

## Oxidation of Fe-(5.3-29.8)%Mn-(1.1-1.9)%Al-0.45%C Alloys at 550-650 °C

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Alloys of Fe-(5.3-29.8)%Mn-(1.1-1.9)%Al-(0.4-0.5)%C were oxidized at 550 °C to 650 °C for 20 h to understand effects of alloying elements on oxidation. Their oxidation resistance increased with increasing Mn level to a small extent. Their oxidation kinetics changed from parabolic to linear when Mn content was decreased and temperature was increasing. Oxide scales primarily consisted of Fe<sub>2</sub>O<sub>3</sub>, Mn<sub>2</sub>O<sub>3</sub>, and MnFe<sub>2</sub>O<sub>4</sub> without any protective Al-bearing oxides. During oxidation, Fe, Mn, and a lesser amount of Al diffused outward, while oxygen diffused inward to form internal oxides. Both oxide scales and internal oxides consisted of Fe, Mn, and a small amount of Al. The oxidation of Mn and carbon transformed  $\gamma$ -matrix to  $\alpha$ -matrix in the subscale. The oxidation led to the formation of relatively thick oxide scales due to inherently inferior oxidation resistance of alloys and the formation of voids and cracks due to evaporation of manganese, decarburization, and outward diffusion of cations across oxides.

**Keywords:** High-Mn steel, Oxidation, Oxide scale, Iron, Manganese

### 1. Introduction

Fe-Mn-Al-C alloys have attracted much interest in the last decades as potential candidates to replace the costly Fe-Ni-Cr stainless steels because of their cost effectiveness, high strength, good formability, tensile strength, low density, and low-temperature properties [1,2]. Their microstructures and mechanical properties have been actively studied for applications in automobiles and oil and gas exploration [3-5]. Previously, oxidation studies were performed in the composition range of Fe-(1-32)Mn-(1.5-16)Al-(0-1)C in a temperature range of 600-1250 °C in air or oxygen [6-11]. All chemistry values are in weight percent unless otherwise specified. The oxidation of Fe-(7.5-30.1)Mn-(5-16)Al-(0, 0.77)C at 600-1000 °C led to the formation of an alumina layer interrupted by nodules that consisted mostly of manganese oxide [6,7], and needle-like AlN precipitated beneath these nodules [7]. The oxidation of Fe-31Mn-9Al-0.87C at 800 - 1100 °C

resulted in formation of the outer (Mn,Fe)<sub>2</sub>O<sub>3</sub>-rich and inner (Mn,Fe)<sub>3</sub>O<sub>4</sub>-rich layer [8,9], whereas that of Fe-32Mn-7.5Al-0.6C at 1000 °C led to the formation of a discontinuous alumina layer interspersed with a less protective MnAl<sub>2</sub>O<sub>4</sub> spinel [10]. Fe-30Mn-5Al-0.5C formed the oxide scale consisting of a thin surface Mn<sub>2</sub>O<sub>3</sub> film, a thick MnFe<sub>2</sub>O<sub>4</sub> layer, and an internal oxidation zone at 600 - 900 °C [11]. As was stated above, diverse kinds of oxides, scale morphologies, and dominant oxidation processes were reported depending on the alloy composition and oxidation conditions.

This study aims at characterizing the oxidation behavior of a new kind of lightweight Fe-Mn-Al-C alloys and comparing the results with the results obtained from oxidation of iron and manganese. It is noted that oxidation studies on manganese are scarce because manganese is usually used as an alloying element [12-14]. In the present work, Fe-(5.29-29.8)Mn-(1.09-1.89)Al-(0.44, 0.45)C alloys were manufactured and oxidized at 550 - 650 °C for 20 h. Such an oxidation condition was selected because stress-relieving temperature of Fe-Mn-Al-C alloys, which were welded with other low alloy steels in automobile applications, lies in the range of 500 - 700 °C for 10-40 h

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**Table 1. Chemical composition of Fe-Mn-Al-C alloys**

Designation	Composition (wt%)
5.3Mn	Fe-5.29Mn-1.89Al-0.45C
10.2Mn	Fe-10.20Mn-1.85Al-0.45C
15.1Mn	Fe-15.06Mn-1.60Al-0.44C
20.4Mn	Fe-20.38Mn-1.49Al-0.45C
25.9Mn	Fe-25.91Mn-1.27Al-0.45C
29.8Mn	Fe-29.80Mn-1.09Al-0.44C

