



Microstructure, Mechanical Characteristics, Wear Behaviour, and Machine Operation of AZ91D-B4C-ZrO₂ Blended Composites

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Abstract. Hybrid composites of AZ91D magnesium alloy with boron carbide (B4C) and zirconia (ZrO₂) were created using the stir casting technique. The microstructure of the composites showed that the distribution of reinforcing phases was heterogeneous. To study the tribological properties of the manufactured composites, including wear and friction coefficient in dry sliding situations, a pin-on-disc test was performed. The dispersion of the reinforcement caused the composites to exhibit increased hardness. When compared to the other specimens, the composite containing 2 wt.% ZrO₂ + 3 wt.% B4C showed much improved yield and tensile strengths. The AZ91D alloy's wear resistance was enhanced by the addition of B4C and ZrO₂, as seen by the reduced wear rate. The specimen with the best wear resistance was the composite reinforced with 2 weight percent ZrO₂ and 3 weight percent B4C. Combining Multiple Design-of-Experiment Methods with the L9 Array. Several WEDM process parameters for AZ91D, B4C, and ZrO₂ optimisation with respect to minimum surface roughness and material removal rate (MRR) have been identified in this study. Surface roughness increases, B4C proportions, and ZrO₂ proportions must have contributed independently to the material removal rate (MRR) decrease. In order to produce HMMC of AZ91D, B4C, and ZrO₂ at different weight percentages, the study's results are utilised to establish the most efficient and accurate WEDM process parameters. This study concludes that improving the mechanical characteristics, MRR, surface roughness, and wear parameters of AZ91 composites by adding B4C and ZrO₂ is a viable option.

Keywords: Stir Casting, Tensile, Wear, Impact, MRR, Surface Roughness

1 Introduction

The field of materials engineering has recently seen a surge in interest in the potential of lightweight materials for energy efficient applications. Magnesium (Mg), the eighth most common metal in the Earth's crust, is gaining favour in this setting because to its low density compared to aluminium (about two-thirds) and steel (about one-fifth). In comparison to more traditional technical alloys, magnesium alloys have a high specific strength, excellent damping capability, and are easy to machine and cast [1]. On the other hand, it becomes more brittle and difficult to shape at ambient temperature, and its mechanical strength drops significantly at higher temperatures [2]. Magnesium matrix composites and low-cost manufacturing methods have been the subject of much study due to the need for lightweight materials with great performance in sophisticated industrial applications.

Many lightweight applications in the aerospace and automotive sectors make use of AZ91D because to its high specific strength, excellent castability, and machinability [3]. However, there are several significant constraints to consider, such as decreased strength, poor creep resistance, and wear resistance. Metal matrix composites (MMCs) are able to circumvent these restrictions by including additional reinforcing phases into AZ91D. Valve covers and housings, brackets, shift actuators, intake manifold blades, and elements of the steering column are among the many automotive sectors that use magnesium matrix composites (Mg-MMCs). Some examples of non-vehicular uses include biodegradable implants, portable oxygen pumps, electrical gadgets, medical equipment, and power tools [4]. Reinforcing phases in MMCs often consist of hard particles, which have remarkable properties including high hardness, refractoriness, and wear resistance. Examples of such particles are B₄C, SiC, Al₂O₃, SiO₂, fly ash, TiO₂, TiC, MoS₂, CNTs, and MgO [5-9]. Changing the quantity of reinforcing elements has been shown to affect the mechanical characteristics, microstructure, and wear resistance of metal-matrix composites, according to many studies [10, 11].

