Concentration Dependence of Cr for Alleviating Environmental

Embrittlement in Fe₃₀Ni₂₀Mn₃₅Al₁₅

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Abstract

Previously, it has been shown that Fe₃₀Ni₂₀Mn₃₅Al₁₅, which consists of alternating submicron B2 and f.c.c. lamellae, exhibits a room temperature yield strength of 770 MPa and an elongation of ~10% at a strain rate of $3 \times 10^{-3} \text{ s}^{-1}$ in air, but at the slower strain rate of $3 \times 10^{-6} \text{ s}^{-1}$ the alloy exhibits an elongation <1% [1]. An addition of 6 at. % Cr has been proven to not only solve this environmental problem, but also to increase the elongation to 15-18% irrespective of strain rate [2]. Since we do not know whether Cr additions less than 6 at. % can suppress this environmental embrittlement, in this paper we examined the room temperature mechanical properties of several alloys based on Fe₃₀Ni₂₀Mn₃₅Al₁₅ with Cr additions ≤ 6 at. %. We show that additions as low as 0.5 at. % Cr alleviate the environmental embrittlement and that additions of ≥ 2 at. % completely suppress the embrittlement with little change in microstructure. X-ray photoelectron spectroscopy examination suggested that the suppression is mostly due to the formation of protective oxide scales on the surface that provide rapid passivation. The lower yield strength when Cr is present may also contribute to the improved ductility, possibly by easing dislocation cross-slip in the deforming f.c.c. phase where most of the Cr resides.

Keywords: Environmental embrittlement, two-phase alloys, mechanical properties, fracture

Introduction

Environmental embrittlement, arising from the production of atomic hydrogen through the reaction of water vapor with aluminum or silicon [3-5], can lead to low ductility in ordered intermetallic compounds at ambient temperatures and can restrict their use as high performance structural materials. In earlier work, we showed that, drop-cast $\text{Fe}_{30}\text{Ni}_{20}\text{Mn}_{35}\text{Al}_{15}$, which consists of alternating (Fe, Mn)-rich f.c.c and (Ni, Al)-rich B2 (ordered b.c.c.) lamellae [6], exhibited an elongation to failure of 10% when strained under tension in air at a strain rate of $3 \times 10^{-3} \text{ s}^{-1}$ [1], but considerably lower ductility at lower strain rates, e.g. only 0.7% elongation at a strain rate of $3 \times 10^{-6} \text{ s}^{-1}$ [1]. It is worth noting the environmental embrittlement observed in $\text{Fe}_{30}\text{Ni}_{20}\text{Mn}_{35}\text{Al}_{15}$ is likely of the f.c.c. phase since during room temperature deformation the harder B2 does not appear to plastically deform [7].

It has been proposed that environmental embrittlement can be alleviated or reduced by control of the surface condition; control of the grain structure; or control of the alloy composition [8, 9]. Among these techniques, the addition of chromium has been shown to be successful for $\text{Fe}_{30}\text{Ni}_{20}\text{Mn}_{35}\text{Al}_{15}$, where it was found that a 6 at. % Cr addition could completely suppress the environmental embrittlement observed in the Cr-free $\text{Fe}_{30}\text{Ni}_{20}\text{Mn}_{35}\text{Al}_{15}$ alloy at low strain rates [2]. Further, the Cr addition increased the elongation to ~18% at all strain rates. There could be a number of reasons for the beneficial effect of chromium on the environmental embrittlement in $\text{Fe}_{30}\text{Ni}_{20}\text{Mn}_{35}\text{Al}_{15}$. First, the Cr may increase the stacking fault energy of the f.c.c. phase, leading to wavy slip [10-12]. This would reduce the ability of gliding dislocations to transport atomic hydrogen into the material. Second, the Cr could enhance the atomic bonding in $\text{Fe}_{30}\text{Ni}_{20}\text{Mn}_{35}\text{Al}_{15}$, resulting in an increase in cohesive strength of the grain boundaries [10, 13]. Third, the Cr addition could lead to a change in oxide chemistry and properties, or a change in the kinetics of oxide formation [2, 12, 14]. Finally, the possible mechanisms responsible for the beneficial effect of Cr addition on alleviating the environmental embrittlement of L1_2 -structured $\text{Ni}_3(\text{Si}, \text{Ti})$ alloys could also be important in the alloy tested here, i.e. blocking site occupation and/or the diffusion of hydrogen along grain boundaries; and affecting both the water decomposition process and the subsequent hydrogen absorption processes [15]. It was found that increasing the amount of chromium in iron [16]

and in iron aluminum [17] decreased the diffusion coefficient and increased the solubility of hydrogen. McKamey et al. [12] indicated that the most important effect of chromium on the room temperature ductility of DO_3 -structured Fe_3Al was to modify the protective surface oxide, resulting in a minimization of environmental embrittlement by reducing the water-vapor reaction. Additionally, the transport of hydrogen within the oxide must occur by the activated jumping of a proton from one oxygen ion to another [18], and the necessity to break the coulombic bond with the initial oxygen ion leads to considerably lower mobility of hydrogen in an oxide compared to that in the metal phase [13].

