

## Wear characteristics and machining studies on AZ31-calcium silicate composites

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**Abstract:** Composites of AZ31 magnesium (Mg) alloy dispersed with calcium silicate were produced through friction stir processing (FSP) to investigate the effects of grain refinement and reinforcement on mechanical performance, wear resistance, and machining characteristics. Microstructural analyses and X-ray diffraction studies revealed significant grain refinement, reducing grain size from  $114 \pm 5.8 \mu\text{m}$  to  $3.5 \pm 1.4 \mu\text{m}$ , along with the development of a basal-dominated texture in the composites. The increased hardness, measured at  $96.6 \pm 7.5 \text{ HV0.1}$ , was higher than that of the base AZ31 alloy, which was  $59.2 \pm 6.4 \text{ HV0.1}$ . This enhancement is attributed to grain refinement and the addition of calcium silicate. Wear tests demonstrated a lower coefficient of friction and improved wear resistance, correlating with the increased hardness of the composite material. Machining studies conducted at drilling speeds of 90 rpm and 180 rpm, with feeds of 15 mm/min and 30 mm/min, showed increased cutting forces for the composite. This increase is likely due to the enhanced mechanical strength resulting from the fine-grained structure and the presence of ceramic phases within the composite. The study concludes that AZ31-calcium silicate composites exhibit superior mechanical properties and wear resistance. However, the increased difficulty in machining presents a challenge, which must be considered when designing processes involving AZ31-calcium silicate structures for machining applications.

**Keywords:** Calcium silicate, Machining, Magnesium, Mechanical behavior, Wear.

### 1. Introduction

Magnetism (Mg) based composites are now gaining more interest as lightweight materials compared with aluminum alloys due to their higher specific strength. Different Mg alloys are used as matrix materials to develop composites [1]. However, Mg alloys suffer from poor formability and inferior wear resistance. Reinforcing hard phases into Mg alloys and producing composites is a potential strategy to enhance the mechanical behavior and tribological performance of Mg alloys [2]. AZ series alloys are the most commonly used group of Mg alloys for automobile and aerospace applications [3]. Among them, AZ31 alloy is the most widely used alloy to manufacture structures and components. Using AZ31 alloy as the matrix material and incorporating different phases such as  $\text{Al}_2\text{O}_3$ , SiC,  $\text{SiO}_2$ ,  $\text{TiO}_2$ , TiC, CNTs, hydroxyapatite, graphene, etc can be seen in the literature to produce composites [4-11]. These composites exhibited improved mechanical performance, wear characteristics due to the addition of hard and brittle reinforcements [12].

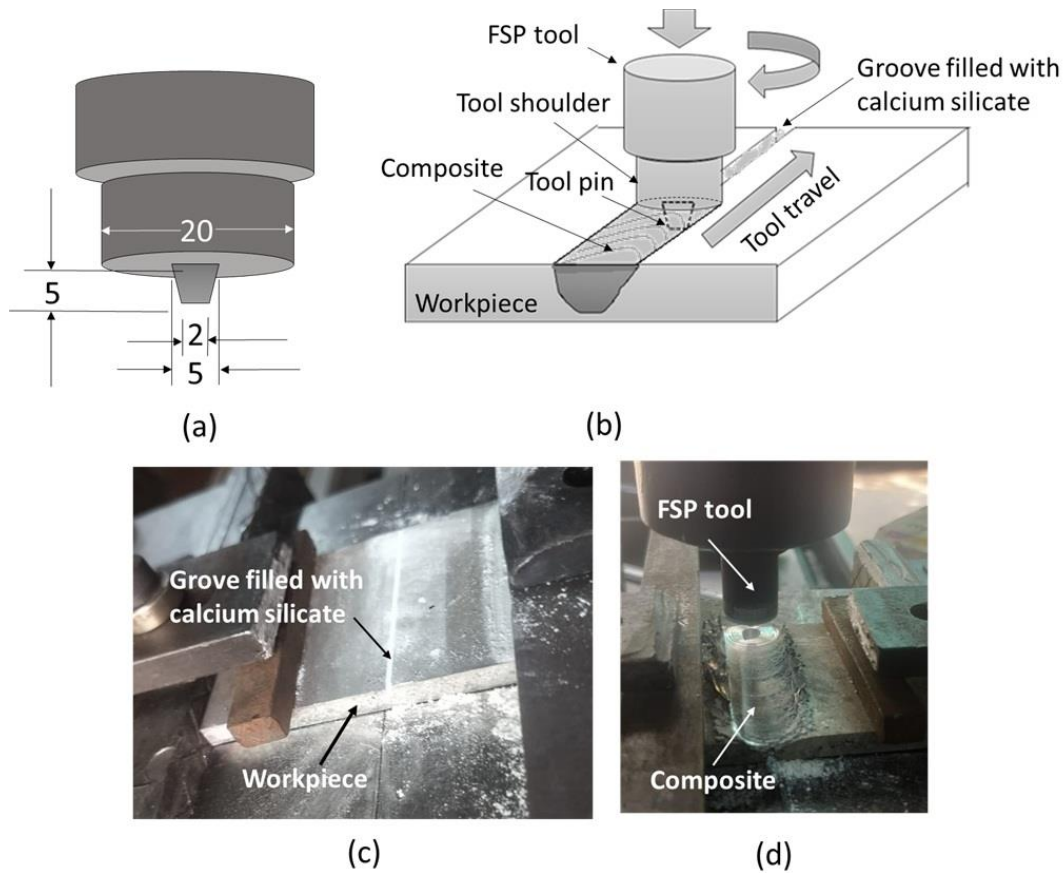
Calcium silicate is a mixture of multi ceramic phases basically contains calcium and silicon compounds. Calcium silicate is a promising structural material exhibits low thermal resistivity and high temperature resistance [13]. Using calcium silicate as reinforcing phase to produce composites for

