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RESEARCH ARTICLE

ASSESSING MICROSTRUCTURE AND MECHANICAL BEHAVIOUR OF Al-15% Mg_2Si IN-SITU COMPOSITE WITH CONCOMINENT ADDITION OF PRASEODYMIUM AND ANTIMONY

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Abstract. Aluminium alloys offer a high strength-to-weight ratio, excellent corrosion and oxidation resistance and significant energy absorption during collision, improving vehicle safety. To strengthen the performance of aluminium-cast alloys, aluminium matrix composites with Mg_2Si reinforcement have been developed. They are consistently promising materials for industries like aerospace and automotive due to their higher specific strength. However, the large size and dendritic morphology of primary Mg_2Si occur in the structure of the unmodified Al-Mg_2Si alloy during solidification, causing the mechanical characteristics of the alloy to deteriorate. This study aims to modify and refine the primary Mg_2Si phase with Pr-Sb simultaneous alloying with varying weight percentages. Hence, the microstructure and mechanical properties were examined with the Al-15% Mg_2Si with the simultaneous addition of Pr and Sb. Optical microscopy and SEM/EDS were used to study microstructure. The region with the smallest average particle size exhibited the maximum density of the Mg_2Si phase, the greatest tensile strength (108.01 MPa vs 84.61 MPa) with elongation (9.42% versus 5.68%) and a hardness value of 58.50 HV was achieved by simultaneously introducing 0.5 wt.% Pr-Sb to the composite compared to the base Al-15% Mg_2Si composite. It was proposed that modification of primary Mg_2Si particles was mainly derived from the formation of Mg_3Sb_2 compounds as heterogeneous nuclei for primary Mg_2Si. Meanwhile, the absorption and poisoning of Pr and Sb can suppress the preferred growth of primary Mg_2Si crystals along <100> direction. Hence, the results showed that the simultaneous addition of Pr and Sb improved the microstructure and mechanical properties of the Al-15% Mg_2Si composite by making the primary Mg_2Si particles finer.

Keywords: Mg_2Si particles, microstructure, refinement, hardness, tensile properties.

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1. INTRODUCTION

Since aluminium matrix composites offer exceptional qualities, including high specific strength, good wear, low density, and low corrosion rate, they are always a potential material for use in the automotive and aerospace sectors. Among these, the Al-Mg_2Si in-situ composite stands out as a novel metal matrix composite with diverse applications in various engineering fields. This is attributed to the abrasive nature of the reinforcement (e.g., Mg_2Si) due to 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_2Si and Mg–Mg_2Si composites used in an automobile's ultralight design [1-2]. However, the specific areas where failure will occur are concentrated stress points, such as needle-shaped eutectic Mg_2Si particles, sharp edges of primary Mg_2Si particles, and narrow sections of elongated and bulky Mg_2Si particles [1]. The composite is susceptible to brittle fracture due to the uneven precipitation of coarse primary Mg_2Si particles. As manufacturing technology advances, enhancing the comprehensive characteristics of Al-Mg_2Si composites to meet high-performance requirements becomes essential. Hence, in order to alter the main Mg_2Si particles in the Al-Mg_2Si composite, many studies have been conducted to alter the microstructure and enhance the comprehensive properties of Al-Mg_2Si composites, through the addition of trace elements including Li [3], Be [4], B [5], et al., a rare-earth element such as Er [1], Y [6], Ce [7], et al., a master alloy such as B_4C [8], by changing the heat treatment et al. [9] and different preparation processes such as equal-channel angular pressing [10], hot extrusion [11], superheat and electromagnetic stirring [12].

