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Investigation of the age hardening behaviour of 6063 aluminium alloys refined with Ti, RE and B

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ABSTRACT

The purpose of this study is to obtain a better understanding on the effect of ageing on the hardness of 6063 aluminium alloys refined with Ti, cerium-rich mixtures of rare earth (RE) and B with the aids of SEM, EDS, TEM, DSC, etc. The following conclusions have been obtained: the 6063 alloy with the joint additions of Ti, B and RE (10 w(Ti)/w(B) mass ratio) has the finest grain, compared to alloys with Ti or Ti and RE additions. Artificial ageing must be performed more than 7 d after extrusion. The 6063 alloys refined with Ti, Ti + RE or Ti + RE + B all have better ageing behaviour. It takes shorter ageing time to obtain a hardness near peak ageing hardness and there is no obvious decrease of ageing hardness of the alloys until ageing for 6 h at 200 °C. The addition of RE forms Al–Si–Mg–RE intermetallic constituents, resulting in lower strength of alloys than that with Ti addition.

1. Introduction

Al–Mg–Si alloys which are age-hardenable are widely used in automotive, architectural and structural applications. Artificial ageing is a universally adopted method for strengthening Al–Mg–Si alloys. So many researches have been done for ageing process of 6063 aluminium alloys (Siddiqui et al., 2000; Liu et al., 2003). 6063 alloys usually adopt Al–Ti or Al–Ti–B master alloys refining α(Al). In the recent years, a number of attempts have been tried to find new grain refiners other than Al–Ti or Al–Ti–B master alloys. Some scholars investigated the grain-refining efficiency of Al–Ti–C and Al–Ti–B–RE (Liu et al., 2002; Lan et al., 2005). Edwards et al. (1998) proposed that β″ precipitate results in maximum age-hardening effect. So it is important to control precipitation during age process in order to get optimum performance (Gavgali et al., 2003.). Age hardening behaviour of 6063 alloys refined with Ti and B has been well investigated, but systematic investigation on that of 6063 alloys refined with Ti, B and RE is lacking. The purpose of this study is to obtain a better understanding on the effect of ageing on hardness of 6063 aluminium alloys refined with Ti, Ti + RE, Ti + RE + B.

2. Experimental materials and procedures

The chemical compositions of the alloys refined with different grain-refining methods in this study are listed in Table 1. Aluminium containing 0.2%Ti was produced with electrolytic method in 82 kA aluminium electrolyzing cell through adding TiO_2 into aluminium electrolyzing cell. 6063 aluminium rotundity ingots were prepared adding Si, Mg, commercial pure aluminium, Al–10%RE or/and Al–5%B master alloys to aluminium containing 0.2%Ti in a 7 kW resistance furnace.

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Table 1 – Chemical compositions of alloys refined with different grain-refining methods (wt%)

Alloy	Grain-refining methods	Mg	Si	Fe	Mn	Ti	RE	B (nominal composition)	Al
1	Ti	0.502	0.372	0.109	<0.030	0.038	–	–	Balance
2	Ti + RE	0.488	0.399	0.081	<0.030	0.019	0.28	–	Balance
3	Ti + RE + B	0.492	0.392	0.115	<0.030	0.024	0.29	0.0024	Balance

