



Communication Microstructure Examination and Sliding Wear Behavior of Al-15%Mg₂Si-xGd In Situ Composites before and after Hot Extrusion

Hamidreza Ghandvar¹, Mostafa Abbas Jabbar², Abdollah Bahador³, Tuty Asma Abu Bakar^{1,*}, Nor Akmal Fadil¹ and Katsuyoshi Kondoh³

- ¹ Department of Materials, Manufacturing and Industrial Engineering, School of Mechanical Engineering, Faculty of Engineering, Universiti Teknologi Malaysia (UTM), Johor Bahru 81310, Malaysia; ghandvar@utm.my (H.G.); norakmal@utm.my (N.A.F.)
- ² Department of Mechanical Techniques, Al-Nasiriya Technical Institute, Southern Technical University, Al-Nasiriya 64001, Thi-Qar, Iraq; m.jabbar@stu.edu.iq
- ³ JWRI, Osaka University, 11-1 Mihogaoka, Ibaraki 567-0047, Osaka, Japan; abdollah@jwri.osaka-u.ac.jp (A.B.); kondoh@jwri.osaka-u.ac.jp (K.K.)
- * Correspondence: tuty@utm.my

Abstract: In current study; the effect of various Gadolinium (Gd) additions on the microstructure and sliding wear behaviour of Al-15%Mg₂Si composite before and after the hot extrusion process was examined. Optical microscopy (OM), scanning electron microscopy (SEM) equipped with EDX facility and X-ray diffraction (XRD) were used to characterize the microstructure. The results showed that with addition of 1.0 wt.% Gd to Al-15%Mg₂Si composite, the primary Mg₂Si particles size reduced from 44 μ m to 23 μ m and its morphology altered from dendritic to polygonal shape. Further refinement of primary Mg₂Si particles was achieved after conducting hot extrusion which resulted in a decrease in its size to 19 μ m with a transfer to near-spherical morphology. The Vickers hardness value increased from 55.6 HV in the as-cast and unmodified composite to 72.9 HV in the extruded 1.0% Gd modified composite. The wear test results revealed that composites treated with Gd possess higher wear resistance in comparison with those of without Gd. The highest wear resistance obtained with the lowest wear rates of 0.19 mm³/km and 0.14 mm³/km in the Al-15%Mg₂Si-1.0% Gd before and after the hot extrusion, respectively. The high wear resistance of extruded Gd-modified Al-15%Mg₂Si composite is due to the refinement of primary Mg₂Si particles with uniform distribution in the composite matrix along with fragmentation of Gd intermetallic compounds.

Keywords: Al composites; Mg₂Si; Gd addition; modification; mechanical properties; wear behavior

1. Introduction

Al–Mg–Si alloys are extensively utilized in the aerospace and automotive applications owing to their high strength to weight ratio, good formability and favorable cast ability [1]. It is claimed that the excellent features of the Al–Mg–Si alloy comes from Mg₂Si particles due to its attractive features which made the Al-Mg₂Si system a replacement for Al–Si alloys [2]. Nevertheless, coarse dendritic primary Mg₂Si particles with sharp corners are formed under low solidification rates of Al-Mg₂Si composites, which could affect the properties of the fabricated composites and constrict their usage in industrial applications [3]. Therefore, modification of primary Mg₂Si particles in Al-Mg₂Si composites is essential to induce adequate mechanical and tribological properties. Palta et al., [4] reported that with the addition of Cu to Al-12Si-20Mg alloy, the size and volume of the Mg₂Si particle, formed within the matrix, decreased which can be due to the increasing the number of nuclei or by poisoning the surface of the in-situ Mg₂Si nuclei owing to the segregation of Cu at the liquid–solid interface. Furthermore, the addition of Cu results in formation of CuAl₂ and CuMg₅-Si₄Al₄ intermetallics within the matrix which caused improvement of the wear



Citation: Ghandvar, H.; Jabbar, M.A.; Bahador, A.; Abu Bakar, T.A.; Fadil, N.A.; Kondoh, K. Microstructure Examination and Sliding Wear Behavior of Al-15%Mg₂Si-xGd In Situ Composites before and after Hot Extrusion. *Lubricants* **2022**, *10*, 3. https://doi.org/10.3390/ lubricants10010003

Received: 8 September 2021 Accepted: 14 December 2021 Published: 24 December 2021

Publisher's Note: MDPI stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.