The major goal of the investigations is to enhance the mechanical characteristics of the Al-15% Mg_2Si composite by altering the size of the primary Mg_2Si particles. Limited information exists about the combined addition of elements on Mg_2Si particle modification capability. Among the addition elements, Sb has been widely utilized as an active element in modifying Mg_2Si particles [4]. Therefore, combining Sb and another element further refined primary and eutectic Mg_2Si particles. Currently, the effect of the combined addition of elements, such as Li–Sb [13], Ca–Sb [14], Sr–Sb [15] and Gd-Sb [16] on the refinement/modification of primary Mg_2Si is investigated, whereby with the addition of Sb, the Mg_3Sb_2 phase is formed, which serves as a heterogeneous nucleation substrate for primary Mg_2Si particles. Furthermore, the growth steps of primary Mg_2Si are suppressed along the <100> direction due to the absorption and poisoning effect of Ca and Li elements [13-14]. However, due to the reaction tendency between two additional elements, new nucleant particles are formed, such as Li_2Sb, $CaSb_2$ and $Sr_{11}Sb_{10}$, in the melt or even fading in modification occurs because the elements consume themselves. Therefore, playing multiple roles in the refinement of primary Mg_2Si 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 achieving the multiple modifying effects. To reach this purpose, the elements with small electronegativity differences that are unlikely to react with each other can be selected because the formation of compounds between these elements can be avoided [7]. 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 [8]. In addition, the formation of compounds between transition metal and Sb usually occurs at relatively higher temperatures than alkaline-earth metals [9]. Therefore, the reaction between two addition elements might be avoided or weakened if a transition metal element such as Pr combines with Sb.

According to the literature, little research has examined the impact of simultaneously adding modifier Sb and Pr rare earth elements (REEs) to the Al-Mg_2Si composite at varying weight percentages. This study aimed to investigate the combined effect of Pr-Sb on the hardness and tensile properties of the Al-15%Mg_2Si composite. This was done by comparing it with a prior study that used REE and Sb [13,16].

2. MATERIALS AND METHODS

The 99.9% pure aluminium ingot, the 99.9% pure magnesium ingot, and the 98.9% pure silicon ingot were used to make the Al-Mg_2Si ingot. Pure aluminium (85 wt.%), pure magnesium (9.5 wt.%), and pure silicon (5.5 wt.%) were used in the production of the Al-15% Mg_2Si ingot. Initially, the graphite crucible, which held the pure aluminium and silicon ingots, was subjected to a temperature of 800 °C in the induction furnace, causing it to melt. Prior to introducing the magnesium into the molten substance, the temperature was reduced to 750 °C to prevent oxidation. 0.25 wt.% Sb and 0.25 wt.% of Pr rare earth elements were added to the molten Al-15% Mg_2Si at a temperature of 750 ±10 °C while stirring slowly. Then, it was cooled after being placed into the heated tensile permanent mould. The proportion of Pr and Sb added to the Al-Mg_2Si composite was based on earlier experiments that simultaneously included Sb and other rare earth elements such as Gd and Y [16-17].

Table 1: The composition of the Al-15% Mg_2Si composite modified by including various Pr-Sb additions

% of Additive	As-cast 1	As-cast 2	As-cast 3	As-cast 4	As-cast 5
Pr-Sb (wt.%)	0	0.5	1.0	1.5	2.0
Pr (wt.%)	0	0.25	0.5	0.75	1.0
Sb (wt. %)	0	0.25	0.5	0.75	1.0

After being cleaned in an ultrasonic bath, the sample was ground using SiC papers ranging in grade from 240 to 4500 grits. After that, alumina and a 0.5µm colloidal silica agent were used to polish the sample until its surface resembled a mirror. The composite samples were etched with 2% HF acid. The material was then examined with a Nikon Midophoto-FXL optical microscope. The main Mg_2Si phase shape in the composite was observed at a magnification of 200x. The software ToupView 3.7 was employed to analyse the morphology of the Mg_2Si phase by computing the particles' average size, density, and average aspect ratio. The cylindrical dog-bone-shaped sample for the tensile test was fabricated by precisely cutting the component, which was formed through in-situ casting, to the specific dimensions specified in accordance with ASTM B577-10 (2010) criteria. The tensile strength of the sample was assessed at room temperature using an Instron Universal Mechanical Testing equipment (5982) equipped with a strain gauge extensometer. The test utilised a strain rate of 0.1 mm/min. The test was carried out for hardness measurement using Vicker's hardness machine (Matsuzawa DVK-2) with an applied force of 5 N within 10 s loading time and 50 mm/s loading speed.

