in the Ti content produces a decrease in lamellar spacing. Further increases in the Ti content to 6 at. % results in the disappearance of the lamellar microstructure.
2. The yield strength and lamellar spacing obey a Hall-Petch-type relationship: $\sigma_y = \sigma_0' + k'\lambda^{-1}$, i.e. the yield strength increases with decreasing lamellar spacing.

3. Softening of the B2 phase at 973 K results in a dramatic decrease of strength but an increase in ductility.
4. The fracture behavior changes from brittle fracture mode at room temperature to ductile fracture mode at 973 K.
5. The addition of Ti has an adverse effect on the ductility of the alloys at both room temperature and elevated temperature.

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Table 1 Lamellar spacing and f.c.c. phase fractions for as-cast $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ ($x=0, 2, 4$ at. %) HEAs and $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_4$ HEA annealed at 1173 K for 20 h. The data for as-cast $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ is from [13].

Alloy	f.c.c. phase fraction (%)	Lamellar spacing (nm)
$\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$	85	1560
$\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_2$	72	541
$\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_4$	66	155
$\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_4$ 1173 K, 20 h	68	577

Table 2 Chemical compositions of phases in as-cast $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ HEAs by EDS (in at. %) from TEM specimens. The compositions of phases in as-cast $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ is from [13]. The error bars indicate the average of five measurements.

Alloy	Phase	Fe	Ni	Mn	Al	Ti
$\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$	f.c.c.	39.6 ± 0.5	14.9 ± 0.5	30.9 ± 0.8	14.6 ± 1.4	--
	B2	11.4 ± 2.5	38.0 ± 3.1	16.5 ± 1.2	34.1 ± 2.6	--
$\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_2$	f.c.c.	42.2 ± 1.6	13.3 ± 0.8	32.0 ± 0.8	11.8 ± 0.7	1.0 ± 0.1
	B2	11.7 ± 1.1	34.2 ± 1.3	15.1 ± 0.4	35.1 ± 0.5	3.9 ± 0.3
$\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_4$	f.c.c.	42.7 ± 1.1	11.6 ± 0.9	33.1 ± 1.1	10.8 ± 1.2	1.7 ± 0.1
	B2	13.3 ± 0.8	33.0 ± 1.6	12.7 ± 1.4	32.7 ± 2.3	8.2 ± 0.6
$\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_6$	f.c.c.	43.5 ± 0.5	9.6 ± 0.3	33.7 ± 0.4	11.3 ± 0.4	2.0 ± 0.1
	B2	13.1 ± 0.3	30.9 ± 1.8	10.0 ± 1.1	33.5 ± 2.4	12.4 ± 0.4

Table 3 Mechanical properties (Vickers hardness, yield stress, fracture strength and elongation) for $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ HEAs in both as-cast and annealed states at room temperature. The hardness results are the average of five measurements, and the tensile test results are the average of three tests. The **error bars** indicate the standard deviation. The elongations were measured directly from the specimens.

Alloy	Vickers hardness	Yield stress (MPa)	Fracture Stress (MPa)	Elongation (%)
$\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$	186.5 ± 2.5	270 ± 18	578 ± 35	22.8 ± 0.9
$\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_2$	279.7 ± 6.4	532 ± 18	876 ± 9	13.9 ± 0.5
$\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_2$ 823 K, 24h	312.9 ± 7.0	563 ± 6	891 ± 10	13.7 ± 1.2
$\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_4$	374.7 ± 7.0	953 ± 21	1145 ± 7	2.3 ± 0.3
$\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_4$ 823 K, 24h	404.6 ± 11.3	996 ± 36	1144 ± 23	1.1 ± 0.2
$\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_4$ 1173 K, 20h	274.1 ± 5.0	511 ± 29	790 ± 14	5.6 ± 0.9

Table 4 The atomic radius of the elements [33].

