JOURNAL OF MATERIALS SCIENCE 29 (1994) 1652–1655

Effect of heat treatment on the insoluble intermetallic phases present in an AA 6063 alloy

S. ONURLU
Department of Materials Research, TÜBİTAK, Gebze, Turkey
A. TEKIN
Faculty of Chemistry and Metallurgy, İ.T.Ü., İstanbul, Turkey

The microstructure of a semi-continuously cast AA 6063 alloy was investigated and the transformation of insoluble intermetallic phases by applied heat treatment was studied by selective dissolution of the matrix. Residual intermetallics were characterized using energy-dispersive X-ray analysis, X-ray and electron diffraction techniques. Transformation of β–AlFeSi phase, which is known to have detrimental effects on the surface finish of the extrudates, to more favourable α-AlFeSi phase by heat treatment was determined.

1. Introduction

Among all aluminium alloys, AA 6063 is the most widely used for extrusion processing. While the extrusion speed of this alloy is determined by the size and distribution of the soluble Mg_2Si phase, the surface finish of the extrudate is determined by the insoluble AlFeSi particles that are usually present at the grain boundaries.

As in most aluminium alloys, iron is present in AA 6063 as an impurity and magnesium and silicon are present as the major alloying elements. Since both iron and silicon are found in most aluminium alloys, extensive studies related to the aluminium side corner of the Al–Fe–Si system have been previously done on pure systems and the phases that form upon equilibrium cooling have been reviewed [1]. However, commercial aluminium alloys contain other elements like copper, manganese, chromium, zirconium either as alloy additions or as impurities. The amount of the elements present in the alloy and the cooling rate will determine the phases that will form upon cooling of an ingot. The phases present in dilute aluminium alloys and their crystallographic data are reviewed by Skjerpe [2], but as the system is complex and is of non-equilibrium character these are difficult to predict.

The monoclinic β-AlFeSi phase that is reported [3, 4] to exist in commercial AA 6063 aluminium alloy ingots will transform into body-centred cubic α-AlFeSi phase by applying suitable heat treatment. Other than β-AlFeSi, various ternary Al–Fe–Si, binary Al–Fe and Mg_2Si precipitates are found to exist in similar alloys [5, 6]. The present investigation is concerned with the determination of insoluble intermetallics present in a commercial AA 6063 and the change of these phases by applied heat treatment.

2. Experimental procedure

The material investigated was commercial purity, semi-continuously cast Al–Mg–Si alloy (Seydişehir Aluminium Plants, Turkey) the composition of which was determined by wet analysis (Table I). The ingot of radius 150 mm was sliced into 20 mm thick discs and the discs were sectioned radially into four equal pieces. Homogenization treatments at four different time intervals of 1, 2, 4 and 8 h were performed at five different temperatures of 550, 560, 570, 580 and 590 °C for each time interval. The pieces were water-quenched after heat treatment. A section from the central part of each piece was metallographically prepared by mechanical polishing, followed by etching in 0.5% HF solution. Pieces were investigated with a scanning electron microscope (SEM) equipped with energy-dispersive X-ray analysis (EDAX). For SEM studies of the as-cast structure two different specimens were prepared, one of which was taken from the mould–casting interface and the second from the centre of the ingot.

For selective dissolution of the matrix, 15 g 8-hydroxyquinoline, 60 g benzoic acid, 60 ml chloroform and 165 ml methanol solution [7] was used as the electrolyte and the alloy itself was used as the counterelectrode. The reaction was carried out under a current density of 10 mA cm^{-2}. Extracted particles were washed with chloroform several times and each time particles were separated from the solvent by centrifugal precipitation. Particles were dispersed in ethanol and taken on glass slides for X-ray diffraction, since the particles obtained after a dissolution time of 8 h were of limited amount. X-ray diffraction was performed using CoK_α radiation. Electron diffraction studies were carried out on 20 particles from each specimen. For this purpose, particles dispersed in ethanol were taken on carbon support grids and the

TABLE I Composition of the alloy investigated (wt %)

Fe	Si	Mn	Mg	Cu	Ti
0.24	0.28	0.014	0.54	0.005	0.009

0022–2461

crystal structures were determined by electron diffraction.