3. RESULTS AND DISCUSSION

3.1 Microstructure Analysis

Figure 1 displays the optical micrographs of the cast Al-15%Mg_2Si composites with varying weight percentages of Pr-Sb (0, 0.5, 1.0, 1.5, and 2.0 wt.%). Figure 1(a) illustrates three large, dark grey particles: whitish-grey particles, α-aluminium, and the primary Mg_2Si, encompassed by the fibrous eutectic Mg_2Si, also referred to as secondary Mg_2Si. In unaltered Al-Mg_2Si, the architecture of primary Mg_2Si was in accordance with other studies; it displayed a coarse dendritic structure with a hole in the middle [18]. The impact of simultaneously adding Pr-Sb at various weight percentages ranged from 0 to 2.0 wt.%, as shown in Figure 1(a) to (e). The microstructure of the unaltered Al-Mg_2Si demonstrated the coarse dendritic form of the original Mg_2Si particles. The initial Mg_2Si particles may be reshaped into a considerably smaller, spherical morphology with just 0.5 wt.% of Pr-Sb added. The addition of 0.5 wt.%Pr-Sb resulted in a reduction in the number of sharp edges present in the main Mg_2Si particles, which were distributed more evenly than in the unmodified Al-Mg_2Si composite. When 1.0 wt.% of Pr-Sb was added, the over-modification problem manifested. Primary Mg_2Si exhibited an increase in average particle size and subsequent coarsening. The mechanical characteristics of the composite deteriorated with each increase in primary Mg_2Si particle size.

