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Influence of high temperature exposure on the properties of alumina short fibre reinforced AA6061 alloy

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Influences of high temperature exposure on the mechanical properties and microstructures of AA6061 alloy matrix composites containing 15 vol % alumina short fibre (FRM) have been examined. When the exposure temperature exceeded 853 K, the degradation in fracture strength of the FRM became remarkable. During the heat treatment, magnesium, which is one of the strengthening elements of the matrix, segregated on the surface of alumina fibres to form a spinel and vaporized from the specimen surface in the vacuum. Therefore, degradation in the strength of the FRM was caused bv weakening of the matrix due to the dissipation of magnesium from the matrix. Appropriate conditions for joining the FRM were also discussed.

1. Introduction

Ceramic fibre reinforced aluminium matrix composites have been applied to a lot of structural components, such as engine parts of cars and aeroplanes, in which the properties of light weight, high modulus, high strength and resistance to wear are required up to high temperatures. For such applications of FRM, complex shaped products cannot be obtained directly in a single process. The products are first divided into simple shaped parts, and then the parts are joined with each other. For the joining, mechanical fastening such as bolting should be avoided because such methods inevitably cause considerable stress concentrations. Thus, suitable joining techniques of FRM are required.

Joining methods of FRM have several requirements. One of the important points is not to disturb the alignment of fibres across a joining interface. Brazings and solid-state joinings seem to be favourable processes. Recently some of the present authors reported on the brazing of alumina short fibre reinforced AA6061 alloy [1], in which joining could be achieved with several kinds of AA4045 brazing sheets above 853 K. In the solid-state joining process temperature conditions of > 873 K were maintained for 1 h [2]. In both cases, FRM must be exposed to high temperatures for a certain period in order to get sound joints. Besides, FRM is sometimes exposed to high temperatures in the other secondary fabrication processes such as extrusion, rolling and forging. In such processes, higher temperatures are better for preventing fibres from breaking up [3].

When the FRM is exposed to high temperatures, degradation of the fibre strength sometimes occurs, due to reactions between the fibre and the alloying elements in the matrix. An alumina fibre is relatively stable in aluminium alloys as compared to other fibres such as boron, carbon and silicon carbide below 773 K [4]. However, exposure to temperatures above 773 K during the secondary fabrication of FRM sometimes degrade the alumina fibre due to reaction with alloying elements, especially magnesium [5, 6]. Magnesium reacts with alumina to form a spinel on the surface of its fibres. However, these works [5, 6] have been focused on reactions during the first fabrication process such as casting above the liquidus temperature of aluminium alloys. Few works have reported on the influence of exposure to high temperatures on the properties of FRM, as in the secondary processes mentioned above. The aim of the present work was therefore to examine the influence of exposure to high temperatures on FRM below the liquidus of alloy matrices and to get a knowledge of appropriate secondary fabrication conditions.

2. Experimental

2.1. Materials

A hot-extruded rod of alumina short fibre reinforced AA6061 alloy (FRM) was used as an FRM. The

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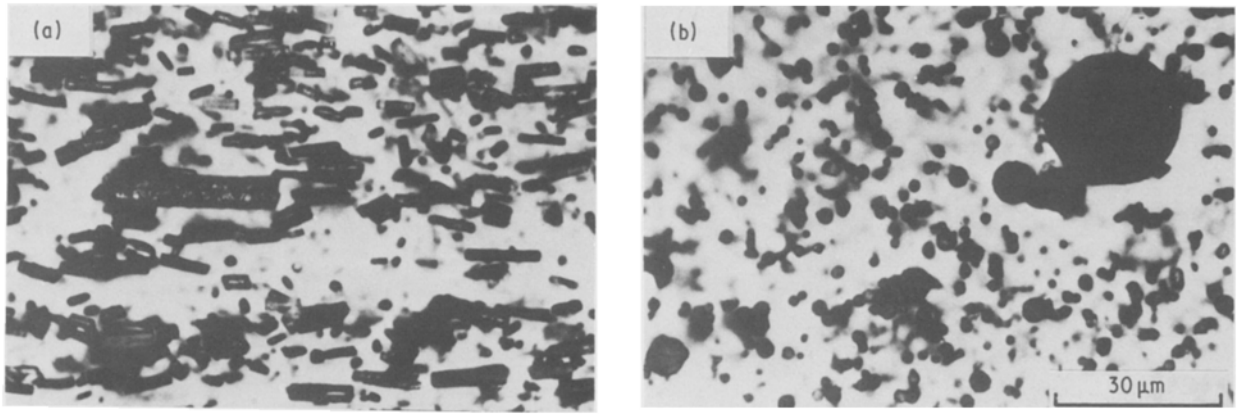


Figure 1 Fibre alignment on (A) longitudinal cross sections and (B) transverse cross sections of FRM (OM).

diameter of the rod was 10 mm. The alumina fibre was 3 μm in average diameter and its aspect ratio was about 5. The volume fraction of the fibre in the FRM was 15 vol %. The fibres were aligned along the extruding direction, as shown in Fig. 1.

