Original Article

Microstructural characterization, solidification characteristics and tensile properties of Al–15%Mg<sub>2</sub>Si–x(Gd–Sb) in-situ composite

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ABSTRACT

In the present study, complex modification of primary and eutectic Mg<sub>2</sub>Si particles in Al–15%Mg<sub>2</sub>Si composite was examined by simultaneous addition of gadolinium (Gd) and antimony (Sb). It was realized that combined addition of 1.5 wt. % Gd–Sb was efficient for modifying Mg<sub>2</sub>Si, in which coarse dendritic primary Mg<sub>2</sub>Si particles were changed into a perfect truncated octahedral morphology with a decrease in average size from 40 μm to 12 μm, an increase in density of Mg<sub>2</sub>Si per area from 495 to 1435 particle/mm<sup>2</sup>, and a reduction in the aspect ratio from 1.34 to 1.17. Furthermore, eutectic Mg<sub>2</sub>Si particles transformed from plate-like to fiber-like structure with an average eutectic Mg<sub>2</sub>Si cell decreasing from 132 μm to 51.22 μm. Thermal analysis revealed that nucleation and growth temperatures of primary Mg<sub>2</sub>Si increased with addition of Gd–Sb. However, it had an opposite effect for eutectic Mg<sub>2</sub>Si. It was proposed that refinement/modification of primary Mg<sub>2</sub>Si particles was mainly derived from formation of Mg<sub>2</sub>Sb<sub>x</sub> compounds as heterogeneous nuclei for primary Mg<sub>2</sub>Si. Meanwhile, the preferred growth of primary Mg<sub>2</sub>Si crystals along <100> direction can be obviously suppressed by the absorption and poisoning of Gd and Sb. The tensile results presented that addition of 1.5 wt. % Gd–Sb to the composite enhanced the UTS and %El from 204.79 MPa and 2.65% in base condition to 242.73 MPa and 3.9% in the modified composite, respectively. This study demonstrated that combined addition of Gd–Sb can provide a better effect on the modification and tensile properties of Al–15%Mg<sub>2</sub>Si composite than addition of Gd or Sb separately.

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1. Introduction

Mg$_2$Si intermetallic compound exhibits low density (1.99 × 10$^3$ km$^{-3}$), high hardness (4500 MN m$^{-2}$), high melting point (1083 °C), high elastic modulus (120 GPa) and low coefficient of thermal expansion (CTE) of (7.5 × 10$^{-6}$ C$^{-1}$). These features make it a promising candidate as a strengthening phase in Al–Mg$_2$Si and Mg–Mg$_2$Si composites that are used in the ultralight design of an automobile [1]. However, the coarse and dendritic shape of primary Mg$_2$Si formation in the composite during solidification process leads to the localization of stress concentration at the sharp ends and angles of Mg$_2$Si particles, which induces low mechanical properties. The mechanical properties, especially ductility, are further impaired by flake-like morphology of eutectic Mg$_2$Si [2]. Therefore, the modification of the MMC is necessary for enhancing the properties. It was reported that the development of microstructure and improvement mechanical properties of Al–Mg$_2$Si composites can be achieved by melting treatment as a practical and cost effective method by using different types of alloying element as the refiner or modifier elements. Recently, P, Sr, Li, Bi, and rare earth (RE), such as cerium (Ce) and neodymium (Nd) as single addition elements have been used to refine or modify Mg$_2$Si during the melting process by modifying the effect on Mg$_2$Si particles morphology [3]. However, there are few studies in regard to co-modification of the addition elements on Mg$_2$Si particles modification in Al–Mg$_2$Si composites. Moreover, there is limited information about the combined addition of elements on Mg$_2$Si particles modification capability with corresponding mechanisms, especially for eutectic structure and solidification behavior and tensile properties of Al–Mg$_2$Si composite. Among the addition elements, Sb has been widely utilized as an active element in the modification of Mg$_2$Si particles [4]. Therefore, the combined addition of Sb and another element results in further refinement of primary and eutectic Mg$_2$Si particles. Currently, the effect of combined addition of elements, such as Li–Sb [5], Ca–Sb [6], and Sr–Sb [7] on the refinement/modification of primary Mg$_2$Si is investigated, whereby with the addition of Sb, the Mg$_2$Sb$_2$ phase is formed, which serves as a heterogeneous nucleation substrates (inclusions) for primary Mg$_2$Si particles [4,7]. Furthermore, the growth steps of primary Mg$_2$Si is suppressed along the <100> direction as a result of absorption and poisoning effect of Ca and Li elements [5,6]. However, due to the reaction tendency between two addition elements, new nucleant particles are formed, such as Li$_5$Sb, Ca$_5$Sb$_2$ and Sr$_5$Sb$_8$ in the melt or even fadin in modification occurs because the elements consume themselves. Therefore, playing multiple roles on the refinement of primary Mg$_2$Si for modifier elements is difficult. As a result, attaining incorporation and advantages of various modifiers and hindering the reaction tendency between two addition elements is the key to achieve the multiple modifying effects. To reach this purpose, the elements with small electronegativity difference that are unlikely to react with each other can be selected because the formation compounds between these elements can be avoided [8]. It was reported that transition metal elements might be good options to combine with Sb because they are less active than Ca, Li and Sr [9].

In addition, the formation of compounds between transition metal and Sb usually occurs at relatively higher temperatures as compared to alkaline-earth metals [10]. Therefore, the reaction between two addition elements might be avoided or at least weakened if a transition metal element such as Gd combines with Sb. The aim of the present study is to provide systematic information about the refinement/modification mechanisms of primary and eutectic Mg$_2$Si, solidification behavior and tensile properties of Al–15%Mg$_2$Si composite with the synergistic effects of co-modification of Gd and Sb additions.