Since we do not know whether additions of Cr less than 6 at. % can suppress the environmental embrittlement in $\text{Fe}_{30}\text{Ni}_{20}\text{Mn}_{35}\text{Al}_{15}$, in this paper we have examined the room temperature mechanical properties of several alloys based on $\text{Fe}_{30}\text{Ni}_{20}\text{Mn}_{35}\text{Al}_{15}$ with Cr additions ≤ 6 at. %. In particular it is of interest to determine whether Cr additions monotonically decrease the environmental embrittlement or whether there is a threshold concentration of Cr below which Cr has no effect. In addition, we have examined the surface condition of Cr-modified $\text{Fe}_{30}\text{Ni}_{20}\text{Mn}_{35}\text{Al}_{15}$ alloys using X-ray photoelectron spectroscopy (XPS). Possible mechanisms for the effect of Cr are suggested based on this study.

Experimental

60g ingots of $\text{Fe}_{30}\text{Ni}_{20}\text{Mn}_{35}\text{Al}_{15}$ without and with Cr additions of 0.5, 1, 2, 3, 4, and 6 at. % were prepared by arc melting a mixture of elemental Fe, Ni, Mn, Al and Cr with purities $>99.8\%$ in a water-cooled copper crucible under an argon atmosphere. The ingots were melted and flipped three times to ensure homogeneity.

Specimens for scanning electron microscope (SEM) and transmission electron microscope (TEM) examination were prepared as described elsewhere [2].

Tensile tests (three per alloy) were performed on flat tensile specimens of dog bone geometry with a gauge length of 10 mm and thickness of ~ 1.27 mm. The specimens were polished using silicon carbide papers followed by $0.3\ \mu\text{m}$ alumina powder to reduce surface defects. Tensile tests were performed at an initial strain rate of $5 \times 10^{-6}\ \text{s}^{-1}$ at room temperature and the resulting fracture surfaces were imaged using secondary electrons (SE) in the SEM.

The oxide on the surface of a previously polished specimen containing 6 at. % Cr was examined using a Thermo Scientific ESCALAB 250Xi angle resolved XPS system. The surface was sputter-cleaned for 2 s to remove carbon contamination before running the scan. Spectra for Fe, Ni, Mn, Al, Cr, C and O were collected at tilt angles of 0°, 30° and 60°.

Results and discussion

Figure 1 shows SE images of Cr-free Fe₃₀Ni₂₀Mn₃₅Al₁₅ and of each of the Cr-modified alloys. All the alloys show a lamellar microstructure consisting of alternating B2 and f.c.c. phases. As demonstrated previously [2, 6], the dark phase is f.c.c and the light phase is B2 in SE images. Previous EDS measurements on the alloy with 6 at. % Cr, also confirmed here on other alloys, showed that most (>85%) of the Cr partitioned to the f.c.c. phase [2]. The lamellar spacings in the Cr-modified alloys are of the order of a few hundred nanometers, which is similar to that of the Cr-free alloy. It was reported earlier that the B2 phase is 200 nm in width and separated by an f.c.c. phase 500 nm in width for Cr-free Fe₃₀Ni₂₀Mn₃₅Al₁₅ [6]. Grain sizes measured from both the Cr-free alloy and alloys with chromium were approximately the same within experimental error at $450 \pm 50 \mu\text{m}$.

Figure 2(a) shows example stress-strain curves for each of the compositions strained under tension at room temperature at a strain rate of $5 \times 10^{-6} \text{ s}^{-1}$, i.e. under conditions where environmental embrittlement occurs [1]. The elongation, yield stress and ultimate tensile stress measured under tension at a strain rate of $5 \times 10^{-6} \text{ s}^{-1}$ at room temperature are shown as a function of atomic % Cr added to Fe₃₀Ni₂₀Mn₃₅Al₁₅ in Figure 2(b).

In Figure 2(b) it is evident that the elongation to fracture, measured directly from the specimens, increases monotonically (almost linearly) with increasing Cr additions, from ~0.7% for the Cr-free alloy to ~15% for 6 at. % Cr modified Fe₃₀Ni₂₀Mn₃₅Al₁₅. For purposes of comparison, Figure 2(b) also shows the elongation obtained at a strain rate of $5 \times 10^{-4} \text{ s}^{-1}$ [from Ref 2], when environmental embrittlement does not occur. Several points can be made. First, even with a chromium addition as low as 0.5 at. %, the elongation to fracture is ~3.2%, indicating reduced environmental embrittlement. Second, an addition of 2 at. % Cr completely suppresses the environmental embrittlement since for Cr additions of 2 at. % and above the elongation is the same when the material is tested under conditions where

environmental embrittlement occurs and under conditions where environmental embrittlement does not occur. In other words, Cr additions <2 at. % do not fully suppress the environmental embrittlement, i.e. there is a threshold level of Cr (2 at. %) required. Third, as noted in Meng et al. [2], increasing Cr (up to 6 at. %) leads to increasing ductility.