Copyright: © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). resistance. In another study, Wu el al., [5] found that by increasing the Bi content to 4 wt.%, the morphology of primary Mg₂Si changes from irregular or dendritic to polyhedral shape with decreasing in its average size significantly which results in enhancement of wear and friction properties of the composite. The reason for the refinement of Mg₂Si crystals by the addition of Bi may be related to the composition supercooling caused by the segregation of Bi at the front of the solid/liquid interface of Mg₂Si during solidification. Farahany et al., [6] claimed that addition of 0.01 wt.% Sr to Al-20%Mg₂Si-2%Cu composite results in the refinement of primary Mg₂Si particles as well as improvement of mechanical and wear behaviour of the Sr-treated composite compared to the untreated one. It is suggested that the refinement of primary Mg₂Si particles as a result of Sr addition can be attributed to the formation of heterogeneous nucleation substrates for primary Mg₂Si particles. Other reasons for enhancement of wear resistance of the composite are good distribution of the fine Mg₂Si particles in the matrix as well as presence of Al₂Si₂Sr intermetallic compound. It has been stated that addition of rare earth elements to Al-Mg₂Si bring about significant influence of refinement of primary Mg₂Si particles and as a consequence the mechanical and tribological properties are enhanced. Wu et al., [7] found that with the addition of 0.5 wt.% Nd addition to Al-18%Mg₂Si composite, the dendritic morphology of primary Mg₂Si changed to polyhedral shape with a decrease in its average size as well as enhancement in wear properties. In fact, Nd segregates at the front of the solid/liquid interface of Mg₂Si during solidification and caused composition supercooling as a responsible mechanism for refinement of Mg₂Si particles. In another study Jafari Nodooshan et al., [8] claimed that addition of 0.5 wt.% Y element as well as conducting a heat treatment process resulted in improvement in hardness and wear resistance of the Al-15%Mg₂Si composite. The formation of Al_2Y intermetallics in the composite matrix hindered the growth of primary Mg₂Si particles through restriction of their growth. In addition, Soltani et al., [9] reported that increasing the extrusion ratio leads to considerable enhancement in hardness and wear resistance of Al-15%Mg₂Si composite. The improvement was related to the role of hot extrusion on refinement and better distribution of primary Mg₂Si particles as compared to the as-cast composite. Although there are some studies in the literature about the influence of rare earth additions and hot extrusion on microstructure and wear behaviour of Al-Mg₂Si composites, there is limitation on combined effect of rare earth element and hot extrusion on microstructure alteration and tribological behaviour of the Al-Mg₂Si composites. Hence, in the present study the effect of various contents of Gd additions and the hot extrusion process on microstructural and sliding wear behaviour of Al-15%Mg₂Si composite is investigated.

2. Experimental Procedures

2.1. Materials Fabrication

Industrially pure Al (99.8%), Mg (99.9%), and Si (99.5%) were utilized to fabricate an ingot of Al-15%Mg₂Si composite. The chemical composition of the fabricated ingot is shown in Table 1. The procedure of Al-Mg₂Si composite preparation is reported elsewhere [10]. An induction furnace was used to melt the composite ingot which was placed inside a graphite crucible with 1 kg capacity. When the temperature of the melt reached 750 °C, 0.3 wt. % Gd was introduced into the melt and then the melt was degassed with tablets containing C_2Cl_6 for 3 min. Once the dross was skimmed the composite melt was poured into a preheated cast iron mold (110 °C). A similar approach was repeated for addition of 0.5 wt. %, 1.0 wt. % and 2.0 wt.% Gd.

Table 1. Al-15%Mg₂Si primary ingot: chemical composition.

Element	Al	Mg	Si	Fe	V	Cr	Ni	Ti	Sn	Mn
Weight%	84.6	8.5	6.7	0.12	0.02	0.01	0.01	0.01	0.01	0.01

2.2. Hot Extrusion

The composite samples with different content of Gd were machined to achieve the dimension of 30 and 29 in length and diameter respectively. The homogenisation treatment was then applied on the samples using an electrical furnace at 520 °C for 4 h followed by furnace cooling prior to hot extrusion. The hot extrusion process was carried out at constant parameters of ram speed (1 mm/s) and extrusion ratio (12:1) at 450 °C using graphite-based oil as lubrication.