Thin foil specimens for TEM investigations were prepared by applying an electrolytic polishing technique to mechanically ground samples. 30% HNO_3 methanol solution at $-20\,^{\circ}C$ was used as electrolyte and the specimens were investigated under transmission electron microscopy (TEM) operating at a voltage of 100 keV.

3. Results and discussion

3.1. SEM investigations

EDAX analyses of the mould–casting interface revealed that the intermetallic components found at the interface were ternary Al–Fe–Si and quaternary Al–Mg–Si–Fe phases (Fig. 1 and Fig. 2, respectively). An accurate analysis of the phases could not be performed due to the fact that precipitate sizes were small with respect to the probe size, and under this condition the aluminium content of the matrix always contributed to the EDAX spectrum acquired.

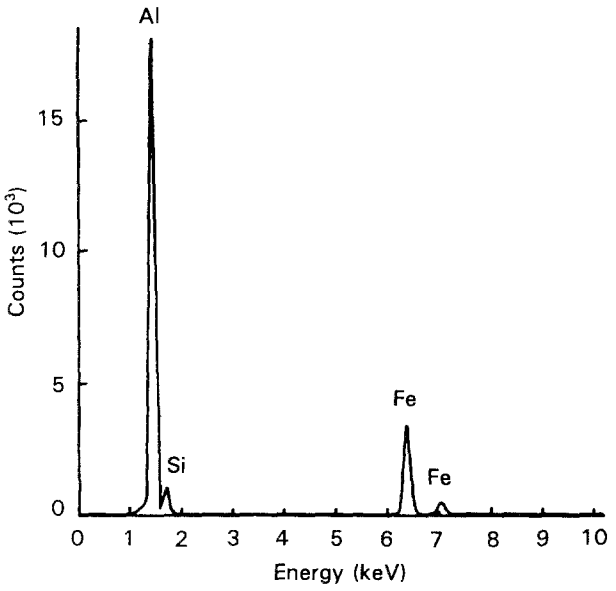

Figure 1 EDAX spectrum of Al–Fe–Si intermetallics present at the mould–casting interface.

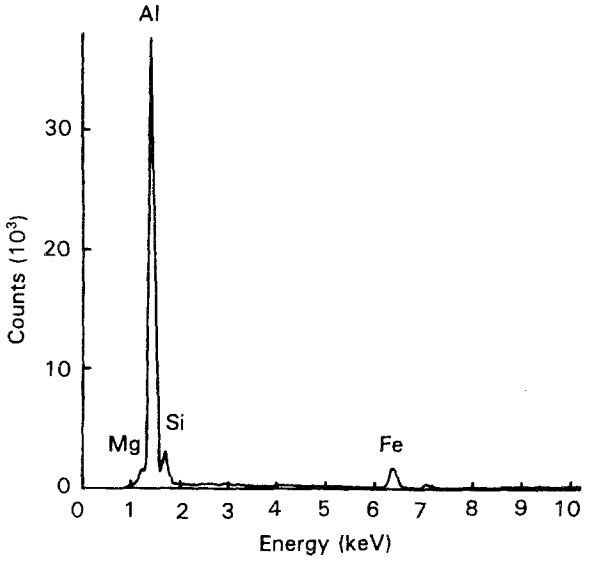

Figure 2 EDAX spectrum of Al–Fe–Mg–Si intermetallics.

The morphology of the phases at the interface (Fig. 3) was different from the morphology in the interior of the ingot (Fig. 4). However, this region of different morphology was at most 50 μm in thickness from the surface. This is a layer which can easily be removed during extrusion and will not affect the extrusion parameters. The morphology of the phases remained the same elsewhere; intermetallic particles were formed mostly at the grain boundaries and few particles existed within the grains.

EDAX analyses of a specimen prepared from the interior of the ingot revealed that as with the phases detected at the interface, the intermetallic particles present were mainly ternary Al–Fe–Si and some quaternary Al–Fe–Si–Mg particles also existed. However, formation of binary Al–Fe phases and equilibrium Mg_2Si phase did not take place during solidification of the ingot.

It was clarified by EDAX analyses of the heat-treated specimens that magnesium could readily be taken into solid solution. Even the sample heat-treated for the shortest time at the lowest temperature, i.e. 1 h at 550 °C, did not contain any Mg-bearing particles.

The surfaces of the pieces to which selective dissolution was applied were examined by SEM. As can be seen from Fig. 5, the matrix was removed during electrolytic dissolution.

