

NATURAL AGING BEHAVIOUR OF AA6111 ALUMINIUM

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Abstract

In the continuing drive for weight reduction in new automobile designs, the 6000 series aluminium alloys have emerged as the most promising age-hardenable body sheet material in the automotive industry. Currently, one of the body sheet alloys used for its combination of strength and formability in the (T4) temper is AA6111, an Al-Mg-Si-Cu alloy. The T4 behaviour of AA6111 has been investigated by means of tensile testing, microhardness measurements, differential scanning calorimetry (DSC), scanning electron microscopy (SEM), and X-ray analysis. The results show that AA6111 attains a monotonic strength after approximately 120 h of natural aging. The microstructure consists of an aluminium matrix and angular intermetallic particles containing mainly Al, Fe, Cu, Si and Mn dispersed within the microstructure. The intermetallic particles are found to possess a structural formula of the type Al_3FeSi . Compared with other 6xxx series of alloys under the same T4 condition, AA6111 shows superior strength properties. Natural aging is observed to affect the kinetic reaction of AA6111.

Introduction

In the continuing drive for weight reduction in new automobile designs, the 6000 series Al-Mg-Si alloys have emerged as the most promising age-hardenable body sheet material in the automotive industry. Currently, the predominant sheet alloy used is the AA6111, an Al-Mg-Si-Cu alloy. This alloy has good combination of strength and formability in the T4 temper. Its strength can be further enhanced by precipitation hardening

Résumé

QUAINOO, G. K. & YANNAKOPOULOS, S.: *Comportement vieillissant naturel d' aluminium AA6111*. Dans la campagne continue pour la réduction en poids dans le nouveau modèle d' automobile, les séries 6000 d' aluminium en alliage ont émergé comme le matériau de feuille de carrosserie la plus prometteuse durcissable avec l'âge dans l'industrie de l' automobile. Actuellement, l' un d' alliage de feuille de carrosserie utilisé pour sa combinaison de solidité et formabilité dans la trempe (T4) AA6111 un alliage de Al-Mg-Si-Cu. Dans cet article, le comportement T4 de AA6111 a été étudié au moyen de l'essai de traction, l'évaluation de microdureté, la calorimétrie de balayage différentiel (CBD), la microscopie électronique à balayage (MEB) et l'analyse radioscopique. Les résultats montrent que AA6111 atteint une solidité monotone après approximativement 120 h de vieillissement naturel. La microstructure consiste d'une matrice d' aluminium et les particules intermétalliques angulaires contenant principalement Al, Fe, Cu, Si, Mn dispersés dans la microstructure. Les particules intermétalliques sont trouvées de posséder une formule structurale du type Al_3FeSi . Comparé avec d' autres séries d' alliage de 6xxx sous la même condition de T4, AA6111 montre les propriétés de solidité supérieure. Les vieillissement naturel est observé d' influencer sur la réaction cinétique de AA6111.

during the final paint bake process in the automotive manufacturing process line.

The effect of precipitation on the strength of various Al-Mg-Si alloys has been reported very extensively in the open literature (Mulazimoglu *et al.*, 1997; Pashley, Rhodes & Sendorek, 1966; Pashly, Jacobs & Vietz, 1967; Panseri & Federighi, 1966; Cersara *et al.*, 1969/70; Moons *et al.*, 1996). Their strength is attributed to the precipitation of the Mg_2Si phase during artificial aging. It is worth

noting that in all these investigations, only the strength increase due to precipitation has primarily been considered.