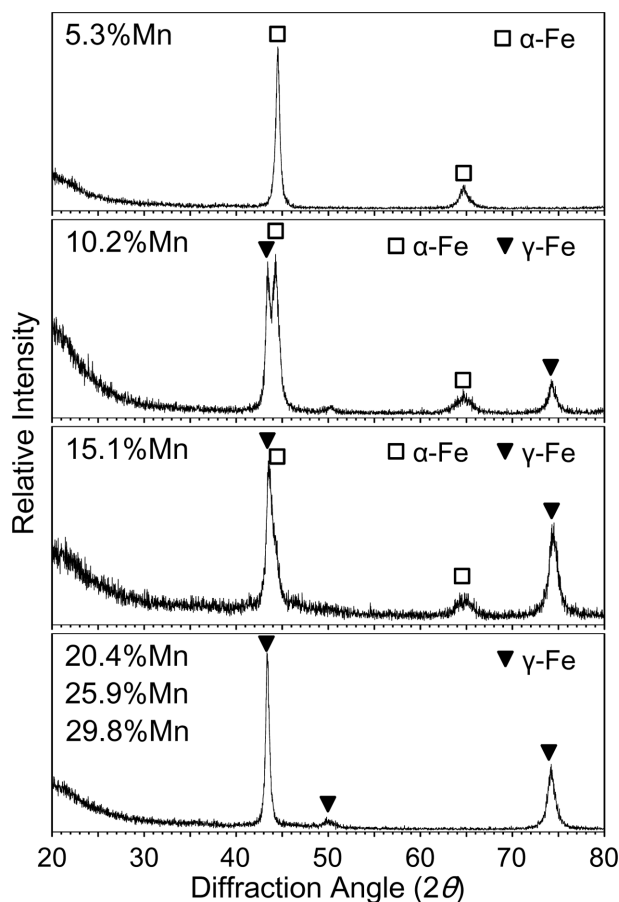
[5,6]. The effect of chemical compositions of Fe-Mn-Al-C alloys on oxidation was discussed in this study.

## 2. Experimental Methods

Table 1 lists the chemical composition of six different Fe-Mn-Al-C alloys. The alloy compositions were designed to achieve high strength and good formability with high elongation for automotive applications. The alloys were induction-melted under vacuum, cast into slabs with a 6 cm thickness, reheated at 1200 °C for 1 h, hot rolled to a 3 mm thickness, water quenched to 650 °C, homogenized at 650 °C for 1 h, and then cooled to room temperature. They were cut into 10 mm × 10 mm × 3 mm coupons, ground to 1000-grit with abrasive paper to remove surface scales, ultrasonically cleaned in acetone and alcohol, and oxidized at 550, 600, and 650 °C for 20 h in atmospheric air using a thermogravimetric analyzer. For comparison purposes, 99.9% pure Fe and Mn sheets were also oxidized. The oxidized specimens were inspected using a scanning electron microscope (SEM) equipped with an energy-dispersive spectrometer (EDS), an electron probe microanalyzer (EPMA), and an X-ray diffractometer (XRD) with Cu-K $\alpha$  radiation at 40 kV and 150 mA.

## 3. Results and Discussion

Fig. 1 shows XRD patterns of Fe-Mn-Al-C alloys. Here, 5.3%Mn was ferritic, and 10.2%Mn was mixed with ferrite and austenite. As the Mn level increased from 10.2% to 15.1%, the diffraction intensity of ferrite decreased, while that of austenite increased. Only austenite was detected for (20.4, 25.9, 29.8)% Mn, because the concentration of austenite-stabilizing manganese increased, whereas that of ferrite-stabilizing



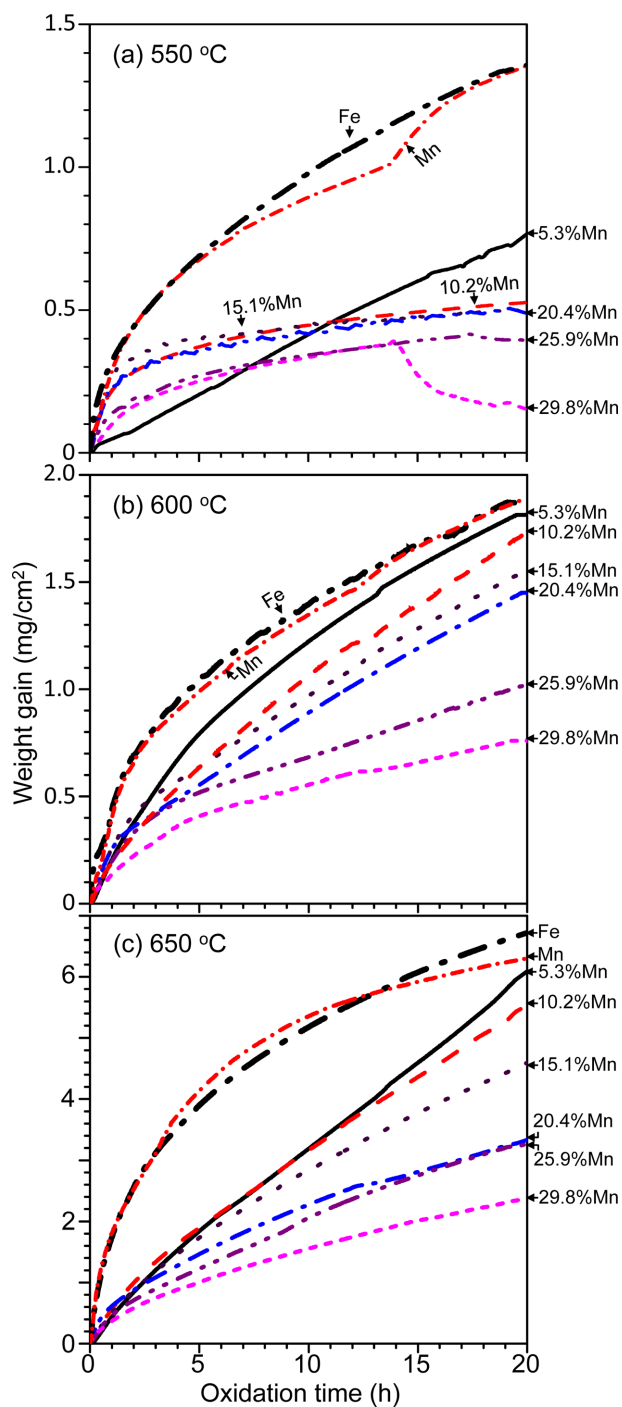
**Fig. 1. XRD patterns of Fe-Mn-Al-C alloys**

aluminum decreased.