Figure 3 SEM micrograph of intermetallics present at the mould–casting interface.

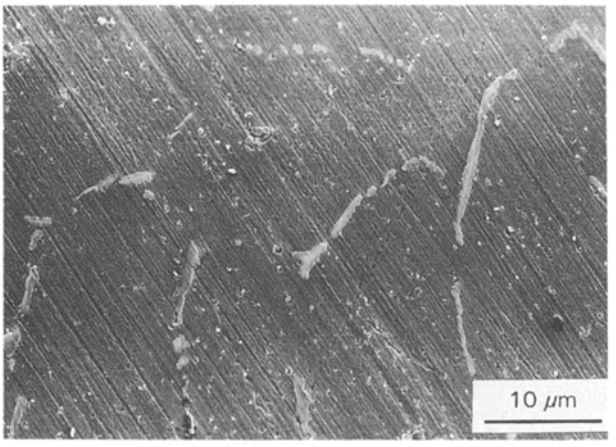

Figure 4 SEM micrograph of the interior structure of the ingot.

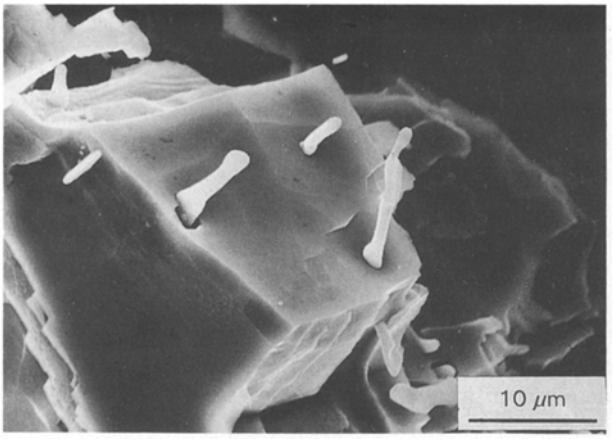

Figure 5 SEM micrograph of the sample surface after selective dissolution was applied. The protruding phases are the insoluble intermetallics.

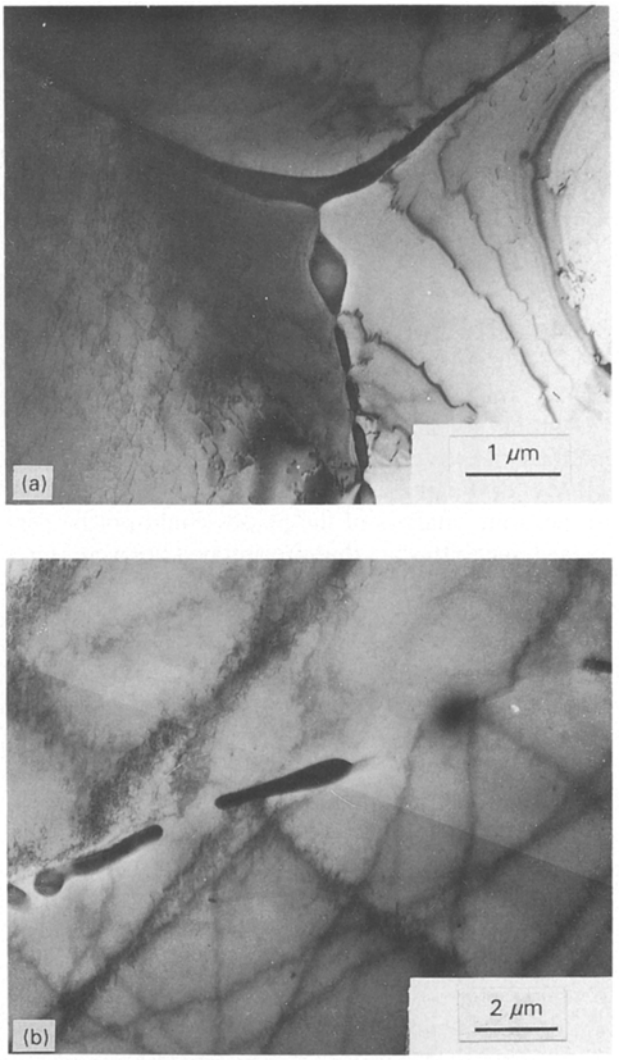

Figure 6 TEM micrograph of (a) as-cast structure, (b) after heat treatment at 550 °C for 1 h. Intermetallics are present at the grain boundary and the grain interiors are free of precipitates.