The effects of natural aging and pre-aging on precipitation of Al-Mg-Si alloys have also been investigated and reported (Poole, Lloyd & Embury, 1997; Tamizifa & Lorimer, 1992; Chatterje & Entwistle, 1973; Livak, 1982; Suzuki, Kanno & Itoh, 1980; Sakurai & Eto, 1992; Chakrabarti, Cheong & Laughlin, 1998; Murayama *et al.*, 1998; Murayama & Hono, 1999). For example, Poole, Lloyd & Embury (1997) investigated the effect of natural aging on the evolution of yield strength during artificial aging of Al-Mg-Si-(Cu) alloys. They observed that with long term natural aging prior to artificial aging, the initial component of yield stress (arising from natural aging) gradually decreased as artificial aging progressed. It is well established that the presence of Cu in Al-Mg-Si alloys enhances the precipitation kinetics of these alloys (Tamizifar & Lorimer, 1992; Chatterjee & Entwistle, 1973; Livak, 1982; Suzuki, Kanno & Itoh, 1980; Sakurai & Eto, 1992; Chakrabarti, Cheong & Laughlin, 1998).

Murayama *et al.* (1998) and Murayama & Hono (1999) have investigated the adverse age-hardening effect due to natural aging on precipitation in Al-Mg-Si alloys using the atom probe field ion microscopy (APFIM) technique. They found that separate Mg- and Si- clusters are present in the as-quenched condition, but the Mg and Si atoms aggregate during natural aging to form Mg-Si co-clusters. It is envisaged that the involvement of Cu-containing clusters during natural aging may alter the precipitation sequence in Al-Mg-Si-Cu alloys and, therefore, affect the

behaviour of these alloys.

Although it has been recognized that natural aging has a deleterious effect on the artificial aging strengthening characteristics of AA6111, no detailed investigation has been carried out to study the natural aging behavior of this particular alloy. In the present investigation, the T4 behavior of AA6111 has been studied with a view to understanding its age-hardening characteristics under this condition. This is very crucial since, in the present and future applications of this alloy for external and internal automobile body panels, natural aging becomes unavoidable in the manufacturing process. The evaluation methods employed in this investigation include tensile testing, microhardness measurements, differential scanning calorimetry (DSC), scanning electron microscopy (SEM) and X-ray analysis.

Experimental

The AA6111 material used in the present study was supplied by Alcan International Limited, Kingston, Ontario, and its composition limit is presented in Table I. The material was manufactured by a special technique, the details of which are reported by Burger *et al.* (1995).

Tensile testing

The tensile specimens were machined from a 1-mm thick stock plate in accordance with ASTM E-8 specifications, with a gauge length of 50 mm and 12 mm gauge width. They were solution heat treated at 560 ± 5 °C for 30 min in a constant temperature air furnace, quenched in laboratory water, and, subsequently, aged at room temperature for various lengths of time. Tensile

TABLE I

Chemical composition limit of AA6111 (weight percent)

	Cu	Fe	Mg	Mn	Si	Ti	Al
Maximum	0.9	0.4	1.0	0.45	1.1	0.10	Balance
Minimum	0.5	0	0.5	0.1	0.6	0	

tests were performed at room temperature using an Instron™ screw-driven machine, model number 1122, at an initial strain rate of 0.025 s^{-1} . The tensile strength and the 0.2 per cent off-set yield strength were determined from the resulting load *versus* extension plots.

Hardness measurements

Microhardness samples measuring $20 \text{ mm} \times 20 \text{ mm}$ were cut from the solution heat treated strips and aged naturally for various lengths of time. They were metallurgically polished using $6 \mu\text{m}$ and $1 \mu\text{m}$ grade diamond paste. Vickers hardness measurements were carried out on the polished samples using a Buehler microhardness (Micromet II) tester with a 50 g direct load applied for 15 s. At least 12 approximately equally spaced measurements were taken for each aging time to ensure representative results.

Differential scanning calorimetry (DSC)

DSC analyses of as-quenched as well as naturally aged samples were carried out using a Mettler TA 4000 thermal analyzer equipped with a plug-in Mettler DSC-20 cell. Three DSC runs were conducted for each natural aging time in order to ensure reproducibility. All DSC runs started at $30 \text{ }^\circ\text{C}$ and ended at $240 \text{ }^\circ\text{C}$ with a constant heating rate of $10 \text{ }^\circ\text{C min}^{-1}$. This temperature range was intentionally chosen to isolate GPI zone reactions since they are the most paramount under the natural aging condition. In order to correct for the additional heat flow arising from the difference in mass of the sample pan and the reference pan, and also to compensate for any asymmetry in the measuring system, a preliminary blank experiment was performed with commercially pure aluminum. Thus, the heat flow obtained was the difference between the measured and the blank values.