The rod was sliced along the longitudinal section into plates of 1.4 mm thickness. All experiments were carried out using the plates. Tensile specimens, 4 mm in the parallel part and 15 mm in gauge length, were prepared from the plates.

2.2. Thermal treatments

Thermal exposure was carried out under vacuum, maintaining a temperature range of 843 to 873 K for 5 h in a 3×10^{-5} torr vacuum. Following thermal exposure, T6 heat treatment was performed. The temperature diagram used is shown in Fig. 2.

2.3. Tensile testing and observations

A tensile test was carried out at a strain rate of $5.6 \times 10^{-5}\,sec^{-1}$ at room temperature. Microstructure and solute concentrations were examined by optical microscopy (OM), electron microprobe analysis (EPMA) and transmission electron microscopy (TEM). A specimen for TEM was prepared both by ion thinning and by electropolishing. TEM was carried out with a Hitachi H600FE operated at 100 kV and with a Hitachi HU650 operated at 500 kV.

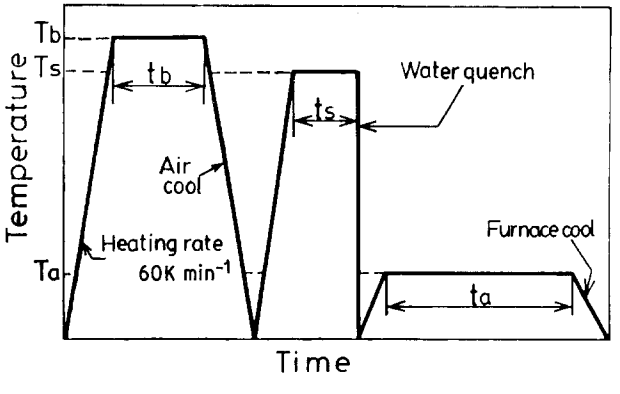


Figure 2 Diagram of thermal treatments. Tb: Exposure temperature; tb: Exposure time; Ts: Solution treatment temperature, 793 K; ts: Solution treatment time, 30 min; Ta: Ageing temperature, 348 K; ta: Ageing time, 8 h.

3. Results and discussion

3.1. Tensile properties

Fig. 3 shows the dependence of fracture strength on the exposure temperature. The fracture strength decreased gradually with increased exposure temperature. Exposed for only 10 min at 873 K, the specimen dropped by 10% in strength from that of the as-received specimen (T6-treated). Fig. 4 shows the dependence of fracture strength on exposure time at 853 K. Suganuma *et al.* [1] determined that this was the most suitable brazing temperature with an AA4045 sheet. Again the strength decreased with increasing exposure time. After 5 h exposure a 40% drop in strength occurred. Fig. 5 illustrates the representative apparent stress–strain curves of the specimen exposed at 853 K for various periods. With increasing exposure time, the strength decreased and the elongation increased, especially after prolonged exposure. Exposure to higher temperatures than 853 K also made elongation increase. Thus, the strength of

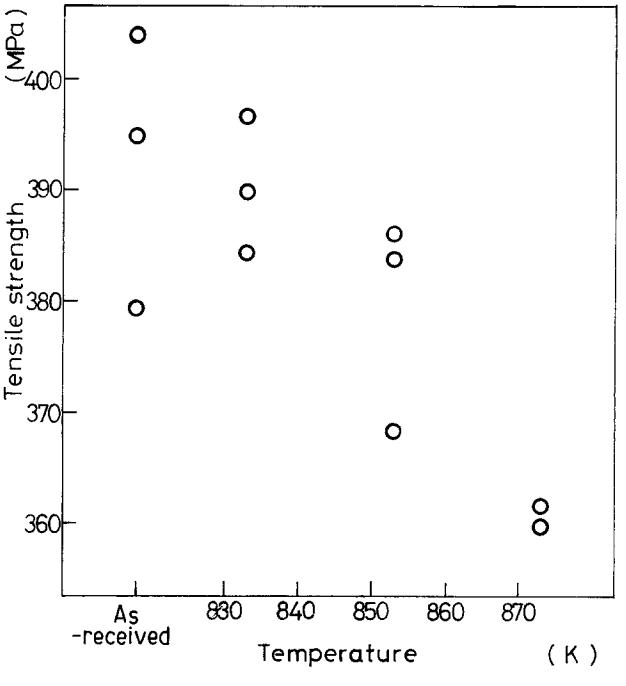


Figure 3 Change in fracture strength as a function of exposure temperature. Exposure time, 600 sec.