2.3. Microstructural Examination

For microstructural observation, the grinding and polishing procedures were conducted on the composite samples using SiC papers and colloidal silica suspension, respectively. To detect the microstructure of Al-15%Mg₂Si composites and perform quantitative analysis on primary Mg₂Si particles size, an optical microscope (Olympus BX60) equipped with an i-Solution image analyzer was utilized, in which the quantitative metallographic characteristic of the microstructure such as mean size was calculated using Equation (1) as follows:

Mean size
$$= \frac{1}{m} \sum_{j=1}^{m} \left(\frac{1}{n} \sum_{i=1}^{n} L_i \right)_i$$
(1)

where Li is the size, n is the number of Mg_2Si particles measured in a single field (0.60126 mm²), and m is the number of the evaluated fields. In addition, the composite samples were inspected using scanning electron microscopy (SEM) (Philips XL40) coupled with energy-dispersive spectroscopy (EDS). To identify the phase formation, X-ray diffraction (XRD) (Siemens-D500) with Cu Ka line produced at 40 kV and 35mA was utilized.

2.4. Hardness and Dry Sliding Wear Test

The hardness of the Al-15%Mg₂Si composite samples comprising different content of Gd before and after hot extrusion was measured by Vickers's hardness machine (Matsuzawa DVK-2). Before conducting the experiment, a standard sample was utilized for machine calibration. A square diamond pyramid as an indenter was used for the Vickers hardness test. In each hardness test, and the applied parameters were a 5 kg loading force and 10 s test time. For conducting the dry sliding wear test of the fabricated composite, a pin on disk wear apparatus was used (Ducom, TR20-LE). The as-cast and extruded samples were machined to produce the wear pins with dimensions of 6 mm in diameter and 25 mm in length. The pins with two different applied loads of 20 N and 40 N were loaded against a steel disk (EN31, hardness = 60 HRC). The dry sliding wear test was conducted at room temperature under a fixed rotating speed of 200 rpm, sliding distance of 2000 m, wear track diameter of 60 mm and sliding time of 1 h. The counter disk open surfaces as well as pin surface were cleaned with acetone before every test. The cumulative wear in µm and frictional force in Newtons were calculated and measured during the sliding wear tests.

3. Results and Discussion

3.1. Microstructural Characterization

Figure 1 depicts the optical images of Al-15%Mg₂Si composites with various Gd concentrations before and after hot extrusion, in which the primary Mg₂Si particles size is depicted in Figure 2. As seen in Figure 1(1a), in the unmodified composite, the primary Mg₂Si particles possess dendritic and irregular morphology which are surrounded with eutectic Mg₂Si cells. In addition, as can be seen there is a hole at the centre of some of the primary Mg₂Si particles which is filled with α -Al as a result of nucleation of α -Al on Mg₂Si particles [2]. When the composite was treated with 0.3 wt.% Gd, the shape of primary Mg₂Si altered to polygon, and reduced in size as illustrated in Figure 1(1b) and Figure 2 compared to unmodified composite. With the addition of 0.5 wt.% Gd the particles changed into a regular polygonal morphology with a further reduction in size, as revealed in Figure 1(1c) and Figure 2. Addition of 1.0 wt.% of Gd displays the most refined primary Mg₂Si particles,

as depicted in Figure 1(1d) and Figure 2. Nonetheless, with an increase of Gd content to 2.0 wt.% the particles started to coarsen as demonstrated in Figure 1(1e) and Figure 2 which can be due to the over-modification phenomenon [11]. A similar behaviour can be detected in the size of primary Mg₂Si articles in unmodified and Gd modified composites after conducting a hot extrusion process as seen in Figure 1(2a–2e) and Figure 2. The decrease in the size of primary Mg₂Si particles after hot extrusion can be attributed to the mechanical fragmentation of Mg₂Si particles caused by hot extrusion [12].



Figure 1. Optical micrographs of Al-15%Mg₂Si composite at different Gd additions: (**1a**,**2a**) 0 wt.%, (**1b**,**2b**) 0.3 wt.%, (**1c**,**2c**) 0.5 wt.%, (**1d**,**2d**) 1.0 wt.% and (**1e**,**2e**) 2.0 wt.% for before (left) and after (right) hot extrusion.



Figure 2. The primary Mg₂Si particles average size in Al-15%Mg₂Si-xGd composite before and after hot extrusion.