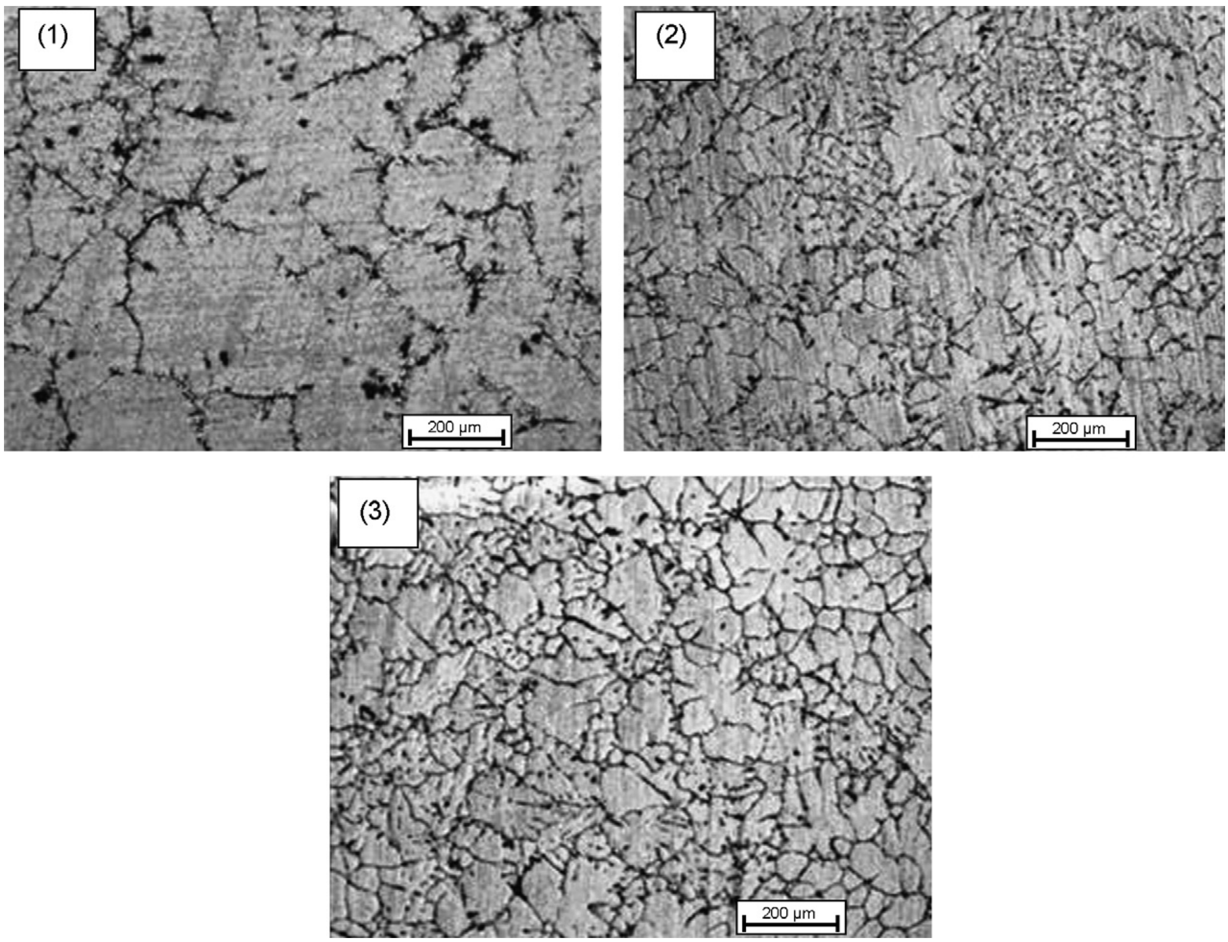

Fig. 1 – As-cast microstructures of alloys 1–3: (1) alloy 1, (2) alloy 2, and (3) alloy 3.

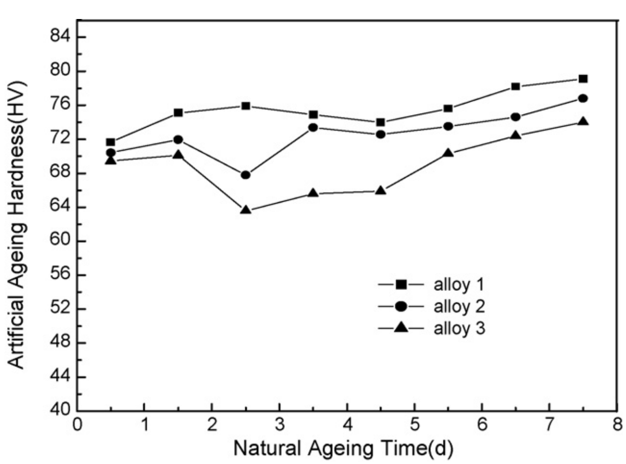

Fig. 2 – Relation between artificial ageing hardness and natural ageing time.