3.2. X-ray results

It was determined by X-ray analyses of the extracts that from the ternary phases that may be present in aluminium alloys only β-AlFeSi had formed during solidification. Since Al–Fe–Mg–Si phases did not appear in the X-ray pattern it was decided that these phase were present only in small amounts. By the application of heat treatment β-AlFeSi phase starts to transform into α-AlFeSi phase. X-ray analysis revealed that this process is rather slow at lower temperatures and the β-AlFeSi phase does not transform into α-AlFeSi completely. The completion of the transformation process with time and temperature is shown in Table II.

3.3. TEM investigations

Thin foil specimens prepared from the as-cast structure revealed that Mg_2Si precipitates did not form during solidification of the ingot and the formation of this phase after heat treatment is prevented by rapid cooling. Electron micrographs of the as-cast structure and the sample heat-treated at 550 °C and water-quenched are shown in Fig. 6a and b, respectively.

Electron diffraction studies on specimens prepared from the extracts were consistent with the X-ray analyses of the specimens. The phase detected in the as-cast structure was β-AlFeSi. Examples of an electron micrograph taken from this phase and the electron diffraction pattern of the particle are given in Fig. 7a and 7b, respectively. With the heat treatment applied, the phase transformed into α-AlFeSi the amount of which increased as the time and temperature of heat treatment increased. An electron micrograph of an α-AlFeSi particle is given in Fig. 8a and the diffraction pattern obtained is seen in Fig. 8b.

TABLE II Al–Fe–Si phases observed after each heat treatment[a]

Time	Temperature (°C)				
(h)	550	560	570	580	590
1	β + α	β + α	β + α	α	α
2	β + α	β + α	β + α	α	α
4	β + α	β + α	α	α	α
8	β + α	β + α	α	α	α

[a] The phase present in the as-cast structure is β-AlFeSi.

4. Conclusions

Of the binary Al–Fe and ternary Al–Fe–Si phases reported [2, 3, 5] to be present in dilute aluminium alloys, monoclinic β-AlFeSi is found to exist in a continuously cast commercial-purity AA 6063 alloy. Other quaternary Al–Fe–Mg–Si phases could not be detected by either X-ray diffraction or electron diffraction techniques, and it is concluded that such phases are present only in trivial amounts. It is revealed that magnesium and silicon did not form a compound under the present cooling conditions for the composition investigated. β-AlFeSi is shown to transform into b.c.c α-AlFeSi by appropriate heat treatment. The times and temperatures determined for completion of the transformation are found to correlate with the heat-treatment processes applied industrially, and the detrimental phase will be transformed into the preferred phase in practical applications.