Fig. 2 shows oxidation curves of Fe, Mn, and Fe-Mn-Al-C alloys at 550 - 650 °C. Generally, weight gain increased with increasing oxidation time and temperature due to the enhanced reaction and diffusion rate. At 550 °C, iron oxidized parabolically, whereas manganese suffered accelerated oxidation at 13.8 h owing to breakage of its oxide scale. At 550 °C, 5.3Mn oxidized linearly, (10.2, 15.1, 20.4)Mn oxidized almost parabolically after initial transient period of 2.5 h, and (25.9, 29.8)Mn suffered partial scale spallation at (17.3, 14) h, respectively, owing to insufficient oxide plasticity. At 600 and 650 °C, oxidation curves were smooth, mainly because the plasticity of oxide scales increased at higher temperatures. Additional minor reason was the eclipse of small weight fluctuations due to local scale breakage/spallation by large weight gains. At 550 - 650 °C, iron and manganese gained more weights than Fe-Mn-Al-C alloys. Completely different oxide scales formed on iron, manganese, and Fe-Mn-Al-

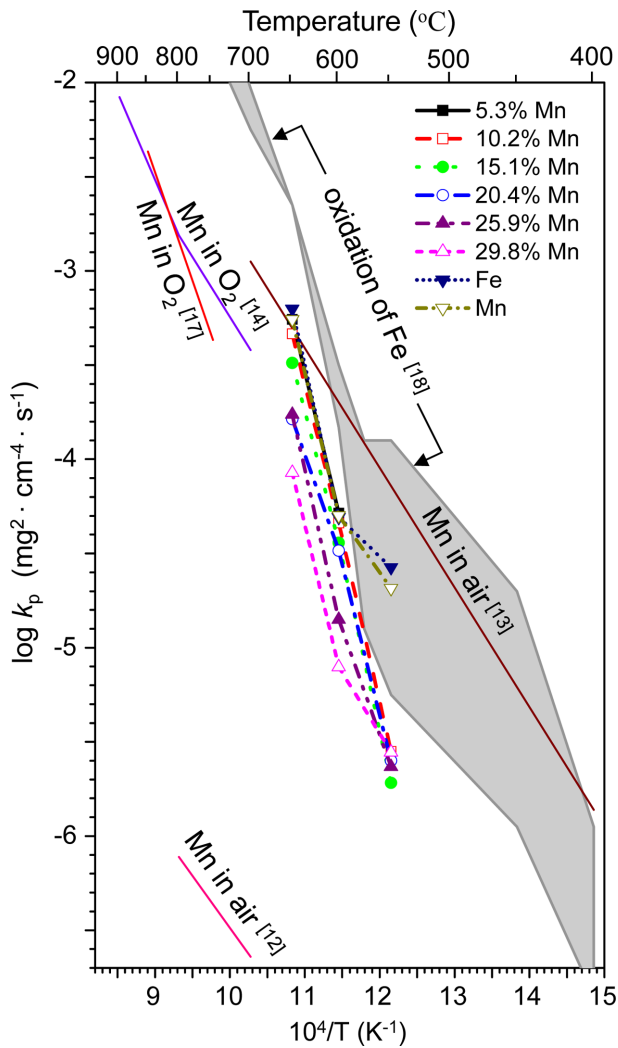
C alloys, as will be shown later. Generally, high-Mn alloys gained less weights than low-Mn alloys, although the former had lower amounts of aluminum than the latter. At each oxidation temperature, high-Mn and low-Mn alloys tended to follow parabolic and linear oxidation kinetics, respectively. Hence, it is seen that Mn beneficially increased the isothermal oxidation resistance of current alloys. However, some authors have suggested that manganese and carbon decreased the oxidation resistance of Fe-Mn(-Al-C(-Cr)) alloys due to decarburization and the selective oxidation of manganese [6,8,15,16]. Such apparent discrepancy between this paper and previous studies is mainly due to the different levels of the alloying elements, which generated dissimilar scale microstructures consisting of diverse oxides, depending on the alloy composition. For example, ours had (1.1-1.9)Al, while others had (5-16)Al. Resultantly, ours did not form any Al-bearing oxides according to the XRD analysis, while others reported the formation of  $\text{Al}_2\text{O}_3$ ,  $(\text{Fe}, \text{Mn})\text{Al}_2\text{O}_4$ , and/or  $\text{MnAl}_2\text{O}_4$  [6,8,15,16]. Previously, manganese was explained to be more oxidation resistant than iron [6]. However, this study indicated that the oxidation resistances of both manganese and iron was poor, and the difference between them was considered to be marginal.