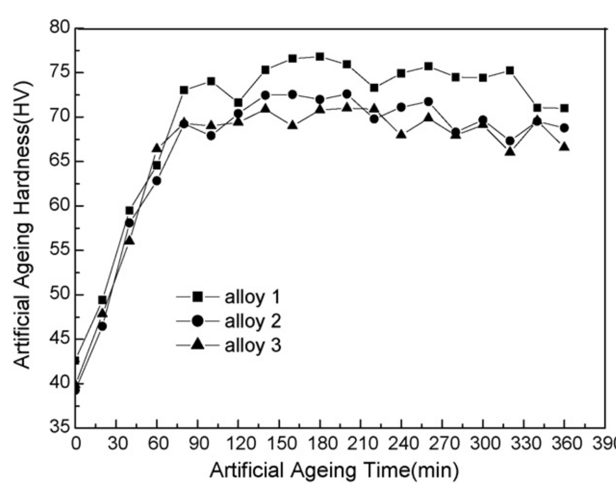

Fig. 3 – Ageing behaviour curves of alloys 1–3 ageing at 200 °C for 0–360 min.

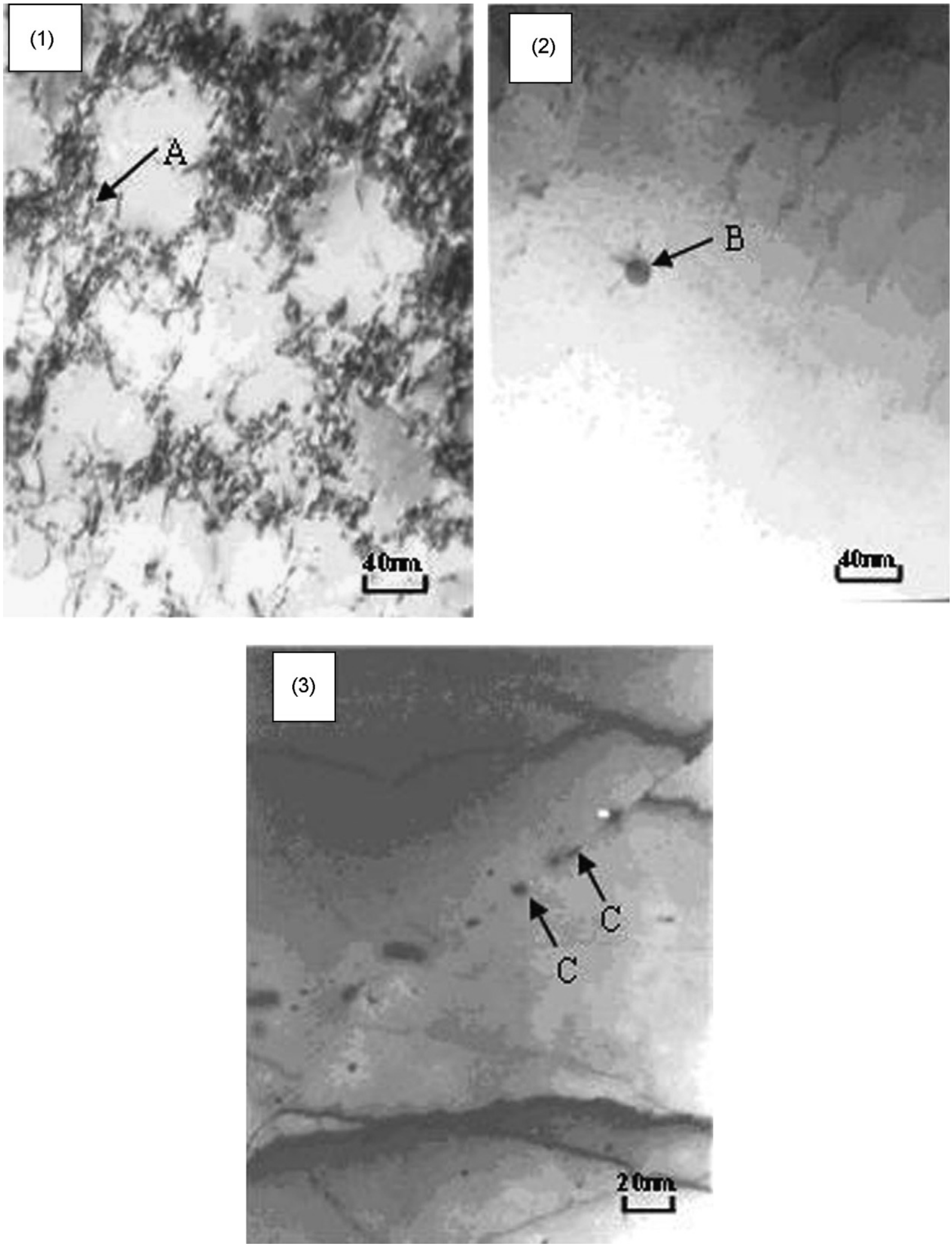

Fig. 4 – TEM bright field images before artificial ageing for alloys 1–3: (1) alloy 1, (2) alloy 2, and (3) alloy 3. Arrow A showing dislocations, arrows B and C showing precipitates containing RE.

