# HIGH-TEMPERATURE OXIDATION BEHAVIOUR OF Fe11Al BASED ALLOYS

**Syahril, Wagiyo H, Bambang Sugeng, and Ari Handayani**
*Research and Development Centre for Materials Science and Technology-Batan*
*Kawasan Puspiptek, Serpong,,Tangerang 15314*

## ABSTRACT

**HIGH-TEMPERATURE OXIDATION BEHAVIOUR OF Fe11Al BASED ALLOYS**. Thermomechanically treated Fe11Al based alloys were isothermally oxidised in air at 900, 1000 and 1100 °C respectively. Their oxidation behaviour was studied using thermogravimetric analyser (TGA), X-ray diffractometer (XRD), and scanning electron microscope (SEM). It was found that the oxide scales formed on alloys studied consisted mainly of $\alpha$- and $\theta$-$Al_2O_3$. Additions of 0.5 wt. % Nb and Mo significantly improved the oxidation resistance of the alloys.

***Key words***: iron aluminium alloys, thermomechanical treatment, ternary alloying additions, oxidation resistance.

## ABSTRAK

**ANALISIS KETAHANAN KOROSI SUHU TINGGI PADUAN BERBASIS Fe11Al**. Telah dilakukan pengujian korosi suhu tinggi terhadap paduan berbasis Fe11Al pada 900, 1000 dan 1100 °C. Uji korosi dilakukan menggunakan *thermogravimetric analyser* (TGA). Lapisan oksida yang terbentuk dikarakterisasi dengan difraktometer sinar X (XRD) dan mikroskop electron sapuan (SEM). Hasil karakterisasi menunjukan lapisan oksida yang terbentuk terdiri dari $\theta$-$Al_2O_3$ dan $\alpha$ – $Al_2O_3$. Penambahan unsur pemadu Nb dan Mo masing-masing sebanyak 0,5 wt. % meningkatkan ketahan korosi bahan secara signifikan.

***Kata kunci*** : paduan Fe11Al, *thermomechanical treatment, ternary alloying additions, oxidation resistance.*

## INTRODUCTION

Iron aluminium alloys are of interest as low cost, structural materials and are currently receiving renewed attention because of their potential for applications where good strength and oxidation resistance at high temperature are required. However, as yet, they have not been commercially exploited due the difficulty in fabrication.

Many studies have been conducted to improve formability but despite some progress, the problem remains incompletely resolved; these alloys can be hot worked but show limited ductility at room temperature [1-5].

Most of the studies were conducted on or around $Fe_3Al$ intermetallic composition and the poor formability and ductility have been commonly associated with ordering that takes place within this compositional range. Development of alloys of lower Al content, containing sufficient Al to ensure good oxidation resistance but without producing long-range ordering, is a potential solution to this problem [6].

This paper reports the oxidation characteristics of such alloys delineating the effect of ternary additions on the oxide scale structure, morphology and its adherence.

## EXPERIMENTAL PROCEDURE

Alloys of nominal compositions (in wt. %): Fe11Al, Fe11Al 0.5Nb, and Fe11Al 0.5Mo were produced from spectrographically pure elements. The raw materials were melted in an arc melter under a reduced-pressure atmosphere. The ingots produced were hot and warm rolled to obtain plate samples of 1 mm thick. Details on the alloy synthesis and thermomechanical treatment were given elsewhere [6].

Specimens of 10x10x1 $mm^3$ were cut from the plates, ground and polished down to 1 mm surface finish, degreased and rinsed in alcohol and dried. Isothermal oxidation tests were performed in a magnetic suspension balance (MSB) thermal analyser at 900, 1000 and 1100 °C respectively. All measurements were carried in ambient atmosphere, each for 100 hours.

The mass changes of the specimens were measured by a magnetic balance with an accuracy of

30 μg. The oxidised specimens were cooled at a rate of 20°C/min to room temperature. The oxide scales were characterised using Cu-Kα XRD and SEM.

## RESULTS

### Isothermal Oxidation Kinetics

The oxidation curves of binary Fe11Al and ternary Fe11Al0.5Nb and Fe11Al0.5Mo alloys are plotted in Fig. 1, showing the mass gains versus time at 900, 1000 and 1100 °C. The binary Fe11Al exhibited a very low oxidation rate at 900 °C, but the rate increased rapidly at 1000 °C and more swiftly at 1100 °C. Additions of Nb and Mo significantly improved the oxidation resistance at 1000 °C, with Mo containing alloy showing the lowest oxidation rate.