Element	Fe	Ni	Mn	Al	Ti
Atomic radius (pm)	140	135	140	125	140

Figure Captions

Figure 1 SEM images of microstructures of (a) as-cast $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ [13], (b) as-cast $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_2$, (c) $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_2$ annealed at 823 K for 24 h, (d) as-cast $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_4$, (e) $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_4$ annealed at 1173 K for 20h, and (f) as-cast $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_6$

Figure 2 Synchrotron XRD patterns of as-cast $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$, showing f.c.c. and B2 peaks.

Figure 3 Lattice constant of f.c.c. and B2 phases in as-cast $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ as a function of Ti content.

Figure 4 BF TEM image of (a) as-cast $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_2$, (b) $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_2$ annealed at 823 K for 24 h, (c) as-cast $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_4$ and (d) as-cast $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_6$; (e and f) X-ray spectra from the f.c.c. and B2 phases in as-cast $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_6$, respectively. The SAD patterns in (b) and (d) show that phase **with light gray contrast is f.c.c and with dark gray contrast is B2**, respectively.

Figure 5 Strain-stress curves of $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ HEAs tested in a strain rate of $5 \times 10^{-4} \text{ s}^{-1}$ at (a) room temperature and (b) 973 K.

Figure 6 Yield stress and elongation of $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ HEAs tensile tested at 973 K.

Figure 7 Room-temperature fracture surfaces of (a) as-cast $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_2$, (b) As-cast $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_4$, and (c) $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_4$ annealed at 1173 K for **20 h**.

Figure 8 Fracture surfaces tensile tested at 973 K of **as-cast** (a) $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_2$, (b) $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_4$, and (c) $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_6$.

Figure 9 Lamellar spacing of alloys as a function of Ti content and Al content. The subscript indicates the content of each element in at. %. The change of lamellar spacing with the varying content of Al is from [13].

Figure 10 Room-temperature yield stress and elongation to failure for lamellar-structured $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ HEAs as a function of (a), (b) $\lambda^{-1/2}$, and (c), (d) λ^{-1} .

Figure 1 SEM images of microstructures of (a) as-cast $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ [13], (b) as-cast $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_2$, (c) $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_2$ annealed at 823 K for 24 h, (d) as-cast $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_4$, (e) $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_4$ annealed at 1173 K for 20h, and (f) as-cast $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_6$.

Figure 2 Synchrotron XRD patterns of as-cast $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$, showing f.c.c. and B2 peaks.

Figure 3 Lattice constant of f.c.c. and B2 phases in as-cast $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ as a function of Ti content.

Figure 4 BF TEM image of (a) as-cast Fe₃₆Ni₁₈Mn₃₃Al₁₃Ti₂, (b) Fe₃₆Ni₁₈Mn₃₃Al₁₃Ti₂ annealed at 823 K for 24 h, (c) as-cast Fe₃₆Ni₁₈Mn₃₃Al₁₃Ti₄ and (d) as-cast Fe₃₆Ni₁₈Mn₃₃Al₁₃Ti₆; (e and f) X-ray spectra from the f.c.c. and B2 phases in as-cast Fe₃₆Ni₁₈Mn₃₃Al₁₃Ti₆, respectively. The SAD patterns in (b) and (d) show that phase with light gray contrast is f.c.c. and with dark gray contrast is B2, respectively.

Figure 5 Strain-stress curves of Fe₃₆Ni₁₈Mn₃₃Al₁₃Ti_x HEAs tested in a strain rate of $5 \times 10^{-4} \text{ s}^{-1}$ at (a) room temperature and (b) 973 K.

Figure 6 Yield stress and elongation of Fe₃₆Ni₁₈Mn₃₃Al₁₃Ti_x HEAs tensile tested at 973 K.

Figure 7 Secondary electron images of room-temperature fracture surfaces of (a) as-cast $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_2$, (b) as-cast $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_4$, and (c) $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_4$ annealed at 1173 K for 20 h.

Figure 8 Fracture surfaces tensile tested at 973 K of **as-cast** (a) $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_2$, (b) $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_4$, and (c) $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_6$.

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Figure 10 Room-temperature yield stress and elongation to failure for lamellar-structured $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ HEAs as a function of (a), (b) $\lambda^{-1/2}$, and (c), (d) λ^{-1} .