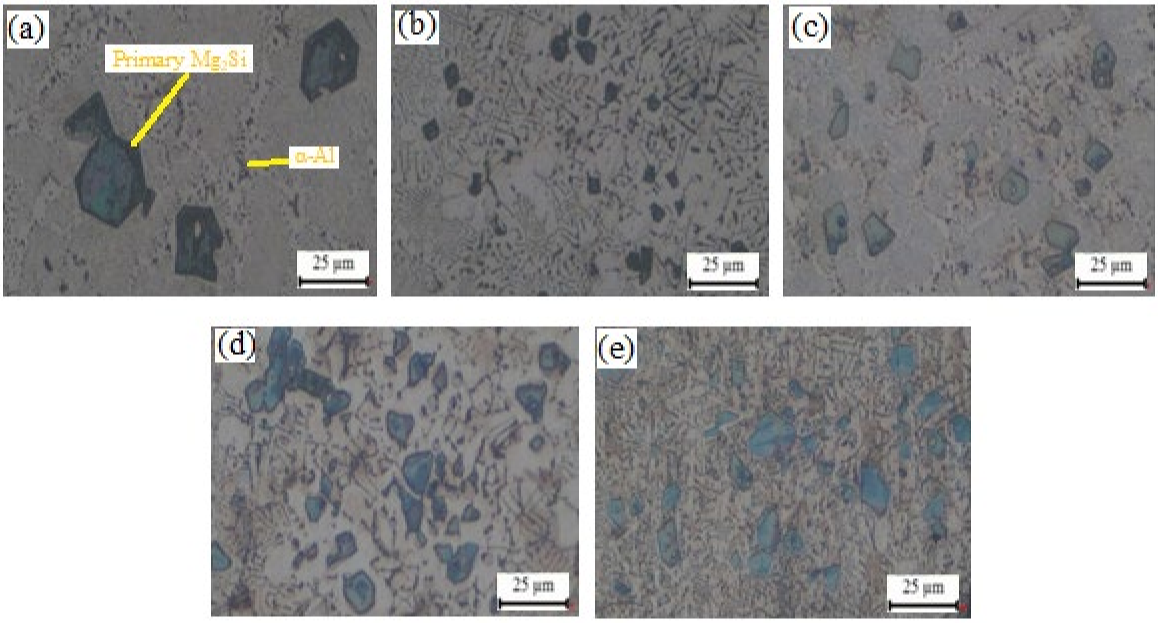

Figure 1: Optical microstructure of Al-15% Mg_2Si with Pr-Sb contents: (a) Pr-Sb 0 wt.%, (b) Pr-Sb 0.5 wt.%, (c) Pr-Sb 1.0 wt.%, (d) Pr-Sb 1.5 wt.%, and (e) Pr-Sb 2.0 wt.%.

The SEM observation in Figure 2 revealed that adding 0.5 wt.% Pr-Sb to the Al-15% Mg_2Si composite resulted in the lowest primary Mg_2Si particle size compared to other Pr-Sb additions. The polygonal form of the basic Mg_2Si particle replaced its previous polyhedral shape, perhaps reducing the concentration of stress on the particle's sharp edges.

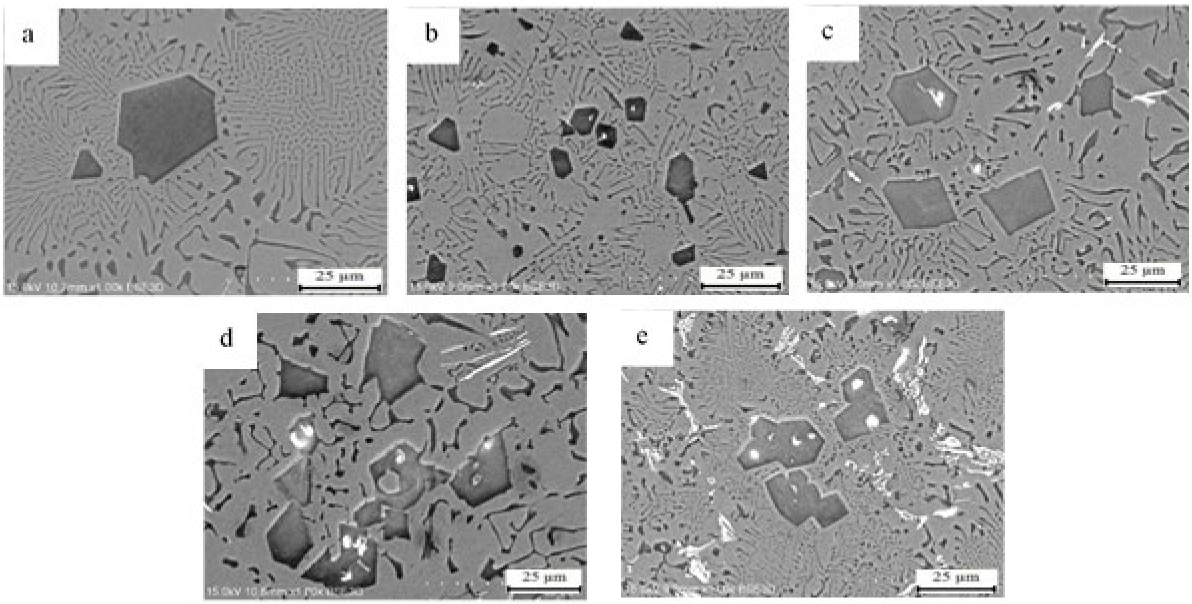

Figure 2: SEM-BSE microstructure of Al-15% Mg_2Si with Pr-Sb contents: (a) Pr-Sb 0 wt.%, (b) Pr-Sb 0.5 wt.%, (c) Pr-Sb 1.0 wt.%, (d) Pr-Sb 1.5 wt.%, and (e) Pr-Sb 2.0 wt.%.