biomedical applications is another research field recently attracted significant interest Yin, et al. [14]. Huan, et al. [15] produced composites of pure Mg-calcium silicate by spark plasma sintering and increased corrosion resistance and bioactivity has been reported. Kumaravelu and Kandasamy [16] developed AZ91D- $\text{Ca}_2\text{SiO}_4$  composites by stir casting route and significant improvement in load bearing capacity was achieved. Furthermore, the addition of  $\text{Ca}_2\text{SiO}_4$  was found to be influential to produce refined microstructure in AZ91D alloy. Recently, it was demonstrated that by adding  $\text{Ca}_2\text{SiO}_4$  to AZ91D alloy, coefficient of friction (COF) and wear rate can be significantly decreased [17].

Machining is an inevitable process to produce engineered structures and components in the manufacturing industry. In order to bring the benefits of the new material systems, adopting different material removal processes is necessary. Due to the presence of heterogeneous phases, composites exhibit different behavior during machining compared with monolithic materials [18-20]. Hence, studying machining characteristics is important in the materials engineering. Mg alloys exhibit good machinability. However, machining of Mg composites is relatively complex due to the reinforced hard and brittle phases. Surya Kiran, et al. [21] demonstrated improved machinability with lower variations in cutting forces in grain refined AZ91 Mg alloy after friction stir processing (FSP). Similarly, Venkataiah, et al. [22] also reported increased cutting forces in ZE41 Mg alloy after FSP due to the increased strength and developed fine grained structure. Singaiah, et al. [23] produced hybrid composites of AZ91-fly ash-granite powder and increased mechanical properties and increased level of difficulty in machining was reported during turning. In the literature, information on using calcium silicate as dispersing phase to produce AZ31 composites is insufficient and particularly machining studies on these composites are lacking. Hence, in the present work, composites of AZ31-calcium silicate were produced by friction stir processing (FSP) with an objective to study the role of reinforcement on enhancing the mechanical properties, wear and machining characteristics targeted for structural applications.

## 2. Experimental Details

AZ31 Mg alloy sheets (2.89 % Al, 0.91 % Zn, 0.15 % Si, 0.1 % Mn, and the balance being Mg by weight) (Exclusive Magnesium, India) were used as raw materials. FSP was done to achieve grain refinement in the AZ31 alloy sheets ( $100 \times 100 \times 6$  mm<sup>3</sup> size). A specially designed workpiece holding attachment was used to fix the workpieces on the work table of vertical milling machine. An FSP tool having tapered pin with circular cross section was adopted in the present work. Figure 1 (a) illustrates the dimensions of the FSP tool. In order to produce composites, a groove having 2 mm depth and 1 mm width was produced on the workpiece (Figure 1 (b)). The workpiece was fixed on the table and the groove was filled with calcium silicate powder of particle size  $77.9 \pm 11.5$   $\mu\text{m}$  (Quality traders, Guntur, India). Initially, the groove was closed by using a pinless-tool and then FSP was carried out on the closed groove to produce the composite at 1400 rpm tool speed and 25 mm/min tool travel speed [24]. Grain refined AZ31 Mg alloy without incorporating calcium silicate powder was also produced by FSP at the same process parameters to compare the effect of grain refinement alone on wear and machining behavior. The photographs obtained while producing the composite and the resulted composite are presented in Figure 1 (c) and (d), respectively. After metallographic polishing, the samples were etched with picric acid reagent for microstructural studies (Leica, Germany). Scanning electron microscopy (SEM, SU1510, Hitachi) of starting materials was also carried out. X-ray diffraction analysis (D8, Bruker, USA) was done by using  $\text{CuK}\alpha$  radiation to characterize the samples.



