Effects of annealing and thermo-mechanical treatment on the microstructures and mechanical properties of a carbon-doped FeNiMnAl multi-component alloy

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ABSTRACT

The microstructures and mechanical properties of both undoped and carbon-doped (1.26 at%) f.c.c./B2 $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ multi-component alloys have been investigated in the as-cast, annealed and recrystallized states. A lamellar structure is present in the undoped alloy, while a tetragonal martensite with irregular shape is present in the carbon-doped alloy. B2-structured precipitates form upon annealing in both alloys, whose size and volume fraction increases with increasing annealing time. Lamellar coarsening also occurs during annealing of the undoped alloy. Two different thermo-mechanical treatments were applied to the carbon-doped alloy in order to decrease the grain size and disperse the martensite, which produced a significant increase in strength. The changes in yield strength are discussed in terms of the underlying strengthening mechanisms, i.e., phase boundary strengthening, grain boundary strengthening, interstitial strengthening, and precipitation strengthening. The carbon addition results in a sharp increase in ductility of the as-cast alloy, a feature ascribed to microband formation during the tensile test arising from the increase of lattice friction stress. Similar to some single-phase f.c.c. alloys, microband-induced plasticity (MBIP) effect is found to present in a two-phase multicomponent alloy in this study.

1. Introduction

A novel f.c.c./B2 dual-phase $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ (in at%) multi-component alloy, which was designed based on earlier studies of lamellar FeNiMnAl alloys [1–6], exhibits an excellent combination of strength and ductility, suggesting promising applications for structural materials [7]. It is well established that the lamellar spacing plays a critical role in the strength of lamellar-structure alloys, as shown in TiAl alloys [8,9], Ni-Fe-Al alloys [10–12], and pearlitic steels [13,14]. Our earlier work [15] revealed that the addition of up to 6 at% Ti to $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ leads to a substantial reduction in lamellar spacing from 1560 nm to 155 nm and thus, produces a sharp increase in yield strength from 270 MPa to 953 MPa, albeit with a reduction in the elongation to failure from 22.8% to 2.3%.

The introduction of precipitates offers a potential strengthening route [16]. For example, Hanna et al. [17] showed that the yield strength of a spinodally-formed B2/b.c.c. $\text{Fe}_{30}\text{Ni}_{20}\text{Mn}_{25}\text{Al}_{25}$ increased from 1510 MPa to 2350 MPa after annealing at 823 K for 115 h due to the strengthening from β -Mn precipitates. Similarly, He et al. [18] produced coherent nano-sized L_{12} precipitates in f.c.c. $(\text{FeCoNiCr})_{94}\text{Ti}_2\text{Al}_4$ after thermo-mechanical treatment, resulting in a yield strength of over 1 GPa.

Grain size reduction is another strengthening route. In dual-phase multicomponent alloys, grain growth of the matrix phase can be hindered by second phase pinning, leading to the formation of alloys with a very fine grain size. Baker et al. [19] cold rolled a f.c.c./B2 $\text{Fe}_{28.2}\text{Ni}_{18.8}\text{Mn}_{32.9}\text{Al}_{14.1}\text{Cr}_6$ alloy to ~65% reduction in thickness, followed by an annealing at 1273 K, which produced a dual-phase alloy with a grain size of 0.5 μm . Wani et al. [20] developed a duplex L_{12} /B2 $\text{AlCoCrFeNi}_{2.1}$ alloy with fine equi-axed grains (~0.60 μm) after cold-rolling and annealing, which showed an high ultimate tensile strength of ~1.2 GPa due to the combination of grain boundary strengthening and strengthening from the B2 phase.