A large mass gain, in particular for the binary Fe11Al oxidised at 1000 and 1100 °C, took place at a very short exposure time. The rest of the curves follow the classical parabolic law, a feature commonly observed in high temperature corrosion, e.g. in b-NiAl [7], FeAl [8] and $Fe_3Al$ [2] intermetallics.

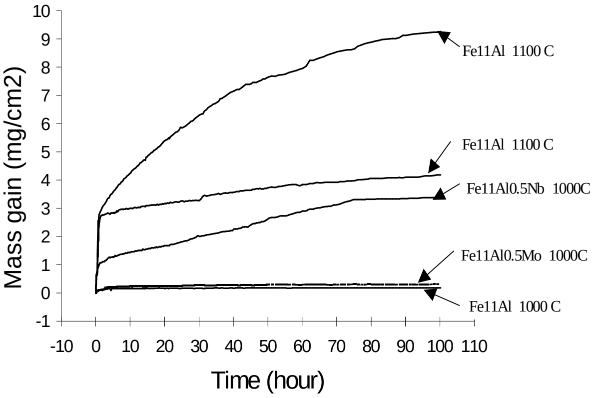

***Figure 1***. Effect of alloying addition and temperature on the oxidation behavior of Fe11Al based alloys.

### Morphologies of Oxide Scales

The binary Fe11Al oxidised at 1000 °C for 100 hours exhibited a non uniform, nodular oxide scales (Fig. 2). The oxide layers had poor adherence, with crater-like cracked oxides dominating the entire surface. The oxide morphologies of the Nb and Mo containing alloys oxidised at the same temperature are shown in Fig. 3 and 4 respectively. The oxide layers in both alloys were very uniform with no spallation observed, though the Mo containing alloy showed a preferential oxide growth at grain boundaries of the substrate. This phenomenon was not observed in the Nb containing alloy. Both alloys exhibited a cigar-like oxide morphology.

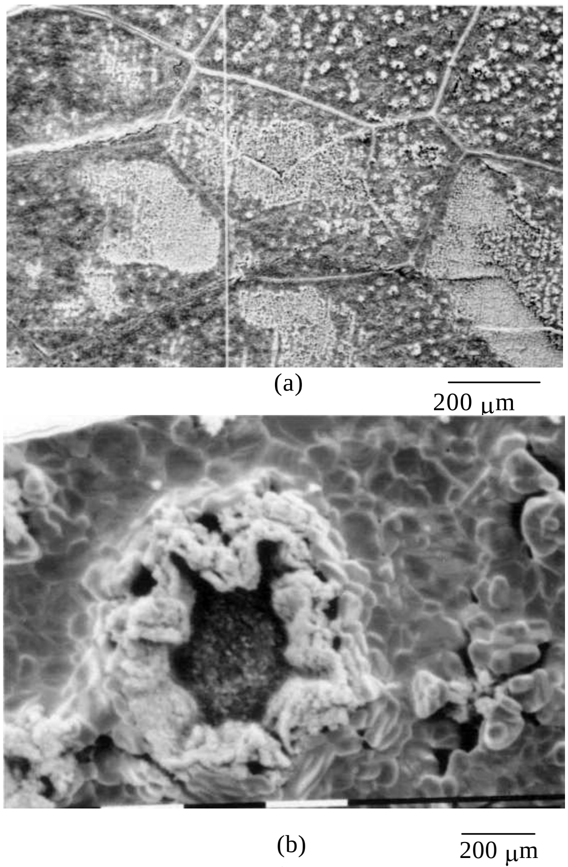

***Figure 2***. (a) Morphology of oxide scale in Fe11Al alloy after oxidation 100 hours. (b) Enlargement of one of the white spot in (a), showing a oxide