Scanning electron microscopy (SEM)

Samples naturally aged for various lengths of time were investigated using a JEOL JSM-5900LV scanning electron microscope (SEM) equipped

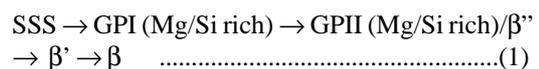
with an INCA energy-dispersive spectrometry (EDS) analysis system. The SEM samples were polished to a high degree of smoothness to ensure high reflectivity during SEM and EDS analysis. To ascertain the elemental composition of particles, which may be found in the alloy and the likely phases they may represent, X-ray point analysis was also performed on a random selection of the particles as well as the matrix.

Results and discussion

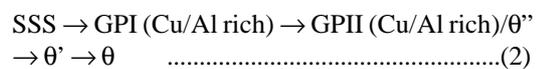
Mechanical properties

Fig. 1, 2 and 3 show the variation of yield strength, tensile strength and hardness with aging time at room temperature, respectively, for AA6111 aluminium. It can be observed that in all these figures the yield strength, tensile strength and hardness all rise very sharply within the first 48 h of aging after which the rate of increase in strength decreases with further aging. They attain an almost monotonic maximum after approximately 120 h of natural aging.

The precipitation sequence for Cu-containing Al-Mg-Si alloys for a long time has been traditionally considered to be analogous to the ternary Cu-free Al-Mg-Si alloys shown in Equation (1):



However, Gupta, Morois & Lloyd (1996) in their study of the precipitation sequence in AA6111 (an Al-Mg-Si-Cu alloy), proposed another possible sequence that could be followed as:



Recently, the precipitation of the quaternary Q phase and its precursor Q' in Cu- containing Al-Mg-Si has been reported (Perovic *et al.*, 1999; Miao & Laughlin, 2000, 1999; Entwistle, Fell & Koo, 1962/63; Gupta & Lloyd, 1992; Lloyd, Evans & Gupta, 2000). As a result, the precipitation

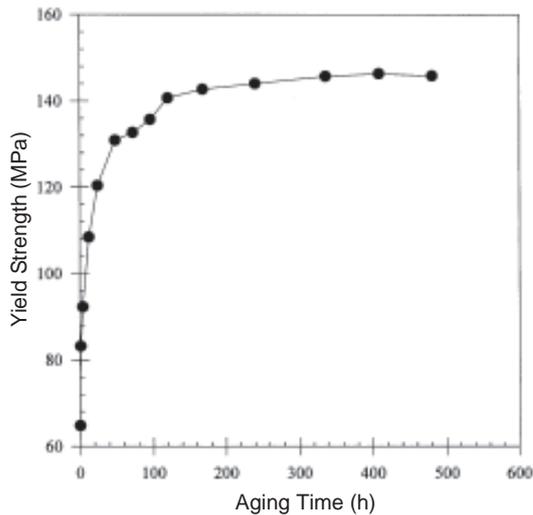


Fig. 1. Variation of yield strength with aging time at room temperature for AA6111

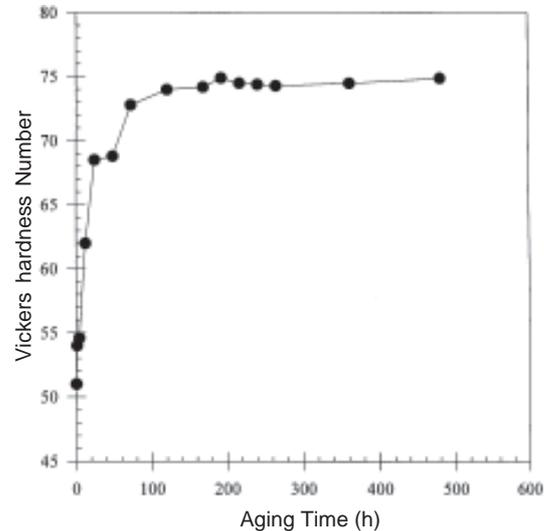


Fig. 3. Variation of Vickers hardness number with aging time at room temperature for AA6111