In addition to the above strengthening mechanisms, carbon has been added to produce interstitial strengthening in multicomponent alloys [21–23]. Wu et al. [21] showed that 0.5 at% carbon addition to a single f.c.c. CoCrFeMnAl alloy increased the room-temperature yield strength from 165 MPa to 225 MPa but with a reduction in elongation from 65% to 38%. Wang et al. [23,24] found that adding up to 1.1 at% carbon to a single f.c.c. $\text{Fe}_{40.4}\text{Ni}_{11.3}\text{Mn}_{34.8}\text{Al}_{7.5}\text{Cr}_6$ alloy increased both the yield strength (from 159 MPa to 355 MPa) and elongation to failure (from 41% to 50%), since the carbon solute induced the formation of

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Fig. 1. BSE images of microstructures of (a) as-cast undoped Fe₃₆Ni₁₈Mn₃₃Al₁₃, (b) as-cast 1.26 at% carbon-doped Fe₃₆Ni₁₈Mn₃₃Al₁₃, (c) undoped Fe₃₆Ni₁₈Mn₃₃Al₁₃ annealed at 1000 K for 1 h, (d) 1.26 at% carbon-doped Fe₃₆Ni₁₈Mn₃₃Al₁₃ annealed at 1000 K for 1 h, (e) undoped Fe₃₆Ni₁₈Mn₃₃Al₁₃ annealed at 1000 K for 72 h, and (f) 1.26 at% carbon-doped Fe₃₆Ni₁₈Mn₃₃Al₁₃ annealed at 1000 K for 72 h.

microbands. Thus, carbon was added to achieve interstitial strengthening in the two-phase alloy Fe₃₆Ni₁₈Mn₃₃Al₁₃ in this study. Annealing and thermo-mechanical treatments were also conducted in attempts to strengthen both undoped and carbon-doped Fe₃₆Ni₁₈Mn₃₃Al₁₃ alloys. The microstructural evolution of the microstructures of alloys was investigated to reveal the underlying strengthening mechanisms. The dislocation substructures after fracture were examined to clarify the effect of carbon on the deformation behavior of the alloys.

2. Experimental

Arc-melting was used to produce ingots of undoped and carbon-doped Fe₃₆Ni₁₈Mn₃₃Al₁₃ alloys from pieces of 99.99% Fe, 99.95% Ni, 99.9% Mn, 99.9% Al and 99% Fe₃C. Detailed arc-melting procedures can be found elsewhere [15]. The chemistry of the carbon-doped alloy

was determined by Chicago Spectro Service Laboratory based on ASTM E1019 and ASTM E1621 method, which showed the carbon content of the alloy is 1.26 at%. The as-cast alloys were annealed at 1000 K for either 1 h or 72 h in argon atmosphere to protect from oxidation.

Two different thermo-mechanical treatments (T1 and T2) were performed on the as-cast carbon-doped alloy to disperse the second phase and refine the grain size.

- **T1 thermo-mechanical treatment:** specimens were cold-rolled to a 70% reduction in thickness with a reduction of ~5% per pass, and then annealed at 1273 K for 1 h, in order to produce a finer microstructure.
- **T2 thermo-mechanical treatment:** In order to further disperse the second phase, two thermo-mechanical treatment were performed. An initial cold-rolling of 50% reduction thickness and

Fig. 2. BSE images at high magnifications of microstructures of (a) undoped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ annealed at 1000 K for 1 h, (b) 1.26 at% carbon-doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ annealed at 1000 K for 1 h, (c) undoped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ annealed at 1000 K for 72 h, and (d) 1.26 at% carbon-doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ annealed at 1000 K for 72 h.

Table 1

The lamellar spacing, precipitate size (Feret diameter), and precipitate volume fraction in f.c.c. phase for undoped and 1.26 at% carbon-doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloys in as-cast and annealed states. The error bars indicate the standard deviation of total measurements.

Alloy	State	Lamellar spacing (μm)	Precipitate size (nm)	Precipitate volume fraction in f.c.c. phase (%)
$\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$	As-cast	1.6 ± 0.5	–	–
$\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$	1000 K, 1 h	2.3 ± 0.5	139 ± 99	14.9 ± 0.2
$\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$	1000 K, 72 h	4.2 ± 0.9	409 ± 272	41.5 ± 1.9
$\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{C}_{1.26}$	1000 K, 1 h	–	149 ± 109	14.4 ± 0.5
$\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{C}_{1.26}$	1000 K, 72 h	–	495 ± 342	46.0 ± 3.1

anneal at 1373 K for 4 h was followed further cold-rolling to a 50% reduction in thickness and annealing at 1273 K for 4 h.