Figure 3a–d illustrates the BSE micrographs of the Al-15%Mg₂Si composites modified with various Gd additions (0.3–2 wt.%). As seen, addition of Gd to the composite results in the formation of new phases with different morphologies in the composite matrix particularly at higher level of Gd. Since the Al-15%Mg₂Si modified with 2.0 wt.% Gd possesses the highest volume fraction of these new phases, in order to distinguish these phases the XRD pattern of unmodified and 2.0 wt.% Gd modified is depicted in Figure 4. The XRD patterns illustrate some new peaks related to AlSiGd, Al₂Si₂Gd and MgGd intermetallic compounds (IMCs).



Figure 3. Back-scattered electron (BSE) micrographs of Al-15 wt.% Mg₂Si composites with different Gd additions: (**a**) 0.3 wt.%, (**b**) 0.5 wt.%, (**c**) 1.0 wt.% and (**d**) 2.0 wt.%.



Figure 4. X-ray diffraction (XRD) patterns of unmodified and 2.0 wt.% Gd modified Al-15%Mg₂Si composite.

There are three different Gd phases in the Al-15%Mg₂Si-2%Gd composite which possess bulky, needle-like and Chinese-script-like morphologies which are shown in Figure 5a–c. The corresponding EDS results are tabulated in Table 2. The EDS analysis on bulky and needle-like phases in Figure 5a,b reveals the various atomic % of Al, Si and Gd correspond to Al₂Si₂Gd and AlSiGd IMCs, respectively. Nevertheless, the EDS result on the Gd phase in Figure 5c which has a Chinese-script structure depicts the composition of MgGd. It is apparent that with addition of Gd, new phase with various morphologies form in the composites microstructure and distributed at the interdendritic regions or grain boundaries. It is expected that the morphologies of Gd IMCs influence the mechanical and tribological properties of the Al-15%Mg₂Si composite; hence, assessment of the Gd IMCs morphologies in Figure 3a–d should be taken into account. As observed in Figure 3a,b, addition of 0.3 wt. % and 0.5 wt.% Gd results in the formation of bulky (Al₂Si₂Gd) and needle-like (AlSiGd) morphology of Gd IMCs. Further addition of Gd to 1.0 wt.% Gd causes formation of script-type structure (Al₂Si₂Gd) as illustrated in Figure 3c. However, when Al-15%Mg₂Si composite is modified with 2.0 wt.% Gd, the size of Gd IMCs has further increased leading to the formation of twin needle-like IMCs in addition to a bulky shape and a Chinese script type (MgGd) as shown in Figure 3d.

Table 2. Atomic ratio for Al, Si, Mg and Gd on 10 different bulky, needle-like and Chinese script-like phases obtained from energy-dispersive spectroscopy (EDS) analysis.

(a) Non Uniform White Phase				(b)	White Nee	dle-like Ph	ase	(c) Chinese Script-like Phase				
Al	Si	Gd	Mg	Al	Si	Gd	Mg	Al	Si	Gd	Mg	
41.65	43.12	12.95	3.76	50.06	52.98	45.36	0.88	6.45	3.10	42.20	46.50	
39.54	43.74	13.52	2.54	60.37	52.50	54.93	2.00	5.36	4.81	35.82	39.71	
37.07	40.52	20.01	4.88	46.03	47.76	40.93	1.01	7.01	6.52	34.02	38.58	
38.17	41.02	20.81	1.98	57.98	46.70	49.51	0.53	3.55	2.09	39.79	38.21	
38.10	43.42	18.48	5.20	50.41	51.14	52.47	0.50	8.22	6.31	39.11	37.88	
38.43	39.20	20.62	3.25	58.92	56.14	48.95	1.97	4.16	3.40	40.93	41.64	
38.47	40.80	20.73	4.41	76.50	62.75	55.22	1.78	6.39	3.55	38.20	41.48	
44.48	37.09	15.88	2.87	54.01	41.29	48.33	2.03	3.75	1.66	42.10	39.99	
37.57	41.13	18.33	5.48	66.24	54.54	55.39	2.45	5.01	4.08	38.32	40.05	
41.48	38.11	17.08	2.06	72.80	68.62	65.61	1.65	3.98	2.79	40.93	39.47	
Al ₂ Si ₂ Gd					AlS	iGd		MgGd				



Figure 5. BSE micrograph of the Al-15%Mg₂Si-2.0%Gd composite, presenting distribution of new phases in the Al matrix: (**a**) bulky shape, (**b**) needle-like shape and (**c**) Chinese script structure of Gd IMCs.