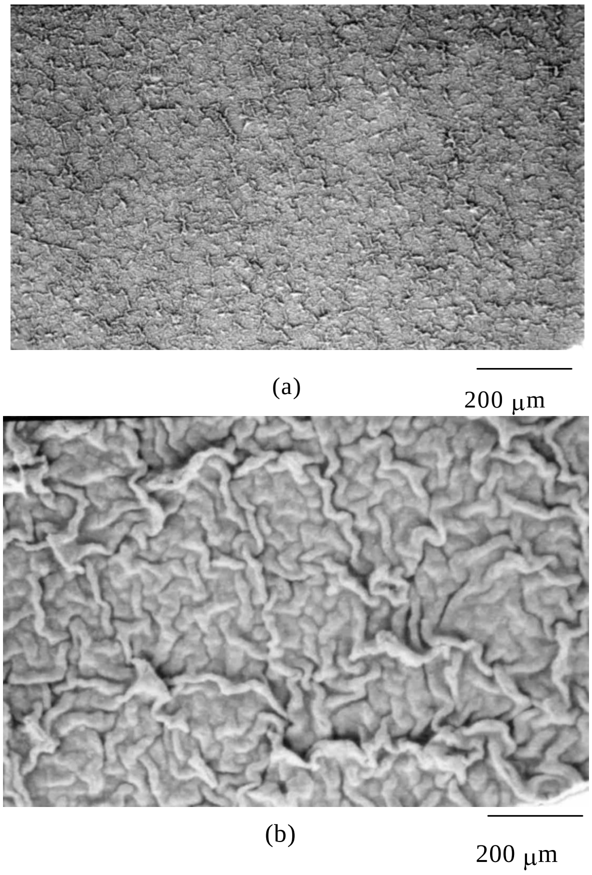

***Figure 3***. Morphology of oxide scale in Fe11Al0.5Nb alloy after oxidation at for 100 hours. (b) Enlargement of some area in (a) showing cigar-like oxide

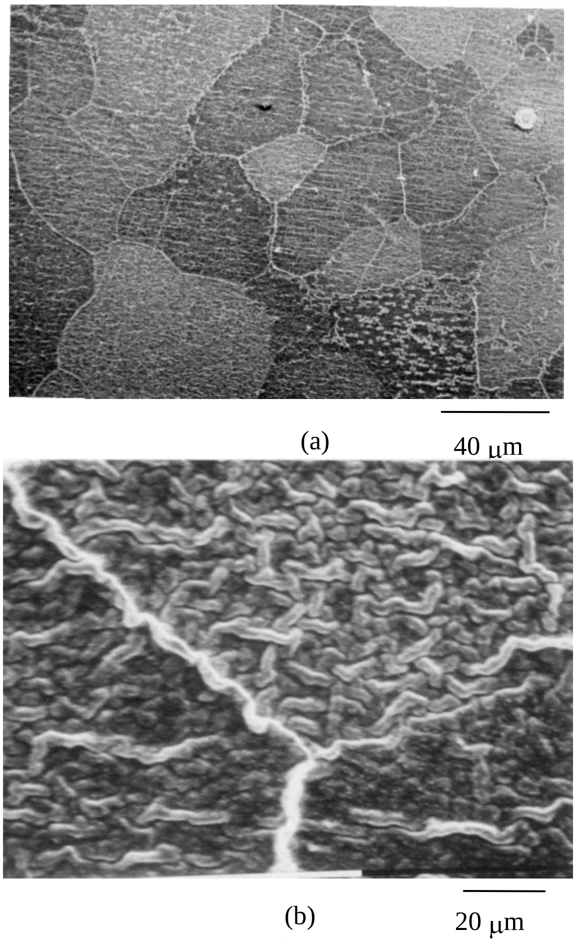

***Figure 4***. (a) Morphology of oxide scale in Fe11Al 0.5Mo alloy after C for 100 hr. (b) Enlargement of a grain boundary area, showing scales decorating grain boundaries of the base metal

## Identification of Oxide Phases

X-ray diffraction patterns of the substrate and oxide scales formed after isothermal oxidation are shown in Fig. 5. The base metal or Fe11Al substrate clearly exhibited an A2 (α-Fe) structure. The same feature remained even after oxidation at 900°C for 100 hour; the XRD did not detect any presence of oxide scales. This is in agreement with the very low oxidation rate observed at this temperature (see Fig. 1). On increasing the temperature to 1000 °C, the base metal was not anymore detectable indicating that the whole surface had been covered by the oxide layer. The scale consisted of two types of alumina, i.e. $\theta$-$Al_2O_3$ and $\alpha$-$Al_2O_3$, with θ predominating over α. The same pattern was observed for Fe11Al samples oxidised at 1100°C with a quite significant increase in α intensities and the presence of the peaks of the base metal. The latter may be ascribed to oxide spallation which may have occurred during the course of oxidation treatment at such high temperature (cracked oxide scales has already been observed at 1000°C as shown in Fig. 2).