A Rigaku D/MAX 2000 diffractometer with Cu-K α radiation was used to perform X-ray diffraction (XRD) analysis on the alloys. The diffractometer was operated at 40 keV and 300 mA, and step scanning was performed for 2θ from 20° to 120° with a scanning rate of 1° per min. The lattice parameter of the f.c.c. phase in the as-cast, carbon-doped alloy was determined based on the XRD result using an extrapolation method [15].

The microstructures of the alloys in the as-cast, annealed and recrystallized states were examined using both a FEI XL-30 field emission gun (FEG) scanning electron microscope (SEM) in both backscattered electron (BSE) and secondary electron (SE) modes operated at 15 keV, and a FEI Tecnai F20 FEG transmission electron microscope (TEM) equipped with energy dispersive X-ray spectrometry (EDS) operated at 200 kV. The sample preparation procedures for SEM and TEM are listed in [15]. The crystal spacing (d) of martensite in the as-cast carbon-doped alloy was obtained from $d = \lambda L/R$, where λL is the

camera constant and R is the distance between the transmitted and diffracted spots. The lattice parameters of the martensite phase were calculated based on the crystal spacing.

The lamellar spacing, size of precipitates and precipitate fraction in the f.c.c. phase were calculated based on analysis of BSE images using Image J. At least 300 measurements for precipitates and 40 measurements for lamellae were performed. The Feret diameter was used to define the particle size of the precipitates. A linear intercept method was used to determine the grain size.

Dog-bone specimens with a gauge length of 10 mm, width of ~ 2.6 mm and thickness of ~ 1.2 mm, polished to mirror surfaces by using 0.3 μm alumina powder, were used for tensile tests. Three tensile tests were conducted for each alloy at room temperature with an initial strain rate of $5 \times 10^{-4} \text{ s}^{-1}$. The elongation to fracture was determined from the change of gauge length from the tensile tests. The work-hardening rate at 1% plastic strain was determined by calculating $d\sigma/d\varepsilon$ at that strain, where σ is the flow stress and ε is the strain. The fracture surfaces were examined in the SEM using SE mode and the dislocation substructures of tensile-fractured specimens were examined in the TEM.

Fig. 3. XRD patterns for carbon-doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ in (a) the as-cast state, and (b) 1 h annealed at 1000 K state. The inserted image in (b) shows the extra (001) peak of B2 phase in detail.

3. Results

Fig. 1 shows the microstructures of undoped and 1.26 at% carbon-doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloys in both as-cast and annealed states: BSE images at high magnification for the annealed alloys are shown in Fig. 2. The as-cast undoped alloy exhibits a lamellar structure. The dark phase is B2 (ordered b.c.c.) and light phase is f.c.c in Fig. 1a based on previous studies [7]. The spacing between the B2 lamellae is 1.6 μm . The addition of carbon caused the lamellar structure to be replaced by a matrix containing a large irregular second phase, see Fig. 1b. The second phase particles consist of a net-like structure, as shown in Fig. 2b and d. After annealing at 1000 K, needle-like precipitates were present in the f.c.c. matrix in both the undoped and carbon-doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloys, see Fig. 1c–f. Table 1 summarizes the size distribution and the volume fraction of precipitates for both undoped and carbon-doped alloys annealed at 1 h and 72 h. When the annealing time increased from 1 h to 72 h, the average size of the precipitates increased from 139 nm to 409 nm for the undoped alloy, and from 149 nm to 495 nm for the carbon-doped alloy, respectively. Meanwhile, the growth of precipitate volume fraction shows the precipitation continued as the annealing proceeded in both undoped and carbon-doped alloys. In addition, lamellar coarsening occurred as the annealing proceeded for the undoped alloys, i.e., the spacing between the B2 lamellae increased from 1.6 μm in the as-cast state to 4.2 μm in 72 h annealing state, as listed in Table 1.