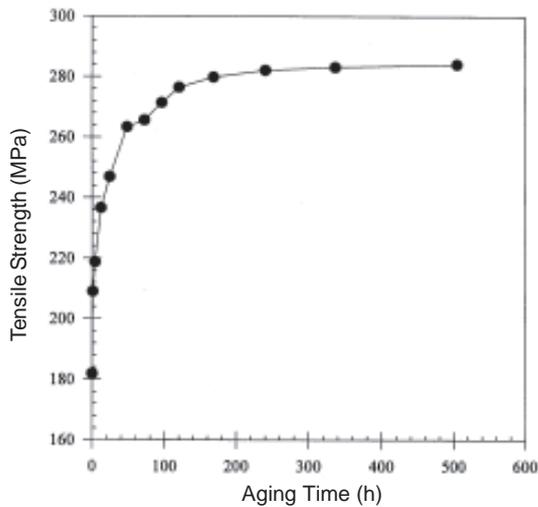
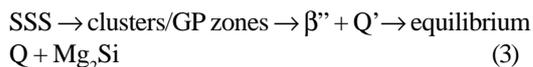


Fig. 2. Variation of tensile strength with aging time at room temperature for AA6111

sequence in AA6111 has come to be accepted as:



Natural aging is generally believed to be due to the occurrence of clusters of solute atoms

followed by the growth of GPI zones. For this particular alloy, the key alloying elements are Mg, Si and Cu. These alloys generally improve the mechanical properties of the alloy by influencing the solution heat treated strength, subsequent aging response, kinetics and corrosion resistance of AA6111 (Gupta, Morois & Lloyd, 1996; Miao & Laughlin, 2000; Miao & Laughlin, 1999), thus making the alloy a suitable candidate material for auto body application.

The process of natural aging in AA6111 entails the formation of separate Mg-, Si-, Cu-, and co-clusters of Mg-Si and / or Cu-Al (Chartergee & Entwistle, 1973; Suzuki, Kanno & Itoh, 1980; Gupta, Morois & Lloyd, 1996) in the matrix at the early stages of natural aging (see Equations (1) and (2)). These coherent GPI zones distort the aluminium matrix, setting up large strain fields around the precipitates, and impede the movement of dislocations, thereby, increasing the strength of the alloy. This stage of precipitation is very complex as the clusters tend to compete with each other, resulting in a lot of dissolution and formation of new clusters. The decrease in the rate of strength increase after the first 48 h is

believed to be due to the dissolution of some of the GPI zones and the formation of new ones.

With the formation and growth of the GPI zones, the contribution from increased internal stresses and the movement of quenched-in vacancies to the GPI zones give rise to a further increase in strength. However, further aging increases the size of the GPI zones, resulting in coherency loss and causing the vacancy concentration in the zones and matrix to reach their equilibrium values. Consequently, the rate of increase of strength becomes sluggish as depicted by the portion of the curve showing monotonic increase in yield strength with further aging. Fig. 4 compares the tensile strength of AA6111 to those of other 6xxx series aluminum alloys (Franz, 1985) subjected to the same condition of natural aging. All the alloys have been subjected to natural aging for 500 h. It can be seen from Fig. 4 that AA6111 shows superior strength compared with alloys studied in Franz, 1985.