According to the research, there are a number of processing pathways that have been used to create Mg based MMCs, and each of these routes has its own set of benefits. The majority of these lightweight MMCs are developed via the casting approach [12]. Cast a hybrid composite of AZ91D, B₄C, and Gr and studied its tribological characteristics. The composite's hardness value was greater than that of the basic alloy. Graphite, when added to the composite, reduced the coefficient of friction and the amount of direct metal contact, acting as a solid lubricant. Using the stir casting technique [13]. Created a magnesium composite that was strengthened with chemosphere's made of fly ash. The microstructure was polished and the hardness was enhanced by the reinforcing. As the fly ash content increased, the matrix's ultimate tensile strength went up and its compressive strength went down. In a similar vein [14]. Created a squeeze cast AZ91D/SiC composite and found that the hardness increased as the volume percentage of SiC in the composite increased. Conversely, when reinforcement levels were raised, surface roughness was reduced and MRR was enhanced. After producing an AZ91/ZrSiO₄ composite using the stir casting process [15]. Studied its mechanical characteristics and tribological behaviour. The AZ91 alloy with 8 wt.% ZrSiO₄ reinforcement outperformed the AZ91 base alloy in terms of wear resistance, hardness (71 HV), and maximum tensile strength

(183 MPa). The mechanical and tribological characteristics of AZ91D composites reinforced with graphite and tungsten carbide were studied [16]. Using the powder metallurgy process. The composites have a higher density (2.64 g/cm³) than the parent alloy (1.81 g/cm³). With an increase from 242 N/mm² to 268 N/mm², the composite outperformed the base alloy in terms of tensile strength. The average elongation was decreased from 2.9% to 2.49 in composites with 6 wt.% WC particles. An Mg composite was also created [17]. Using the stir casting process. The coefficient of friction (COF) decreased over time when nanoparticles were added to the base metal, and there were also noticeable changes in anti-frictional behaviour. The COF value was found to be the lowest in a solution of 2 weight percent SiC in soluble oil, at 0.109[18]. Studied machining-induced surface defects in an AZ91/15 wt.% SiCp composite and found voids, matrix cracking, and particulate fracture due to matrix ductility failure or particle de-bonding. The tiny size of the reinforcing particles made it impossible to detect particulate fracture.

In contrast, Mg based MMCs have been developed using a variety of alternative processing techniques, including powder metallurgy, equal channel angular pressing (ECAP), friction stir processing, and others [19, 20]. A high-strength AZ91 Mg alloy reinforced with SiC was developed[21]. Using ECAP followed by rolling. Among the several effective methods for creating Mg-based composites, friction stir processing stands out [22, 23]. Research shows that adding different kinds of reinforcement to a Mg matrix significantly enhances its mechanical and wear characteristics. The inclusion of carbides improved the wear resistance, while the addition of B₄C enhanced the density of the basic alloy. The addition of oxides improves the composite's mechanical properties.

Features may be further improved by a deeper understanding of the comparative quantity's structure and the distribution of each component inside a compound, as well as the conditions of dispensation. A WEDM method is singular for several mechanistic practices. This method is very adaptable for complicated and intricate geometries, making it the most suitable production procedure for challenging structures composed of composite materials. Despite the efforts of academics to categorize various MMC using the WEDM procedure, very little tedious work has been accomplished[24]. Materials used for tools in WEDM processes are consistently made from copper and brass, with cable thickness varying between 0.1 and 0.25 mm. Despite the absence of physical engagement between the workpieces and the cutting wire during this procedure, sparks are nevertheless generated inside the material of the workpieces, aided by the dielectric liquid. The sparks are consistently generated throughout the equipment working locations, and the manufacturing process concluded due to the excessive spark production [25,26]. The production phase does not result in rejection stress or resistance between the wire and the material objects due to the design of the procedure. The manufacturing of tools, gauges, dies, and fittings often use this procedure. Wire EDM involves several intricate process parameters that depend on its material, the dielectric fluid, and the efficiency of the machining operation. The selection of the suitable progression consideration outline, including MRR and Ra, might be problematic for a research scholar or engineer, since even little alterations can significantly affect machining performance [26].

Literature review revealed a lack of investigation into how B4C and ZrO2 interact to affect the AZ91D alloy's tribological behaviour. Therefore, this study's objective is to create AZ91D/B4C/ZrO2 hybrid composites by means of the stir casting technique and to examine their mechanical and tribological characteristics when subjected to dry sliding.