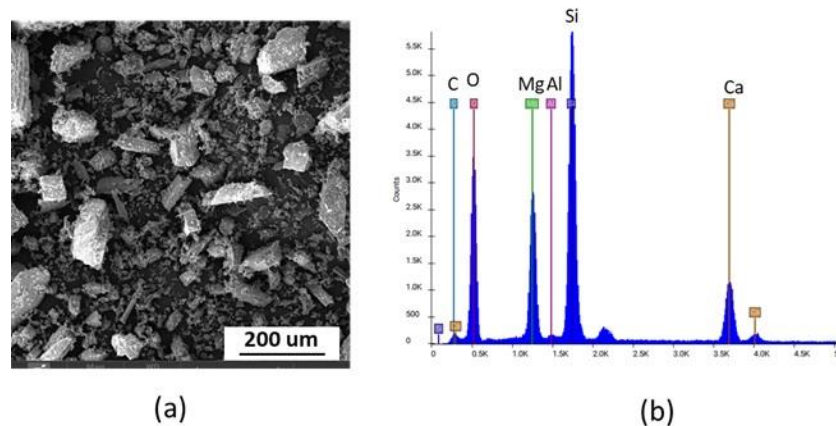
**Figure 1.** (a) schematic illustration of tool dimensions, (b) FSP to produce composites, (c) photographs showing groove filled with workpiece and (d) photograph of the produced composite.

Hardness of the samples was assessed by Vicker's indentation by applying a load of 100 g. Measurements were done for every 1 mm on the polished surfaces of the samples. Wear properties of the samples were assessed by pin on disc method against a stainless-steel disc. Experiments were done by applying 10 N load for 900 s at the ambient conditions. The disc was operated at 1000 rpm speed and wear track diameter of 40 mm was selected. Test pins (10 mm diameter for 6 mm length) were machined and collected from AZ31, FSP AZ31 and the composite. From the test data, COF and wear values was measured for all the samples. Machining studies were carried out by drilling experiments at 90 rpm and 180 rpm rotational speeds and at 15 mm/min and 30 mm/min feeds. A drill bit of 6 mm diameter was used to conduct the experiments. The workpieces were fixed on a load cell (Kistler, Germany) fixed on the worktable of drilling machine (hmt, India). Experiments were done in dry conditions.