Assuming that the parabolic oxidation law is obeyed for the sake of simplicity, the oxidation curves shown in Fig. 2 were fitted to the equation  $\Delta W^2 = k_p t$ , where  $\Delta W$  is the weight gain per unit area,  $t$  is the oxidation time, and  $k_p$  is the parabolic rate constant. The deviation of Fig. 2 from the parabolic oxidation law resulted from experimental scattering, partial scale detachment/spallation, and gradual transition from parabolic to linear oxidation with increasing the temperature and decreasing Mn level in Fe-Mn-Al-C alloys. Values of  $k_p$  of Fe-Mn-Al-C alloys were measured for the oxidation period of 2.5-20 h, excluding the initial transient period. Also, the oxidation data of (Mn after 13.8 h), (25.9Mn after 17.3 h), and (29.8Mn after 14 h) at 550 °C were disregarded due to accelerated oxidation, or partial spallation of oxide scales. Fig. 3 indicate that manganese oxidized slightly more slowly than iron. The oxidation resistance of Fe-Mn-Al-C alloys increased with increasing Mn level to a small extent. Our  $k_p$  values of iron and manganese were reasonably located around those of iron and manganese



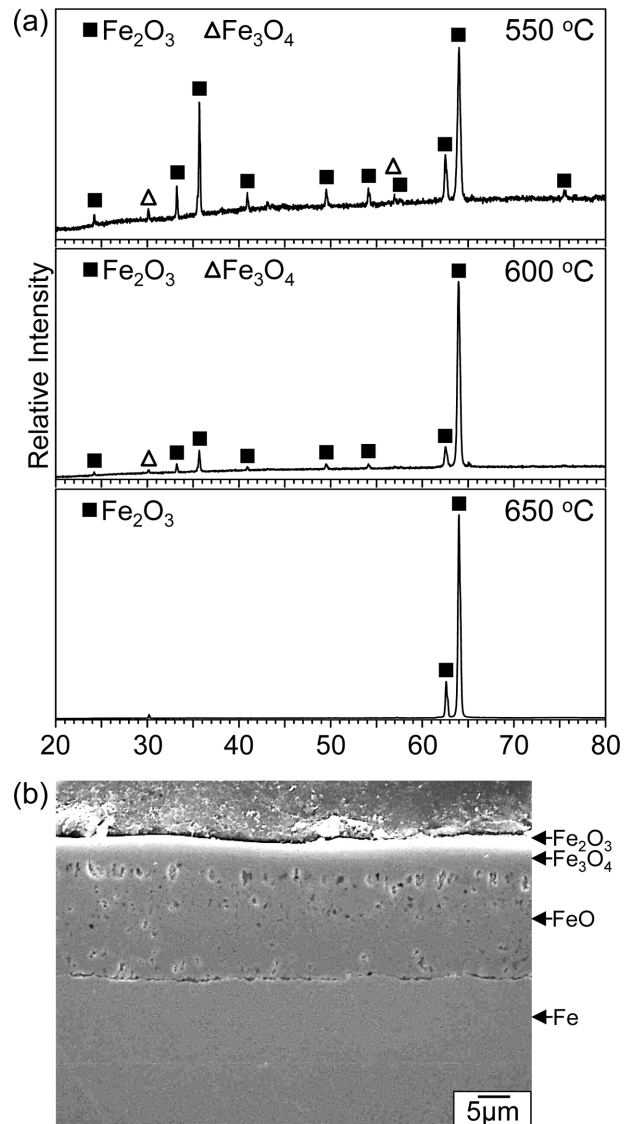
**Fig. 2.** Oxidation curves of Fe, Mn, and Fe-Mn-Al-C alloys at (a) 550 °C, (b) 600 °C, (c) 650 °C

reported previously [13,14,17,18]. Values of  $k_p$  of manganese in air reported by Buscail and Larpin were too low [12]. Those of iron in 1 atm of oxygen or air, which were compiled by Bertrand et. al. [18] indicated that they were broadly independent of oxygen pressure.



**Fig. 3.** Arrhenius plot of parabolic rate constants for the oxidation of Fe, Mn, and Fe-Mn-Al-C alloys. The  $k_p$  values for the oxidation of manganese in 1 atm of air [12,13] and oxygen [14,17] as well as iron in 1 atm of air and oxygen [18] are superimposed