X-ray diffraction (XRD) patterns for the carbon-doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ in the as-cast state and 1 h annealed state are presented in Fig. 3. The carbon-doped alloy contains both f.c.c. and b.c.t. (body-centered-tetragonal structure, further confirmed by TEM, see later) martensite phases in the as-cast state (Fig. 3a), while, as

Fig. 4. BF TEM images of 1.26 at% carbon-doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloys annealed at 1000 K for (a) and (b) 1 h, and (c) 72 h. (a) shows the microstructure of the martensite phase and (b) shows precipitates (ppt.) in the f.c.c. matrix after 1 h annealing, and (c) shows the microstructure of the precipitates after 72 h annealing. The inserted SAD patterns confirm that the second phase is tetragonal martensite (MS), the matrix phase is f.c.c. structure, and the precipitates are B2 respectively.

noted earlier, the undoped alloy consists of f.c.c. and B2 phases. The additional peaks appearing in the annealed carbon-doped alloy (Fig. 3b), indicate the precipitation is a B2 phase.

The bright field (BF) TEM images in Fig. 4 show the microstructures of the carbon-doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ annealed at 1000 K for 1 h and 72 h. Fig. 4a and b shows the microstructure of the irregular second phase and precipitates in 1 h annealed specimens. Selected area diffraction (SAD) patterns in Fig. 4a and b confirm that the irregular second phase is a tetragonal martensite and the matrix is f.c.c. structure. The converge beam electron diffraction (CBED) pattern in Fig. 4c from a precipitate after the 72 h anneal, after which the precipitates have grown significantly shows a B2 structure, which is

Fig. 5. X-ray spectra from the (a) f.c.c. matrix, (b) martensite and (c) B2 precipitates (ppt.) in the 1.26 at% carbon-doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloys annealed at 1000 K for 1 h.

Table 2

Chemical compositions of phases determined by EDS (in at%) from TEM specimens in undoped and carbon-doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloys in as-cast, annealing and thermo-mechanical treated states. The chemical compositions of phases in undoped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloys are from [7]. Note that the carbon content is not listed due to the limited detection accuracy of EDS for light elements. MS and ppt. indicate the martensite and precipitate, respectively. The error bars indicate the standard deviation from five measurements.

Alloy	Phase	Fe	Ni	Mn	Al
$\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$	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
$\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ 1000 K, 1 h	f.c.c.	44.6 ± 0.1	11.5 ± 0.2	32.9 ± 0.2	11.0 ± 0.5
	B2	7.6 ± 0.8	39.8 ± 0.9	15.5 ± 0.5	37.2 ± 0.4
	ppt.	6.8 ± 0.5	34.2 ± 0.7	14.0 ± 0.7	41.1 ± 0.5
$\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{C}_{1.26}$	f.c.c.	43.3 ± 1.5	14.9 ± 0.6	30.2 ± 0.6	11.7 ± 0.5
	MS	10.7 ± 2.2	37.5 ± 1.9	22.9 ± 1.6	28.9 ± 2.0
$\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{C}_{1.26}$ 1000 K, 1 h	f.c.c.	43.3 ± 1.0	14.0 ± 0.4	30.9 ± 1.2	11.8 ± 0.4
	MS	5.3 ± 0.9	40.0 ± 0.1	15.3 ± 0.5	39.3 ± 1.2
	ppt.	4.1 ± 0.2	37.5 ± 1.2	13.0 ± 1.2	44.8 ± 2.6
$\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{C}_{1.26}$ T1 treatment	f.c.c.	43.1 ± 1.0	11.9 ± 0.4	34.7 ± 1.0	10.2 ± 1.7
	MS	9.7 ± 0.1	34.9 ± 0.1	16.5 ± 0.5	38.9 ± 0.5
	ppt.	8.6 ± 0.3	34.7 ± 0.6	15.5 ± 0.3	41.1 ± 0.8