Adding 1 at. % Cr to Fe₃₀Ni₂₀Mn₃₅Al₁₅ decreased the yield strength from 772 ± 30 MPa for the Cr-free alloy to 691 ± 35 MPa, but further increases in Cr appeared to have little effect on the yield strength, e.g. the yield strength of the 6 at. % Cr alloy was 674 MPa. One possible mechanism for the effect of Cr on the reduction in yield strength might be easier cross-slip in the f.c.c. phase, thereby reducing the lengths of dislocation pile-ups [2]. The ultimate tensile stress exhibits an increase from 812 MPa for the Cr-free alloy to 941 MPa for 0.5 at. % Cr modified alloy, and is nearly constant for all Cr-containing alloys. The lower ultimate tensile stress of Cr-free alloy is the result of the environmental embrittlement that occurs at low strain rate ($5 \times 10^{-6} \text{ s}^{-1}$) in air [1]. In related work, McKamey et al. [10, 11] also noted a slight reduction in the yield strength with the addition of 2-6% Cr to Fe-28% Al (228-248 MPa for Fe-28% Al-(2-6)% Cr alloys compared to 279 MPa for Fe-28% Al alloy), but that the ductility increased by a factor of at least two, from 4% in the base alloy to 8-10% in Cr-containing alloys at room temperature.

The fracture modes of the alloys with 0% Cr and 6 at. % Cr tested at $5 \times 10^{-6} \text{ s}^{-1}$ have been reported previously (see Figure 8 in ref. 2). The alloys tested here are all very similar with largely transgranular fracture with ductile tearing and microvoids more evident as the amount of Cr increased. Interestingly, it was shown that the fracture of unalloyed Fe-28% Al occurred by transgranular cleavage, while the fracture mode of Fe-28% Al-4% Cr changed to one of approximately 50% transgranular cleavage and 50% intergranular failure, suggesting that chromium enhanced the cleavage strength and partially suppressed cleavage fracture [10, 11].

XPS spectra of a Cr2p scan (a) and an Al2p scan (b), which are their primary XPS regions, at different tilt angles from a previously polished surface of a specimen of the 6 at. % Cr modified Fe₃₀Ni₂₀Mn₃₅Al₁₅ are shown in Figure 3. Only spectra for Cr and Al are shown because other metal elements are nearly identical for different tilting angles, indicating that they aren't involved in the passivation at the surface. At 0° tilt, strong Cr metal peaks are

present with weak shoulders on the Cr peaks, implying oxide states. At tilts of 30° and 60°, there are strong Cr oxide peaks, see Figure 3a. Similar oxidation conditions are shown for the Al2p scan in Figure 3b. A set of synthetic peaks was fitted to the XPS data at 30° and 60° after the background subtraction, as shown in Figure 4. The oxide ratios (in atomic percentage) were calculated based on the fitted peaks. It was found that at 30°, Al/Cr = 79.05/20.95, Al/Al oxide = 16.68/83.32 and Cr/Cr oxide = 0.1/1. At 60°, Al/Cr = 79.43/20.57, Al/Al oxide = 0.12/1 and Cr/Cr oxide = 1/0.85. The oxide ratios suggest that substantial amounts of aluminum oxide and chromium oxide are on the surface. It is evident that both Cr and Al form protective oxides at the surface and, thus, provide rapid passivation of the surface, thereby reducing hydrogen liberation rates on the surface and minimizing the environmental embrittlement [14, 19]. This phenomena has been theoretically explained in iron aluminides based on the mixed potential theory by Balasubramaniam et al. [14]. It was noted that the overpotential or the extent of polarization of the hydrogen reaction in pure Fe₃Al was lower compared to that in Fe₃Al+Cr, implying that much higher hydrogen partial pressure were generated on Fe₃Al surfaces than on Fe₃Al+Cr intermetallics [14]. The lower hydrogen partial pressures on Fe₃Al+Cr surfaces led to reduced reaction rates and a lower amount of nascent hydrogen available to diffuse into the material to cause embrittlement [14, 20]. McKamey et al. [12] performed tensile tests on Fe-28 Al and Fe-28% Al-4% Cr with different surface conditions. They noted an increase in ductility for the Cr-containing alloys with oxide coating (elongation of 7.8%) compared to both the binary (elongation of 4.1%) and to Cr-containing specimens which had been electropolished (elongation of 4.0%). A recent study by Zamanzade et al. [13] showed that the thickness of oxide layer in freshly electropolished Fe-26Al-5Cr was very thin (~3 nm) and the chromium and aluminum oxide signals exhibited a maximum at a depth of ~1.5 nm. It was also found that the Cr-containing specimens which were annealed in air had comparable ductilities (7 - 8%) whether they were electropolished or left with the oxide coating. This suggested that the heat treatment in air produced a thin protective oxide coating on previously electropolished specimens. Therefore the addition of chromium to Fe₃Al resulted in a change in the nature of the protective oxide coating [12]. It has also been observed that Cr additions accelerated the formation of a continuous Al₂O₃ scale in Ni-30Fe-10Al-Cr alloy [21]. XPS analysis by Velon et al. [22]