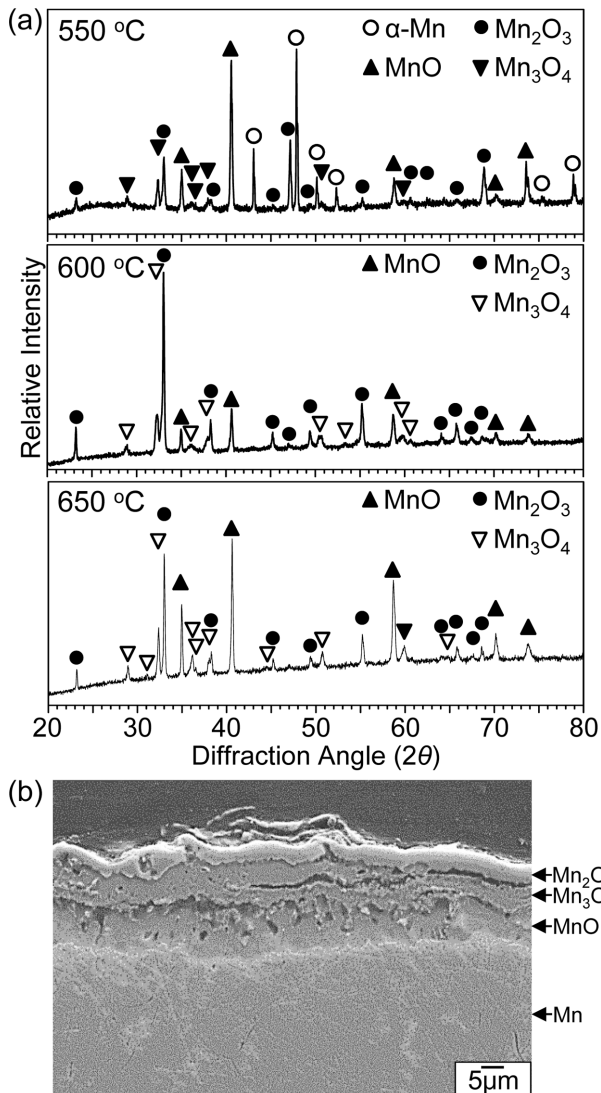
When iron is exposed to air above 570 °C, it grows a scale consisting of an inner, thick FeO layer, an intermediate, thin Fe<sub>3</sub>O<sub>4</sub> layer, and an outer, extremely thin Fe<sub>2</sub>O<sub>3</sub> layer, because nonstoichiometric Fe<sub>1-x</sub>O grows much faster than stoichiometric Fe<sub>3</sub>O<sub>4</sub> and Fe<sub>2</sub>O<sub>3</sub> [18,19]. Below 570 °C, FeO is unstable so that an inner, thin Fe<sub>3</sub>O<sub>4</sub> layer, and an outer, thinner Fe<sub>2</sub>O<sub>3</sub> layer form. The growth of FeO, Fe<sub>3</sub>O<sub>4</sub> and Fe<sub>2</sub>O<sub>3</sub> is primarily governed by the diffusion of Fe<sup>2+</sup>, (Fe<sup>2+</sup>, Fe<sup>3+</sup>), and (Fe<sup>3+</sup>, O<sup>2-</sup>) ions, respectively [18,19]. At 550 - 650 °C, the intensity of the XRD pattern of the outer Fe<sub>2</sub>O<sub>3</sub> layer was stronger than that of the underlying Fe<sub>3</sub>O<sub>4</sub> layer (Fig. 4a). FeO was not detected in this figure because of its instability at 550 °C, and the presence of



**Fig. 4.** Iron oxidized for 20 h. (a) XRD patterns at 550, 600, and 650 °C, (b) cross-sectional SEM image at 600 °C

overlying Fe<sub>3</sub>O<sub>4</sub>/Fe<sub>2</sub>O<sub>3</sub> layer at 600 - 650 °C. The outward diffusion of Fe<sup>2+</sup> ions from the FeO/Fe interface toward the FeO/Fe<sub>3</sub>O<sub>4</sub> interface accompanies the counter-diffusion of cation vacancies toward the FeO/Fe interface [19]. Hence, the fast growing FeO layer had numerous voids, reflecting a high cation-vacancy concentration (Fig. 4b).

Manganese oxidizes in a manner similar to iron, forming multi-layer scales. It forms an inner, thick MnO layer, an intermediate, a less thick Mn<sub>3</sub>O<sub>4</sub> layer, and an outer, thin Mn<sub>2</sub>O<sub>3</sub> layer [12]. Mn<sub>2</sub>O<sub>3</sub> becomes unstable above 880 °C. Like FeO, MnO exhibits a relatively large deviation from stoichiometry. Mn<sub>1-x</sub>O grows faster than



**Fig. 5. Manganese oxidized for 20 h. (a) XRD patterns at 550, 600, and 650 °C, (b) cross-sectional SEM image at 600 °C**

Mn<sub>3-3</sub>O<sub>4</sub> or Mn<sub>2</sub>O<sub>3-z</sub> [20]. Fig. 5a indicates the formation of MnO, Mn<sub>3</sub>O<sub>4</sub>, and Mn<sub>2</sub>O<sub>3</sub> at 550 - 650 °C. At 550 °C, strong α-Mn peaks were additionally detected due to partial spallation of the oxide scale during the scaling and subsequent cooling stage. A non-uniform, and loosely adherent oxide scale was shown in Fig. 5b. The outer Mn<sub>2</sub>O<sub>3</sub> layer convoluted and partially detached due to the developed growth and thermal stress. When manganese is oxidized primarily by the outward diffusion of cations, cation vacancies form to generate voids, especially in the MnO layer [21]. Manganese, whose vapor pressure is much higher than that of iron or aluminum, also diffuses outward through voids. For example, the equilibrium

vapor pressures of Mn, Fe, and Al are calculated as  $1.86 \times 10^{-10}$ ,  $1.16 \times 10^{-17}$ , and  $1.88 \times 10^{-13}$  (atm) at 600 °C, respectively, from the thermodynamic data [22]. Fast oxidation, void formation, local cracking, and spallation of the oxide scales were the characteristics of Mn oxidation (Fig. 5b).