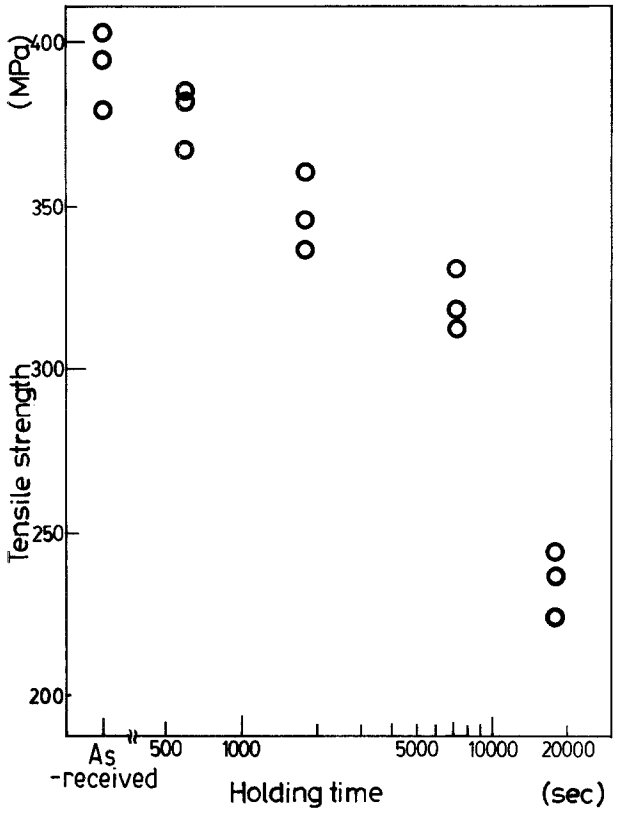


Figure 4 Change in fracture strength as a function of exposure time. Exposure temperature, 853 K.

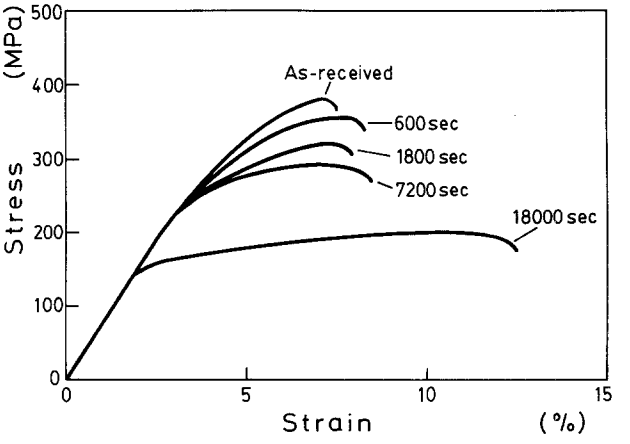


Figure 5 Apparent stress–strain curves of FRM exposed to 853 K for various times.

the FRM decreased with increasing exposure temperature or time, which was accompanied by an increase in elongation.

3.2. Change in microstructure

Fig. 6 shows the SEM images of two specimens and the characteristic X-ray images of the main alloying elements, magnesium and silicon in AA6061. The distribution of these elements in the specimens are shown. The as-received T6 treated specimen showed minor segregation of magnesium around the alumina fibres;