2. Experimental procedures

2.1. Material fabrication

The starting materials for preparing composite ingot were Al (99.98 purity), Mg (99.85 purity), Si (99.7 purity), pure Gd (99.8 purity) and Sn (98.00 purity). The Al–15%Mg$_2$Si composite ingot was prepared by melting pure Al and Si in a graphic crucible by using a resistance furnace at 800 °C. Subsequently, pure Mg was added into the melt once the temperature decreased to 750 °C and finally cast in a metal mould. The composite ingot was then subjected to cutting into small pieces and re-melting in the induction furnace with a weight of 200 g at 750 °C. The melt was added with 0.5 wt. % Gd–Sb and after holding for 3 min at 750 °C and degassing with dry tablets containing C2Cl6, it was poured into a preheated (220°C) cast iron mold. The same method was followed for 0 wt. %, 1.0 wt. %, 1.5 wt. % and 2.0 wt. % of Gd–Sb additions. Table 1 illustrates the composition of used Gd–Sb additions, in which the designed atomic ratio of Gd–Sb was 1:1. In addition, Table 2 shows the tested composition of fabricated composites which were analyzed by a glow discharge spectrometer (LECO GDS-850A). The alloy compositions represented the average values taken over three measurements made on each sample.

2.2. Cooling curve thermal analysis (CCTA)

To record the cooling curves and monitor the characteristic temperatures of Mg$_2$Si phases and other information during the solidification process of the composites without and with different content of Gd–Sb, the cooling curve thermal analysis technique was carried out. For each casting, thermal analysis was set up by placing K-type thermocouples at the center of the ceramic mold that was preheated at 750 °C for 15 min. The thermocouples were then connected to an acquisition system with high-speed data (EPAD-TH8-K, EPAD-BASE2; Dewesoft-7.5-Lit software) to continuously record the temperature-time variations during the cooling process of the molten composites. The recorded data was then imported into Flexpro 10 for data analysis. The calculated cooling rate for all thermal analysis tests was about 0.8 °C/s.

Table 1 – The designed composition of different Gd–Sb additions added into the Al–15%Mg$_2$Si composite.

<table>
<thead>
<tr>
<th>Gd–Sb addition (wt. %)</th>
<th>0</th>
<th>0.5</th>
<th>1.0</th>
<th>1.5</th>
<th>2.0</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gd content (wt. %)</td>
<td>0</td>
<td>0.25</td>
<td>0.5</td>
<td>0.75</td>
<td>1.0</td>
</tr>
<tr>
<td>Sb content (wt. %)</td>
<td>0</td>
<td>0.25</td>
<td>0.5</td>
<td>0.75</td>
<td>1.0</td>
</tr>
</tbody>
</table>

2.3. Tensile test

Tensile test was conducted on the composite specimens that were made in accordance with ASTM B557M-02a standard in a computer-controlled SANTAM tensile testing machine at a constant cross-head speed of 1 mm/min. To ensure repeatability of results, the average tensile results were achieved from four specimens for each test.

2.4. Microstructural characterization

Specimens for microstructure characterization and crystallography analysis were cut from the gauge length of the tensile test and prepared according to standard metallurgy procedures, including grinding, polishing and etching with 2% HF acid. To observe the 3D morphology of primary and eutectic Mg-Si particles, the Al matrix was removed by deep etching of the samples for 6 h in a solution of 5% HCl acid and 95% ethanol. Quantitative analysis of the microstructure was conducted by using an optical microscope (Nikon-MIDROPHOT-FX1) equipped with an I-Solution image analyzer. At least eight optical images were used for each condition to calculate the mean size, density and aspect ratio, as reported elsewhere [11]. The specimens also underwent scanning electron microscopy, SEM (XL-40) equipped with EDX facility to investigate the morphology evolution of primary and eutectic Mg-Si particles, as well as Gd intermetallic compounds (IMCs) as a function of different concentrations of Gd-Sb addition. To identify phases that existed in the samples, x-ray diffraction (XRD) was accomplished on a Siemens-D500 diffractometer by using CuKα line generated at 40 KV and 35 mA. For crystallographic analysis of primary Mg-Si particles and eutectic Mg-Si grains, the EBSD analysis was accomplished with the samples.

3. Results and discussion

3.1. Effect of Gd-Sb addition on composite microstructure

Table 2 – Analyzed chemical compositions of Al–15Mg2Si composites (wt. %).

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Mg</th>
<th>Si</th>
<th>Gd</th>
<th>Sb</th>
<th>Fe</th>
<th>Cr</th>
<th>V</th>
<th>Mn</th>
<th>Ti</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gd–Sb-00</td>
<td>9.71</td>
<td>5.09</td>
<td>0.00</td>
<td>0.00</td>
<td>0.18</td>
<td>0.02</td>
<td>0.01</td>
<td>0.01</td>
<td>Bal.</td>
<td></td>
</tr>
<tr>
<td>Gd–Sb-05</td>
<td>9.50</td>
<td>5.10</td>
<td>0.23</td>
<td>0.24</td>
<td>0.11</td>
<td>0.01</td>
<td>0.01</td>
<td>0.02</td>
<td>Bal.</td>
<td></td>
</tr>
<tr>
<td>Gd–Sb-10</td>
<td>10.12</td>
<td>5.03</td>
<td>0.48</td>
<td>0.47</td>
<td>0.16</td>
<td>0.01</td>
<td>0.03</td>
<td>0.01</td>
<td>Bal.</td>
<td></td>
</tr>
<tr>
<td>Gd–Sb-15</td>
<td>9.68</td>
<td>4.98</td>
<td>0.72</td>
<td>0.74</td>
<td>0.22</td>
<td>0.03</td>
<td>0.02</td>
<td>0.03</td>
<td>Bal.</td>
<td></td>
</tr>
<tr>
<td>Gd–Sb-20</td>
<td>9.85</td>
<td>5.06</td>
<td>0.97</td>
<td>0.99</td>
<td>0.13</td>
<td>0.02</td>
<td>0.01</td>
<td>0.01</td>
<td>Bal.</td>
<td></td>
</tr>
</tbody>
</table>

Fig. 1 elucidates variation in the composite microstructure, corresponding to primary and eutectic Mg-Si crystals (cells) as a result of 0 wt. %, 0.5 wt. %, 1.0 wt. %, 1.5 wt. % and 2.0 wt. % Gd-Sb addition at a molar ratio of 1:1. In addition, the 3D morphologies of eutectic Mg-Si crystals as a function of different Gd–Sb additions are illustrated in Fig. 2. As observed in Fig. 1(a), the primary Mg-Si particles in unmodified condition exhibit a coarse dendritic structure with a hole at the center. Introducing Gd–Sb addition into the composite melt are shown to induce a substantial effect on the primary Mg2Si structure as the particles were converted to polygonal morphology with a change in their average size. When the composite was treated with 0.5 wt. % Gd–Sb, the corresponding sharp ends of primary Mg2Si particles started to fade, and the morphology of primary particles changed to polyhedral with a decrease in size, as shown in Fig. 1(b). With the addition of 1.0 wt. % Gd–Sb into the composite, the appearance of particles changed as the polyhedral shape transformed into a polygonal structure, as shown in Fig. 1(c). When the Gd–Sb concentration was increased to 1.5 wt. %, the primary Mg2Si particles were observed to have undergone refinement of the fine polygonal morphology (Fig. 1(d)). Nevertheless, increasing the concentration of Gd–Sb to 2.0 wt. % had a reverse effect on the refinement of primary Mg2Si particles in which their size increased slightly as observed in Fig. 1(e). This effect was due to an over modification phenomenon [12]. A similar result was reported in cast A–20MgSi–2Cu when the Sb addition exceeded 0.8 wt. % [4].

