Effects of Mn Contents on the Microstructure and Mechanical Properties of the Fe–10Al–xMn–1.0C Alloy

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The microstructures and mechanical properties of the Fe–10mass%Al–(5–40)mass%Mn–1.0mass%C alloys sheet castings have been investigated by using optical microscope (OM), transmission electron microscope (TEM), scanning electron microscope (SEM), experimental model analysis, tensile test and hardness test. Based on the present studies, the macroscopic microstructure of the present alloys are a mixture of austenite (γ) and ferrite (α) duplex phases, and the α phase would decrease with the Mn content increased. In the meantime, according to the examination of TEM, the macroscopic γ phase is a mixture of the γ + fine(Fe, Mn)3AlC1(κ) + (Fe, Mn)3AlC1(κ') phases and the macroscopic α phase is a mixture of the D03 + coarse(Fe, Mn)3AlC1(κ”) phases. The mechanical properties such as the ultimate tensile strength (UTS), yield strength (Y.S.), elongation of these alloys were in the range of 646–887 MPa, 575–824 MPa, and 16.3–29.2%, respectively. The hardness addition, the damping ratios and Young’s modulus of the present alloys were in the range of 0.0603–0.0417, and 139–165 GPa, respectively. It is noted here that the results have never been observed by other research workers in the Fe–Al–Mn–C alloy system.

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1. Introduction

Recently, the microstructures, oxidation resistance and mechanical properties of the Fe–Al–Mn–C alloys system have been extensively studied.1–36) The Fe–Al–Mn–C alloy has a number of features that distinguish it from the other iron-based metals, which make its physical metallurgy both complex and interesting, with low density (nearly 6.6–6.9 g/cm3), no or low magnetism, good atmosphere corrosion resistance, etc. In addition, the alloys could possess high strength and high ductility at room temperature with the fully austenite structure1–19) and some coherent κ phase precipitates. Moreover, in order to improve the corrosion and high temperature oxidation resistance as well as strength, some alloy elements such as Si, Cu, Ti, V, Nb, Mo, W, etc. are added.16–23)

Based on these studies, being solution heat-treated and then quenched, the microstructure of the Fe–Al–Mn–C alloys were a single γ phase or γ phase containing α phase dispersions. After aging processes, some fine L12-type κ phase carbide precipitates8–11,24,25) within the γ matrix. Prolonged the aging times, some κ phase carbides, D03 phase and/or the β-Mn phase started to precipitate on the twin boundaries and grain boundaries.5,11–13,24,25) And some other carbide such as M23C6, M7C3, M2C, would be observed during the aging processes if alloy elements were added.8–11,24,25)

In addition, the mechanical properties of the Fe-Al–Mn–C alloys aged in the temperature range from 450 to 750°C by an optimized heat treatment condition, such as the ultimate strength, yield strength and elongation, were about 590–1765 MPa, 490–1550 MPa, and 10–65%, respectively. Classically, the Fe–Al–Mn–C alloys are prepared with the chemical compositions in the range of Fe–(5–11)mass%Al–(23–35)mass%Mn–(0.5–1.3)mass%C–(Ti, V, Nb, Mo, Si, Cu) by a conventional casting with or without subsequent plastic deformation.7,19,20,27,28) In contrast to the alloys prepared by a conventional casting, the microstructures of as-rapidly solidified Fe–Al–Mn–C alloys with higher carbon contents in the range of 0.74–4.17 mass% have also been examined by many workers,25,27,28) The Mn content of the alloy should be above 23.5% for obtaining the fully austenitic structure,22) and below 35.0% for inhibiting the embrittlement phase precipitated. However, up to date, information concerning the mechanical properties and microstructures of Fe–Al–xMn–C alloys, prepared by a sheet casting, is deficient with various Mn contents. Therefore, the purposes of the present study are to investigate the microstructures and mechanical properties of the as-cast Fe–10Al–(5–40)Mn–1.0C alloy sheet castings.

2. Experimental Procedure

The alloys, Fe–(10 ± 0.2)mass%Al–(5, 10, 15, 20, 25, 30, 35, 40)mass%Mn–(1.0 ± 0.05)mass%C, were melted in an air induction furnace under the N2 atmosphere protection by using AISI 1008 low carbon steel, 99.7% pure electrolytic aluminum, 99.9% pure electrolytic manganese, and pure carbon powder. After melting, the alloys were poured at 1500–1520°C into the investment casting preheated at 1160–1180°C with the 2.5 mm × 15 mm × 125 mm thick sheets. The chemical compositions were identified by atomic emission spectrometry (AES) and are listed in Table 1.