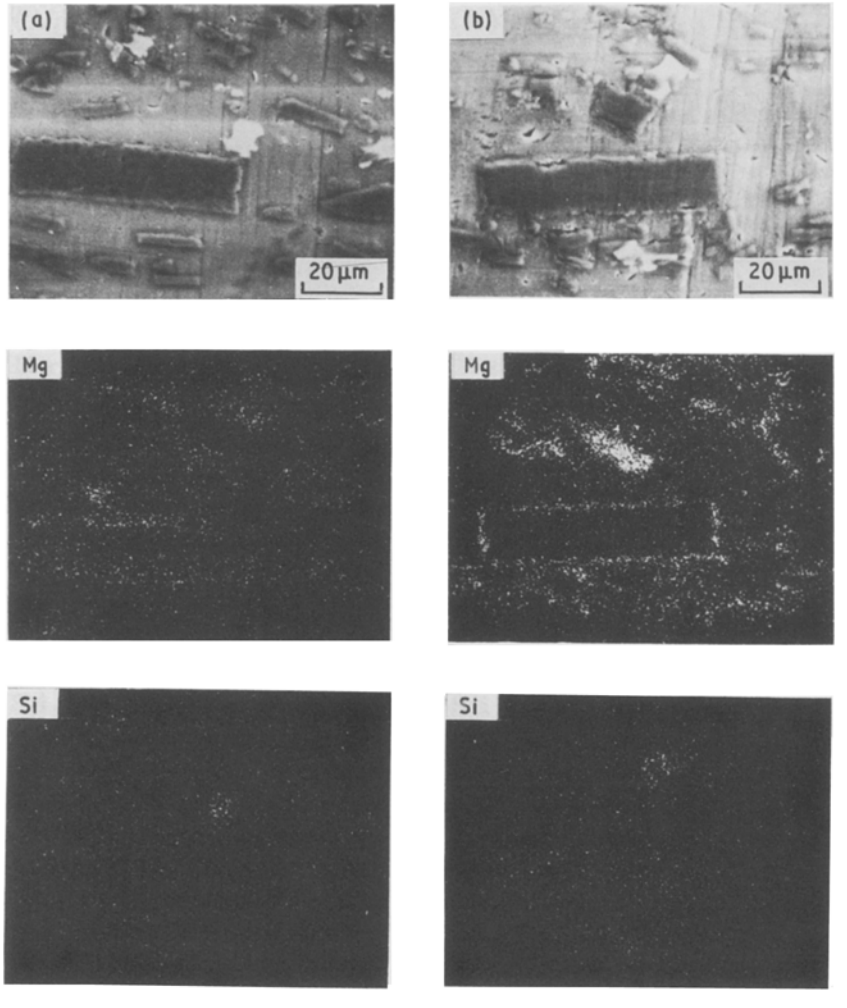


Figure 6 SEM images and characteristic X-ray images of longitudinal cross sections of (A) as-received and (B) exposed to 853 K for 5 h FRM.

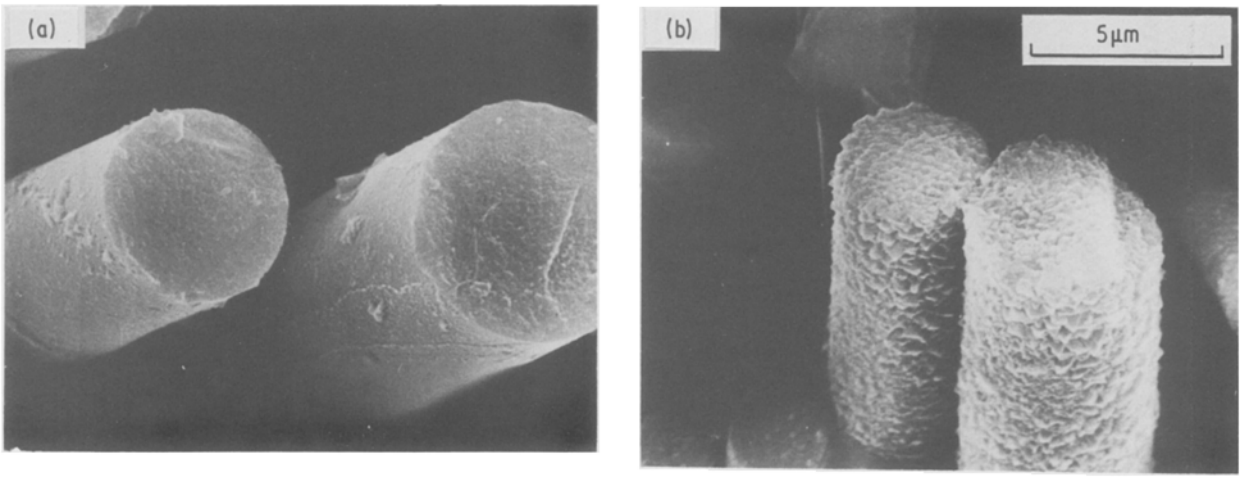


Figure 7 SEM images of extracted alumina fibres (A) as-received and (B) exposed to 853 K for 5 h.

silicon showed little segregation. On the other hand, the specimen exposed to 853 K for 5 h experienced severe magnesium segregation; but little segregation of silicon was recognized.

Fig. 7 shows the surface appearances of electrically extracted alumina fibres for the two specimens. Although the fibre in the as-received T6 treated specimen had a relatively smooth surface, those in the thermally exposed specimen had a very irregular surface which seemed to be covered by fine sharp precipitates.

The TEM photograph of the interface between the alumina fibre and the matrix is shown in Fig. 8. The alumina grains in the fibre were finer than 50 nm. The reaction products were recognized at the interface. They were observed as a layer consisting of fine sharp precipitates about 100 nm in size, which were the same as those seen in Fig. 7. Fig. 9 is an enlarged photograph of the interface between the precipitates and the alumina fibre. The lattice spacings of the precipitates in the photograph are 0.24 nm and 0.23 nm. Fig. 10 shows the representative electron diffraction patterns of precipitates on the electrically extracted fibres, which had no damage due to ion irradiation during thinning treatment [7]. The electron diffraction pattern indicated that the precipitate was a spinel, $MgAl_2O_4$. This result is consistent with the lattice spacings obtained above. The values of 0.24 nm and 0.23 nm corresponded to the spinel lattice spacings of (3 1 1) and of (2 2 2).