In accordance with the binary phase diagram of Al–Mg-Si [8], binary eutectic Al–Mg-Si phase was formed by further solidification of the cast composite. As stated, the eutectic Mg2Si phase existed as flake-like structure as observed in Figs. 1(2a) and 2(a). Figs. 1(2b) and 2(b) illustrate the changes of eutectic Mg2Si structure with the addition of 0.5 wt. % Gd–Sb. It can be seen that the morphology had revolved into small flakes in various directions. When the content of Gd–Sb addition reached 1.0 wt. %, the eutectic Mg2Si crystals transformed from flake-like to rod-like, as seen in Figs. 1(2c) and 2(c). With increased Gd–Sb addition to 1.5 wt. %, the morphology of eutectic Mg2Si altered to fibrous form (Figs. 1(2d) and 2(d, e)) and the eutectic Mg2Si exhibits the most refinement effect. As observed in Fig. 1(2e) and Fig. 2(f), with further addition of Gd–Sb addition to 2.0 wt. %, over modification occurs, which resulted in increasing the eutectic Mg2Si size and transition of the short flake-like morphology into rod-like. Therefore, from the aforementioned micrographs, it can be found that 1.5 wt. % Gd–Sb is the best concentration to achieve such refinement of eutectic structure. Ceschini et al. [13] claimed that the mechanical properties of Al alloys can be improved when the eutectic Si owned a fibrous morphology. Therefore, it is believed that the transformation of eutectic Mg2Si structure from flake-like to fiber-like improves the mechanical properties of Al–15%Mg2Si composite.

3.2. Quantitative analysis of microstructure with the addition of Gd–Sb

The characteristics of primary Mg2Si particles, including the grain size (μm), aspect ratio and number of particles formed per unit area (mm²) as a function of Gd–Sb addition are presented in Fig. 3 (a). As seen, increasing the Gd–Sb...

addition from 0.5 wt. % to 1.5 wt. % caused a significant refinement/modification effect on the characteristics of primary Mg2Si particles. With the addition of 1.5 wt. % Gd–Sb the most average size reduction is achieved by about 70%, from 40 μm (unmodified) to 12 μm. The aspect ratio also indicated an enhancement since it was reduced by about 13 % from 1.34

Fig. 1 – Optical micrographs of primary and eutectic Mg2Si particles (cells) at various Gd–Sb contents: (1a, 2a) 0; (1b, 2b) 0.5; (1c, 2c) 1.0; (1d, 2d) 1.5 and (1e, 2e) 2.0 wt. %.
in the unmodified composite to 1.17 with 1.5 wt. % Gd–Sb. In addition, 1.5 wt. % Gd–Sb caused the formation of the highest number of primary Mg₂Si particles per unit (mm²) with 1435 as compared to 495 in the unmodified composite, in which the morphology shown in Fig. 1 reflects these changes. Furthermore, Fig. 3 (b) depicts the alteration in average eutectic Mg₂Si cell size as a function of the amount of Gd–Sb addition. As shown, the eutectic grains have decreased from 132 μm (unmodified) to 51.22 μm with the addition of 1.5 wt. % Gd–Sb. These results were consistent with the observed microstructure shown in Fig. 1, which reflected the influence of Gd–Sb addition to the growth evolution of eutectic Mg₂Si particles, and resulted in refinement of eutectic cells (grains) by treating the composite with 1.5 wt. % Gd–Sb. It is believed that Gd/Sb compounds are responsible for such refinement effect by breaking the larger eutectic cells into smaller ones.

Fig. 4 (a–d) illustrates the BSE micrographs of Al–15%Mg₂Si composite, treated with 0.5, 1.0, 1.5 and 2.0 wt. % Gd–Sb additions. It was observed that the chemical interaction between the elements and intermetallic compounds (bright contrast) are formed in the microstructure of the composite, which was mostly distributed as a network at the interdendritic regions and grain boundaries. The magnified BSE micrograph and corresponding elemental mapping spectra of Al–15%Mg₂Si treated with 2.0 wt. % Gd–Sb are depicted in Fig. 4 (e) and (f), respectively. As observed, these white particles were probably Al–Si–Gd intermetallic compounds that existed in various morphologies, which owned non-uniform and needle-like white shapes, in which the volume fraction of these phases increased with increasing concentration of Gd–Sb addition.

To detect these compounds, the XRD pattern of Al–15%Mg₂Si composite treated with 2.0 wt. % Gd–Sb is presented in Fig. 5 (a). According to the results, several peaks presented certain phases, particularly the primary and eutectic Mg₂Si phases were altered in terms of intensity. By increasing the amount of Gd–Sb in the melt, new diffraction angles were occurred which indicated that new compounds were spotted around the diffraction angle of ~24° and ~28°, as shown in the magnified area (Fig. 5(b)) marked as (iii). This was believed that no peak corresponds to the Sb compounds and only Gd compounds were presented in the composite, in which the diffraction angles of 24.48° and 28.47° belonged to A₂Si₃Gd and Al₅Si₃Gd which corresponded to the non-uniform and needle-like white particles, respectively (Fig. 4 (e)), similar to the compounds that existed in the Al–15%Mg₂Si composite modified with 1.0 wt. % Gd addition [12].

3.3. Effect of Gd–Sb addition on cooling curves

As shown in Fig. 6, Gd–Sb addition at different concentrations leads to the alteration of cooling curves as a result of phase transformation or variation during the solidification process. Consequently, the characteristic temperatures (\(T_N\) and \(T_C\)) of the primary and eutectic Mg\(_2\)Si phases have also changed as a function of different Gd–Sb additions.