Micro-hardness Rockwell instrument was used to measure the hardness of the alloy sheet castings using ten tests for an average value of each alloy. Tensile specimens with a gauge length of 25 mm and a cross section of 6.25 mm × 2.0 mm were machined from these plates in the direction parallel to their solidification direction. Tensile tests were carried out on an HT-9102 machine at room temperature, with an initial
strain rate of $3.5 \times 10^{-4} \text{s}^{-1}$ and used five specimens to obtain their average. After ultrasonic cleaning in acetone, the fracture surfaces of tested specimens were examined by Hitachi S-2500 SEM operated at 20 kV. The specimens for OM observations were prepared by sectioning, mounting, polishing and etching. The etching solution used was a 5%-nital solution and etching. The etching solution used was a 5% nital solution.

TEM observation was performed on a JEOL 2000FX STEM operated at 20 kV. The specimens for OM observation were prepared by grinding, polishing and etching. The etching solution used was a 5% nital solution.

The Young’s modulus and damping ratio were examined by using the experimental model testing method. Eight types of sheet castings were constructed as a cantilever beam structure, and tested to obtain the system frequency response function (FRF). The natural frequencies and damping ratios can then be extracted through the FRF by the half-power method. Model analysis of the cantilever beam is also performed via finite element analysis to fit the natural frequencies by adjustment via Young’s modulus. Therefore, the damping ration and Young’s modulus of the alloys could be determined.

3. Results and Discussion

3.1 Microstructural analysis

The typical OMs of the present alloys are shown in Fig. 1(a) through 1(h), revealing the dendritic solidification occurred. Macroscopically, there is a mixture region of $\alpha$ phase (white region) and $\gamma$ phase. The $\alpha$ phase consisted in the alloys decreases with increasing Mn content, and the alloy with 40 mass%Mn content is essentially a single $\gamma$ phase. Based on the image software analyzing, the fraction of the $\alpha$ phase using ten specimens for average value of each alloy was examined, as shown in the Table 2. Neglected the effects of the aluminum and carbon contents, it could be also found that the ratios between the percentage of the $\alpha$ phase and Mn mass% contents for various alloys is $\alpha/\% = 0.0244(Mn%)^2 - 2.744(Mn%) + 68.65$ with the error of about 0.93%.

A typical TEM examination is described as following. Figure 2 shows a bright field (BF) electron micrograph of the Fe–10Al–15Mn–1.0C alloy (alloy C). It is obvious that it consists of two regions as marked A and B, where the “A” phase and the “B” region is a $\gamma$ phase by comparing with the Fig. 1(c). Figure 3(a) shows a BF electron micrograph taken from the B region in Fig. 2, revealing that some precipitates marked as C and extremely fine precipitates marked as D were formed within the $\gamma$ matrix. Figures 3(b) and (c), two [001] selected-area diffraction patterns (SADP) taken from the $\gamma$ matrix for different size of precipitates, reveal that in addition to the reflection spots of the $\gamma$ phase, the SADPs also consist of small superlattice spots. Since the diffraction patterns for the $L1_2$ structure phase and the $L1_2$ structure phase are very similar, it is necessary to compare the intensities of diffraction spots from each phase. The structure factor of the $L1_2$-phase precipitate in Fe–Al–Mn–C alloy system can be written as $F_{hkl} = f_{Al} + f_{Fe/Mn} \times (\cos(h + k)\pi + \cos(k + l)\pi + \cos(l + h)\pi)$, where $f_{Al}$ is the atomic scattering amplitude for Al atoms, $f_{Fe/Mn}$ is that for Fe or Mn atoms corrected by their fraction. Comparing to the $L1_2$-structure phase, for the randomly distributed carbon atom within the structure, the structure factor of the $L1_2$-phase precipitate is simply given by $F_{hyd} = f_{Al} + f_{Fe/Mn} \times (\cos(h + k)\pi + \cos(k + l)\pi + \cos(l + h)\pi) = f_{Al} \pm f_{Fe/Mn}$. By taking the chemical composition of the present study into account, the structure factor ratio for (100) and (110) reflections of the $L1_2$-structure is near 9.1 and of $L1_2$-structure being near 1.1. For the intensity of diffraction spot approximating to the square of the structure factor, the intensity of (100) spot for $L1_2$ structure phase is much stronger than that of (110) reflection. And the intensity for (100) and (110) reflections of the $L1_2$-structure is similar.