3.2 SEM/EDS Analysis

The SEM microstructure and the point analysis EDS spectra are displayed in Figure 3. The main Mg_2Si was observed as a dark black polygonal form surrounded by α-aluminium in the matrix, as shown by the SEM image. Figure 3 shows energy dispersive spectrometry (EDS) of white particles. According to Figure 3, BSE elemental mapping, the distribution of Pr and Sb particles was homogenous both within and along the matrix of the primary Mg_2Si particles. In addition, a new phase has been found inside the primary Mg_2Si particles. It was round and white. The EDS examination revealed that Al, Pr, and Sb proportions in the primary Mg_2Si particles have changed, forming a new phase within.

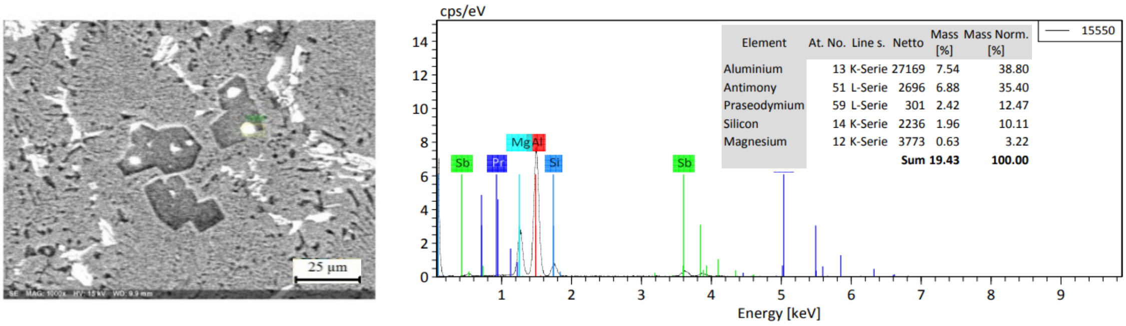

Figure 3: Energy dispersive spectrometry (EDS) of white particles

3.3 Size Analysis of the Primary Mg_2Si Particles

The mean dimensions of primary Mg_2Si alters with varying Pr-Sb wt.%, as displayed in Figure 4. When 0.5 wt.% of Pr-Sb was used as additives, the average size of the main Mg_2Si particles decreased significantly from 186.03 to 44.79 µm. Adding 0.5 wt.% of Pr-Sb resulted in the biggest percentage decrease 75.92% when compared to other Pr-Sb additions. Perhaps because of over-modification, the average size of main Mg_2Si in reinforcement samples (1.0, 1.5, and 2.0 wt.%) exhibited increments. The addition of Gd-Sb to the Al-15% Mg_2Si alloy has resulted in a significant reduction in the average size of primary Mg_2Si particles, from 40 to 12 µm, representing a 70% decrease [16]. Hence, the incorporation of Pr-Sb had a significant impact on decreasing the size of primary Mg_2Si particles in the Al-15% Mg_2Si composite.

By introducing a refining agent of Pr, the equilibrium solidification and growth process of the corresponding phase may be altered. This was achieved by the modification of the surface energy of the liquid melt [16]. Sb could be collected on the interface between the melt and the Mg_2Si, which limited the formation of primary Mg_2Si when Sb is added [19]. The reduction in the size of primary Mg_2Si in the composite may be significantly influenced by the addition of two refining agents and one modifying agent. The size of primary Mg_2Si could be decreased by a negligible quantity of Pr and Sb.

When 0.5 wt.% of Pr-Sb was added to the Al-15% Mg_2Si composite, the aspect ratio of primary Mg_2Si decreased from 0.80 to 0.78, representing a reduction of about 2.5%. By including 0.5 wt.% of Pr-Sb, the morphology of the primary Mg_2Si underwent a substantial transformation, transitioning from a coarse dendritic structure to a much smaller and spherical shape. Prior studies have shown that adding Sb and rare earth elements at the same time will have a synergistic effect on refining and changing the primary Mg_2Si. Research indicated that rare earth elements can impede the development of crystals by accumulating at the boundary between solid and liquid phases. This hinders the growth mechanism of primary Mg_2Si particles and leads to the creation of an intermetallic compound in the region surrounding the grain boundaries. The equilibrium form of the particle is determined by the minimal total surface energy, which is based on the Gibbs-Curie Wulff theorem and competition mechanism

[17]. A change in the surface anisotropy of some crystal facets might result in a change in the crystal's morphology by altering the Sb element [18].