Refinement or modification of primary Mg\(_2\)Si can be associated with nucleation stage of the particles, in which the particles nucleate easily and obtain stable nuclei and continue to grow. As seen in Fig. 6, the starting temperature for primary Mg\(_2\)Si was shifted up to higher temperature once Gd–Sb content was increased from 0.5 wt. % to 2.0 wt. %. The changes in nucleation temperature (\(T_N\)) of primary Mg\(_2\)Si phase were further elucidated in Fig. 7. This figure clearly shows that the addition of Gd–Sb in various amount caused the nucleation temperature of primary Mg\(_2\)Si (\(T_N\)) to increase from 607.3 °C for the unmodified composite to 610 °C, 612.2 °C, 614.5 °C and 618 °C with additions of 0.5 wt. %, 1.0 wt. %, 1.5 wt. % and 2.0 wt. % of Gd–Sb, respectively. A similar pattern was also observed in \(T_C\). Similar to the effect of Gd addition in the previous study [12], the start of solidification at a higher temperature indicated that the primary Mg\(_2\)Si was susceptible to nucleation and growth. Besides, Fig. 6 illustrates the change in undercooling with respect to nucleation of primary Mg\(_2\)Si particles. With the addition of Gd–Sb addition up to 1.5 wt. % the undercooling, \(\Delta U\) had decreased as compared to unmodified composite. However, further addition of Gd–Sb to 2.0 wt. % caused the \(\Delta U\) to increase. It was reported that finer particles were achieved when there were more nucleation sites for primary Mg\(_2\)Si phase; hence, little undercooling was required. Nevertheless, the particles became coarser when there were few nucleation substrates for the particle. Therefore, significant undercooling could occur [14,15]. Accordingly, the most refinement of the primary Mg\(_2\)Si particles with the most formation of the
Fig. 4 – BSE micrographs of Al–15%Mg₂Si composite treated with different Gd–Sb contents; (a) 0.5 wt. %, (b) 1.0 wt. %, (c) 1.5 wt. %, (d) 2.0 wt. % showing intermetallic compounds (white contrast), (e) magnified micrograph of Al–15%Mg₂Si–2.0%Gd–Sb and (f) corresponding elemental mapping indicating Gd element.
Fig. 5 – (a) XRD patterns of the Al-15%Mg2Si composite treated with 2.0 wt. % Gd–Sb addition (b) Magnified Gd–Sb treated results.

particles per unit area was obtained when the composite was treated with 1.5 wt. % Gd–Sb as in Fig. 3 (a). Fewer drops in temperature (undercooling) facilitated the nucleation process of primary Mg2Si nuclei. Therefore, the primary Mg2Si are formed easily. This was in good agreement with other research works, which stated that decreases in undercooling degree (ΔΤ) were accompanied with easier phase nucleation [14,16].

Fig. 8 depicts the duration of the formation of primary Mg2Si phase and it was mentioned that although the nucleation of primary Mg2Si occurred at higher temperatures, the duration for the complete solidification of the respective phase into the solid was less, which refined the primary Mg2Si particles. In fact, the growth of these refined particles was controlled by the simultaneous addition of Gd and Sb and accelerated the solidification process of primary Mg2Si particles simultaneously.

The eutectic Mg2Si phase modification was related to a decrease in eutectic growth temperature (ΔΤcMg2Si) and eutectic depression temperature (ΔΤcMg2Si) [17]. As observed in Fig. 9, the addition of Gd–Sb from 0.5 wt. % to 1.5 wt. % decreased the ΔΤcMg2Si and consequently increased the ΔΤcMg2Si. The turning point of the eutectic Mg2Si phase was 1.5 wt. %, which was because of the exceeded level to 2.0 wt. % that increased and decreased the characteristic temperatures TcMg2Si and ΔΤcMg2Si, respectively. This trend was consistent with the results discussed in Section 3.1, which showed that increase in the concentration of Gd–Sb in the composite caused a reverse effect on the refinement of the eutectic structure. Based on Fig. 2, the refined fibrous form of eutectic Mg2Si was obtained when the content of Gd–Sb was 1.5 wt. %. Therefore, considering the aforementioned thermal analysis results and microstructure observations in Figs. 2 and 3 (b), it could be concluded that composite treated with 1.5 wt. % Gd–Sb addition exhibits refined fibrous morphology of eutectic structure. As presented in Fig. 9 (b), adding Gd–Sb at concentrations of 0.5 wt. %, 1.0 wt. % and 1.5 wt. %, the TcMg2Si had decreased to 591.6 °C, 590.9 °C and 589.7 °C, respectively, as compared to 591.8 °C in unmodified composite, indicating that the eutectic morphology has further refined. Indeed, as shown in Fig. 8, the duration of the eutectic Mg2Si formation had gradually decreased from 23 s (unmodified) to 18 s (0.5 wt. % Gd–Sb), 15.5 s (1.0 wt. % Gd–Sb) and 13 s (1.5 wt. % Gd–Sb), indicating the refinement effect was further improved. As mentioned in the previous study [12], absorption of Gd atoms in the solid-liquid interface changed the growth mode, in which Gd and Sb were added into the melt. Therefore, in

addition to Gd effect, Sb element can induce alteration in the growth mode of eutectic Mg$_2$Si. In fact, the solubility of Sb in liquid Al was less than 0.01 wt.%. Therefore, until the precipitation of eutectic Mg$_2$Si at 593.3°C, Sb was pushed into the solid-liquid interface. As a result, the Sb addition with high concentration decreased the surface tension of aluminium around the Mg$_2$Si crystals, which reduced the contact angle between molten Al and Mg$_2$Si, which in turn suppressed the growth of Mg$_2$Si crystals and the eutectic Mg$_2$Si was modified. Therefore, more activation energy was required by the system to start the growth phenomenon, which was provided by decreasing the temperature. As shown in Fig. 2 (d), several fibers are growing out from flake plates, which indicate the initiation of fibrous growth. Furthermore, another considerable matter in this issue was constitutional supercooling similar to the role of Sb on the refinement of eutectic Si in Al-Si alloys [18]. With the addition of 2.0 wt. % Gd-Sb into the composite, the T$_2$Mg$_2$Si and eutectic formation time increased to 590.2°C and 14 s, respectively. The morphology of 2.0 wt. % Gd–Sb (Figs. 1 and 2) depicted that the eutectic structure was coarse as compared to that of the 1.5 wt. % Gd–Sb treated composite. This clearly revealed that the 1.5 wt. % Gd–Sb was the optimum refinement effect of eutectic Mg$_2$Si phase. It was observed that although the difference in T$_C$ of eutectic Mg$_2$Si for all composites was only ±2°C, the results provided information on the level of refinement effect in the respective eutectic Mg$_2$Si phase.