Therefore, from these superlattice spots and the fact that superlattice 100 spots are much stronger than the 110 spot, it is deduced that the precipitates are the (Fe, Mn)$_3$AlC carbidest with the error of about some precipitates marked as C and extremely fine precipitates marked as D were formed within the $\gamma$ matrix. Figures 3(b) and (c) show the superlattice spots and the 100 spots in the [001] direction.
Fig. 1 Typical optical micrographies of the alloys composed of, (a) 5 mass%Mn, (b) 10 mass%Mn, (c) 15 mass%Mn, (d) 20 mass%Mn, (e) 25 mass%Mn, (f) 30 mass%Mn, (g) 35 mass%Mn and (h) 40 mass%Mn, respectively.

\( \kappa'' \) carbide, same as observed in the matrix. However, from the same strength of superlattice 100, 120 and 110 spots, it is deduced that the particle is having an ordered L1_2-type structure with the lattice parameter \( a = 0.387 - 0.389 \) nm. It also means that the \( \kappa'' \) carbide having L1_2 structure may be formed at a high temperature resulted in more random, disorder car-
bon distribution.\textsuperscript{3,13,16} Figures 5(a) and (b) are two SADPs taken from the matrix marked as A in Fig. 2, revealing that in addition to the reflection spots of the $\alpha$ phase, the SADPs also consist of some superlattice spots. From these superlattice spots, it is deduced that the $\alpha$ matrix has an order D0\textsubscript{3} type structure with the lattice parameter $a = 0.586–0.588$ nm. Figure 6 is a SADP taken from the D0\textsubscript{3} matrix and the particle $\kappa''$, indicating that the orientation relationship between the D0\textsubscript{3} matrix and the $\kappa''$ phase is $[1\tilde{1}1]_{\kappa} \parallel [220]_{\text{D0}_3}$ and $(011)_{{\kappa}} \parallel (001)_{\text{D0}_3}$. Figures 7(a) and (b), two 111 and 200 D0\textsubscript{3} DF electron micrographs, clearly exhibit the presences of $a/4(200)$ and $a/4(111)$ anti-phase boundaries (APDs), respectively. From the fact that the $a/4(111)$ APDs are small than the $a/4(200)$ APDs, it indicates that the crystal structure of the matrix at high temperatures should be a disordered
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The α phase and the D0₃ domains were formed during quenching through a α → B2 → D0₃ continuous ordering transition. Similarly, based on the TEM examination, the α region of the Fe–10Al–xMn–1.0C alloys is all the D0₃ phase containing some coarse κ″ phase precipitates. To sum up, the microstructure of the various Fe–10Al–xMn–1.0C sheet castings is a mixture of (γ + κ + κ″) region and (D0₃ + κ″) region and only the alloy with 40% Mn content is a single γ phase. Moreover, based on TEM examination, it would be observed the amount of κ″ precipitates along the γ/α boundaries, as shown in Fig. 2, are higher than within the D0₃ matrix. However, any β-Mn phase could not be observed in the present study. It is noted here that the results have never observed by other research workers in the Fe–Al–Mn–C alloy system.

3.2 Tensile and hardness test

Tensile properties are shown in Table 3. When the Mn was added, the UTS and Y.S. of the present alloys significantly increased. With the 15 mass%Mn content, the alloy sheet casting possesses maximum UTS = 887 MPa and Y.S. = 824 MPa. However, the UTS and Y.S. gradually decreased as the Mn content more than 20 mass%, as shown in Fig. 8(a). In Table 3 or Fig. 8(b), it is also found that the elongation of the present alloy are in the range of 16.3–29.2%. In addition, based on the hardness test shown in Table 3, it is found that the hardness of the present alloy are in the range of HR C 31–44, and the relationship between the strength and hardness is nearly UTS ≒ 20.93HR C with the error about 2.1%. Figures 9(a) through (c) are three SEM electron mi-
crographs showing the fracture surfaces of specimens taken from the Fe–10Al–10Mn–1.0C alloy (alloy B), the Fe–10Al–25Mn–1.0C alloy (alloy E) and the Fe–10Al–35Mn–1.0C alloy (alloy G), respectively. Compared with the microstructures of the alloys, it implies that the \((D0_3+\kappa''\) two-phase region would play an important role to the fracture mechanism.