Fig. 6 shows the XRD/SEM/EPMA results of 10.2 Mn after oxidation at 650 °C for 20 h. The intensity of the XRD pattern shown in Fig. 6a decreased in the order of Fe<sub>2</sub>O<sub>3</sub>, Mn<sub>2</sub>O<sub>3</sub>, and MnFe<sub>2</sub>O<sub>4</sub>. Hematite reacted partially with MnO to yield the MnFe<sub>2</sub>O<sub>4</sub> spinel. Higher manganese levels formed more manganese oxides and MnFe<sub>2</sub>O<sub>4</sub>. The MnFe<sub>2</sub>O<sub>4</sub> spinel might grow more slowly than manganese- or iron-oxides. It might play a major role in improving the oxidation resistance of Fe-Mn-Al-C alloys (Fig. 2). Nonetheless, oxidation resistance of Fe-Mn-Al-C alloys was inferior, because the oxide scales were mixed with Fe<sub>2</sub>O<sub>3</sub>, Mn<sub>2</sub>O<sub>3</sub>, and MnFe<sub>2</sub>O<sub>4</sub>. The platinum wire, both end of which was spot welded on the alloy surface prior to oxidation for good contact, was found above the inner oxide scale (Fig. 6b). This marker test indicates that the outer and inner oxide scale were formed by the outward diffusion of cations and oxygen ions, respectively. The 40 μm-thick outer scale was detached from the 30 μm-thick inner scale to relieve thermal and growth stresses generated during oxidation and subsequent cooling. The enlarged cross-sectional EPMA image and corresponding elemental maps of the oxide scale are shown in Fig. 6c and d, respectively. Detachment of the outer scale from the inner scale was accompanied by the cracking at the bottom of the outer oxide scale (Fig. 6c). The outer scale was enriched with Fe, Mn, and Al (Fig. 6d). Manganese diffused outward strongly into the outer scale, which was assisted by its evaporation and relatively rapid rate of diffusion across the scale. Strong outward diffusion of manganese was similarly reported from the high-temperature oxidation of Fe-30.1Mn-9.7Al-0.77C alloy [7], Fe-30Mn-5Al-0.5C alloy [11], and Fe-28Cr-1Mn alloy [23]. Aluminum diffused outward weakly due to its low concentration in the alloy, and low diffusivity in oxides resulting from its high oxygen affinity. On the other hand, the absence of any Al-bearing oxides in Fig. 6a was attributed to their small amount, or dissolution in other oxides. For example, Al<sub>2</sub>O<sub>3</sub> can dissolve in Fe<sub>2</sub>O<sub>3</sub> and Mn<sub>2</sub>O<sub>3</sub> up to ~ 5 and 10%, respectively. Inwardly diffusing

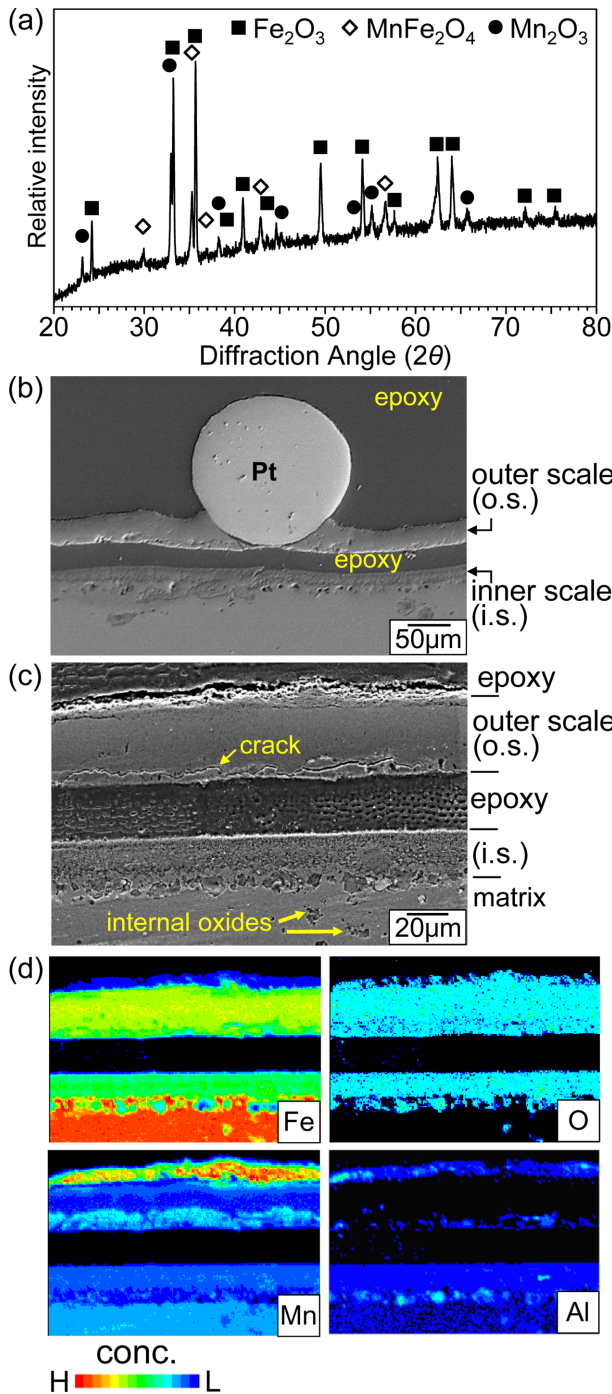


Fig. 6. Fe-10.2Mn-1.9Al-0.5C alloy after oxidation at 650 °C for 20 h. (a) XRD pattern, (b) SEM cross-sectional image, (c) EPMA cross-sectional image, (d) EPMA maps of (c)

oxygen ions formed the (Fe, Mn, Al)-mixed inner oxide scale and internal oxides. Internal oxidation of aluminum hindered the outward diffusion of aluminum and the buildup of the Al-bearing protective oxide layer. Spallation and delamination of the oxide scale facilitated internal oxidation.