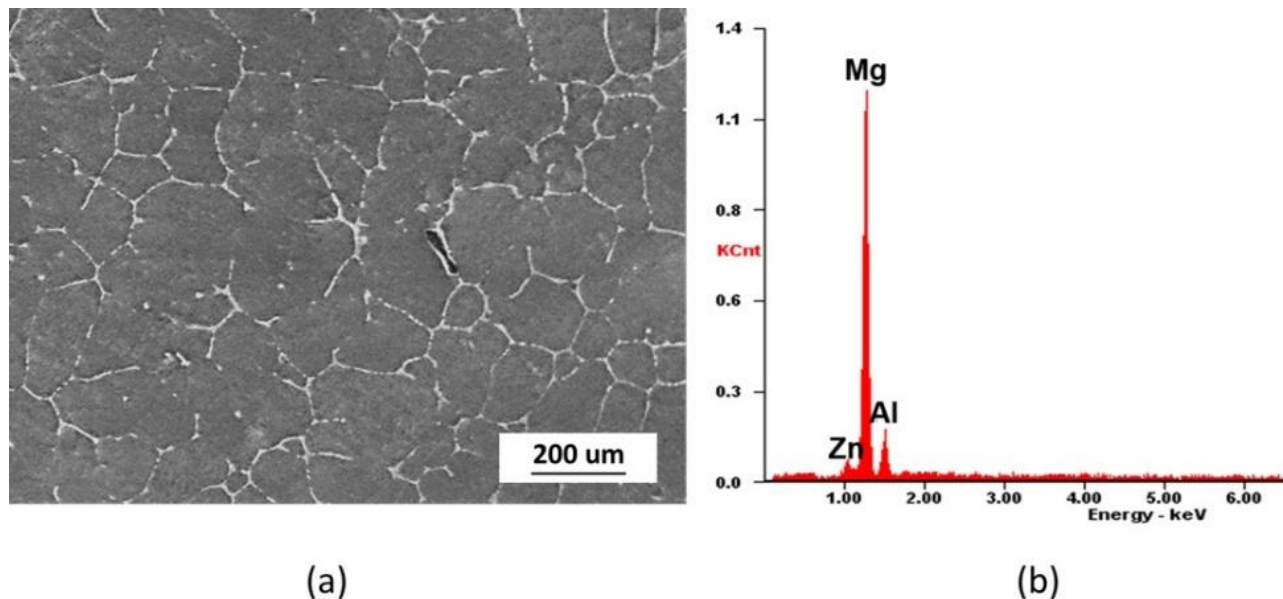
### 3. Results and Discussion

The SEM image and corresponding EDS analysis of calcium silicate powder used in the present work are presented in Figure 2. Very fine particles were also noticed along with the large calcium silicate particles. The presence of Ca, Si, Mg, and O elements from the chemical composition as observed from the EDS spectra was observed as expected in calcium silicate powder. Figure 3 presents the microstructure and EDS analysis of AZ31 base alloy. Due to the presence of more Al, hard and brittle intermetallic ( $Mg_{17}Al_{12}$ ) phase is formed in AZ31 alloy as shown in Figure 3 (a). Compared with AZ61 and AZ91 Mg alloys, AZ31 alloy possess lower amount of  $Mg_{17}Al_{12}$  phase which makes AZ31 relatively

low brittle and more ductile within the AZ series alloys and can be a suitable matrix material to develop Mg based composites [25, 26]. Base alloy has an average grain size of  $77.9 \pm 11.5 \mu\text{m}$  (Figure 3 (a)). The two types of regions i)  $\alpha$ -Mg grains and ii)  $\text{Mg}_{17}\text{Al}_{12}$  phase at the grain boundary are a typical observation in AZ series Mg alloys. Solid solution  $\alpha$ -Mg grains exhibit soft nature and the intermetallic phase exhibits hard and brittle nature [3]. Hence, altering the size of the solid solution  $\alpha$ -Mg grains and intermetallic quantity modifies the mechanical, wear and machining properties. In addition to the microstructural changes, if external phases are incorporated into the matrix in the form of reinforcements, the bulk properties of the composites are significantly altered.



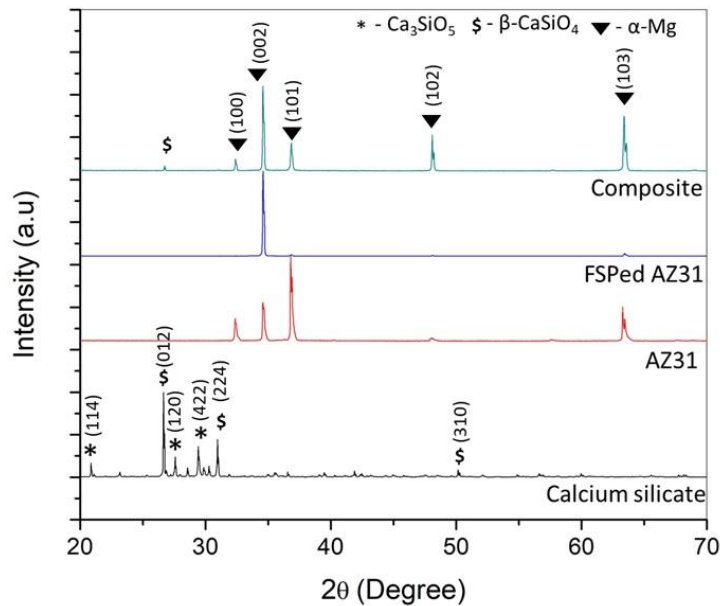
**Figure 2.**  
Characterization of calcium silicate powder: (a) SEM image and (b) EDS analysis.