Another analysis for detecting the modification level of eutectic phase was through depression growth temperature, T$_C$Mg$_2$Si [16,17]. Considering T$_C$Mg$_2$Si, $\Delta$T$_C$Mg$_2$Si was the dif-

Fig. 6 – Cooling curves and associated first and second derivatives for the Al–15%Mg$_2$Si composite with different Gd–Sb contents: (a) 0, (b) 0.5, (c) 1.0, (d) 1.5 and (e) 2.0 wt. %.
Fig. 7 – Change of nucleation temperature ($T_N$) and growth temperature ($T_G$) of primary Mg$_2$Si phase corresponding to various Gd–Sb additions.

Fig. 8 – Duration of Mg$_2$Si particles to nucleate and grow ($t_G^{Mg_2Si} - t_N^{Mg_2Si}$) as a function of Gd–Sb addition.

Fig. 9 – (a) Decrease in eutectic nucleation $T_N^{Mg_2Si}$ and (b) growth $T_G^{Mg_2Si}$ temperature and (c) corresponding increases of depression eutectic temperature, $\Delta T_G^{Mg_2Si}$.

From this relation, it can be found that the appropriate concentration level for refinement of eutectic Mg$_2$Si phase was 1.5 wt. % Gd–Sb, in which the value of $\Delta T_G^{Mg_2Si}$ was around 2.1 °C.

Fig. 10 – (a) SEM micrograph of Al–15%Mg$_2$Si composite modified with 1.5 wt. % Gd–Sb and (b) corresponding elemental mapping indicating Mg–Sb compound.
involved mechanism would be discussed later. Nonetheless, the concentration of Mg and Sb elements in the nuclei was considerable. As a result, it was proposed that Mg-Sb compound behaved as a heterogeneous nucleation substrate for primary Mg$_2$Si particles in the modified composite. Similarly, it was stated that in Al-Si alloys and Al-Mg$_2$Si composites treated with Sb addition, the Mg$_2$Sb$_2$ intermetallic compound was formed, which acts as nuclei for eutectic silicon and primary Mg$_2$Si crystals [18,19]. Based on the EDS result in Fig. 10, the Gd element was not detected inside the nuclei, which confirmed that there was no chemical interaction between Gd with Mg and/or Sb to make a compound as the nuclei of primary Mg$_2$Si particles. However, it was shown that the development of nucleation of primary Mg$_2$Si phase in Al melt was due to the formation of Al$_2$Gd compound, which behaved as a heterogeneous nucleation site for primary Mg$_2$Si [20]. Therefore, in addition to Mg$_2$Sb$_2$ compound, Al$_2$Gd particles contributed in the refinement of primary Mg$_2$Si particles. As displayed in Fig. 7, with the addition of Gd-Sb into the composite melt, the nucleation temperature ($T_N$) of primary Mg$_2$Si phase increased as compared to the unmodified composite. In accordance with the classical nucleation theory, the required energy for heterogeneous nucleation was less than homogeneous nucleation, and as a result, smaller undercooling degree was needed [15]. Nucleation of primary Mg$_2$Si with decrease in undercooling degree obviously approved the existence of heterogeneous nuclei in the modified composite melt, which was consistent with the SEM/EDS result in Fig. 10. Therefore, during solidification of the composites, the formation of Mg-Sb compound (Mg$_2$Sb$_2$) in the melt, as well as Al$_2$Gd, could provide efficient nucleation substrates for primary Mg$_2$Si at higher temperature. Therefore, the number of nuclei increased, which increased the density of primary Mg$_2$Si particles (Fig. 3(a)) and consequently decreased in primary Mg$_2$Si particle size (Fig. 3(a)).

3.4. Mechanisms of refinement/modification of Gd-Sb addition

3.4.1. Heterogeneous nucleation

Fig. 10(a) shows the BSE micrograph of Al–15%Mg$_2$Si treated with 1.5 wt. % Gd-Sb. Small fine particles (white color), showed by arrows were detected inside primary Mg$_2$Si particles. Elemental mapping of this phase (nuclei) showed that Sb and Mg elements were detected inside the nuclei, while there was no signal from Al atoms detected from the nuclei (Fig. 10(b)). In addition, there was no concentration of Gd element in the nuclei, representing that Gd was likely to embed into the primary Mg$_2$Si particles rather than existing in the nuclei. The

![Fig. 11](image-url) - (a) BSE micrograph of Al–15%Mg$_2$Si-1.5 wt. % Gd–Sb composite and (b) EDS line scan of the Mg$_2$Si particle in (a).
was dependent on the minimum total surface energy. Furthermore, according to the competition mechanism, introducing of modifier elements changed the surface anisotropy of some significant crystals facets, which led to a transition of crystal morphology [23].