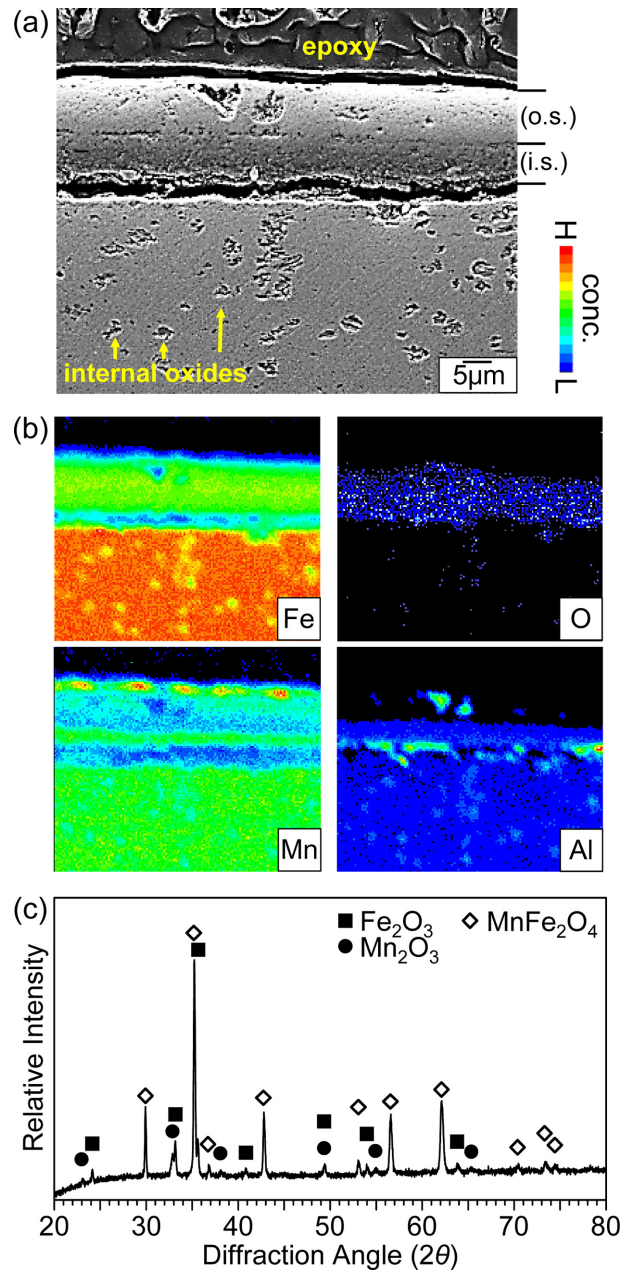


Fig. 7. Fe-10.2Mn-1.9Al-0.5C alloy after oxidation at 600 °C for 20 h. (a) EPMA cross-sectional image, (b) EPMA maps of (a), (c) XRD pattern of the oxide scale

Fig. 7 shows the EPMA/XRD results of 10.2Mn after oxidation at 600 °C for 20 h. The thickness of the oxide scale decreased to 17 μm (Fig. 7a). The oxide scale consisted of Fe, Mn, and a lower amount of Al (Fig. 7b). This figure indicates that more Mn diffused outward to the top surface than Al, making the figure analogous to Fig. 6d. Again, the outward diffusion of Al was limited due to the low Al levels in the alloy and the strong oxygen

affinity of Al. Manganese was unevenly distributed in the scale, and it was depleted in the subscale because of its strong tendency to diffuse outward. Both the oxide scale and internal oxides consisted of Fe, Mn, and Al. Unlike Fig. 6b, Fig. 7a shows that the bi-layered oxide scale was not yet split into two, because a thinner oxide scale formed. The oxide scale was loosely adherent, due to accumulated growth and thermal stress, voids that formed by the outward diffusion of cations, evaporation of Mn, and decarburization of the alloy [9]. The XRD pattern shown in Fig. 7c indicates that the intensity decreased in the order of  $\text{MnFe}_2\text{O}_4$ ,  $\text{Fe}_2\text{O}_3$ , and  $\text{Mn}_2\text{O}_3$ . Again, Al-bearing oxides such as  $\text{Al}_2\text{O}_3$ ,  $\text{FeAl}_2\text{O}_4$ , and  $\text{MnAl}_2\text{O}_4$ , which were found in Fe-31Mn-9Al-0.87C alloys after oxidation at 800 - 1100 °C [13] were absent due to their

small amount, and/or the dissolution of  $\text{Al}_2\text{O}_3$  in isostructural  $\text{Fe}_2\text{O}_3$ .