3.3. Quantitative analyses of magnesium and silicon

From the results mentioned above, it is clear that the concentration of alloying elements in the aluminium matrix would change upon heating because the heat treatments brought about precipitation of magnesium compounds on the fibre surfaces. Fig. 11 and Fig. 12 show the semi-quantitative analyses of magnesium and silicon in the alloy matrix, measured by a point counting method using EPMA as functions of heat treatment, temperature and time. Although the concentration of silicon did not change during heat treatment, that of magnesium decreased both with increasing

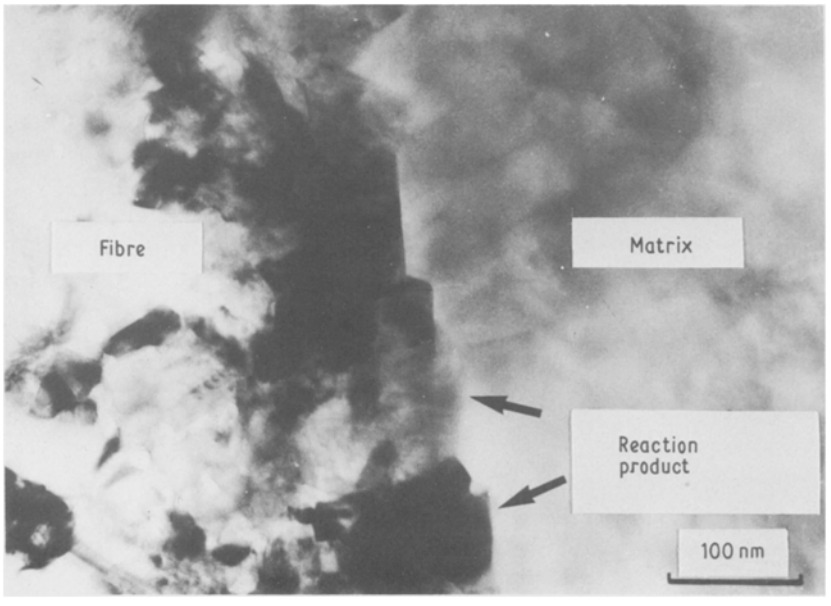
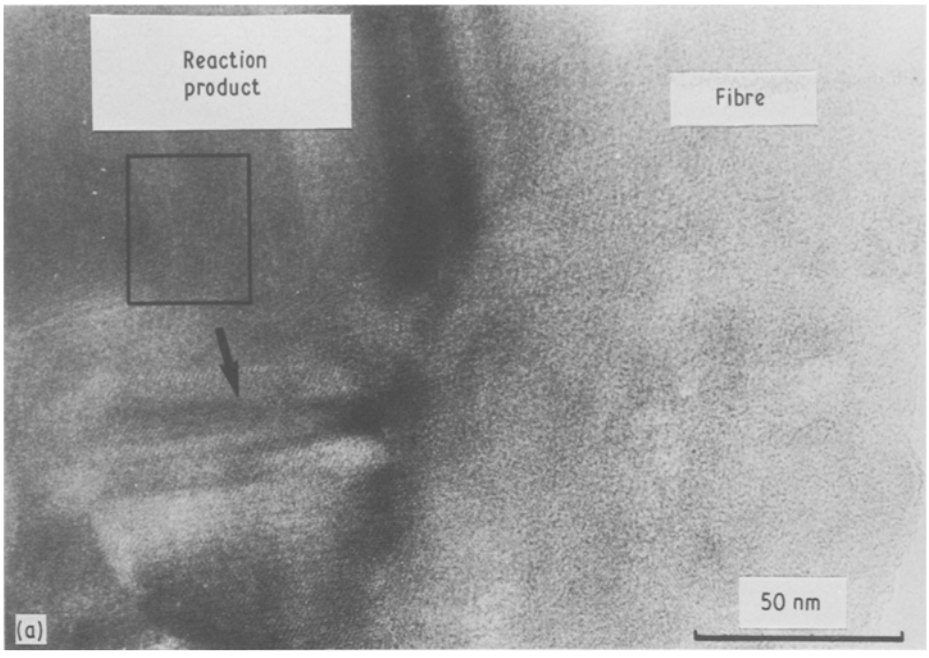


Figure 8 TEM image of the interface between the alumina fibre and the matrix of FRM exposed to 853 K for 5 h.