Dimensions of 6063 aluminium alloys rotundity ingots are Ø80 mm × 175 mm. Alloys were cast in the laboratory, and then extruded to 3 mm-thick sheet in 5000 kN extruding machine. Alloys were held at room temperature for natural ageing for 0–7.5 d, and then for artificial ageing at 200 °C for 2 h. Another ageing treatment was performed at 200 °C for various times. The ageing behaviour curves were obtained by measuring the Vickers hardness number. The hardness measurements were carried out by using a 49 N load. Microstructures were observed using an optical microscope (OLYMPUS BX51) and Video TEST image analysis system. Microstructural examination was carried out in a JEOL, JSM-6700F SEM with energy-dispersive X-ray spectrometer (EDS). The foils for TEM studies were prepared by electropolishing in a 30 vol.% nitric acid in methanol solution at −25 °C, and were examined in a Philips EM400 electron microscope operated at 100 kV. Differential scanning calorimetry (DSC) was carried out with a heating rate of 10 °C/min in the range between 50 °C and 600 °C, protected by an argon atmosphere.

3. Results and discussion

3.1. *As-cast microstructures of alloys with different grain-refining methods*

Fig. 1 shows as-cast microstructures of alloys 1–3. The 6063 alloy with Ti and RE additions has finer grain than that with Ti addition. The 6063 alloy with the joint additions of Ti, B and RE (10 w(Ti)/w(B) mass ratio) has the finest grain. During the solidification of aluminium melt, Ti and RE gather in solid/liquid interface forward because of small solubility of Ti and RE in α(Al), resulting in composition undercooling and growth inhibition (Fu et al., 2002). So the 6063 alloy with Ti and RE additions has finer grain than that with Ti addition. Joint additions of Ti, RE and B to 6063 alloy improve the grain-refining efficiency, obtaining the finest microstructure. RE is a kind of surface-activated element, RE addition promotes the forming of TiB_2 and prevents TiB_2 and other impurity phases acting as heterogeneous nucleating core from assembling, which improves grain-refining efficiency (Fu et al., 2003.).

3.2. *Artificial ageing behaviour of alloys with different natural ageing time*

Alloys 1–3 were held at room temperature for natural ageing, and then for artificial ageing at 200 °C for 2 h. Fig. 2 shows the artificial ageing behaviour of alloys with different natural ageing time. Artificial ageing hardness varies with the natural ageing time. After natural ageing for more than 7 d, artificial ageing hardness is higher enough. Artificial ageing must be performed more than 7 d after extrusion in order to get higher artificial ageing hardness. Clusters of Si atoms and clusters of Mg atoms form extremely rapidly after quenching. But Mg atoms dissolve in solid solution with time during natural ageing, resulting in decreasing of hardness. Subsequently the dissolving Mg atoms combine with the clusters of Si atoms to form co-clusters of Si and Mg atom. Co-clusters of Si and Mg atom form gradually during natural ageing with the help of quenched-in vacancies, and the longer the natural ageing time, the higher the number density of clusters (Zhen and Kang, 1998.). Co-clusters of Si and Mg atoms have the similar compositions with intermediate phases, which act as nuclei for intermediate phases, nucleate the β″ precipitates and affect the distribution of β″ precipitates (Marioara et al., 2003.).