The alloy outlined in Figs. 6 and 7 was oxidized at 550 °C and analyzed using XRD/EPMA. The results are shown in Fig. 8. The  $\alpha$ -Fe matrix was strongly detected since the oxide scale consisting of  $\text{Fe}_2\text{O}_3$  and  $\text{MnFe}_2\text{O}_4$  was thin (Fig. 8a).  $\text{Mn}_2\text{O}_3$  was no longer detected, due to its small amount or dissolution in  $\text{Fe}_2\text{O}_3$ , which can dissolve up to 5%  $\text{Mn}_2\text{O}_3$  [24]. No Al-bearing oxides were detected. The oxide scale was 4  $\mu\text{m}$ -thick and did not yet develop into bi-layered oxide scale (Fig. 8b). It consisted of Fe, Mn, and a lower amount of Al (Fig. 8c). Again, manganese diffused outward into the oxide scale more strongly than aluminum, reflecting its high diffusivity in the oxide scale. This resulted in the enrichment of aluminum and the depletion of manganese around the scale/matrix interface, below which oxygen dissolved to a certain extent. In this study, competitive oxidation of Fe, Mn, Al, and possibly carbon was unavoidable due mainly to absence of a protective barrier oxide layer.

#### 4. Conclusions

The Fe-5.3Mn-1.9Al-0.5C alloy was ferritic, Fe-(10.2-15.1)Mn-(1.6-1.9)Al-(0.4-0.5)C alloys consisted primarily of ferrite and austenite, while Fe-(20.4-29.8)Mn-(1.1-1.5)Al-(0.4-0.5)C alloys were austenitic. All these alloys oxidized relatively fast to  $\text{Fe}_2\text{O}_3$ ,  $\text{Mn}_2\text{O}_3$ , and  $\text{MnFe}_2\text{O}_4$  at 550 - 650 °C. They oxidized more slowly than iron and manganese. Manganese that formed  $\text{MnO}$ ,  $\text{Mn}_3\text{O}_4$ , and  $\text{Mn}_2\text{O}_3$  oxidized slightly more slowly than iron that formed  $\text{FeO}$ ,  $\text{Fe}_3\text{O}_4$ , and  $\text{Fe}_2\text{O}_3$ . High-Mn alloys displayed better oxidation resistance than low-Mn alloys, indicating that manganese was beneficial due to the formation of an increased amount of  $\text{MnFe}_2\text{O}_4$  spinel. The growth of outer and inner oxide scales was governed by the outward diffusion of ( $\text{Fe}^{2+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Mn}^{2+}$ ,  $\text{Mn}^{3+}$ ,  $\text{Al}^{3+}$ )-ions and inward diffusion of  $\text{O}^{2-}$  ions, respectively. The outward diffusion of cations, volume expansion caused by the formation of oxides, vaporization of manganese, and decarburization generated pores and cracks in scales. Inward diffusion of anions resulted in the formation of the inner oxide layer and internal oxides consisting of Fe, Mn, and Al. These elements also made up the outer oxide layer.

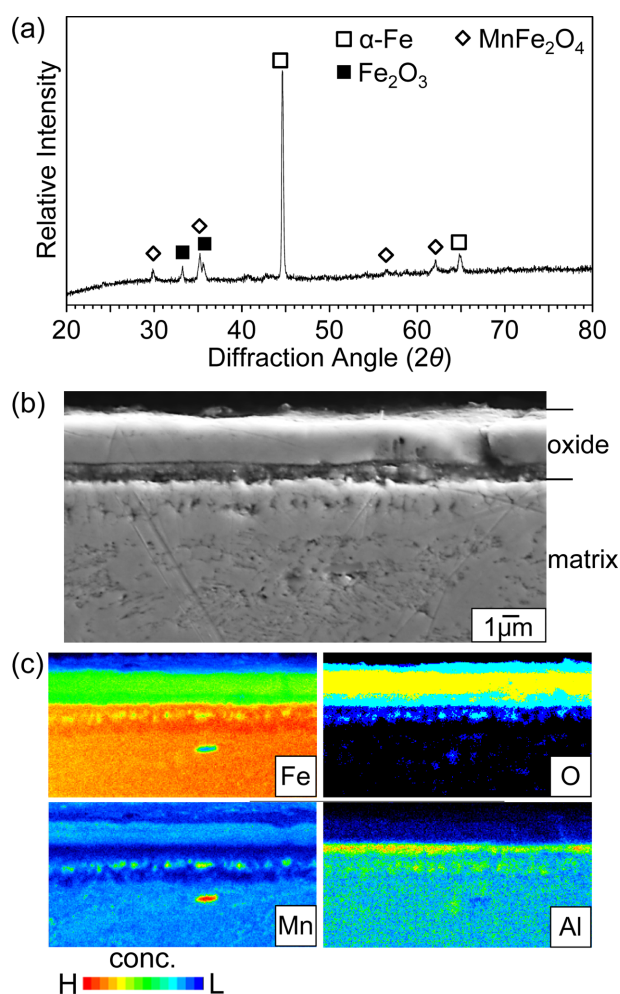


Fig. 8. Fe-10.2Mn-1.9Al-0.5C alloy after oxidation at 550 °C for 20 h. (a) XRD pattern, (b) EPMA cross-sectional image, (c) EPMA maps of (b)

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