2. Experimental Procedure

In order to create the Mg hybrid composite, the stir casting technique was used. Table 1 shows the chemical make-up of the basic alloy that was used to create the composites. Table 2 details the ingredients used to make the composites. Small chunks of the AZ91 Mg alloy were cut to the necessary weights in order to make the composites. Before adding the metal components to the stir casting setup's crucible, the furnace was warmed to 4200C. Then, the appropriate amounts of ZrO2 and B4C powders were added to the metal. To melt the mixture, the furnace was sealed and brought to a temperature of 850°C. The vortex technique was used to synthesise the composite. The metal in its molten state was spun at 400 revolutions per minute for ten minutes. This ensures that the liquid metal is evenly distributed and wets the reinforcing particles. The next step was to pour the molten composite mixture into a steel mould and let it cool. The manufactured composite samples are shown in Fig 1.

Table.1 basic substance of AZ91D base alloy in weight percent

Material	Al	Zn	Mn	Si	Cu	Fe	Ni	Mg
Wt.%	8.3	1	0.15	0.1	0.03	0.005	0.002	rest

Table.2 Composition of the materials used to construct the composites

S. No.	AZ91D [%]	B4C [%]	ZrO2 [%]
1	100	0	0
2	97	3	0
3	95	3	2

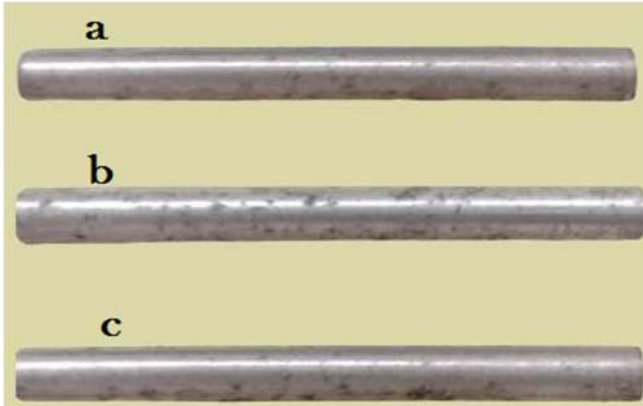


Fig. 1. Produced composite samples: a) AZ91D, b) AZ91D+3%B4C c) AZ91D+3%B4C+2%ZrO₂

The samples were prepared for the microstructural analysis by polishing them according to the conventional metallographic procedure, which comprises emery paper, diamond paste, and picric acid reagent etching. A Leica light microscope from Germany was used to study the microstructures. Energy dispersive X-ray spectroscopy (EDS) was used to record the specimens' chemical composition, while scanning electron microscopy (SEM) was used to analyse the morphologies of the reinforcing powders. A Rockwell instrument was used to test the hardness of the specimens. A thorough cleaning was performed on the specimen's surface to eliminate any dirt or oxide scales. A 100 kgf force was applied to a 1/16" ball indenter. The pointer was then set to the correct place when a little load was applied. Then the whole force was slowly brought to bear. The cursor came to rest, releasing the primary burden. When the arrow stopped going in the other way, that is when the Rockwell hardness was determined. Three materials were subjected to tensile testing using Unitech's (India) uniaxial tensile test equipment. Test data was used to quantify yield strength, ultimate tensile strength, and percentage of elongation, with a strain rate of 0.1/s applied.



Fig. 2. Pin-on-disc equipment

The material wear under dry sliding circumstances was measured using a pin-on-disc instrument. While the disc was being rotated, the stationery pin (sample) was forced against it using a steel counter disc. When making the composite pins, we thought about the ASTM G99 standard. The wear experiments were conducted using the pinon-disc equipment, as shown in Figure 2. Acetone was used to clean the pin and disc surfaces prior to testing. The test was run for 15 minutes with the starting wear and friction forces set to zero. The tests were conducted with a 2 kilogramme weight and 200 rpm speed, taking into account a radius of 0.07 m. Organic solvents were used to clean the disc surfaces after each test, ensuring that no worn particles remained. At each step, the linear wear measurements and frictional force were recorded, and the coefficient of friction and wear rate were computed. The wear parameters were calculated using the following formulas:

Surface speed (s) = $(\pi DN/60)$ [m/min] Frictional torque (T) = $F \times R$ [Nm] Frictional power (P) = $(2\pi NT)/60$ [Watts]

Wear rate = Linear wear / time period [microns/min]

Following that, the machining process obtain place on a Wire Electrical Discharge Machining (WEDM) platform, with a 0.25 mm dimension bronze thread serving as the primary cutting component. This machining procedure resulted in the fabrication of MMCs with dimensions of 100 x 100 x 10 mm, which is achieved via the use of the complex stir casting technique.