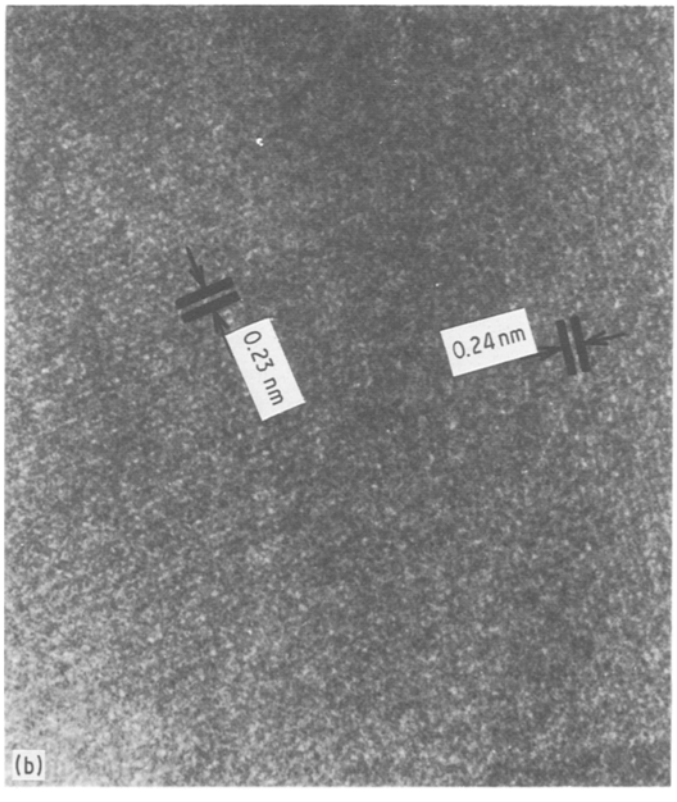


Figure 9 Enlarged TEM image of the interface between (A) the alumina fibre and the reaction product, and (B) the lattice image of the reaction product when the FRM was exposed to 853 K for 5 h.

temperature and with time. The concentration of magnesium especially, dropped to the impurity level when the FRM was treated at 853 K for 5 h.

There is one more reason for the decrement of magnesium to be considered. Because the heat treatment was carried out in a vacuum, vaporization of magnesium from the surface of the specimen could occur. Fig. 13 and Fig. 14 show the change of magnesium and silicon concentrations in an AA6061 sheet, of the same dimensions as the FRM specimen, during heat treatment. Again the concentration of silicon did not show any change. However, that of magnesium decreased apparently. Because these specimens did not contain alumina fibres, the decrease of magnesium from the matrix occurred by vaporization of the element from the specimen surface. Fig. 15 shows the profiles of magnesium and silicon line analyses near the free surface. The concentration of magnesium decreased upon approaching the free surface, which indicates that vaporization of magnesium from the specimen surface occurred. In addition, the concentration of silicon increased near the surface. This increase seems to be caused by vaporization of magnesium near the free surface, which prevented the formation of Mg_2Si and, hence, silicon remained in the matrix.

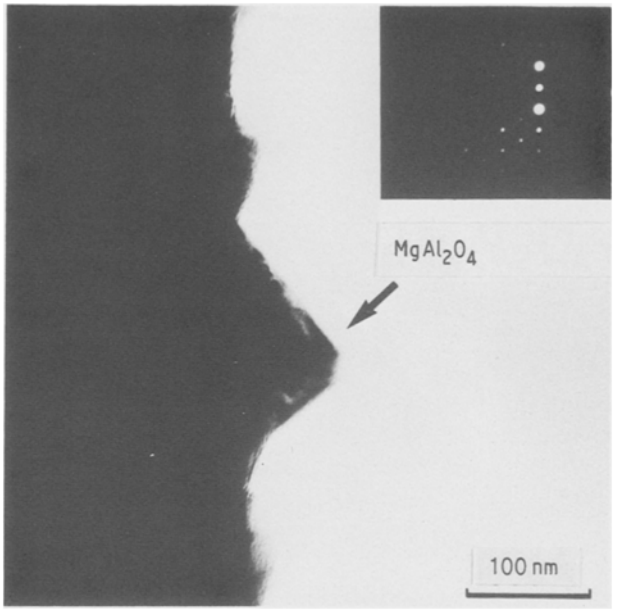


Figure 10 TEM image of the surface of an extracted alumina fibre exposed to 853 K for 5 h and the diffraction pattern taken from the reaction product.

3.4. Optimization of conditions for high temperature secondary fabrication

Summarizing the present results, we obtained that the vacuum heated treatments (> 850 K) brought about the precipitation of magnesium on the alumina fibre surface as a spinel and that vaporization of magnesium from the specimen surface occurred. Subsequently the aluminium matrix became depleted in magnesium. This lack of magnesium weakened the matrix because the precipitates of Mg_2Si in the matrix became less. In addition, the rough alumina or spinel surface will produce stress concentrations under an applied load, causing the strength of the FRM to decrease. Thus, heat treatments weakened the FRM. Finally from this point of view, the optimum thermal conditions for joining or extrusion at elevated temperatures could be derived.

Lower temperatures and shorter times are recommended because they prevent the precipitation of a spinel on the alumina fibre surface and prevent the vaporization of magnesium from the specimen surface. However, in order to tightly join the AA6061 alloy or the AA6061 based composites with each other or to extrude the FRM without damage to the fibres, exposure to a certain temperature and time is required.