3.3. *Artificial ageing behaviour of alloys with different grain-refining methods*

Fig. 3 shows the artificial ageing hardness curves of the 6063 alloys aged at 200 °C for 0–360 min. Fig. 4 shows the TEM bright field images of alloys 1–3 before artificial ageing. The ageing hardness of the alloy 1 refined with Ti increases to the hardness near peak hardness after ageing for 80 min at 200 °C. 6063 alloys refined with Ti, Ti + RE, or Ti + RE + B all have better ageing behaviour. It takes shorter ageing time to obtain a hardness near peak ageing hardness and there is no obvious decrease of ageing hardness of the alloys until ageing for 6 h at 200 °C. The TEM results reveal that the shortening of time of obtaining peak hardness for alloy 1 is due to the contributions of high density of dislocations in the sample before artificial ageing (Fig. 4(1)). The high density of dislocation provides a number of sites for the heterogeneous nucleation of precipitates, which accelerates the precipitation processes, because of solute atoms diffusion accelerated along dislocations and the increasing of vacancies by dislocation movement (Yao et al., 2001; Cai et al., 2004.). It was found that round precipitates containing RE were formed within grains and on grain boundaries in (2) and (3).

Fig. 5 shows the DSC thermograms of alloys 1–3 before artificial ageing. Exothermic peaks B and B″ at about 250 °C in alloys 1 and 3 and exothermic peak B′ in alloy 2 at about 285.5 °C are associated with β″ precipitates. This kind of precipitates is responsible for the peak hardness of alloys. So it takes longer ageing time for alloy 2 to attain the peak ageing hardness than alloys 1 and 3. Fig. 5 also shows that alloy 1 has more β″ precipitates than alloys 2 and 3. So alloy 1 obtains

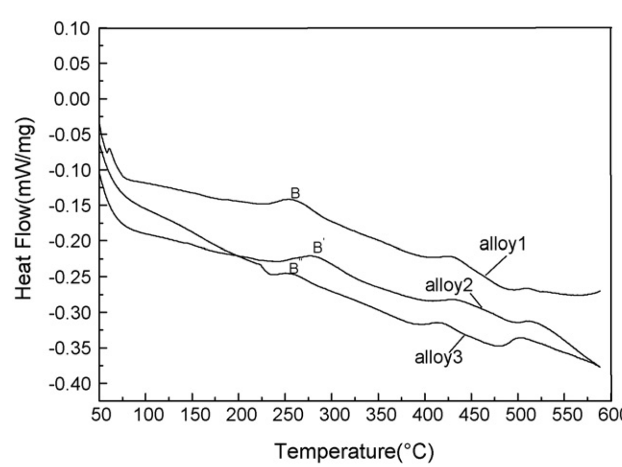

Fig. 5 – DSC thermograms of alloys 1–3 before artificial ageing.