The choice of dielectric medium that are used in order to promote the sparking phenomena that occurred between the tool and the workpieces is the novel component of this method." Within the context of this situation, water is designated as the dielectric medium. The WEDM process is strengthened in terms of its accuracy and efficiency as a result of this selection, which brought a novel component into the equation of machining. Figure 2 presents a schematic depiction of the WEDM configuration, which may be used to get a better understanding of the system. In order to shed light on the complexities of the process, this graphic provides an explanation of the location and interaction of the components. To summarize, the swirl casting technique, in conjunction with the careful selection of materials and techniques, made it possible to produce complicated MMCs that had dimensions and qualities that are exceptional.

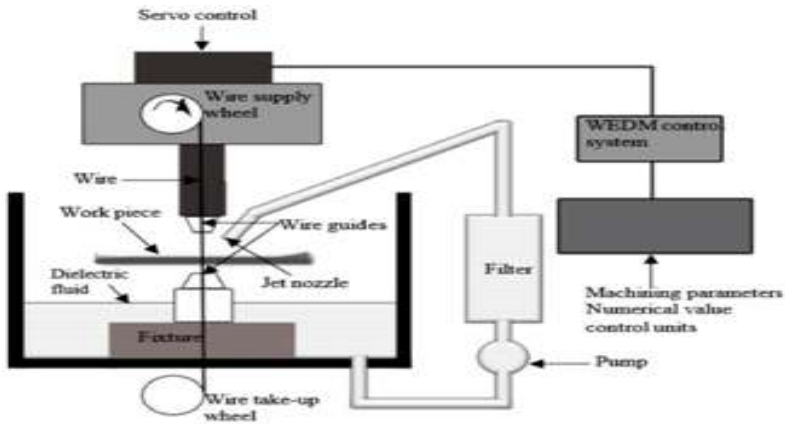


Fig. 3. Wedm Set Up

After that will utilize a design of experiments (DOE) that makes use of a L9 orthogonal array to modify the other four variables, including pulse-off time, after that have listed all of the distinct variables that may be altered during the procedure in Table 3. For the purpose of achieving the desired effect, nanoscale components are first cut, and then the surfaces of the sample pieces are honed using emery paper with varying grits. It is possible for us to achieve a mirror-like quality on both a specimen and an alumina postponement amid rubbing material by using disc polishing equipment.

Table. 3. Defining the WEDM Procedures

Levels	Process parameters			
	Pulse on Time-TON	Pulse OFF Time-TOFF	Voltage Gap-V	Wire Feed Rate-F
1	5	9	55	6
2	7	7	75	8
3	9	5	95	10

A combination of optical and scanning electron microscopes (SEMs) are used in the process of microstructure creation. By using Keller's reagent, it is possible to etch samples of composite fabrics. A Vickers hardness test is performed on the samples using a weight of 50grammes in order to measure the amount of toughness. This test is performed after the microstructure pictures have been taken. The smoothness of the exterior that had been machined using EDM is evaluated with the use of a surface area measuring smoothness device manufactured by Mitutoyo[27,28].

3. Results and discussion

Reinforced B4C and ZrO2 particle shape and chemical composition are shown in Figure 4. While the ZrO2 particles were spherical in shape, the B4C ones were more amorphous. The particles' sizes were found to be less than 10 μm , with the ZrO2 particles measuring less than 1 μm in particular.