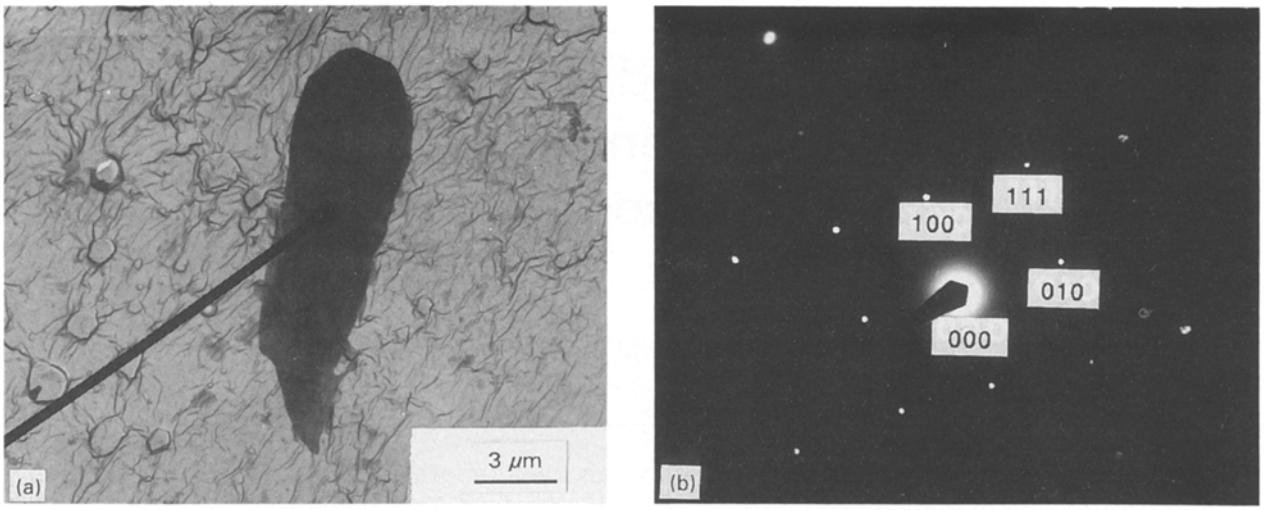

Figure 7 (a) TEM micrograph of a monoclinic β-AlFeSi phase taken on carbon support grid, (b) diffraction pattern of the particle (zone axis [0 0 1]).

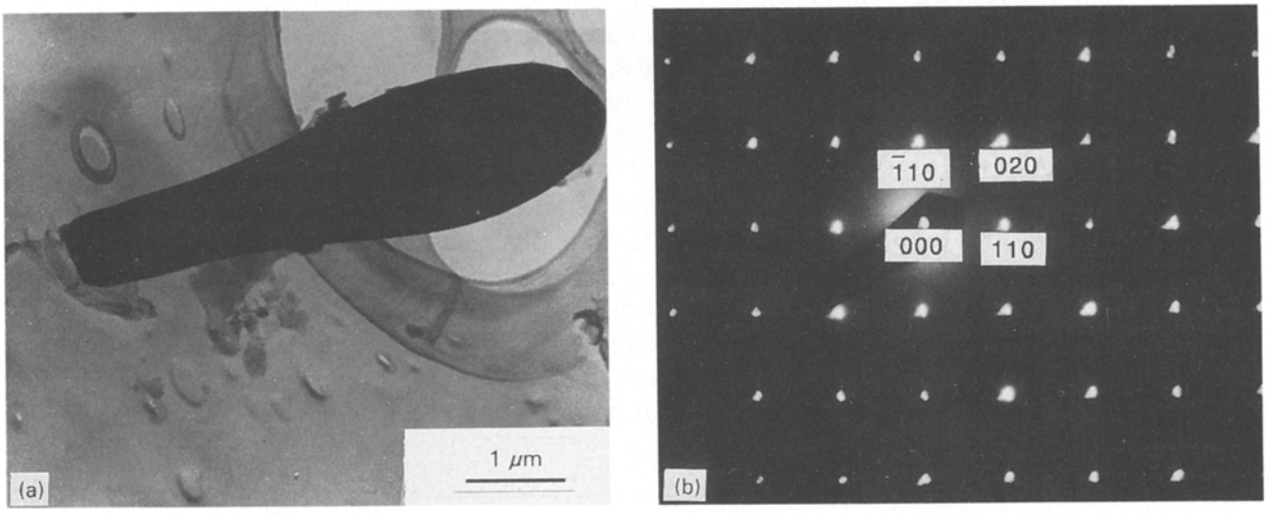

Figure 8 (a) TEM micrograph of a b.c.c α-AlFeSi phase taken on carbon support grid, (b) diffraction pattern of the particle (zone axis [0 0 1]).

Acknowledgement

The authors thank Dr Hüseyin Yörücü for his valuable contribution during the research.

References

1. V. G. RIVLIN, G. V. RAYNOR, *Int. Met. Rev.* **26** (1981) 133.
2. P. SKJERPE, *Met. Trans.* **18A** (1987) 189.
3. M. RYVOLA and L. R. MORRIS, *Microstr. Sci.* **5** (1977) 203.
4. N. C. PARSON and H. L. YIU, in Proceedings of Light Metals Conference, Las Vegas, February 1989, edited by P. G. Campbell (TMS/AIME, Warrendale, Pennslvania, 1989) p. 713.
5. A. L. DONS, *Z. Metallkde* **75** (1984) 170.
6. E. HIDVEGI and A. GRIGER, *Aluminium* **60** (1984) 603.
7. F. H. COCKS, M. L. SHEPARD and H. G. CHILTON, *J. Mater. Sci* **12** (1977) 494.

Received 26 February
and accepted 14 September 1993