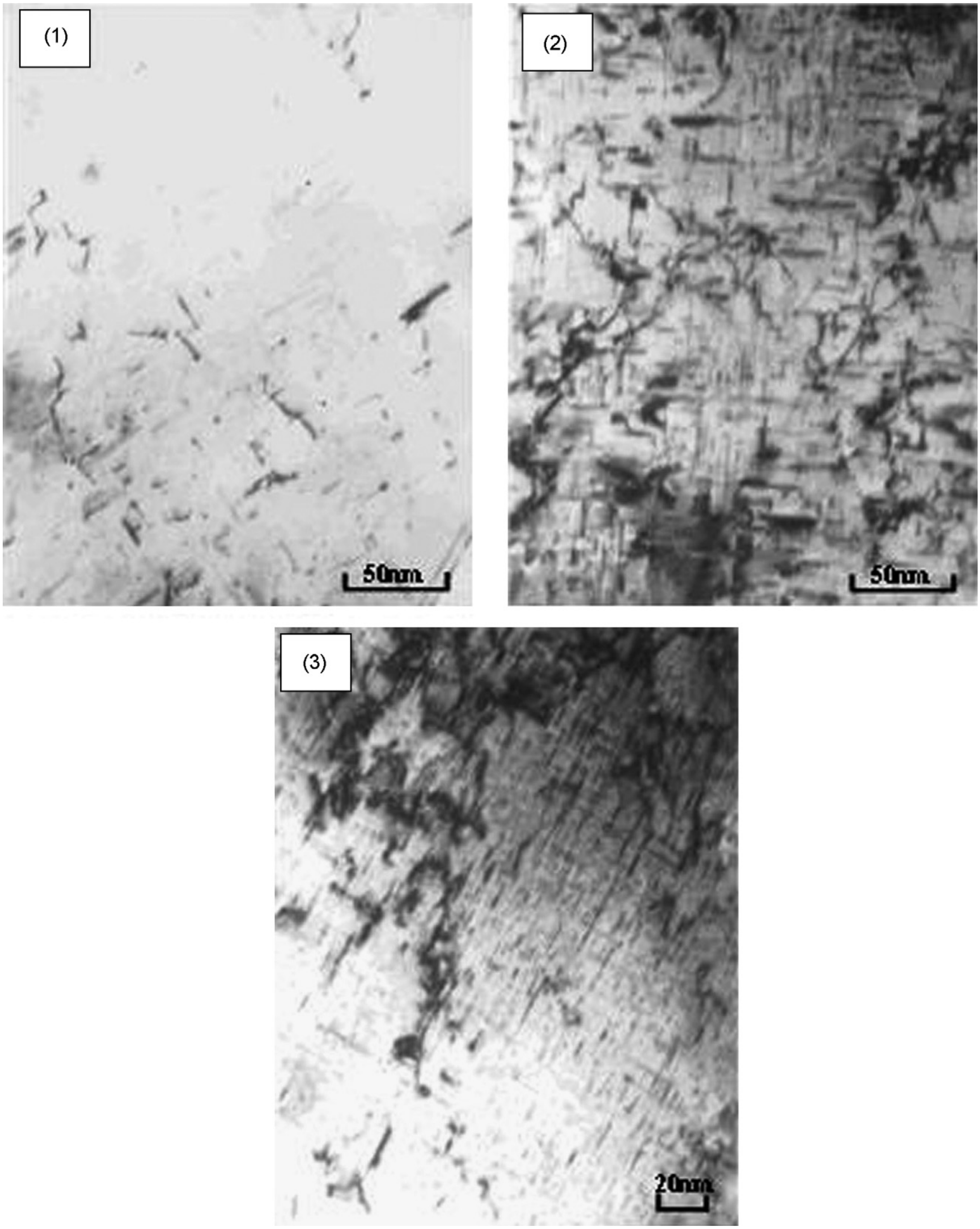

Fig. 6 – TEM bright field images of alloys 1–3 aged at 200 °C for 140 min: (1) alloy 1, (2) alloy 2, and (3) alloy 3.

higher peak aged-hardness than alloys 2 and 3. Alloy 3 has the least amount of β″ precipitates, resulting in the lowest peak aged-hardness. Fig. 6 shows the TEM bright field images of alloys 1–3 aged at 200 °C for 140 min. The increment of hardness of 6063 aluminium alloys is due to the contributions of refined β″ precipitates (Fig. 6).

Fig. 7 shows the SEM photomicrographs of as-cast alloys 1–3. Table 2 shows the results of EDS of precipitated phases