The addition of 0.5 wt.% of Pr-Sb to the Al-15% Mg_2Si resulted in an increase in the density of primary Mg_2Si particles from 69 to 370 particles/mm^2, as depicted in Figure 4. By increasing the weight percentage of Pr-Sb from 0.5 to 2.0 wt.%, the average density of primary Mg_2Si particles decreased from 370 to 324 particles/mm^2. The rise in the synthesis of large Pr intermetallic compounds might be responsible for the decrease in the density of primary Mg_2Si particles. Furthermore, the combination of Mg and Sb resulted in the formation of a complex compound called Mg-Sb (Mg_3Sb_2), while the combination of Al and Pr formed a compound known as Al_3Pr. When present in the molten state at elevated temperatures, these compounds act as a highly effective surface for forming primary Mg_2Si particles. Producing these compounds will increase the density and number of nuclei in initial Mg_2Si particles with reduced particle sizes [16].

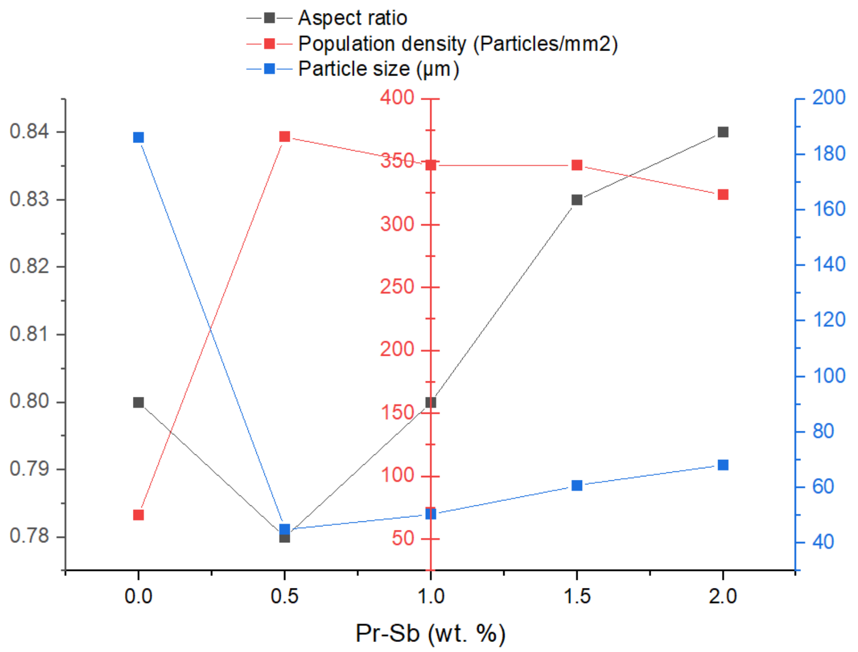

Figure 4: Geometric aspects of primary Mg_2Si particles in Al-15% Mg_2Si composite with different Pr-Sb wt.%.

3.4 Tensile Properties

Figure 5 demonstrates that the inclusion of 0.5 wt.% of Pr-Sb in the Al-15% Mg_2Si alloy resulted in a generally favourable outcome, characterised by increased ductility and maximum ultimate tensile strength (UTS). The addition of Pr-Sb to the composite resulted in an increase in the ultimate tensile strength (UTS) of the Al-15% Mg_2Si alloy from 84.61 to 108.01 MPa.