Additions of Mo and Nb changed the XRD pattern significantly. In Fe11Al0.5Nb alloy oxidised at 1000°C, $\alpha$-$Al_2O_3$ was predominant over $\theta$-$Al_2O_3$ . The latter actually began to decease. A striking feature was observed in Fe11Al 0.5Mo oxidised at 1000 °C, in which the oxide scale consisted in $\alpha$-$Al_2O_3$. A2 (base metal) peaks were also observed in both alloys. This give an indication that the oxide layers were very thin, which is consistent with the low oxidation rate as observed in Figure 1.

## DISCUSSION

Fe11Al alloys exhibited good resistance at temperature of up to 900°C. At higher oxidation temperatures, i.e. at 1000 and 1100 °C, a rapid increase in oxidation rate was observed. Poor oxide adherence was already observed at 1000°C and the spallation seems to have taken place at 1100°C. The latter can be inferred from the oxidation rate, which was much higher at this temperature, and the presence of A2 (base metal) peak in the XRD pattern. This observation is in agreement with those observed by Xu et.al [8] and Chan et.al [9]. Addition of Mo and Nb significantly improved the oxidation resistance of the alloys, with the Mo bearing alloy showing excellence oxidation resistance at 1000 °C. Although showing higher oxidation rate than Fe11AlMo, the Nb bearing alloy exhibited better oxide adherence and more uniform oxide scale.

The oxide scales in the binary Fe11Al consisted of mixed $\alpha$-$Al_2O_3$ and $\theta$-$Al_2O_3$ , the latter being the metastable one. Transformation from θ to α seems to take place slowly even at a temperature as high as 1100°C. Mo and Nb additions appeared to accelerate this transformation, with Mo being the more effective one. The appearance of the base metal peaks in these alloys indicates that the scale formed after oxidation at 1000°C was very thin. This is considered advantageous for applications at high temperature because thin oxide layer has been shown to have a better mechanical stability toward thermal cycling [2,10].

## CONCLUSIONS

Binary Fe11Al alloys showed good oxidation resistance at 900°C, but further increase in the oxidation temperature caused the oxide scale to break away. Improvement in the oxidation resistance can be achieved by adding ternary elements, in which Nb and Mo addition has been shown to be very effective. These elements also accelerate the transformation form metastable $\theta$-$Al_2O_3$ to stable $\alpha$-$Al_2O_3$ . The mechanism by which these ternary elements affects the transformation and oxidation resistance has yet to be explored and will be the subject of the future work.