consistent with our previous observation that B2-structured precipitates were formed in the undoped alloy during annealing [25]. Fig. 5 displays X-ray spectra from the f.c.c. matrix, the martensite phase and the B2 precipitates in the carbon-doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloy annealed at 1000 K for 1 h. The Fe and Mn are concentrated in the f.c.c. matrix, while the martensite phase was enriched in Ni and Al. The composition of the B2 precipitates is close to that of the martensite, as illustrated in Fig. 5c. Table 2 summarizes the compositions for each phase in the undoped and carbon-doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloys in both as-cast and annealed states. The EDS results show that the f.c.c. matrix, second phase (martensite phase), and precipitates have similar compositions in both the undoped and carbon-doped alloys.

The SE images in Fig. 6a and b show the microstructures of the recrystallized carbon-doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloy after the T1 and T2 thermo-mechanical treatments (indicated as T1 and T2 alloy hereafter for simplicity), respectively. The grain size for the as-cast alloy was in the range of 100–300 μm [7], while it decreased to 1.9 μm in the T1 alloy and 4.5 μm in the T2 alloy. The grain size of T2 alloy is larger than that of T1 alloy due to the longer annealing time. The martensite phase was dispersed during the cold-rolling and distributed among the grain boundaries of the f.c.c. phase. As shown in Fig. 6, precipitates were also formed inside the f.c.c. matrix after both T1 and T2 treatments. Fig. 7 presents BF TEM images for the recrystallized carbon-doped alloys after the T1 treatment. The SAD pattern in Fig. 7b confirms that the precipitate inside the f.c.c. grain is a B2 structure. As shown in Table 2, the compositions for each phase in T1 alloy are similar to that in the annealed alloys.

Representative engineering stress-strain curves for the undoped and carbon-doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloys in both as-cast and annealed states are shown in Fig. 8a. The corresponding variations of yield strength (Fig. 8b), ultimate tensile strength (UTS) (Fig. 8c), work-hardening rate at 1% strain (Fig. 8d), and elongation to fracture (Fig. 8e) for alloys in the different conditions are also displayed. The yield strength, UTS, and work-hardening rate at 1% strain for both undoped and carbon-doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloys increase significantly with increasing annealing time, i.e., the yield strength increased from 270 MPa to 483 MPa for the undoped alloy, and from 260 MPa to 611 MPa for the carbon-doped alloy, after annealing for 72 h at 1000 K. Conversely, the elongation to fracture for both undoped and carbon-doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloys decreases significantly as the annealing time increases, see Fig. 8e, but even the alloy with the lowest ductility shows 10% elongation.

Fig. 9a shows typical engineering stress-strain curves for carbon-doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloys after T1 and T2 thermo-mechanical

Fig. 6. SE images of 1.26 at% carbon-doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloys after (a) the T1 treatment and (b) the T2 treatment. The grain size of T1 alloy is 1.9 μm and that of the T2 alloy is 4.5 μm . The martensite (MS) resides along the grain boundaries, while the B2 precipitates are inside the grains (see arrowed).

Fig. 7. (a) BF TEM images of 1.26 at% carbon-doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloys after the T1 treatment. (b) SAD pattern indicating the precipitate inside the grain is a B2 structure.

treatments, and Fig. 9b shows the variations of yield strength and elongation to fracture. The reduction of grain size to 1.9 μm in the T1 alloy and 4.5 μm in the T2 alloy produces an increase in strength but a decrease in ductility, i.e., the yield strength increased from 260 MPa for the as-cast alloy to 532 MPa for the T1 alloy and 426 MPa for the T2 alloy, but the elongation decreased from 39.3% for the as-cast alloy to 23.8% for the T1 alloy and 32.8% for the T2 alloy.

The dislocation substructures in the as-cast undoped alloy, as-cast carbon-doped alloy and 1 h annealed carbon-doped alloy strained to failure are shown in Fig. 10. For the undoped and annealed carbon-doped alloys, a high density of randomly distributed dislocation were present in the f.c.c. phase. At these high levels of strain, microbands were also observed in the f.c.c. phase in the as-cast carbon-doped alloys, but not in the undoped alloy or the carbon-doped alloy containing precipitates. Slip trace analysis indicates that these microbands are oriented along $\{111\}$, which is consistent with our recent observations in a single-phase f.c.c. high entropy alloy [23].