Figure 6a,b illustrates the BSE micrograph of Al-15%Mg₂Si-1.0% Gd in as-cast and after extrusion, respectively. As observed, the Gd IMCs are fragmented throughout the composite matrix after extrusion. The higher magnification of Gd IMCs and corresponding elemental mapping is depicted in Figure 6c which clearly illustrates the role of hot extrusion in significantly altering the size and morphology of Gd IMCs in comparison with the as-cast composite.

3.2. Mechanical Properties

Figure 7 illustrates the Vickers hardness property of the Al-15%Mg₂Si composite modified with various content of Gd. As seen in Figure 7, the hardness value of the composites increased as a result of Gd addition as compared to unmodified composite. The improvement can be attributed to the modification of Mg₂Si phase and better distribution of particles that could withstand stress positively. The hardness value of the as-cast materials increased from 55.6 HV in unmodified composite to 56.00 HV when 0.3 wt.% Gd was added. The hardness value further increased to 67.80 HV and 68.6 HV when the composite is added with 0.5 wt.% Gd and 1.0 wt.% Gd, respectively. The similar trend is observed for extruded composites. In addition, conducting the hot extrusion process caused increasing hardness value compared to as-cast composites, in which the hardness values for extruded unmodified composite, 0.3 wt.% Gd, 0.5 wt.% Gd and 1.0 wt.% Gd were 60.4 HV, 61.8 HV, 69.38 and 72.9 HV, respectively, showing that 1.0 wt.% Gd possesses the highest value due to further refinement of Mg₂Si particles, better particle distribution in the composite, reduction of porosity content, as well as fragmentation of Gd IMCs after a hot extrusion process (Figures 1 and 6). In fact, the presence of hard primary Mg₂Si particles and Gd IMC particles act as a barrier against the dislocation motion which

caused higher resistance to indentation [13]. However, the hardness value was found to decrease when the Gd concentrations exceeded the 1.0 wt.%. In fact, with the addition of 2.0 wt.% Gd into the composite, hardness value reduced to 65.9 HV and 67.8 HV for as-cast and extruded composite, respectively. This phenomenon happened due to the over-modification of Gd addition whereas the formation of primary Mg₂Si particles per unit area (density) decreased; hence, the number of barriers against dislocation motion and consequent resistance to indentation is decreased which results in hardness value lessening. A similar behaviour was observed by [14] whereas the Na addition exceeded 1.0 wt.% in Al-Mg₂Si composite.



Figure 6. BSE image of Al-15%Mg₂Si-1.0% Gd composite: in as cast (**a**), after hot extrusion (**b**) and corresponding elemental mapping of hot-extruded composite displaying broken Gd intermetallic compounds (IMCs) (**c**).



Figure 7. Vickers hardness values of Al-15%Mg₂Si-xGd composite for before and after hot extrusion.

3.3. Wear Properties

Figure 8a,b shows the wear rates of Al-15%Mg₂Si-xGd composites before and after hot extrusion process under the loads of 20 and 40 N, respectively. As observed, for the applied load of 20 N, with the addition of Gd up to 1.0 wt.% the wear resistance of the composite increased with decreasing wear rate from 0.34 mm³/km in unmodified composite to 0.19 mm³/km in the 1.0 wt.% Gd modified Al-15%Mg₂Si composite. The enhancement in the wear resistance can be due to the role of Gd addition on refinement/modification of primary Mg₂Si particles (Figures 1 and 2) as well as formation of Gd-IMCs (Figure 3). In fact, refinement of Mg₂Si particles and formation of Gd IMCs lead to increasing the hardness of the composites (Figure 7) which in turn caused the reduction in the composites wear rate. Indeed, with increasing the hardness of the composite the depth of penetration caused by hardened steel disk asperities govern the results in higher wear resistance [15]. Further improvement of the wear resistance of Al-15%Mg2Si composites with lower wear rate was obtained after performing a hot extrusion process compared to the as-cast composites as observed in Figure 8a,b irrespective of the effect of Gd addition. This was because the extrusion process has a substantial effect on the reduction of Mg₂Si particles size (Figure 2), porosity content as well as breakage of Gd IMCs in the matrix (Figure 6) which in turn results in higher hardness. It was reported that hot extrusion not only promoted a decrease of average size but also caused a better distribution of the fine Mg_2Si particles [9]. Indeed, the fragmented primary Mg₂Si particles and Gd IMCs had a considerable influence in reducing the extent of materials removal by supporting the contact stresses through lessening high plastic deformation and abrasion between the pin and disc [16]. However, as observed in Figure 8 with further addition of Gd to 2.0 wt.%, the wear rate increased for both as-cast and extruded composites, which was due to the coarsening of primary Mg_2Si particles (Figures 1 and 2) and decreasing of the hardness value (Figure 7) due to over-modification phenomenon. Another reason for increasing the wear rate of as-cast and extruded composite modified with 2.0 wt.% Gd was the formation of a high fraction of brittle Gd IMCs in the composite matrix (Figure 3d) which own low load-supporting ability during the wear test. The similar result is reported by [7] in the Al-18%Mg₂Si treated with Nd addition. Thus, combination of 1.0 wt.% Gd addition and the hot extrusion process promotes the lowest wear rate, 0.14 mm³·km⁻¹ in the Al-15%Mg₂Si composite under applied load of 20 N. Another indicative feature in Figure 8b was that with increasing the