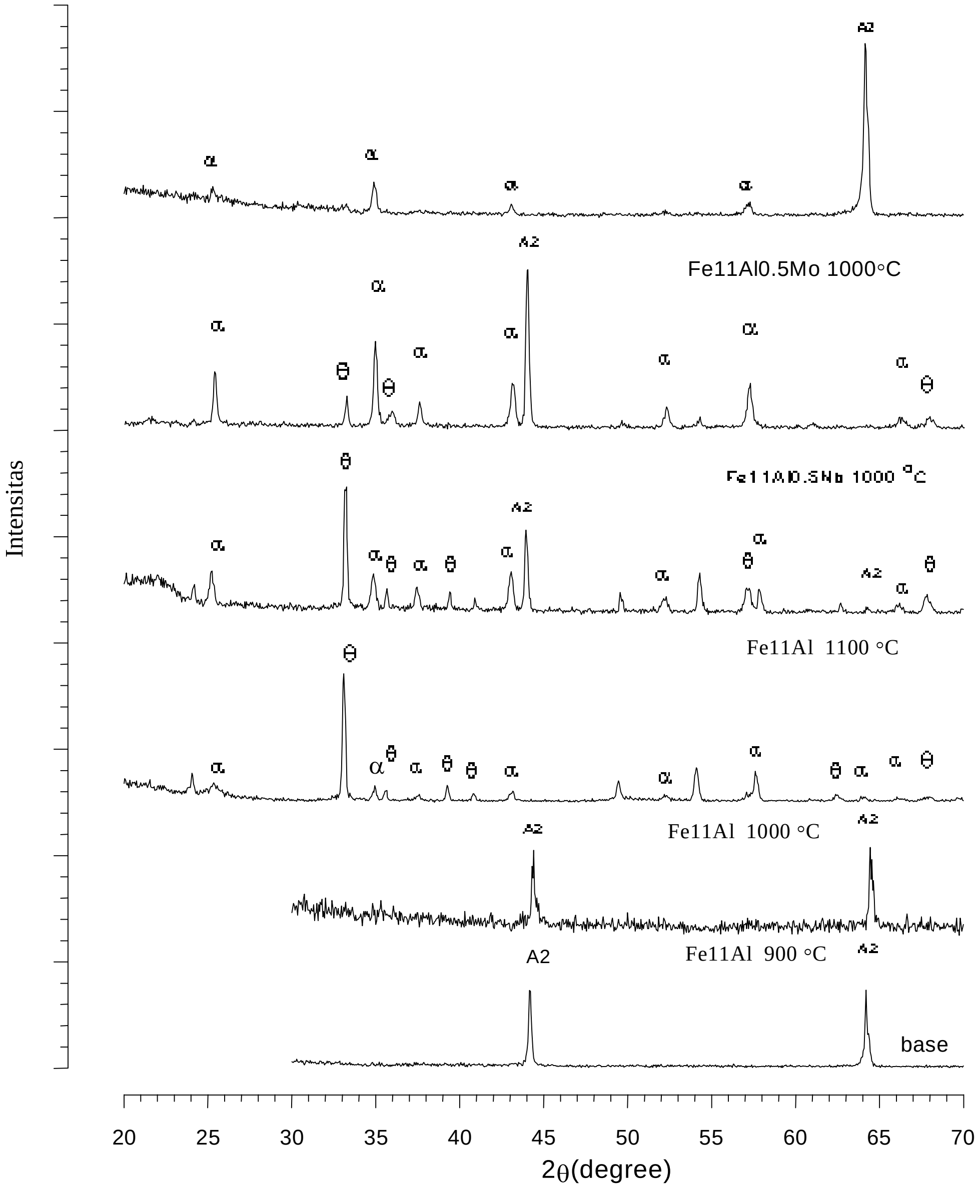

***Figure 5***. XRD patterns of the surfaces of Fe11Al based alloys after 100 hours at various oxidation

## ACKNOWLEDGEMENTS

The authors would like to thank Dr. R. Major and Dr. D. Kendall of Carpenter Technology (UK) Ltd. for the provision of raw materials and to Professor R. D. Rawlings of Imperial College, London, United Kingdom for all the support and encouragement. The high-temperature oxidation measurements have been carried out using the Magnetic Suspension Balance (MSB) Thermal Analyser provided by BMBF Germany through a bilateral project with Batan (Project IDN 99/004).

## REFERENCES

[1]. C. G. MCKAMEY, J. A. HORTON and C. T. LIU, *J. Mater.* Res. **4**, (1989), 1156.

[2]. C. G. MCKAMEY, J.H DEVAN, P.F.TORTORELLI and V. K. SIKKA, *ibid*. **6**, (1991), 1779

[3]. C. G. MCKAMEY and D. H. PIERCE, *Scripta. Metall. Mater*. **28**, (1993), 1173.

[4]. D. G. MORRIS and M. A. MORRIS-MUNOZ, *Intermetallics*. **7**, (1999), 1121.

[5]. C. T. LIU, E. H. LEE and C. G. MCKAMEY, *Scripta. Metall*. **23**, (1989), 875.

[6]. SYAHRIL and R. D. RAWLINGS, *J. Mater. Sci*. **37**, (2002), 1823.

[7]. V. JACOB, R. BACCINO, F. MORET, C. SURYANARAYANA, F.H. FROES and J. MISHURDA, *Proc. 2$^{nd}$ Intern. Conf. on Mechanical Alloying for Structural Applications*, Vancouver, **33**, (1993).

[8]. C. H. XU, W. GAO and H. GONG, *Intermetallics,* **8** (2000) 769

[9]. C. DANG NGOC CHAN, C. HUVIER and J.F. DINHUT, *Intermetallics*, **9**, (2001) 817

[10]. M. CEDERGREN and K. GORANSSON, Conf. Proc. in Materials Aspect in Automotive Catalytic Converter (Edited by H. Bode), Munich, (2001) 128.