The fracture surfaces for the carbon-doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloy in the as-cast, 72 h annealed state and T2 thermo-mechanical treatment state are shown in Fig. 11. Dimples can be found in all three cases, of which the larger dimple size corresponds to higher elongations obtained in tensile test. Cleavage was also present along with the dimples in the alloy annealed for 72 h, suggesting a mixture of ductile and brittle fracture mode.

4. Discussion

4.1. Microstructures

The lamellar structure of $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ exhibits a strong dependence on chemical composition and processing conditions [15]. In the present study, when 1.26 at% a carbon was added to $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$, the f.c.c./B2 lamellar was replaced by a f.c.c. matrix with a, b.c.t. martensite phase with an irregular shape. On one hand, the carbon dissolved in the f.c.c. phase, leading to the increase of lattice parameter from 3.6468 Å in the undoped alloy [15] to the 3.6511 Å in the carbon-doped alloy. On the other hand, the carbon addition caused the B2 (ordered b.c.c.) phase to change to clumps of b.c.t. martensite. The transformation from b.c.c. structure to b.c.t. structure is widely seen in the quenched Fe-C steels [26]. A similar phase transformation from B2 to 7 R martensite has also been observed in undoped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$, after cold-rolling and annealing [25]. The carbon atoms presumably occupy octahedral interstices, which produces lattice parameters of $a=2.8802$ Å and $c=2.9515$ Å i.e. a c/a ratio of 1.03.

In b.c.c. iron, carbon atoms sitting in octahedral sites on the unit cell edge lengthen the c -axis and dilate the a -axis and the c/a ratio of the resulting martensite increases with increasing carbon [26]. The situation in B2 phases is somewhat different since the size of the edge

Fig. 8. (a) Engineering stress-strain curves for undoped (Al_{13}) and 1.26 at% carbon-doped (Al_{13}C) $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ in both as-cast and annealed states; variation of (b) yield strength, (c) ultimate tensile strength (UTS), (d) work-hardening rate at 1% strain, and (e) elongation to fracture for undoped and carbon-doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloys for different annealing conditions.

octahedral site is different to the size of the octahedral in the center of the unit cell with their relative sizes depending on the ratio of the radii of the constituent atoms [27].

Needle-like B2-structured precipitates were found after annealing at 1000 K for both the undoped and carbon-doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloys. Table 1 shows that the size and volume fraction of precipitates in undoped and doped are similar under the same annealing condition. Though the phase morphology in this as-cast carbon doped alloys has been completely changed, the compositions for each phase are very close, as seen in Table 2. Thus, it is not surprising to observe the similar precipitation kinetics in both undoped and carbon-doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloys during annealing. Except for the precipitation, the lamellar coarsening occurred for the undoped alloys upon annealing at 1000 K, of which the lamellar spacing increased from the 2.3–

4.2 μm with an increase in annealing time from 1 h to 72 h. This is consistent with observations for $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}\text{Ti}_x$ alloys, where significant coarsening of the lamellae was found upon annealing at 1173 K [15].

The recrystallized carbon-doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloys show a very fine microstructure with grain sizes of 1.9 μm for the T1 alloy and 4.5 μm for the T2 alloy, see Fig. 6. The fine-grained structure after recrystallization can be ascribed to the pinning effect on grain boundaries from the martensite during recrystallization. The dispersed martensite particles are capable of exerting a drag force on migrating grain boundaries, and thus, impede the grain growth during the recrystallization process [28]. Baker et al. [19] showed that a very fine grain size of 0.5 μm can be achieved in a f.c.c./B2 $\text{Fe}_{28.2}\text{Ni}_{18.8}\text{Mn}_{32.9}\text{Al}_{14.1}\text{Cr}_6$ alloy after recrystallization owing to the drag effect of the B2 phase.