**Figure 3.**  
Characterization of AZ31 alloy: (a) SEM image and (b) EDS analysis.

Figure 4 presents the XRD patterns of the samples. XRD analysis of calcium silicate powder used in the current work confirms the significant phases present in the powder. Similarly, XRD analysis of AZ31 Mg alloy also confirms the condition of the matrix material. No peaks corresponding to any other

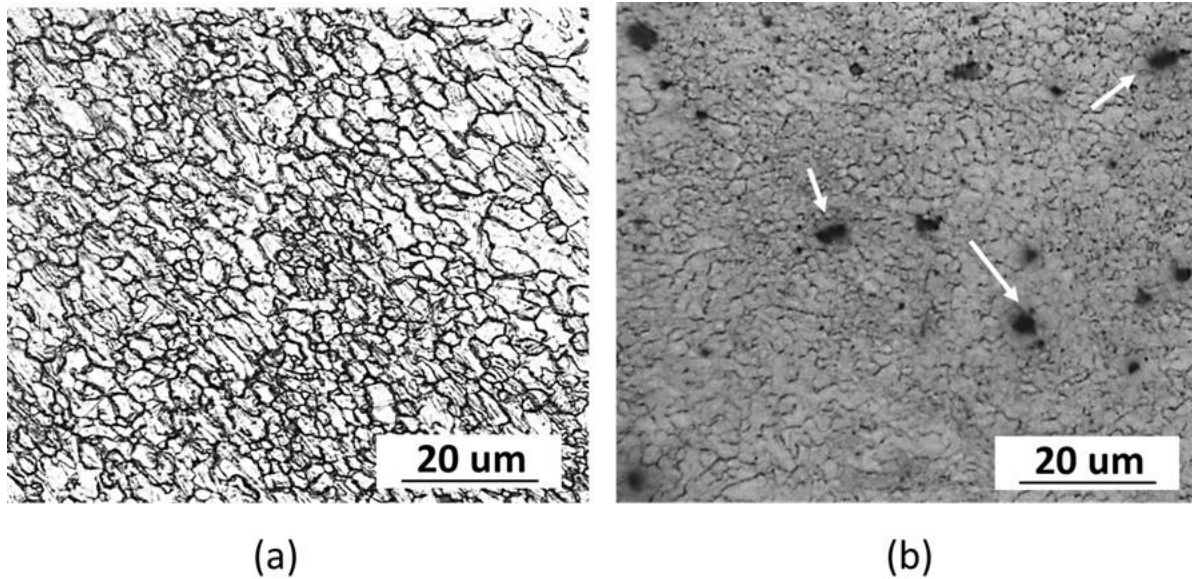
phases were identified. XRD pattern of FSPed AZ31 demonstrate altered intensities of peaks compared with AZ31 base alloy. Intensity of (002) peak was significantly increased after FSP compared with all other peaks which is an indication for the developed basal dominated texture during dynamic recrystallization, a mechanism by which grain refinement happens in FSP [27]. Similarly, XRD analysis of the composite also shows change in the peak intensities. However, change in the intensity of (002) peak is relatively lower for FSPed AZ31. The added calcium silicate powder affected the flow of material in the FSPed region and resulted in lower change in the peak of the composite. However, peak intensities were clearly changed for FSPed AZ31 compared with AZ31 which confirms the texture change in the composites. The presence of peaks corresponding to calcium silicate also can be seen in the XRD pattern of the composite.