For joining of FRM, although several methods have been applied, a solid-state joining and brazing will produce a tight and sound FRM joint, preventing disturbance of the fibre arrangement across the interface. In addition, breakage of the oxide film formed on

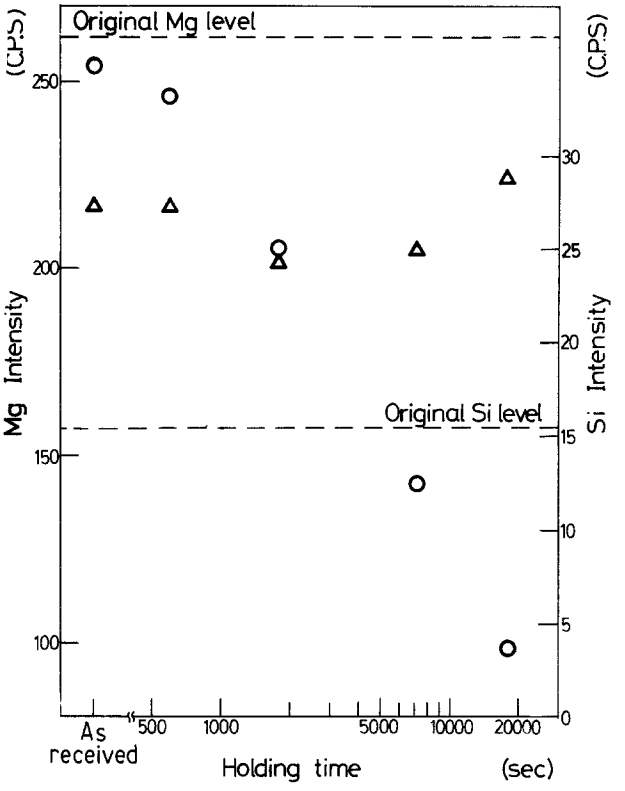


Figure 12 Change in content of magnesium and silicon in the FRM matrix as a function of exposure time, measured by EPMA. The magnesium level of the AA1050 pure aluminium was 70 cps. Exposure temperature = 853 K. Mg . . . ○, Si . . . △.

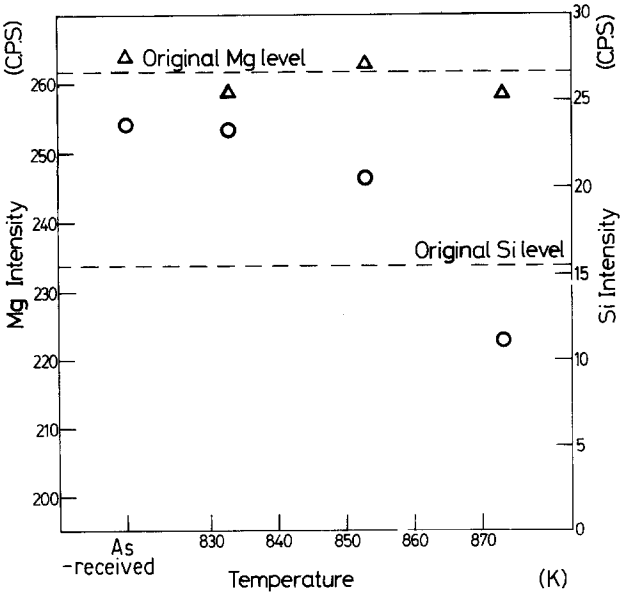


Figure 11 Change in content of magnesium and silicon in the FRM matrix as a function of exposure temperature, measured by EPMA. Where the exposure time = 600 sec. Mg . . . ○, Si . . . △.

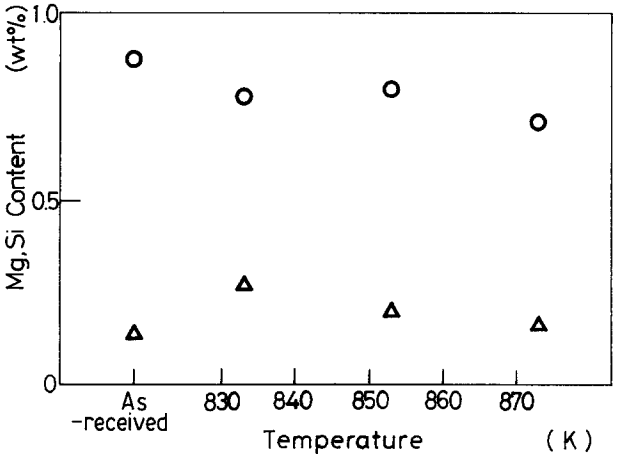


Figure 13 Change in content of magnesium and silicon in the AA6061 alloy as a function of exposure temperature, measured by chemical analyses. Exposure time = 600 sec. Mg . . . ○, Si . . . △.