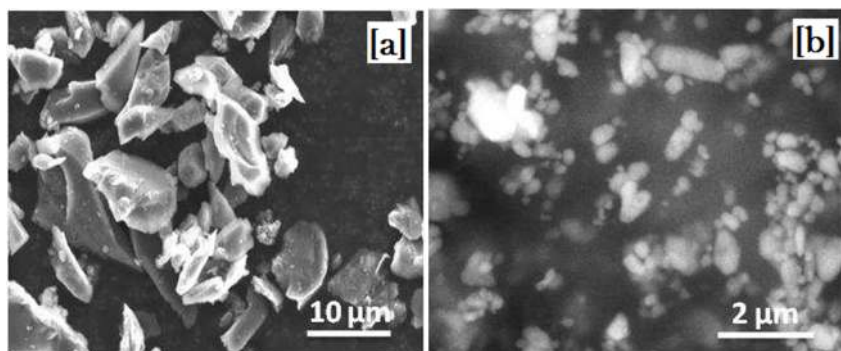


Fig. 4. SEM analysis of reinforcements: a) SEM micrograph of B₄C, b) SEM micrograph of ZrO₂

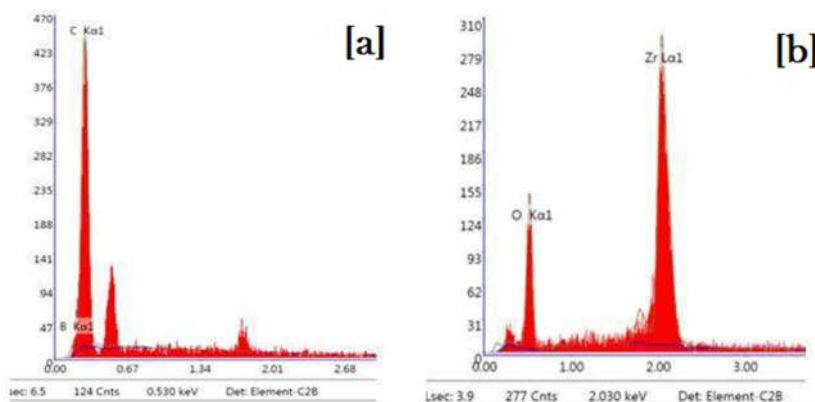


Fig. 5. EDS analysis of reinforcements a) EDS analysis of B₄C, b) EDS of ZrO₂

In Figure 6, we can see the sample microstructures. In the AZ91D base alloy, the β -phase is the darker part, which is a mixture of magnesium and aluminium (Mg₁₇Al₁₂), and the α -phase is the lighter part, which is a solid solution of the same elements. Improving the base alloy's overall mechanical characteristics is mostly accomplished via the intermetallic β -phase. The inclusion of the B₄C particles caused this phase to be dispersed equally. Adding ZrO₂ and B₄C further improved the dispersion uniformity.

The liner intercept technique was used to measure the average grain size of the base alloy, which was found to be $76 \pm 5.4 \mu\text{m}$. Grain size measurements for the AZ91D + 3% B₄C composite are $64 \pm 3.1 \mu\text{m}$ and $61 \pm 5.3 \mu\text{m}$, respectively, after the reinforcements have been added. Another benefit is that the composites' grain size was somewhat decreased due to the reinforcements. Figure 5 shows that the reinforced composites are harder than the original material. The composite of 3% B₄C and 2% ZrO₂ had the greatest hardness value of 52 HRB, whereas AZ91D had an average hardness of 45HRB. The addition of the ceramic reinforcements was crucial in raising the hardness. Increased resistance to indentation in hardness tests is indicative of improved resistance to plastic deformation, which occurs when a reinforcing phase is introduced into the AZ91D matrix via the dispersion

strengthening process. Composites, in comparison to the basic metal, are hence harder.