The addition of Pr-Sb to the Al-15% Mg_2Si alloy resulted in a significant improvement of around 27.66% in ultimate tensile strength, as compared to previous research that employed Gd-Sb as an additive. This demonstrated a notable impact. Prior research indicated that the utilisation of Gd-Sb to strengthened Al-15% Mg_2Si yielded a modest 18% rise in ultimate tensile strength (UTS) [16]. The addition of 0.5 wt.% Pr-Sb resulted in a significant increase in the elongation at maximum stress of the unmodified Al-15% Mg_2Si composite. Specifically, the elongation increased from 5.68 to 9.42%, 65.84% higher than the unmodified composite. By increasing the maximum tensile strength of the Mg_2Si particle, the addition of 0.5 wt.%Pr-Sb can decrease the size of the primary particle. A decrease in the size of the primary Mg_2Si particles could lead to an increase in the number of grain boundaries, which could account for the observed enhancement in the tensile strength of the composite material. The dislocation theory offers an elucidation for the enhancement of the material's tensile strength. The presence of large crystal sizes in the Al-15%Mg_2Si alloy led to the accumulation of dislocations and their movement along the crystal lattice. Utilising Pr-Sb inhibited the aggregation of crystallite and decreased the size of the primary Mg_2Si particles [18, 20]. Hence, the addition of 0.5 wt.% Pr-Sb to the Al-15% Mg_2Si alloy augmented the number of grain boundaries characterised by smaller crystal sizes and impeded the movement of dislocations.

Figure 5 shows a reduction in ultimate tensile strength (UTS) when Al-15% Mg_2Si alloy was reinforced with 2.0 wt.% Pr-Sb. The elongation of Al-15% Mg_2Si-2.0% Pr-Sb decreased from 9.42 to 4.2%, while the ultimate tensile strength (UTS) decreased from 108.01 to 93.05 MPa. The UTS value of the modified composite might decrease due to excessive modification caused by the addition of 2.0 wt.% Pr-Sb. Figure 5 demonstrates the role of the Pr intermetallic compound in acting as a barrier to hinder dislocation and achieve enhanced ductility. However, excessive alteration led to the formation of a significant amount of fragile Pr IMCs, decreasing the ductility of the composite. Increased amounts of Sb led to the creation of a rigid and fragile phase of Mg_3Sb_2 at the interfaces between grains. This phase caused the separation of Mg_2Si and decreased the ability of the Pr-Sb-modified composite to deform plastically [15-16].

Based on the stress-strain curve trends shown in Figure 5, adding only 0.5wt.% of Pr-Sb is necessary to enhance the mechanical properties of Al-15% Mg_2Si. Previous research determined that the optimal amount of Gd-Sb to be added was 1.5 wt.%. Thus, by utilising less amount of Pr-Sb, the mechanical properties of the Al-15% Mg_2Si composite could be enhanced. This finding indicates the strong co-modification effect of Pr and Sb on the structure and mechanical properties of primary Mg_2Si particles compared to Gd and Sb in the Al-15 Mg_2Si composite.

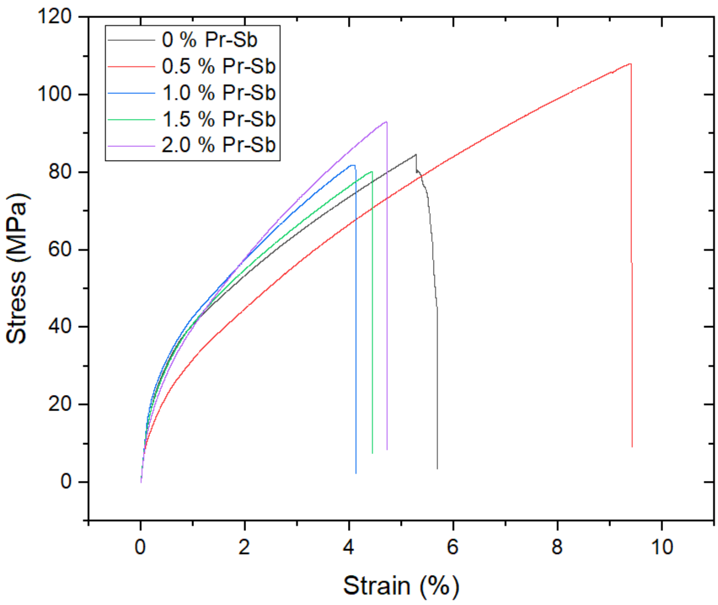

Figure 5: Stress-Strain Curve.