**Figure 4.**  
XRD patterns of the samples.

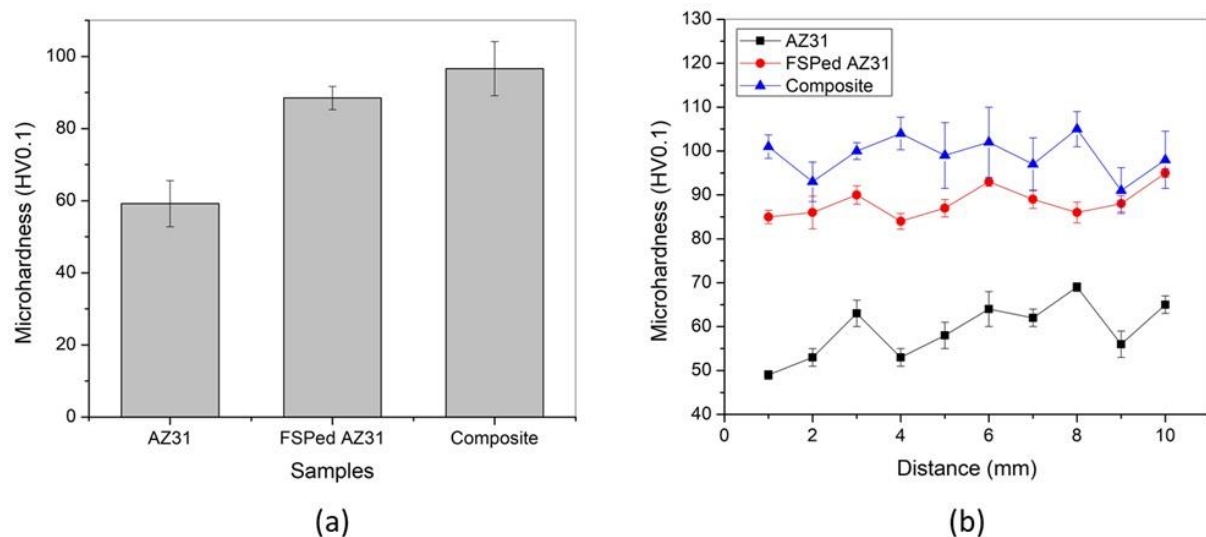
Figure 5 compares the microstructures of FSPed AZ31 and the composite. Composite has  $3.5 \pm 1.4 \mu\text{m}$  grain size which is lower compared with FSPed AZ31 ( $6.2 \pm 2.5 \mu\text{m}$ ) which is significantly lower than AZ31. Furthermore, intermetallic phase was also significantly decreased after FSP which is similar to earlier reports in which FSP decreases the intermetallic phase in Mg alloy [21, 22, 28]. Both the microstructures (Figure 5) show significant refined grain size and greatly decreased secondary phase after FSP. Furthermore, the microstructure of the composite also shows the agglomerated calcium silicate powder as clusters as indicated with white arrows (Figure 5 (b)). From the XRD analysis and microstructural studies development of fine grains and texture change was observed in FSPed AZ31 and the composite. The grain refinement in the composite was higher compare with FSPed AZ31.





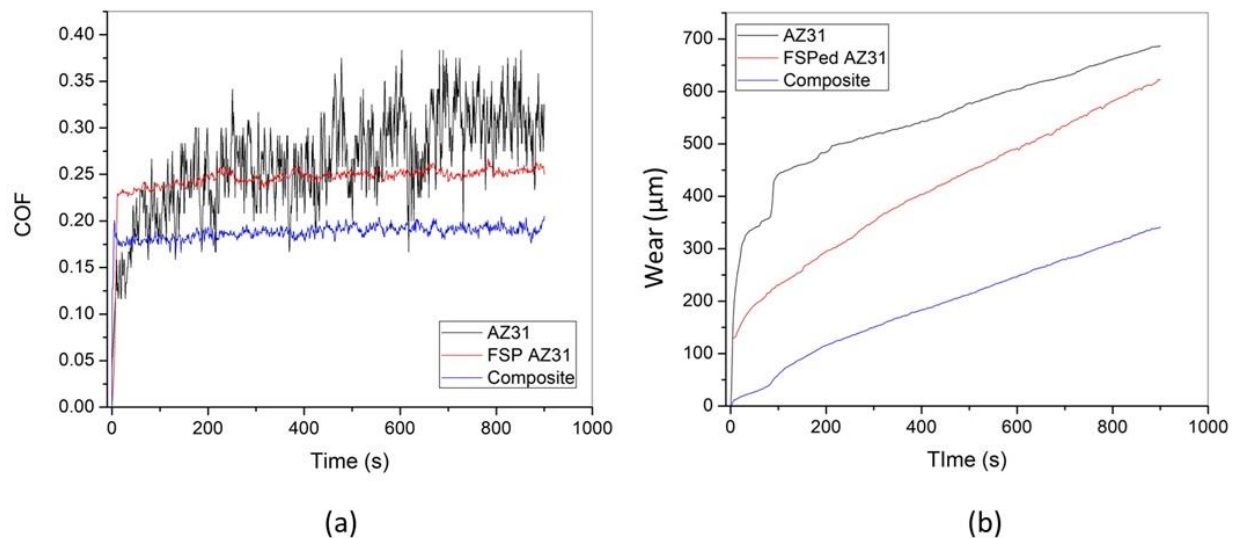
**Figure 5.**  
Microstructural studies of processed samples: (a) FSP AZ31 and (b) composite.