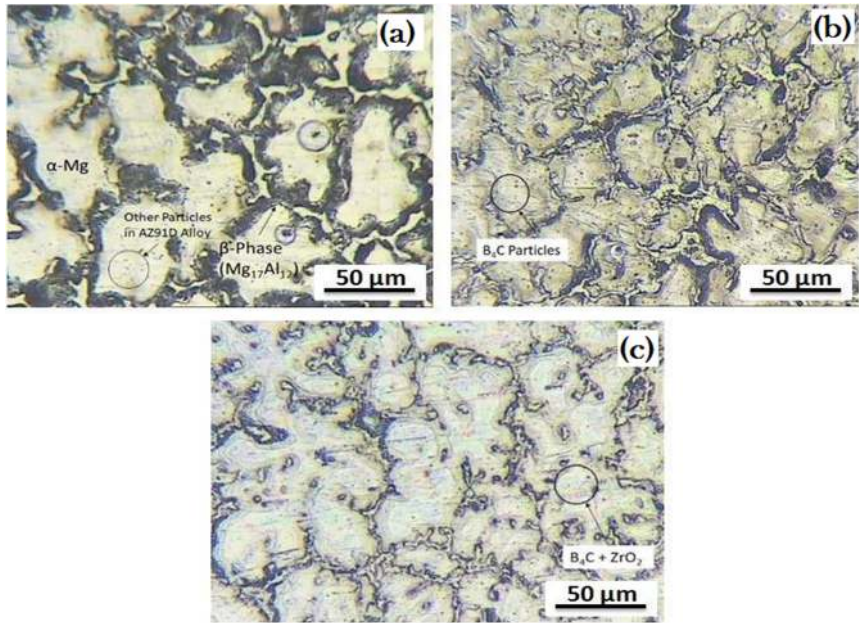


Fig. 6. Microstructure of: a) AZ91D, b) AZ91D+3% B4C, c) AZ91D+ +2% ZrO₂ + 3% B4C (recorded at 200 $\times$ magnification)

Table 4 displays the results of the tensile tests, which show that the addition of ZrO₂ and B4C to the AZ91 matrix significantly increases the composite's strength. The composites' strength was greatly enhanced by the reinforcements, yet their ductility was maintained, as seen by the comparable percentage of elongation compared to the basic alloy. It was found that the tensile characteristics of the composites were significantly improved with the inclusion of multi-phases.

Table. 4. Tensile test data results of specimens

Test/Specimen	AZ91 D	AZ91D+ +3% B4C	AZ91D + 2% ZrO ₂ + 3% B4C
Ultimate tensile strength (UTS) [MPa]	103.07	111.01	129.06
ield strength (YS) [MPa]	41.47	44.38	51.72
% of Elongation	4.53	4.22	4.82

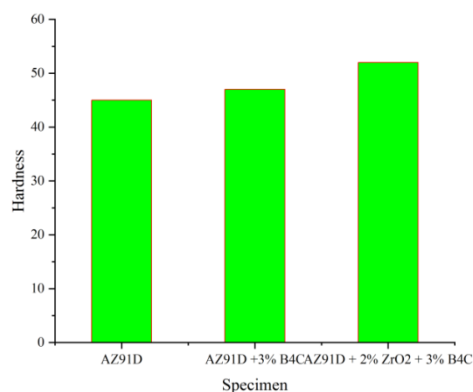


Fig. 7. Comparison of hardness of specimens: a) AZ91D, b) AZ91D+ +3% B4C, c) AZ91D + 2% ZrO₂ + 3% B4C

The specimens' wear rates are compared in Figure 8. The addition of B4C reduced the wear rate from 0.0356 to 0.0328 micron/min, in comparison to the basic alloy. Wear decreased by 7.7 percent, proving that B4C is an effective wear-resistant reinforcement. Further reduction in wear to 0.0304 micron/min was achieved with the use of ZrO₂. When compared to the composite using B4C alone, wear decreased by 6.1%; however, when ZrO₂ was added, wear decreased by an additional 13.32% compared to the base alloy test.

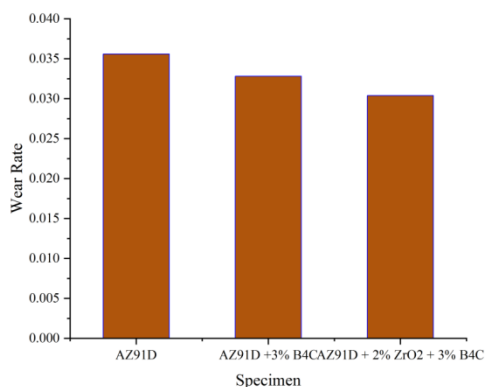


Fig. 8. Wear rate comparison of specimens: a) AZ91D b) AZ91D+ +3% B4C, c) AZ91D+3% B4C+2% ZrO₂

The zirconium particles refine the grain, making the composite far more resistant to wear, which may explain why adding ZrO₂ reduces wear even more. Regardless of the strengthening, the coefficient of friction was almost constant throughout all test circumstances. You may see a summary of the specimens' wear statistics in Table 5.