The SEM images and corresponding EBSD inverse pole figure (IPF) of Al–15%Mg2Si composite in unmodified and modified with 1.0 wt. % Gd–Sb are shown in Fig. 12 (a–d). As observed in the unmodified composite, the crystallographic planes of polyhedral primary Mg2Si are symmetrical to {111} planes (Fig. 12 (a)). According to EBSD inverse pole figure of the selected primary Mg2Si in Fig. 12 (b), the intensity of {111} facets were much more than {001} and {101} crystal planes, meaning that the crystal morphology was polyhedral. As reported in the previous study [12], Gd atoms were absorbed on {100} facets of primary Mg2Si during its growth manner, the growth of {100} facets was hindered, and expanded the facets. Therefore, faster growth of {111} facets of the primary Mg2Si that occurred during solidification altered the final morphology of primary Mg2Si into octahedral shape. Similarly, Fig. 12 (c) illustrates that with the addition of 1.0 wt. % Gd–Sb to the composite melt, the morphology of primary Mg2Si has altered in truncated octahedral shape without holes (growth defects). Accordingly, as depicted in Fig. 12 (d), the EBSD inverse pole figure (IPF) showed that the intensity of both {100} and {111} crystalline planes was concentrated, which indicated that the formation of primary Mg2Si with truncated octahedral morphology with both {111} and {100} planes. However, perfect truncated octahedral primary Mg2Si crystal with large {100} facets as crystal planes, as compared to {111}, was not formed, which was probably due to an insufficient concentration of Gd and Sb atoms in the melt. In fact, with the addition of Gd–Sb into the composite melt, the Sb atoms preferentially replaced the Si position of the primary Mg2Si crystal which promoted the exposure of {111} facets in order to minimize the total surface energy [24]. Furthermore, it was found that preferential absorption of the Sb element on {100} retarded the growth rate along <100> direction and formed the truncated octahedral outline of primary Mg2Si crystal [25]. Besides, based on
the kinetic point of view, the geometric shape of the crystal was determined by the velocity of the growth along the \( <100> \) and \( <111> \) directions, i.e., \( R = V_{<100}> / V_{<111>} \), where \( V_{<100>} \) and \( V_{<111>} \) were the growth rates along the \( <100> \) and \( <111> \) directions, respectively [26]. Therefore, the crystal will grow into a perfect octahedron if \( R = \sqrt{3} \). The crystal will grow into a truncated octahedron if \( \sqrt{3} < R < \sqrt{3} \). However, a perfect cubic primary \( \text{Mg}_2\text{Si} \) could be achieved if \( R = \sqrt{3} \).

In general, the growth rate of main growth facets of the crystal was changed by the selective absorption of impurities atoms, which resulted in the alteration of growth habit [27]. Therefore, it was proposed that with combined addition of 1.5 wt. % Gd–Sb into the composite melt, both Gd and Sb atoms were absorbed preferentially on the \( <100> \) facets of primary \( \text{Mg}_2\text{Si} \) crystal and restrained the growth velocity along with the \( <100> \) direction. With the absorption of Gd and Sb on the \( <100> \) faces, the \( <100> \) surface growth of the primary \( \text{Mg}_2\text{Si} \) crystals was inhibited, resulting in a reduced \( V_{<100>} \) and finally a perfect truncated octahedral primary \( \text{Mg}_2\text{Si} \) was obtained (Fig. 13(f)). Therefore, it could be observed that Gd–Sb addition had influenced the morphology of primary \( \text{Mg}_2\text{Si} \) particles. The truncated octahedral \( \text{Mg}_2\text{Si} \) crystals were formed when \( V_{<100>} \) fell to a value in the range of \( \sqrt{3} < R < \sqrt{3} \). Therefore, the refinement/modification mechanisms of \( \text{Mg}_2\text{Si} \) as a result of Gd–Sb addition could be classified as heterogeneous nucleation, restricted growth theory, including adsorption and poisoning.

Due to different undercooling and composition segregation during the solidification of the alloy, various morphologies of primary \( \text{Mg}_2\text{Si} \) crystals could be achieved in the same sample. As a result, observing the various particles obtained from the same sample can speculate the growth process of primary \( \text{Mg}_2\text{Si} \). Fig. 13 (a–f) depict the typical morphologies of primary \( \text{Mg}_2\text{Si} \) crystal extracted from the Al–15%\( \text{Mg}_2\text{Si} \)–1.5 wt. % (Gd–Sb) composite at different growth stages from the cast iron mold. As clearly observed, a typical frame style was the growth manner of primary \( \text{Mg}_2\text{Si} \) namely truncated octahedral frame of \( \text{Mg}_2\text{Si} \), which formed preferentially.
Subsequently, the growth and perfection of primary \( \text{Mg}_2\text{Si} \) proceeded continuously by enriching the existing frame with Si and Mg atoms. With the proceeding of the growth, Si and Mg atoms gradually filled the frame and produced smaller hollows and finally with the complete formation of \{111\} planes, the hollows disappeared. Besides, the \{100\} facets were gradually exposed with the absorption of Gd and Sb atoms on them, which led to the formation of perfect truncated octahedral \( \text{Mg}_2\text{Si} \) crystals with six \{100\} planes and eight \{111\} planes.

To achieve the flat truncated octahedral primary \( \text{Mg}_2\text{Si} \) and observe its growth process, the cooling rate was reduced to decrease the growth rate of primary \( \text{Mg}_2\text{Si} \) by pouring the melt into a copper mold preheated at 200°C. The growth stage of flat truncated octahedral primary \( \text{Mg}_2\text{Si} \) in Al–15%\( \text{Mg}_2\text{Si} \) composite modified with 1.5 wt. % \text{Gd–Sb} was presented in Fig. 14 (a–f). In the early stage of growth, the primary \( \text{Mg}_2\text{Si} \) existed in an equiaxed-dendrite with four branches (Fig. 14 (a)), then the four branches join together and the two restricted directions begin to grow (Fig. 14 (b)). Due to the slow cooling rate along the restricted direction, it gradually connected with the first four branches, in which flat truncated octahedral outline with hollow and gaps were formed (Fig. 14 (d)). Finally, disappearing of the gaps and hollows resulted in the formation of flat truncated octahedral primary \( \text{Mg}_2\text{Si} \) (Fig. 14 (f)). Therefore, a skeleton type growth of flat truncated octahedral primary \( \text{Mg}_2\text{Si} \) was observed during its growth process.

Fig. 15 (a–c) shows the SEM micrograph and corresponding EBSD map and pole figure of Al–15%\( \text{Mg}_2\text{Si} \) composite modified with 1.5 wt. % \text{Gd–Sb}. As seen, the distribution of \( \text{Mg}_2\text{Si} \) particles was almost along the \{001\} crystallographic planes with a high intensity of 23.02 MUD. It indicated that the Gd and Sb atoms absorbed preferentially on the \{100\} facets of primary \( \text{Mg}_2\text{Si} \) (Fig. 15 (b, c)). Once the content of Gd–Sb increased to 1.5 wt. %, more Gd and Sb atoms were absorbed on the \{100\} facets of primary \( \text{Mg}_2\text{Si} \) and bigger \{100\} facets were achieved. With serious restriction on the growth rate along the \{001\} direction, the flat truncated octahedral primary \( \text{Mg}_2\text{Si} \) particle was achieved (Figs. 14 (f) and 15(b)).