Differential scanning calorimetry (DSC)

Fig. 5 shows DSC thermograms of AA6111

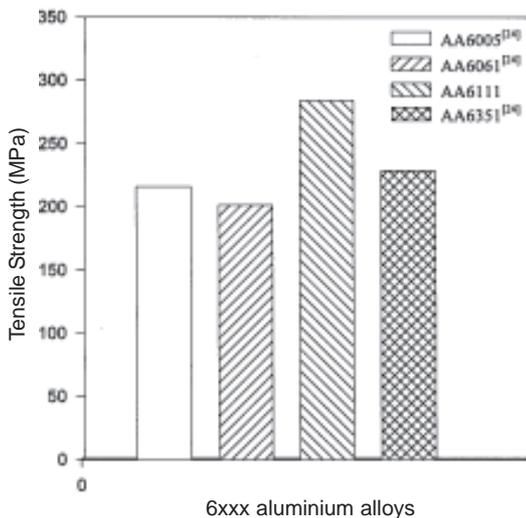


Fig. 4. Comparison of tensile strength of AA6111 with other 6xxx series Al alloys in Franz, 1985

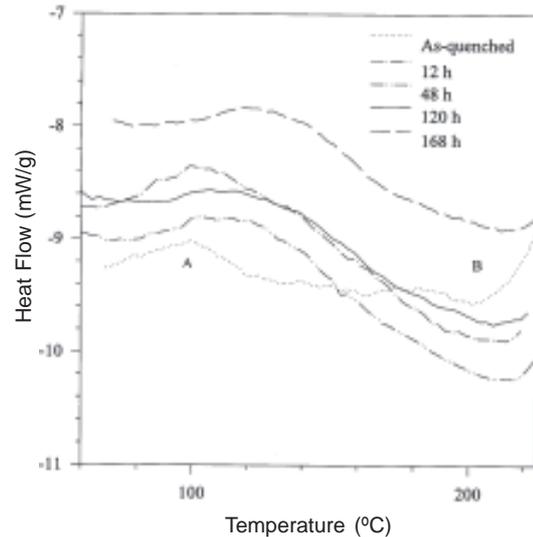


Fig. 5. DSC thermograms of as-quenched and naturally aged samples of AA6111

samples in the as-quenched and naturally aged conditions for various lengths of time. The exothermic peak A indicates the formation of GPI zones while the endothermic trough B indicates their dissolution. The variation of DSC peak reaction temperature (T_p) with aging time at room temperature is plotted in Fig. 6 for the formation and dissolution of GPI zones. It can be observed that peak reaction during GPI zone formation and dissolution occurs at higher temperature as the length of natural aging time increased. A visual evaluation of the area under peak (Fig. 5) shows that A is larger for the as-quenched sample than for the naturally aged samples, indicating a decrease in the volume fractions of the GPI zones with aging time. This may be attributed to the formation of initial clusters of Mg, Si, and/or Cu clusters following quenching and the speed with which these transform into stable GPI zones. This is consistent with the reported results of DSC scans conducted on other Al-Mg-Si alloys (Lloyd, Evans & Gupta, 2000; Dutta & Allen, 1991; Kovacs, Lendvai & Nagy, 1972). It is also possible that Mg-Si co-clusters may have formed

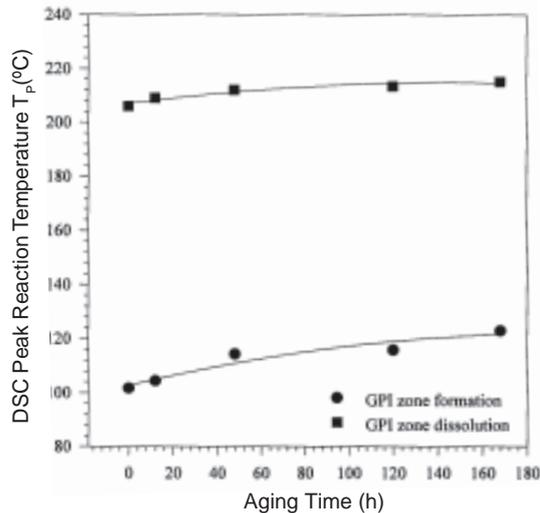


Fig. 6. Variation of DSC peak reaction temperature (T_p) with aging time at room temperature

(Murayama *et al.*, 1998; Murayama & Hono, 1999; Edwards *et al.*, 1998).