The μ coefficient of friction, which is the ratio of frictional force (F) to normal force (N), remained unchanged by the additional reinforcements, according to the wear test results. But as compared to the base alloy, the composites had a far higher frictional. The fact that the coefficient of friction remained constant indicates that the same fraction of increased normal force was responsible for this finding. Reinforcements in the composites enhance the normal frictional force; the AZ91D +3 wt.% B4C+2 wt.% ZrO₂ composite showed the greatest increase. The enhanced mechanical performance of the composites had an effect on the wear resistance, as is evident from their increased mechanical characteristics. Figure 7 shows a common comparison between hardness and wear rate. Greater wear resistance was achieved as a result of the increased hardness. Hardness is an important factor in wear resistance as it is a surface phenomena. Composites' hardness, tensile strength, and resistance to surface weight loss from wear were all enhanced by the insertion of reinforcements. Therefore, the findings show that incorporating B4C and ZrO₂ into the AZ91D Mg alloy might be a great way to make high performance composites with better wear and mechanical properties.

Table 5. Wear test data of specimens

Specimen material	AZ91D	AZ91D +3% B4C	AZ91D + 3% B4C+ 2% ZrO ₂
Load on pin [N]	19.62	19.62	19.62
Speed[rpm]	200	200	200
Frictional force (F) [N]	4.345	5.237	5.412
Linear wear[μ m]	0.53	0.49	0.46
Coefficient of friction (μ) [F/N]	0.22	0.23	0.22
Wear rate [microns/ min]	0.0356	0.0328	0.0304

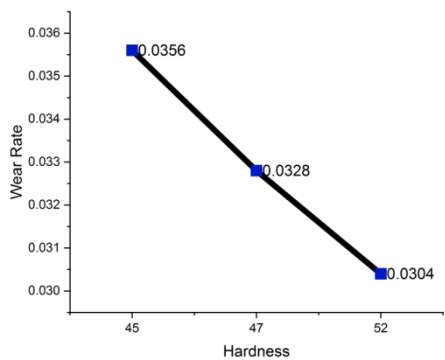


Fig. 9. Role of hardness in wear.

3.1. Manufacturing variables affect effectiveness

The MRR and Ra experimental results are shown in Table 6, which may be accessed here. Additionally, the source process parameters are displayed in this table. Controlling four factors the Voltage Gap, the Pulse On Time, the Pulse Off Time, and the Wire feed Rate is the responsibility of the task that is now being done. The performance of the machine are influenced by a number of different process factors throughout the whole of the production process for making accurate products. Every parameter has the potential to be given a value, and those values may be placed into one of several preset categories[29,30].

Table. 6. Process variables and the interactions with MRR and RA are investigated.

Ex. No	Input Process variables				Output Response of MRR(mm ³ /min)			Surface roughness, RA		
	Pulse on Time	Pulse off Time	Voltage Gap	Wire Feed Rate	AZ91D	AZ91D +3% B4C	AZ91D + 3% B4C+ 2% ZrO2	AZ91D	AZ91D +3% B4C	AZ91D + 3% B4C+ 2% ZrO2
1	5	9	55	6	24.52	23.98	24.15	0.62	0.98	1.65
2	5	7	75	8	18.12	20.24	21.72	1.32	1.62	1.71
3	5	5	95	10	16.28	20.52	17.68	1.41	1.62	1.44
4	7	9	75	10	19.12	22.45	21.23	1.62	1.32	1.65
5	7	7	95	6	18.58	16.25	14.56	1.12	1.22	1.56
6	7	5	55	8	33.24	28.21	21.28	1.25	1.76	1.75
7	9	9	95	8	18.54	21.15	15.28	1.44	1.68	1.85
8	9	7	55	6	29.51	28.04	22.38	0.94	1.52	1.18
9	9	5	75	10	19.02	21.45	21.12	1.02	1.12	1.32

3.2. Material Removal Rate (MRR)

According to the data shown in Figure 9a-d, the standard Material Removal Rate (MRR) investigative significances caption towards existence are as follows: 20.97, 20.87, and 18.15 mm³/min, respectively. Using an AZ91D metal matrix composite, the average Material Removal Rate (MRR) is essentially beginning inside and maintaining with 0, 2, and 3%, of weight percent B₄C and ZrO₂.