Fig. 9. (a) Engineering stress-strain curves and (b) yield strength and elongation for as-cast and recrystallized 1.26 at% carbon-doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloys after T1 and T2 treatment.

4.2. Yield strength and work-hardening rate

The yield strength (σ_y) of the $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloys can be described by the following expression:

$$\sigma_y = \sigma_{fr} + \sigma_b + \sigma_{ppt} \quad (1)$$

where σ_{fr} is the lattice friction stress, σ_b is the strength increase associated with the phase boundaries and grain boundaries, and σ_{ppt} is stress from the precipitate strengthening. In $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ multicomponent alloys, we can not define the solute and solvent atoms since the constituents are distributed randomly in the lattice. Hence, the solid solution strengthening (σ_{ss}) is commonly embedded in the lattice friction stress in multicomponent alloys, as seen in $\text{Fe}_{28.2}\text{Ni}_{18.8}\text{Mn}_{32.9}\text{Al}_{14.1}\text{Cr}_6$ alloy [29] and FeNiCoCr alloy [30]. Both the grain boundaries and the martensite phase are impediments to dislocation movement, which produces a boundary strengthening effect. The grain boundary strengthening follows a Hall-Petch relationship [31,32] $\sigma_{gb} \sim \lambda^{-0.5}$, while the phase boundary strengthening is described by a modified Hall-Petch type relationship in FeNiMnAl alloy system [7,15] $\sigma_{pb} \sim \lambda^{-1}$; where λ is the grain size or phase spacing. The precipitate strengthening effect results from dislocations bowing around the precipitates, which for large precipitates can be expressed by [33]:

$$\sigma_{ppt} = \frac{Gb}{L} \quad (2)$$

where G is the shear modulus, b is the Burgers' vector, and L is the precipitate spacing. In the following, the variation of yield strength for undoped and 1.26 at% carbon-doped alloys in different conditions are discussed based on the strengthening mechanisms noted above.

Fig. 10. Dislocation substructures in the (a) undoped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloy, and in the 1.26 at% carbon-doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloys in (b) the as-cast state and (c) after annealing at 1000 K for 1 h strained to failure. The slip trace shows that the microbands are oriented along $\{111\}$.

Fig. 11. Fracture surfaces of 1.26 at% carbon-doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloys in (a) as-cast state, (b) annealed at 1000 K for 72 h, and (c) recrystallized state after T1 treatment. Some cleavage steps are arrowed in (b).

The yield strength of carbon-doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloy (~26 MPa) is close to that of the undoped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloy (~27 MPa) in as-cast state, as shown in Fig. 8. For the as-cast and annealed alloys, the contribution from grain boundary strengthening is likely to be very small due to the large grain size in the range of 100–300 μm . Therefore, the dominant contributions to the yield strength for both the undoped and carbon-doped alloys results from the lattice friction and phase boundary strengthening. For the carbon-doped alloys, the carbon atoms are dissolved in the lattice, improving the lattice friction stress via the interstitial strengthening. The transformation of phase morphology from lamellar-structured to particle-structured, however, reduced the effectiveness of phase boundary strengthening. The decrease in phase boundary strengthening counteracted the increase in lattice friction stress. As a result, the yield strength is close for

undoped and carbon-doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloys in as-cast state.

The yield strength increases significantly with increasing annealing times for both undoped and doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloys because of the B2 precipitation. As listed in Table 1, a growing number of precipitates are formed after longer annealing time, causing the enhancement of precipitate strengthening effects in alloys. But for undoped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloys, the lamellar spacing increases as annealing proceeds (see Table 1), which reduces the phase boundary strengthening. As a consequence, the increase in yield strength after annealing in the carbon-doped alloys is higher than that of the undoped alloy, i.e., the improvement of yield strength after 1 h annealing is 274 MPa for the doped alloy and 148 MPa for the undoped alloy, respectively, even though the size and volume fraction of the precipitate are similar in both alloys.