The dissolution reaction peak B (Fig. 5) also shifts to higher temperatures with increasing natural aging time. This is attributed to a greater thermal stability of GPI zones resulting from their increased size. Thus, the strength of the alloy will

be influenced by the growth of GPI zones.

Scanning electron microscopy (SEM)

Scanning electron microscopy investigation was carried out to study the microstructure, distribution, and elemental composition of the phases present in the alloy with a view to identifying the factors affecting the strength of this alloy. Fig. 7 shows the SEM micrograph of a typical sample of AA6111 aged naturally for 500 h. The microstructure reveals the presence of angular intermetallic particles randomly distributed in the alloy. Gupta, Morois & Lloyd (1996) have documented the presence of insoluble Al(Fe,Mn,Cu,Cr)Si particles in the microstructure of as-quenched samples of AA6111 and indicated that these are free of quenched-in defects, such as dislocation loops and helices.

Fig. 8(a) and (b) show the EDS spectra for the matrix and particle, respectively. A summary of the elemental composition and likely phases present is presented in Table 2. It can be observed from the table that the particles contain mainly Si, Mn, Fe, Cu, and Cr, which is very consistent with the results of Gupta, Morois & Lloyd (1996). The

atomic ratio of Fe and Si is practically consistent at unity indicating that Fe and Si have approximately the same solubility in Al at the solution heat treatment temperature used for this alloy. The atomic ratio of Al and Fe-Si differed to varying degrees and so, in order to represent them chemically, it was decided to denote the phases by the general formula Al_xFeSi .

The level of hardening in age-hardenable Al alloys is determined primarily by the amounts of solute atoms available to form precipitate phases as well as the distribution of the resulting phases in the