showed that the oxide on Cr-containing Fe_3Al consisted of a continuous layer of $(\text{Al}, \text{Cr})_2\text{O}_3$ underneath a layer of Fe_2O_3 and FeAl_2O_4 .

It is worth noting that Zamanzade et al. [13] showed that for Fe_3Al , the presence of hydrogen decreased the shear modulus, the energy for dislocation nucleation and the dislocation mobility. This is consistent with the observations that for B2 FeAl , the yield strength is lower in air than in vacuum [23] and that the yield strength in air (but not vacuum) decreases with decreasing strain rate [23-25]. In contrast, the yield strength of $\text{Fe}_{30}\text{Ni}_{20}\text{Mn}_{35}\text{Al}_{15}$ does not show any strain rate dependence in air at room temperature [1].

Thus, in the present work, it appears that the role of Cr is to suppress the environmental embrittlement by aiding the rapid formation of protective oxide scales on the surface that provide rapid passivation. The lower yield strength when Cr is present may also contribute to the improved ductility, possibly by easing dislocation cross-slip.

Conclusions

We show that an addition of Cr as low as 0.5 at. % alleviates the environmental embrittlement observed in $\text{Fe}_{30}\text{Ni}_{20}\text{Mn}_{35}\text{Al}_{15}$ and that additions of ≥ 2 at. % completely suppress the embrittlement with little accompanying change in microstructure. X-ray photoelectron spectroscopy examination suggested that the suppression is mostly due to the formation of protective oxide scales on the surface that provide rapid passivation. Cr additions also decrease the yield stress, presumably due to softening of the f.c.c. phase where the Cr mostly resides, which may also contribute to the improved ductility.

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Figure Captions

Figure 1. SE images of as-cast $\text{Fe}_{30}\text{Ni}_{20}\text{Mn}_{35}\text{Al}_{15}$ with different amount of Cr. All the alloys show lamellae microstructures with alternating B2 and f.c.c. phases.

Figure 2. (a) Strain-stress curves of Cr-modified $\text{Fe}_{30}\text{Ni}_{20}\text{Mn}_{35}\text{Al}_{15}$ tested at a strain rate of $5 \times 10^{-6} \text{ s}^{-1}$ at room temperature, and (b) elongation, yield stress and UTS as a function of atomic % Cr added to $\text{Fe}_{30}\text{Ni}_{20}\text{Mn}_{35}\text{Al}_{15}$ tested at a strain rate of $5 \times 10^{-6} \text{ s}^{-1}$ at room temperature. The error bars were calculated from three tests performed at each condition. The elongation obtained at a strain rate of $5 \times 10^{-4} \text{ s}^{-1}$ from Ref. 2 is shown for comparison.

Figure 3. XPS spectra at different tilting angles from the surface of 6 at. % Cr modified $\text{Fe}_{30}\text{Ni}_{20}\text{Mn}_{35}\text{Al}_{15}$: (a) Cr2p scan and (b) Al2p scan. It is evident that both Cr and Al form protective oxides at the surface.

Figure 4. Peak fitting of the XPS results in Figure 3 after applying background subtraction: (a) Cr2p scan at 30° ; (b) Al2p scan at 30° ; (c) Cr2p scan at 60° and (d) Al2p scan at 60° .

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Graphical Abstract

Comments to journal office:

This manuscript describes the effects of different amounts of Cr on alleviating the environmental embrittlement of eutectic Fe₃₀Ni₂₀Mn₃₅Al₁₅ in air at room temperature. Environmental embrittlement has been reported in a number of ordered intermetallic alloys and is one of the key problems to solve in order to develop intermetallics as high performance structural materials. In this paper, it is shown that the environmental embrittlement of Fe₃₀Ni₂₀Mn₃₅Al₁₅ decreases monotonically with increasing Cr addition from 0.5 at. % to 6 at. %. Possible mechanisms for the effect of Cr are suggested based on a study of the surface oxides of the Cr-modified Fe₃₀Ni₂₀Mn₃₅Al₁₅ alloys using XPS.