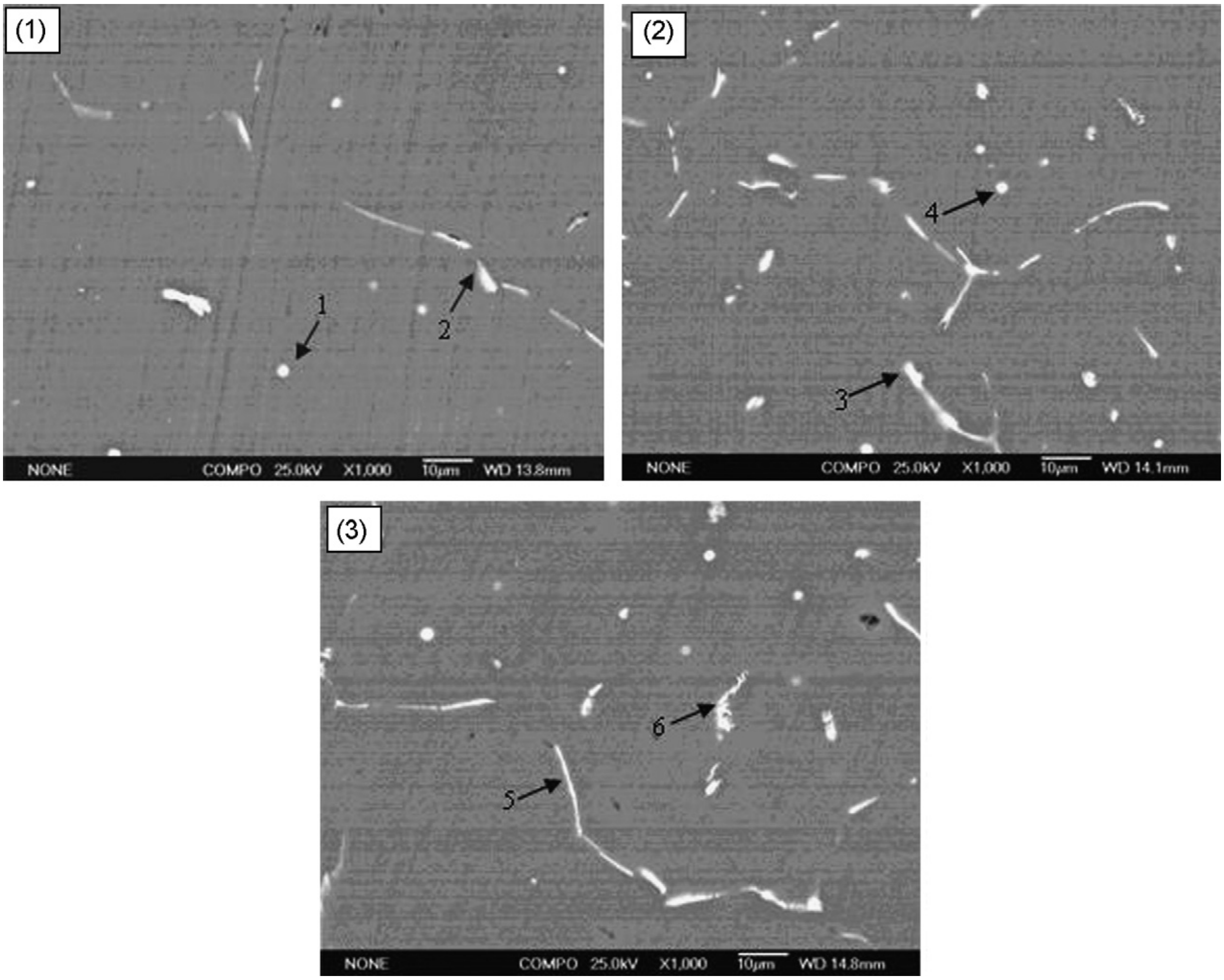

Fig. 7 – SEM photomicrographs of as-cast alloys 1–3: (1) alloy 1, (2) alloy 2, and (3) alloy 3.

in Fig. 7. The alloys 2 and 3 with the RE additions contain a lot of white block particles, the results of EDS analysis indicate that they contain higher RE, Mg and/or Si, whereas alloy 1 without RE addition has only less Al–Fe–Si–Mg precipitates. From the results shown above, it is possible to suggest that the reason for the highest strength in alloy 1 is more strengthening phases than that in alloys 2 and 3 with the RE addition. This is because RE and Si have strongly interaction, forming a lot of Al–Si–Mg–RE intermetallic constituents. So the addition of RE makes Mg and Si content forming Mg_2Si decrease, resulting in decreasing of strength of alloys 2 and 3.

Table 2 – Results of EDS of precipitated phases in Fig. 7 (wt%)

Tested place	Mg	Si	Fe	La	Ce	Al
1	1.61	5.75	10.49	–	–	Balance
2	0.77	4.30	3.10	–	–	Balance
3	–	3.92	–	3.51	5.52	Balance
4	1.14	7.91	3.98	2.65	4.56	Balance
5	0.63	2.55	1.24	–	2.24	Balance
6	3.13	9.18	–	3.72	7.05	Balance

4. Conclusions

The following conclusions have been obtained:

(1) The 6063 alloy with Ti and RE additions has finer grain than that with Ti addition. The 6063 alloy with the joint additions of Ti, B and RE (10 w(Ti)/w(B) mass ratio) has the finest grain, compared to alloys with Ti or Ti + RE additions.
(2) Artificial ageing must be performed more than 7 d after extrusion for 6063 alloys refined with Ti, Ti + RE or Ti + RE + B.
(3) The 6063 alloys refined with Ti, Ti + RE or Ti + RE + B all have better ageing behaviour. It takes shorter ageing time to obtain a hardness near peak ageing hardness and there is no obvious decrease of ageing hardness of the alloys until ageing for 6 h at 200 °C.
(4) The addition of RE makes Mg and Si content forming Mg_2Si decrease, resulting in lower strength of alloys with

Ti + RE and Ti + RE + B additions than that of alloy with Ti addition.

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