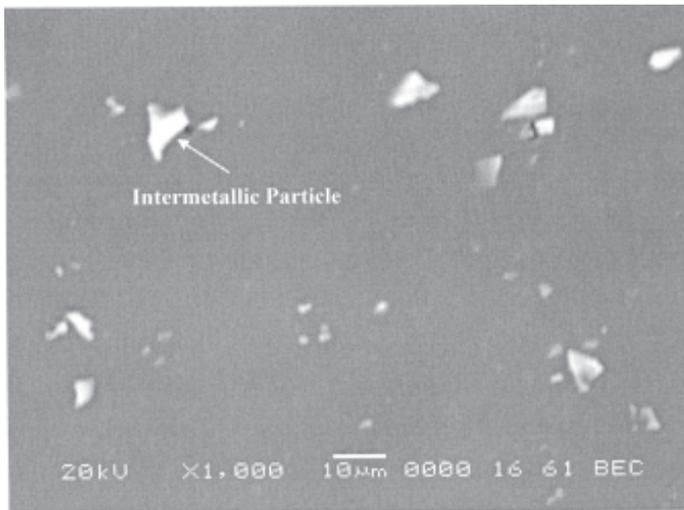


Fig. 7. SEM micrograph of 500 h naturally aged AA6111

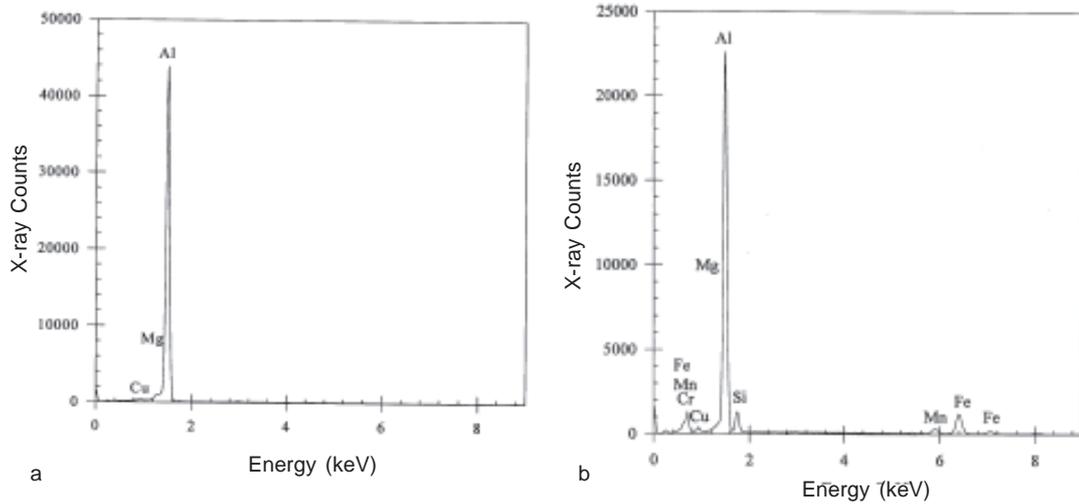


Fig. 8. EDS spectrograph of AA6111 (a) matrix, (b) typical intermetallic particle

TABLE 2

EPMA point analysis from intermetallic particles

Particle #	Element – wt %							Total	Likely phases
	Si	Mg	Mn	Fe	Cu	Cr	Al		
1	4.76	0.43	1.87	7.26	1.42	0.21	84.05	100.00	$Al_{24}FeSi$
2	6.22	0.16	2.93	13.80	1.97	0.38	74.55	100.00	$Al_{11}FeSi$
3	7.01	0.14	3.80	18.46	2.00	0.28	68.31	100.00	$Al_{10}FeSi$
4	7.41	0.12	3.77	19.63	2.21	0.57	66.29	100.00	Al_9FeSi
5	7.52	0.00	3.97	21.32	2.40	0.37	64.43	100.00	Al_9FeSi
6	5.95	0.25	2.84	13.30	1.61	0.44	75.60	100.00	$Al_{13}FeSi$

matrix. Fig. 9(a) – (e) show X-ray maps of the intermetallic particles for samples of AA6111 aged naturally for up to 500 h. These show the distribution of Fe, Si, Mg, Mn, respectively. It can be seen that the distribution of Fe, Si, and, to some extent, Mn is very similar, indicating that the intermetallic particles do not contain Mg and Cu. The formation of the intermetallic particles is believed to have consumed some amounts of the solute atoms which, otherwise, would have been available for the formation of the precipitate phases in the alloy upon subsequent artificial

aging. Since a very negligible amount of Cu was found in the intermetallics, indicating that most of the Cu is in the matrix, the possibility of Cu-clusters and co-clusters of Al-Cu forming at the early stages of the precipitation reaction during natural aging *via* Equation (2) is very high. This is consistent with the suggestion by some investigators (Sakurai & Eto, 1992; Gupta, Morois & Lloyd, 1996) that Cu-containing precipitates such as θ and its precursor metastable phases θ' may form in Cu containing Al-Mg-Si alloys.