The hardness data of the samples is compared in Figure 6. The average hardness comparison clearly shows the improved hardness in FSPed AZ31 compared with base alloy. Grain boundary strengthening helped to increase the hardness of FSPed AZ31. For the composite, further increment in the hardness was observed which is claimed to the effect of smaller grain size and also dispersion strengthening. Marginally higher variations within the measured hardness values were observed in the composite due to the added reinforcement (Figure 6 (b)). Compared with base alloy, FSPed AZ31 has shown lower variation in the hardness values that is claimed to the uniform and decreased grain size and intermetallic. However, due to the added reinforcement, in spite of having fine and uniform grains, hardness values were observed as distributed in a wide range compared with FSPed AZ31.



**Figure 6.**  
Microhardness data: (a) mean values and (b) variations in the measured data.

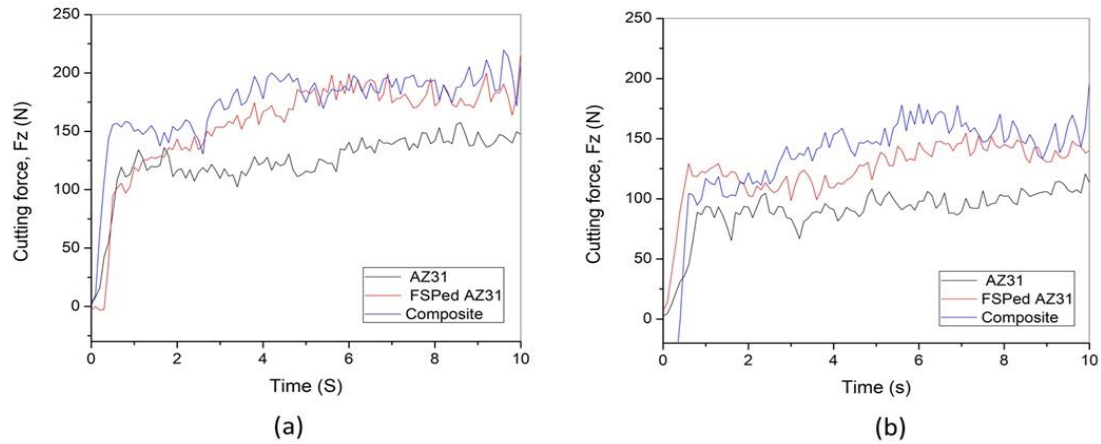
Wear test data of the samples are compared in Figure 7. The COF vs time graphs clearly demonstrate lower COF for the composite among all the samples. Similarly, COF of FSPed AZ31 was measured as lower than AZ31 sample. At the same test conditions, more variations were observed for the base alloy. Coarse grains and the presence of hard  $Mg_{17}Al_{12}$  phase at the grain boundaries resulted wide range of COF values in AZ31 sample. Both the FSPed AZ31 and composite samples have shown lower variations in COF resulted from grain refinement and decreased  $Mg_{17}Al_{12}$  phase due to FSP [29]. From the wear data (Figure 7 (b)), lower wear observed in the FSPed AZ31 and composite samples compare with AZ31 sample. Grain refinement has a predominant role on improving the wear resistance in FSPed AZ31 sample. Furthermore, the addition of calcium silicate has positive role on improving the wear resistance in the composite.



**Figure 7.**

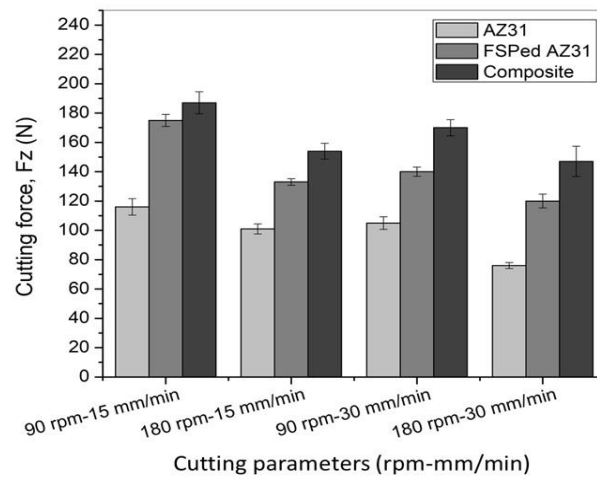
Wear characteristics of the samples: (a) COF and (b) wear rate of the samples.