### 3.3 Experimental model test

Figures 10(a) through (h) are the frequency response function of the present alloys. The natural frequencies of the Fe–10Al–xMn–1.0C alloy containing Mn of 5.12, 9.85, 14.89, 20.06, 24.87, 30.45, 34.88, 39.73 mass\%, with various thickness are 226, 238, 232, 226, 236, 234, 242 and 242 Hz, respectively. The damping ratios are in the range between 0.06 and 0.04 as shown in Fig. 11, which are extracted through the FRF by the half-power point method. In addition, it is found that damping ratios of the present alloys are decreased as the Mn content increased. Model analysis of the cantilever beam was also performed via finite element analysis to fit the natural frequencies by adjustment of Young’s modulus. Based
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Table 4 The damping ratio and Young’s modulus of the Fe–10Al–xMn–1.0C alloy sheet castings.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
<th>E</th>
<th>F</th>
<th>G</th>
<th>H</th>
</tr>
</thead>
<tbody>
<tr>
<td>Natural frequencies $\omega$ (Hz)</td>
<td>226</td>
<td>238</td>
<td>232</td>
<td>226</td>
<td>236</td>
<td>234</td>
<td>242</td>
<td>242</td>
</tr>
<tr>
<td>Damping ratio ($\xi$)</td>
<td>0.0603</td>
<td>0.0589</td>
<td>0.0561</td>
<td>0.0539</td>
<td>0.0518</td>
<td>0.0491</td>
<td>0.0446</td>
<td>0.0417</td>
</tr>
</tbody>
</table>

Fig. 10 The figures of the frequency response function taken from the alloys consisted of, (a) 5 mass%Mn, (b) 10 mass%Mn, (c) 15 mass%Mn, (d) 20 mass%Mn, (e) 25 mass%Mn, (f) 30 mass%Mn, (g) 35 mass%Mn and (h) 40 mass%Mn. (x axis: Hz; y axis: mass s$^{-2}$N$^{-1}$).
on the present analysis, the Young’s modulus of the present alloys is in the range from 139 to 165 GPa as shown in the Table 4. With the Mn contents between 25 to 30 mass%, the alloy sheet castings possess the maximum Young’s modulus about 159 to 165 GPa. It is noted here that such results have never been reported by other workers in the Fe–Al–Mn–C alloy system.

4. Conclusions

(1) The microstructures of the Fe–10Al–(5–40)Mn–1.0C alloy sheet castings are a mixture of the (γ + κ + κ′) regions and the (D0₃ + κ′) regions, and the (D0₃ + κ′) phase region will decreases with the Mn content increased. With 40 mass% Mn content, the microstructure of the alloy would be a macroscopic single γ phase.

(2) The κ or κ′ phase has an ordered L1₂-type structure, and formed during quenching by, presumably, spinodal decomposition, and the chemical fluctuations were occurred between the (κ + κ′) carbides and the austenite matrix. On the other hand, the κ″ carbide has an ordered L1₂-type structure, and the carbon solution of the κ″ carbides would be lower than the κ′ and κ carbides, and being more random disorder distribution.

(3) The α matrix has an order D0₃-type structure formed during quenching through a α → B2 → D0₃ continuous ordering transition. The orientation relationship between the D0₃ matrix and the κ″ phase is [111]ₜ ↔ [220]₀ and (011)ₜ ↔ (001)D₀₃.

(4) The hardness, UTS, Y.S., elongation, damping ratio and Young’s modulus of the Fe–10Al–(5–40)Mn–1.0C alloys were in the range of HRc 31–44, 646–887 MPa, 575–824 MPa, 16.3–29.2%, 0.0603 and 0.0417, and 139–165 GPa, respectively. With near the 15 mass% Mn content, the alloy sheet casting possesses maximum UTS and Y.S.

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REFERENCES