loads from 20 N to 40 N the wear rate was increased for all samples. In fact in the higher applied load the generated stress on the wear surface was greater and caused an increase in the number of contact asperities, which resulted in faster wearing of the material surface.



Figure 8. Variation of wear rates with the varying contents of Gd before and after hot extrusion under different applied loads of 20 N (**a**) and 40 N (**b**).

Figure 9a,b illustrates the friction coefficient (μ) of the Al-15%Mg₂Si composites modified with different Gd additions before and after hot extrusion under applied loads of 20 N and 40 N, respectively. As observed, the relationship of the friction coefficient with Gd content is like those of the wear rates as shown in Figure 8, in which the coefficient friction decreased at first from 0.48 in the unmodified Al-15%Mg₂Si composite to 0.4 in the Al-15%Mg₂Si-1.0 wt.% Gd composite and then increased to 0.41 in the Al-15%Mg₂Si-2.0% Gd composite under applied load of 20 N (Figure 9a). The coefficient friction further decreased to 0.38 after conducting hot extrusion. The decrease in the friction coefficient of the Al-Mg₂Si-1.0% Gd composite compared to the unmodified one was due to the presence of fine primary Mg₂Si particles and fragmented Gd IMCs with large ability of load-supporting during the sliding wear test to decrease the touching area between the pin and disc. Nevertheless, when the Gd content exceeded 1.0 wt.%, the friction coefficient of the composites increased which was due to the existence of large primary Mg₂Si particles and brittle Gd-IMCs in the composite matrix. Similar behaviour is reported in mischmetal (MM) treated Al-7.0 wt.% Si-0.3 wt.% Mg alloy [17]. In addition, Figure 9b illustrates that increasing the load from 20 N to 40 N did not induce considerable alteration in the friction coefficient value. However, under the applied load of 40 N the friction coefficient was slightly higher.

The wear behaviour and its corresponding mechanism (s) can be revealed by examination of the worn surfaces of the fabricated composites under SEM. Figure 10a–f shows the SEM images of worn surfaces of the Al-15%Mg₂Si-x Gd (x = 0, 1.0 and 2.0 wt.%) composites before and after hot extrusion process tested at a load of 20 N. As seen in Figure 10a, the worn surface of unmodified Al-15%Mg₂Si composite shows deep and wide plowing grooves parallel to the motion direction. This demonstrates that a combination of abrasion wear and delamination wear appear to be the main wear mechanism of the composite. When conducting the hot extrusion process, the groove become narrow and the pits shallower than those as-cast composites which results in alteration of the wear mechanism to abrasion and adhesive wear (Figure 10b). When the Al-15%Mg₂Si composite is modified with 1.0 wt.% Gd, the worn surface of the composite possesses a rather smooth appearance with grooves which is indicative of a single abrasion wear mechanism (Figure 10c). The grooves become narrower when the composite undergone a hot extrusion process, in which mild abrasion wear is the governing wear mechanism as can be observed on the worn surface as shown in Figure 10d. However, abrasion and pits appear again when increasing the content of Gd to 2.0 wt.% as illustrated in Figure 10e. Performing the hot extrusion on Al-15%Mg₂Si-2% Gd composite caused enhancement as it lessened the grooves and pits from the worn surface of the composite (Figure 10f). In fact, the presence of coarse primary Mg₂Si particles and brittle Gd IMCs in Al-15%Mg₂Si-2% Gd composite caused worsening of the worn surface compared to Al-15%Mg₂Si-1% Gd composite. During the wear test these hard particles fractured and were entrapped between the composite and counterface, acting as third body abraders which in turn damage the worn surface. As a result, the plowing grooves formed on the worn surface of the composite (Figure 10e) and results in reduction of wear resistance of the composite which is consistent with a higher wear rate and friction coefficient (Figures 8 and 9).