The typical cutting force vs time graphs at the combination of lower cutting speed - lower feed (90 rpm-15 mm/min) and higher cutting speed - higher feed (180 rpm - 30 mm/min) are shown in Figure 8. Mean cutting force ( $F_z$ ) values were measured and compared in Figure 9. With the increased cutting speed,  $F_z$  values were decreased for all the samples. Furthermore, with the increased feed from 15 to 30 mm/min,  $F_z$  values were marginally increased for the samples. At all the machining conditions, FSPed AZ31 sample has higher cutting force values compared with AZ31 sample. Composite has shown higher cutting forces compared with FSPed AZ31 and AZ31 base alloy. The increased cutting force is due to the increased resistance towards the material removal against the cutting-edge during drilling resulted from grain refinement and the presence of calcium silicate as schematically shown in Figure 10. While drilling FSPed AZ31 sample, smaller grains resulted higher resistance against the cutting edges to remove the material compared with AZ31 sample. The combined effect of fine grains and added calcium silicate increased the cutting forces in drilling of the composites.



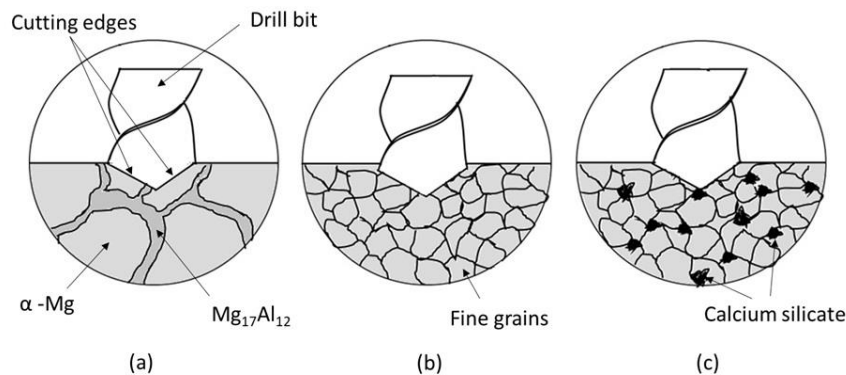
**Figure 8.**

Cutting force vs time graphs during drilling at different combination of parameters: (a) 90 rpm with 15 mm/min feed and (b) 180 rpm with 30 mm/min feed.



**Figure 9.**

Mean cutting force values of the samples.



**Figure 10.**

Schematic illustration of machining of samples: (a) AZ31, (b) FSPed AZ31 and (c) composite.



From the results, it is summarized that achieving grain refinement and incorporating calcium silicate in AZ31 alloy increases the mechanical properties and wear resistance which are the benefits from grain refinement and added reinforcement. However, in order to yield the benefits from these composite materials, manufacturing structures by different machining operations is necessary. As observed from the current study, the cutting forces were increased which increases the difficulty in machining. In order to develop high performing light weight structures, designing machining operations considering special concerns while machining is required.

#### 4. Conclusions

The present study aims to study the role of grain refinement and incorporating calcium silicate into AZ31 alloy by FSP on mechanical performance, wear resistance and machining characteristics during drilling. FSP resulted significant grain refinement in AZ31 alloy. Addition of calcium silicate helped to produce smaller grain size in AZ31. XRD analysis confirms the basal dominated texture in FSPed AZ31 and the composite. Fine grain structure coupled with reinforcement improved the hardness in FSPed AZ31 ( $88.5 \pm 3.2$  HV0.1) and adding calcium silicate further increased the hardness of the composites ( $96.6 \pm 7.5$  HV0.1 ) compared with AZ31 sample ( $59.2 \pm 6.4$  HV0.1 ). Wear studies clearly demonstrated the role of grain refinement and calcium silicate addition to increase the wear resistance as reflected from lower COF and wear. Machining studies done by conducting drilling experiments at 90 rpm and 180 rpm speeds; and at 15 mm/min and 30 mm/min feeds demonstrated higher cutting force for FSPed AZ31 and also for the composite than AZ31 alloy because of the increased hardness which resulted from the grain refinement and incorporated calcium silicate. The results suggest decreased machinability for grain refined AZ31 and the composite. It is concluded that increased mechanical and wear properties can be obtained for AZ31-calcium silicate composites developed by FSP. However, decreased machinability as reflected from increased cutting forces needs a special consideration while planning machining operations to produce structures using the developed composites.

#### Transparency:

The authors confirm that the manuscript is an honest, accurate, and transparent account of the study; that no vital features of the study have been omitted; and that any discrepancies from the study as planned have been explained. This study followed all ethical practices during writing.

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