The yield strengths of the T1 and T2 alloy is much higher than that of the as-cast carbon-doped alloy (see Fig. 9b), since the grain boundary strengthening is significant for recrystallized T1 and T2 alloys. For the T1 and T2 alloy, both second phase and precipitate strengthening occur; however, the grain size of the T1 alloy (1.9 μm) is smaller than that of the T2 alloy (4.5 μm), which produces a stronger grain boundary strengthening in the T1 alloy. Thus, the yield strength of the T1 alloy is higher than that of the T2 alloy, as shown in Fig. 9b.

The work-hardening rate at 1% strain for both undoped and C-doped alloys increases with increasing of annealing time (see Fig. 8d), showing the same trend as the yield strength. The work-hardening rate for both alloys increased up to ~700 MPa after 72 h annealing at 1000 K, which exhibited a similar value to $\text{Fe}_{28}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{21}$ alloys after a long annealing (100–250 h) at 1173 K [34]. More precipitates form as the annealing time increases, which exerts a stronger retard to dislocation motions during deformation and thus, leads to the higher work-hardening.

4.3. Ductility

Generally, the ductility of both undoped and doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloys decreases as the strength increases, which is a typical behavior for most metallic materials [35]. It is worth noting that, although the yield strength is similar for the doped and undoped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloy in the as-cast state, the ductility of the carbon-doped alloy is far better than that of the undoped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloy, i.e., an elongation of 39.3% for the carbon-doped alloy versus 22.8% for the undoped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloy. As shown in Fig. 10b, microbands were formed in the f.c.c. matrix of the carbon-doped alloys at a high strain. Previous studies indicated that the formation of microbands allows relaxation of the local strain and provides extra accommodation of plasticity, which leads to an increase of ductility [23]. Thus, microband formation may account for the remarkable increase of ductility in the carbon-doped alloy, as also seen in austenite high Mn steels [36,37] and a f.c.c. high entropy alloy [23,38]. The occurrence of microband is related to the increase of lattice friction stress due to the addition of carbon solutes [23]. Microbands were observed in the carbon-doped alloy (Fig. 10b) but not the undoped alloy (Fig. 10a). However, for the carbon-doped alloy annealed at 1000 K for 1 h, microbands are no longer seen and a random distribution of dislocation is found in f.c.c. phase, see Fig. 10c. The formation of microbands relies on the subdivision of grains during deformation [23]. In the annealed carbon-doped alloy precipitates distributed inside the f.c.c. phase hinders the grain subdivision as the strain is increased and, thus, impedes the formation of microbands. A similar phenomenon was observed in a thermo-mechanically processed 1.1 at% carbon-doped HEA with a very fine distribution of grain boundaries and carbides [38]. Correspondingly, the annealed carbon-doped alloy exhibits much lower ductility than the as-cast alloy, i.e., the elongation reduced sharply from 39.3% in the as-cast state to 14.7% in the 1 h annealed state.

5. Conclusions

The effects of carbon and thermo-mechanical treatment on the microstructure and room temperature mechanical properties of lamellar-structured B2/f.c.c. $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ were studied and the following observed:

1. The addition of carbon altered the microstructure from a lamellar structure to randomly-distributed, coarse two-phase structure, and further induced a transformation from a B2 (ordered b.c.c.) phase to the b.c.t. martensite phase due to the occupation of octahedral interstices by carbon atoms.
2. B2-structure needles precipitate in both undoped and carbon-doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ alloys upon annealing, leading to a sharp increase in strength but a decrease in ductility. With increasing annealing time, the precipitation continues and the coarsening for individual precipitate occurs in both alloys.
3. Thermo-mechanical treatments of the carbon-doped alloy reduced the as-cast grain size from 100 to 300 μm to 1.9–4.5 μm depending on the processing, resulting in a significant improvement in strength via grain boundary strengthening.
4. The carbon solute increases the lattice friction stress of f.c.c. phase in as-cast carbon-doped $\text{Fe}_{36}\text{Ni}_{18}\text{Mn}_{33}\text{Al}_{13}$ and thus, leads to the formation of microbands during deformation, which further results in a remarkable increase in the ductility of the alloy.

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