3.5 Hardness Property

The value of hardness of the Al-15% Mg_2Si composite may be efficiently increased by adding Pr-Sb, as Figure 6 illustrates. The unaltered Al-15% Mg_2Si composite's hardness value rose from 54.07

to 65.48 Hv upon the addition of 2.0 wt.% Pr-Sb. The rise in the composite's hardness value was around 21.10%, as seen in the graph in Figure 6. The production of Pr intermetallic compound may cause the composite's hardness value to increase. The amount of Pr intermetallic compound formed was increased as the weight % of Pr increased. Pr intermetallic compound has hard qualities that can raise the composite's hardness value [18].

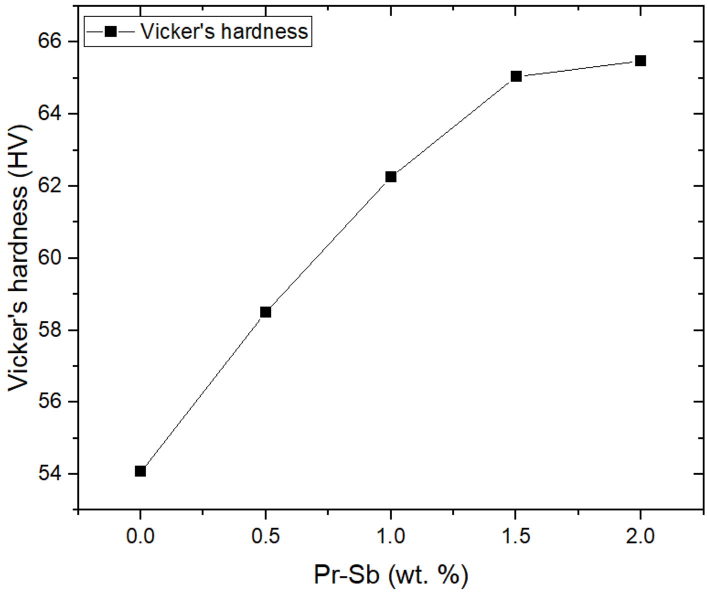

Figure 6: Vicker's hardness of Al-15%Mg_2Si composite with different Pr-Sb wt.%.

4. CONCLUSIONS

The conclusions of this research are summarized below:

- The inclusion of 0.5 wt.% of Pr-Sb to Al-15% Mg_2Si composite resulted in the transformation of the large and irregular dendritic structure of the original Mg_2Si particles into smaller and spherical shapes. The incorporation of 0.5 wt.% Pr-Sb enhanced the refinement and modification of the coarse dendritic structure of the primary Mg_2Si in the unaltered Al-15%Mg_2Si composite, forming particles with reduced sharp edges. The morphology of the primary Mg_2Si particles transformed from a coarse dendritic structure to a polygonal shape, accompanied by a 2.5% rise in density and a significant 75.92% reduction in average size and aspect ratio.
- The addition of merely 0.5 wt.% Pr-Sb to the in-situ composite increased its tensile strength, demonstrating an improvement in the mechanical characteristics of the material. Tensile strength increased by around 27.66% from 84.61 to 108.01 MPa, and elongation increased from 5.68 to 9.42%, 65.84% higher than the unmodified Al-15%Mg_2Si composite. Due to the formation of a harder intermetallic compound containing Pr, the hardness value of the composite was raised from 54.07 Hv to 65.48 Hv.

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Author Contributions

All authors contributed toward data analysis, drafting and critically revising the paper and agree to be accountable for all aspects of the work.

Disclosure of Conflict of Interest

The authors have no disclosures to declare.

Compliance with Ethical Standards

The work is compliant with ethical standards.

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