Figure 9. Variation of friction coefficient with varying content of Gd in Al-15%Mg₂Si composite before and after hot extrusion under 20 N (**a**) and 40 N (**b**) applied loads.

As discussed above, performing a hot extrusion process on the composite samples results in improvement of wear resistance by reducing the wear rate and friction coefficient compared to as-cast composites. Another reason for such an enhancement is the creation of a mechanically mixed layer (MML) on the worn surfaces which is clearly observed on the worn surfaces of the extruded composites with and without Gd addition (Figure 10b,d,f). This layer shields the underlying material from severe plastic deformation or brittle fracture but simultaneously is delaminated to from the wear particles and again restored by intermixing and transfer of components from both pin and disc. In this study, hard fragmented Gd intermetallic compounds plays an important role as initial structural components together with other elements from both pin and disc which are deteriorated and transformed into the MML. Therefore, formation of this stable and compacted layer controls the wear mechanism of the fabricated composites. Formation of the MML protective layer with a mixture of materials (Al, Si, Fe and Cr) (face and counterface) has been detected on the worn surface of A356-10%B₄C ex-situ composite [18].



Figure 10. Comparison of scanning electron microscopy (SEM) micrographs of Al-15%Mg₂Si composite treated with (**a**,**b**) 0 wt.% Gd, (**c**,**d**) 1.0 wt.% Gd, and (**e**,**f**) 2.0 wt.% Gd before (left) and after hot extrusion (right) under applied load of 20 N.

4. Conclusions

The following conclusions can be drawn based on the experimental work conducted:

- (1) For both as-cast and extruded composites, 1.0 wt.% Gd was found to be the best content to induce refinement/modification of primary Mg₂Si particles in Al-15%Mg₂Si composite. However, exceeding this amount has an adverse influence on the modification of primary Mg₂Si particles.
- (2) Addition of Gd element up to 1.0 wt.% to Al-15%Mg₂Si composite as well as conducting hot extrusion results in improvement of the hardness value by 31% as compared to the as-cast unmodified composite.
- (3) The wear properties including wear rate and coefficient of friction of as-cast Al-15%Mg₂Si composite modified with 1.0 wt.% Gd was found to be considerably higher than the unmodified composite due to refinement/modification of primary Mg₂Si particles and formation of Gd IMCs. Further improvement of the wear properties was achieved after conducting hot extrusion due to fragmentation of primary Mg₂Si particles and Gd IMCs.

(4) When the as-cast Al-15%Mg₂Si composite was modified with 1.0 wt.% Gd, the wear mechanism changed to abrasion and adhesion compared to abrasion and delamination in the as-cast unmodified composite. The wear mechanism changed to mild abrasion after performing the hot extrusion on Al-15%Mg₂Si-1.0% Gd composite.

Author Contributions: H.G., M.A.J. and A.B. methodology, formal analysis, investigation, writing original draft; H.G. and N.A.F. conceptualization, methodology, formal analysis, investigation, writing—original draft; T.A.A.B., K.K. validation, resources, supervision, writing—review and editing. All authors have read and agreed to the published version of the manuscript.

Funding: This research is financially supported by Universiti Teknologi Malaysia (UTM) under the TDR Grant using vote number 06G22.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Not applicable.

Conflicts of Interest: The authors declare that they have no known competing financial interest or personal relationship that could have appeared to influence the work reported in this paper.