To indicate the role of Gd–Sb addition on the refinement of eutectic \( \text{Mg}_2\text{Si} \) grains, Fig. 16 (a–d) depicts the SEM micrograph and corresponding EBSD analysis of eutectic \( \text{Mg}_2\text{Si} \) in 1.5 wt. % Gd–Sb modified Al–15%\( \text{Mg}_2\text{Si} \) composites solidified in
a preheated copper mould. As seen in Fig. 16 (a) the microstructures with large eutectic cells (grains) consist of fine eutectic Mg$_2$Si crystals with fibre-like morphology are embedded in the continuous eutectic α-Al matrix. Consequently, Fig. 16 (b) shows small size eutectic grains with different crystal orientations (different colors), in which this result is consistent with the results of Figs. 1 and 3(b). Furthermore, Fig. 16 (c) shows high-angle grain boundaries in misorientation angles of the composite sample which elucidate the refinement of eutectic Mg$_2$Si crystals due to the grain refining effect of Gd-Sb addition.

In Al–Si alloys, different mechanisms were responsible for refinement/modification of eutectic Si [28–30] in which the addition of Sr and Na caused nucleation of eutectic Si in the melt. Accordingly, the impurity induced twinning (ITT) mechanism [29] which was based on the layer growth model and the restricted TPRE growth mechanism [31] led to the growth of eutectic Si, in which both mechanisms were well accepted. It was reported that there existed great similarities between Mg$_2$Si and eutectic Si in terms of properties and solidification behavior [32]. Therefore, it can be suggested that the refinement/modification of eutectic Mg$_2$Si crystals due to Gd–Sb addition can be associated with the aforementioned mechanisms, and Gd/Sb atoms/compounds are believed to cause such refinement effect. However, more research is required to find out the exact modification mechanism(s) of eutectic Mg$_2$Si with chemical modification. From the obtained results, it was evident that 1.5 wt. % Gd–Sb was the best concentration to induce proper refinement/modification effect on both primary and eutectic Mg$_2$Si particles in Al–15%Mg$_2$Si composite. Indeed, the addition of Gd–Sb into the composite melt caused the absorption of Gd and Sb atoms/compounds into the growth steps of Mg$_2$Si crystals, which enriched Mg$_2$Si growing steps with Gd/Sb layer, resulted in surface energy change of the crystals and suppressed the preferred growth manners of Mg$_2$Si crystals. Therefore, it caused difficulties for the external Mg and Si atoms to continue diffusing into Mg$_2$Si. Nonetheless, exceeding the concentration of Gd–Sb to 2.0 wt. % caused the reverse effect by increasing the size of primary Mg$_2$Si and eutectic Mg$_2$Si cells due to over modification mechanism, while the shape of primary and eutectic Mg$_2$Si was preserved as polygonal and rod-like, as shown in Figs. 1 (e) and 2 (f), respectively. In fact, increasing the concentration of Gd–Sb addition by 2.0 wt. % to increase of the volume fraction of Gd (IMCs) by consuming more Gd atoms, resulted in insufficient Gd atoms in the melt and lowered the diffusion of Si and Mg atoms into the Mg$_2$Si growth front. Therefore, Mg$_2$Si particles coarsened again. Furthermore, the growth restrictive role of Gd (IMCs) decreased by their formation at grain boundaries and interdendritic regions as a network since they cannot restrict the

Fig. 15 – (a) SEM secondary image, (b) EBSD map and (c) corresponding EBSD pole figure of Al–15%Mg$_2$Si composite modified with 1.5 wt. % Gd–Sb along {111}, {001} and {101} crystal planes.
growth of Mg$_2$Si particles which caused the Mg$_2$Si particles to be coarsened. Furthermore, according to the diffusion and phase transition theory, the coarsening rate of the precipitate particles is associated with the solute atom concentrations. Coarsening of the particles is higher when the concentration of solute atoms is high. By increasing the Sb content, the Mg$_3$Sb$_2$ compound can aggregate and grow, which results in decreasing the heterogeneity of nuclear-point. Therefore, the nuclear rate is affected and enlarges the Mg$_2$Si particle size.

To indicate the effect of combined addition of 1.5 wt. % Gd–Sb on refinement/modification of Mg$_2$Si particles in Al–15%Mg$_2$Si composite as compared to separate addition of Gd or Sb, 0.75 wt. % Gd and 0.75 wt. % Sb additions were separately added into the composite melt in which the resulting micrographs of Al–15%Mg$_2$Si composite and corresponding deep etch images of primary and eutectic Mg$_2$Si particles are tabulated in Fig. 17. Accordingly, Fig. 18 (a) depicts the primary Mg$_2$Si and eutectic Mg$_2$Si cell sizes. As observed in Fig. 17, with addition of 0.75 wt. % Sb the morphology of primary and eutectic Mg$_2$Si particles changed from dendritic and flake-like structure into truncated octahedral and rod-like morphologies, respectively. With the addition of 0.75 wt. % Gd, a similar transformation of morphologies occurred for primary and eutectic Mg$_2$Si particles. However, as observed in Fig. 17 (h), when the composite was treated with 0.75 wt. % Gd the expansion of {100} facets of primary Mg$_2$Si was more than {100} facets when 0.75 wt. % Sb was added into the composite, which specified the effective role of Gd to produce more perfect truncated octahedral primary Mg$_2$Si particles. The perfect truncated octahedral primary Mg$_2$Si and fiber-like eutectic Mg$_2$Si crystals were achieved when the composite was treated with 1.5 wt. % Gd–Sb (Fig. 17 (k, l)) with smallest particle/cell sizes (Fig. 18 (a)). Furthermore, Fig. 18 (b) shows that with the addition of 0.75 wt. % of Sb or Gd, the duration of Mg$_2$Si particles to nucleate and grow decreased which established the refinement of primary and eutectic Mg$_2$Si particles as compared to unmodified composite and the most decrease in the duration of Mg$_2$Si particles to nucleate and grow (refinement of Mg$_2$Si particles) occurs when the composite is treated with 1.5 wt. % Gd–Sb.