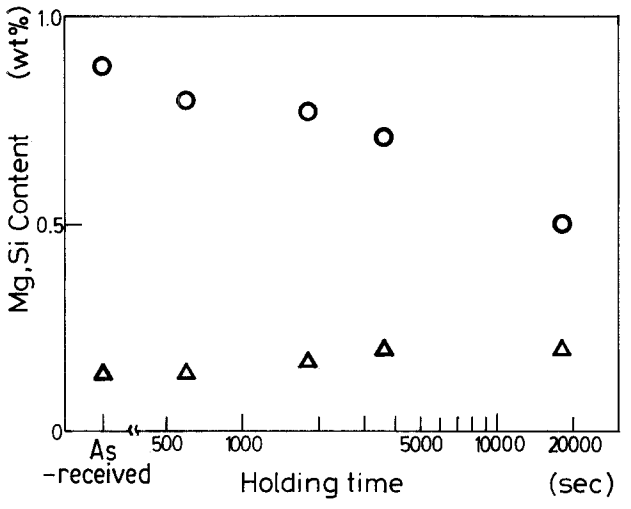


Figure 14 Change in content of magnesium and silicon in the AA6061 alloy as a function of exposure time measured by chemical analysis. Exposure temperature = 853 K. Mg . . . ○, Si . . . △.

the aluminium alloy surface is needed for joining of the matrix in both methods. For solid-state joining of the AA6061 matrix, temperatures > 873 K, maintained for > 1 h are required [2]. Beyond this condition the oxide film on the alloy surface can be broken. However, this condition will yield more than a 10% drop in strength as shown in Fig. 3. To get a strong FRM joint, this method has to be modified somewhat. The brazing method can lower the temperature and shorten the time by selecting an appropriate brazing material. For instance, brazing of the FRM used in the previous study with a AA4045 sheet or with a BA03 brazing sheet can be performed for only 10 min at low temperatures, 853 K [1]. This condition kept the strength drop within 2% (Fig. 4). Therefore, the brazing method is recommended for joining of the FRM.

Recently hot-extrusion of FRMs with ceramic short fibres has been focused on, because of its mass-producibility. However, fibres are so damaged during hot extrusion that the extruded FRMs cannot have the same strength level as that obtained by continuous fibre reinforcement. Such FRMs are now used only for abrasive purposes. To prevent the breakage of fibres during extrusion, especially under semi-solid conditions, the higher temperature extrusion process is now expected to be adopted. For instance, the semi-solid extrusion of the same as-cast FRM used in the present study at 883 K raised the strength of the FRM by 20% at room temperature [3]. However, the present results indicate the importance of temperature on the weakening of the matrix and the fibre. The optimization of this condition requires further experimentation on extrusion, which is beyond the scope of the present study.

4. Conclusion

This paper dealt with the changes in microstructure and strength during high temperature exposure of the alumina short fibre reinforced AA6061 composite and showed the importance of temperature and time optimization for secondary fabrications such as joining and extrusion.

The matrix strength decreased upon heat treatment at > 850 K and was accompanied by a decrease in the concentration of magnesium following precipitation of magnesium as a spinel on the alumina fibre surface. Vacuum treatment also contributed to the decrement of magnesium concentration by vaporization from the specimen surface. The present results recommend the use of the brazing method at short intervals and at lower temperatures for joining FRMs.

Acknowledgement

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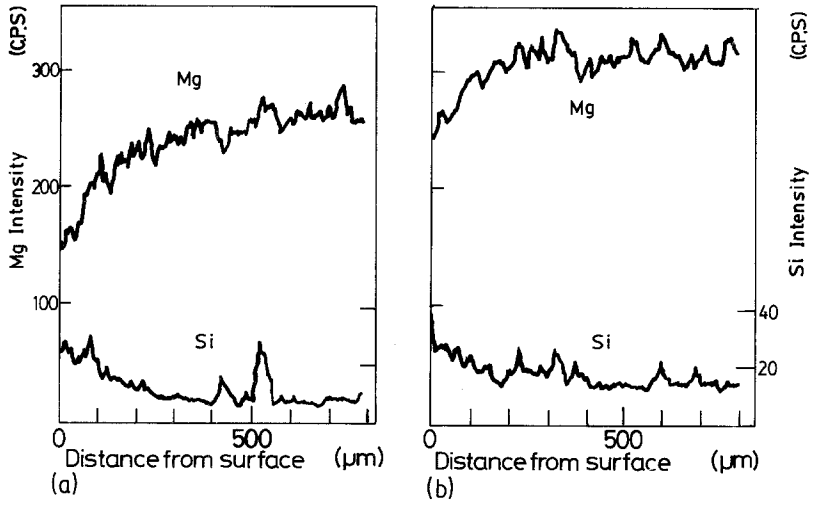

Figure 15 Line profiles of characteristic (A) magnesium (exposure temperature = 853 K, exposure time = 18 000 sec) and (B) silicon (exposure temperature = 833 K, exposure time = 600 sec) X-rays in FRM near the free surface of specimens exposed to high temperatures.