References

- Palta, A.; Sun, Y.; Ahlatci, H. Effect of copper addition on wear and corrosion behaviours of Mg₂Si particle reinforced composites. *Mater. Des.* 2012, *36*, 451–458. [CrossRef]
- Li, C.; Liu, X.; Wu, Y. Refinement and modification performance of Al–P master alloy on primary Mg₂Si in Al–Mg–Si alloys. J. Alloys Compd. 2008, 465, 145–150. [CrossRef]
- Emamy, M.; Khorshidi, R.; Honarbakhsh-Raouf, A. The influence of pure Na on the microstructure and tensile properties of Al-Mg₂Si metal matrix composite. *Mater. Sci. Eng. A* 2011, 528, 4337–4342. [CrossRef]
- 4. Ghandvar, H.; Idris, M.H.; Abu Bakar, T.A.; Nafari, A.; Ahmad, N. Microstructural characterization, solidification characteristics and tensile properties of Al-15%Mg₂Si-x(Gd-Sb) in-situ composite. *J. Mater. Res. Technol.* **2020**, *9*, 3272–3291. [CrossRef]
- Ghandvar, H.; Idris, M.H.; Ahmad, N. Effect of hot extrusion on microstructural evolution and tensile properties of Al-15%Mg₂SixGd in-situ composites. *J. Alloys Compd.* 2018, 751, 370–390. [CrossRef]
- Jafari Nodooshan, H.R.; Liu, W.; Wu, G.; Bahrami, A.; Pech-Canul, M.I.; Emamy, M. Mechanical and Tribological Characterization of Al-Mg₂Si Composites After Yttrium Addition and Heat Treatment. J. Mater. Eng. Perform. 2014, 23, 1146–1156. [CrossRef]
- Lashgari, H.R.; Zangeneh, S.; Shahmir, H.; Saghafi, M.; Emamy, M. Heat treatment effect on the microstructure, tensile properties and dry sliding wear behavior of A356–10%B₄C cast composites. *Mater. Des.* 2010, *31*, 4414–4422. [CrossRef]
- Lu, L.; Lai, M.; Hoe, M. Formaton of nanocrystalline Mg₂Si and Mg₂Si dispersion strengthened Mg-Al alloy by mechanical alloying. *Nanostruct. Mater.* 1998, 10, 551–563. [CrossRef]
- Montoya-Dávila, M.; Pech-Canul, M. Effect of bi-and trimodal size distribution on the superficial hardness of Al/SiCp composites prepared by pressureless infiltration. *Powder Technol.* 2007, 176, 66–71. [CrossRef]
- 10. Farahany, S.; Ghandvar, H.; Bozorg, M.; Nordin, A.; Ourdjini, A.; Hamzah, E. Role of Sr on microstructure, mechanical properties, wear and corrosion behaviour of an Al–Mg₂Si–Cu in-situ composite. *Mater. Chem. Phys.* **2020**, *239*, 121954. [CrossRef]
- 11. Soltani, N.; Jafari Nodooshan, H.R.; Bahrami, A.; Pech-Canul, M.I.; Wencai, L.; Wu, G. Effect of hot extrusion on wear properties of Al–15 wt. % Mg₂Si in situ metal matrix composites. *Mater. Des.* **2014**, *53*, 774–781. [CrossRef]
- 12. Qin, Q.D.; Zhao, Y.G.; Zhou, W.; Cong, P.J. Effect of phosphorus on microstructure and growth manner of primary Mg₂Si crystal in Mg₂Si/Al composite. *Mater. Sci. Eng. A* **2007**, *44*, 186–191. [CrossRef]
- 13. Straffelini, G. Experimental Observations of Subsurface Damage and Oxidative Wear in Al-Based Metal-Matrix Composites. *Wear* **2000**, 245, 216–222. [CrossRef]
- 14. Vencl, A.; Bobic, I.; Arostegui, S.; Bobic, B.; Marinkovic, A.; Babic, M. Structural, mechanical and tribological properties of A356 aluminum alloy reinforced with Al₂O₃, SiC and SiC+ Graphite particles. *J. Alloys Compd.* **2010**, *506*, 631–639. [CrossRef]
- 15. Wang, K.Y.; Zhao, R.D.; Wu, F.F.; Wu, X.F.; Chen, M.H.; Xiang, J. Improving microstructure and mechanical properties of hypoeutectic Al-Mg₂Si alloy by Gd addition. *J. Alloys Compd.* **2020**, *813*, 152178. [CrossRef]
- Wu, X.F.; Zhang, G.G.; Wu, F.F. Microstructure and dry sliding wear behavior of cast Al–Mg₂Si in-situ metal matrix composite modified by Nd. *Rare Met.* 2013, 32, 284–289. [CrossRef]
- 17. Wu, X.F.; Zhang, G.A.; Wu, F.F. Influence of Bi addition on microstructure and dry sliding wear behaviors of cast Al-Mg₂Si metal matrix composite. *Trans. Nonferrous Met. Soc. China* **2013**, *23*, 1532–1542. [CrossRef]
- 18. Zhu, M.; Jian, Z.Y.; Yao, L.J.; Liu, C.X.; Yang, G.C.; Zhou, Y.H. Effect of mischmetal modification treatment on the microstructure, tensile properties, and fracture behavior of Al–7.0 % Si–0.3 % Mg foundry aluminum alloys. *J. Mater. Sci.* 2011, 46, 2685. [CrossRef]