3.5. Effect of Gd–Sb addition on tensile properties

The typical stress-strain curve of Al–15%Mg$_2$Si composites with various compositions is illustrated in Fig. 19 (a, c). Accord-
Fig. 17 – Optical micrographs of Al–15%Mg2Si composite with corresponding deep etch SEM images of primary and eutectic Mg2Si crystal in (a–c) unmodified and modified with (d–j) 0.75 wt. % Sb, (e–i) 0.75 wt. % Gd and (j–l) 1.5 wt. % Gd–Sb.

ingly, variation trends of the ultimate tensile strength (UTS) and percentage elongation (El %) values are shown in Fig. 19 (b–e). As observed in Fig. 19 (a), with increasing Gd–Sb content from 0 to 1.5 wt. %, the tensile properties of the composite increases. In comparison, between the strength results in Fig. 19 (b) and microstructure in Fig. 1, it is obvious that refinement of primary Mg2Si particles has a substantial effect on the tensile strength in which the UTS values increases from 204.79 MPa in the unmodified composite to 242.73 MPa in the composite modified with 1.5 wt. % Gd–Sb, approximately 18% increment. In fact, lessening of the negative influence of coarse dendrite morphology of primary Mg2Si with big size on tensile properties can be achieved after the introduction of Gd–Sb additions into the composite melt, which led to refinement of crystallite and decreasing in crystal size. By introducing Gd–Sb addition, not only the size of primary Mg2Si particles decreased but also the aggregation of crystallite was hindered. As a result, the study of the relationship between the crystal size and tensile strength with various Gd–Sb contents can reveal the refined-grain strengthening on Mg2Si phase in Al–15%Mg2Si composite. Based on dislocation theory, the relation between tensile strength and crystal size can be analyzed [33]. The essence of plastic deformation is the ultimate stress to be overcome, which leads to a mass of moving dislocation. Nonetheless, dislocation glides are hindered by grain boundary and even dislocation pile-up occurs. When the content of dislocation pile-up increased to a certain value, it will be a driving force for dislocation glide [34]. When the crystal size is large, there is a high content of dislocation pile-up; consequently, a bigger driving force for dislocation glide. Therefore, the composites with smaller crystal size display the high strength. Meanwhile, the number of crystal boundary increases with small crystal size (Fig. 3) which hinders dislocation motion. As a result, the crystal size is based on the ultimate stress for the plastic deformation to be formed. However, there was a reduction in UTS to 231 MPa when the Gd–Sb addition increased to 2.0 wt. % which is due to the increase in the size of primary Mg2Si particles. Fig. 19 (b) illustrates the percentage elongation of Al–15%Mg2Si composites with various contents of Gd–Sb additions as well. As seen, the elongation values increase from 2.65 in unmodified composite to 3.90 as a maximum value in 1.5 wt. % Gd–Sb modified Al–15%Mg2Si composite, which is about 1.5 times higher than unmodified composite and then decreased to 3.55 within the composite treated with 2.0 wt. % Gd–Sb. As presented in Fig. 2, with the Gd–Sb addition of up to 1.5 wt. %, the flake-like morphology of eutectic particles were altered to fiber-like. Therefore, difficulties occur for the propagation of

cracks through the refined primary and eutectic Mg$_2$Si particles. Moreover, enhancement of the ductility compared to unmodified composite can be attributed to the existence of Gd (IMCs) in 1.5 wt. % Gd–Sb modified composite since these IMCs act as the obstacle to the crack propagation. However, according to Fig. 19 (b), increasing the Gd–Sb addition by more than 1.5 wt. %, the elongation to fracture values of the composite is decreased. The reduction in the elongation to failure value of the fabricated composite is due to the existence of brittle Gd-rich IMCs in the matrix matrix with a high-volume fraction (Fig. 4 (d)), which induce low ductility in comparison with the composite modified with 1.5 wt. % Gd–Sb. Another reason for decreasing ductility of Al–15%Mg$_2$Si composite treated with 2.0 wt. % Gd–Sb is that with the addition of more Sb, high and brittle hard-phase Mg$_3$Sb$_2$ is formed at the grain boundaries and causes segregation of Mg$_2$Si at the grain boundaries to decrease the plasticity of Gd–Sb modified composite. Furthermore, Fig. 19 (c, d, e) depict that the composite treated with 1.5 wt. % Gd–Sb has the highest tensile properties in terms of UTS and El (%) compared to composites added with single additions of 0.75 wt. % Gd or Sb in which this enhancement is attributed to the finer size and enhanced morphologies of primary and eutectic Mg$_2$Si particles.

4. Conclusions

The influence of combined addition of Gd–Sb on microstructural characteristics, solidification behavior and tensile properties of Al–15%Mg$_2$Si composite were examined. The following conclusions can be drawn:

1. The simultaneous addition of 1.5 wt. % Gd–Sb exhibits apparent refining and modifying effects on both primary and eutectic Mg$_2$Si particles in Al–15%Mg$_2$Si composite in which the morphology of primary Mg$_2$Si is altered from coarse dendrite to perfect truncated octahedral with a decrease in the average size and aspect ratio by 70% and 12%, respectively, with an increase in density of about 1.0%. In addition, the morphology of eutectic Mg$_2$Si changed from plate-like to fibrous-like with a reduction in eutectic cell size of about 61%.

2. The mechanism of refinement and modification of primary Mg$_2$Si particles is a synergistic effect of the simultaneous addition of Gd and Sb, in which Mg$_3$Sb$_2$ phase is formed in the composite melt and behaves as heterogeneous nuclei of primary Mg$_2$Si crystals, leading to a decrease in the undercooling degree. Therefore, the number of nuclei increased, which obviously leads to refinement of primary Mg$_2$Si particles. Moreover, Gd and Sb elements could promote the growth of primary Mg$_2$Si by absorbing on the (100) facets, which govern the final morphology of primary Mg$_2$Si crystals as perfect truncated octahedral.

3. Transformation of eutectic Mg$_2$Si crystal from plate to fibrous morphology is synergistically affected by the combined addition of Gd and Sb by controlling the growth of eutectic Mg$_2$Si, which was aligned with the reduction in
growth temperature obtained from the cooling curves. In addition, the formation of Gd (IMCs) in the composite structure has an indispensable role in the refinement of primary and eutectic Mg$_2$Si by restricting the growth of the Mg$_2$Si phases.

4 Primary and eutectic Mg$_2$Si crystals modification by simultaneous addition of Gd–Sb resulted in the enhancement of tensile properties of Al–15%Mg$_2$Si composite, which is crucial in the fabrication of light-weight composites with high strength and toughness.

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