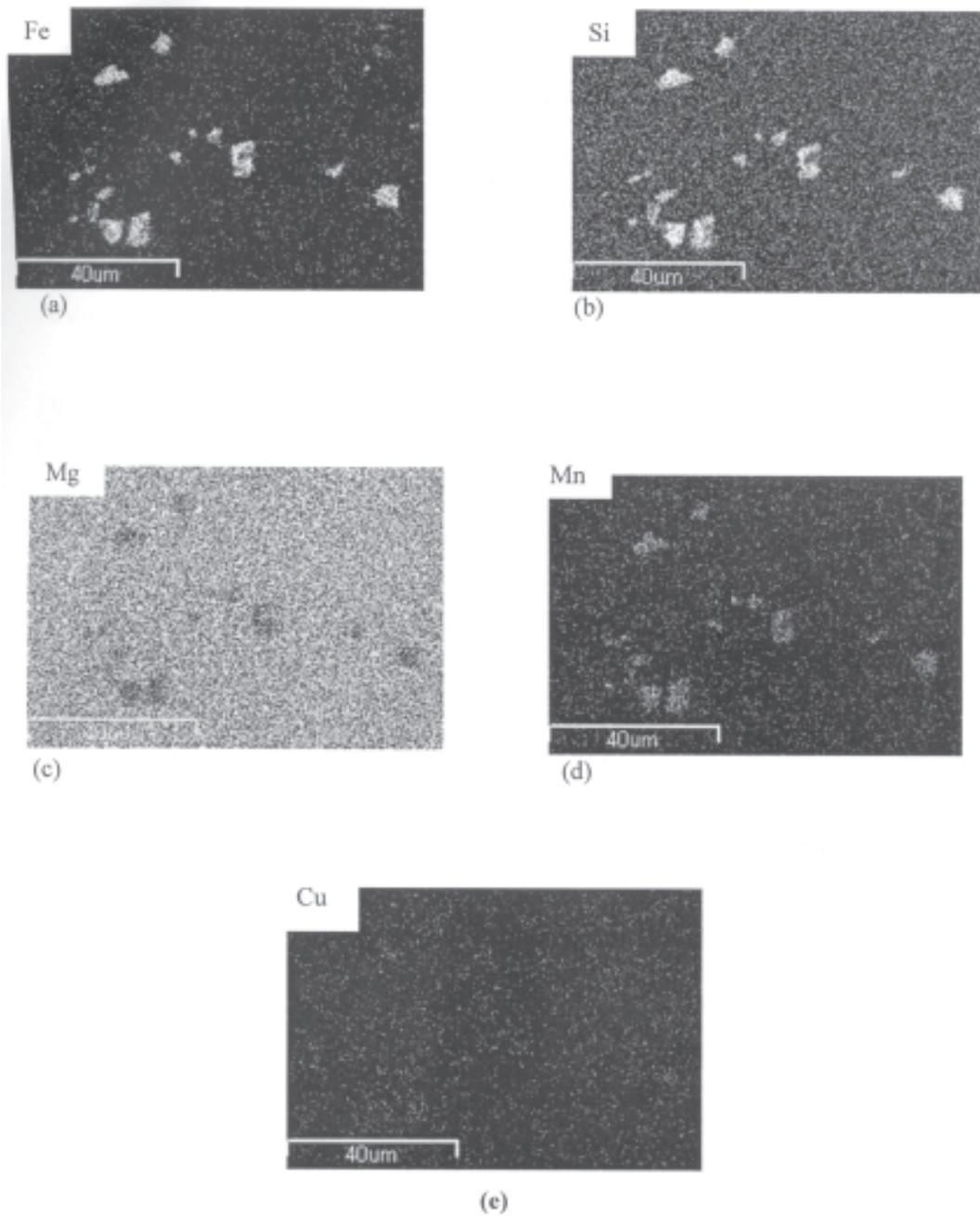


Fig. 9. X-ray map showing (a) iron, (b) silicon, (c) magnesium, and (d) manganese

Conclusion

The AA6111 alloy attains an almost monotonic strength level after approximately 120 h aging at room temperature. Natural aging affects the GPI zone formation and dissolution kinetics in AA6111. DSC and EPMA analyses of naturally aged samples of AA6111 alloy suggest the possibility of Cu clusters forming during the early stages of natural aging since a very negligible amount of Cu was found in the intermetallics. The microstructure of AA6111 consists of angular intermetallic particles of the Al_xFeSi type.

Acknowledgement

The authors thank Dr A. K. Gupta of Alcan International Limited, Kingston, Ontario, for his technical contribution. The Government of Ghana Scholarship grant through the University of Cape Coast to G. K. Quainoo is gratefully acknowledged. Financial assistance from the Natural Sciences and Engineering Research Council of Canada (NSERC) in the form of a research grant to S. Yannacopoulos is also acknowledged.

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