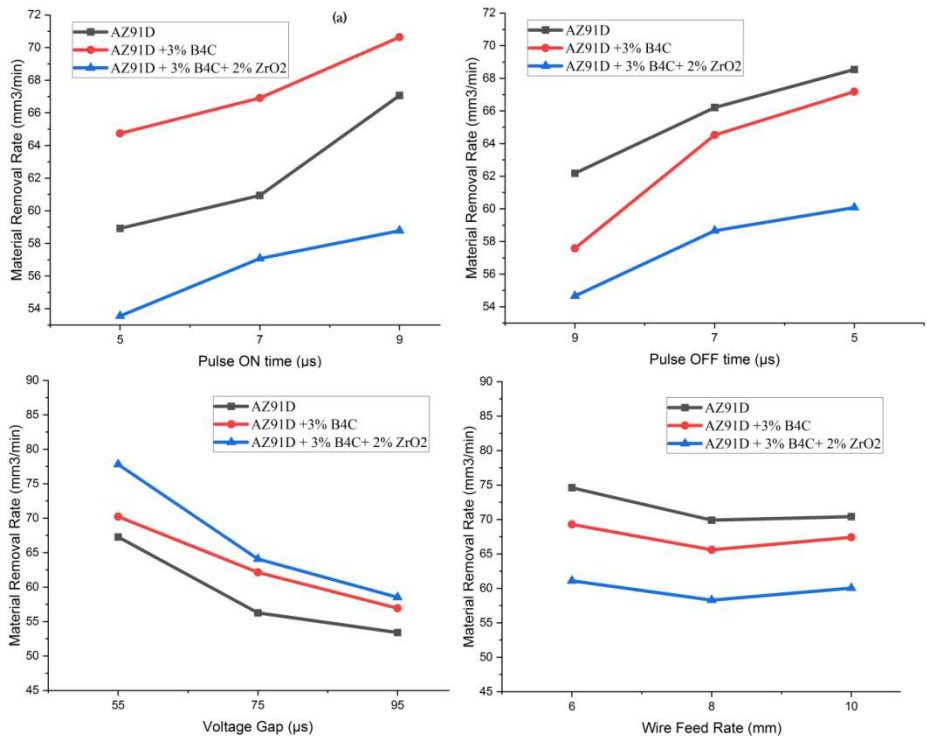


Fig. 9. Graphs Showing the Effect of Material Subtraction

In the case of metal matrix composites (MMCs), it is discovered that a reduction in the material removal rate (MRR) may be achieved by increasing the weight proportion using B4C and ZrO₂. Among the B4C and ZrO₂ particles that are included inside the MMCs, the MRR has the capability to soften them. Because of their high heat conductivity and stiffness, respectively, reinforcements and B4C and ZrO₂ both contribute to a decrease in MRR. This contributes to the overall reduction in MRR. The results of this research indicate that MRR values may often be identified by the enhancement with Pulse on Time. This is due to the fact that an increase in pulse-on time indicates that a spark has happened during the process of wire electric discharge machining variables. The superior pulse on time addresses ejections, pulse during rises in deliberation, and the development of deep deprived position that takes place when specimen material is compared to superior substance[31,32].

3.3. Surface Roughness-Ra

A surface roughness investigation are carried out on AZ91D via B4C and ZrO₂, and Figure.10 a-d illustrates the exit variables that are observed throughout the study. The weights of 0, 2, and 3%, together with a constituent portion that serves to reinforce

the structure, are the components that make up these reactions. Figures 10a through 5d illustrate a degree of surface roughness that is consistent throughout the whole surface.

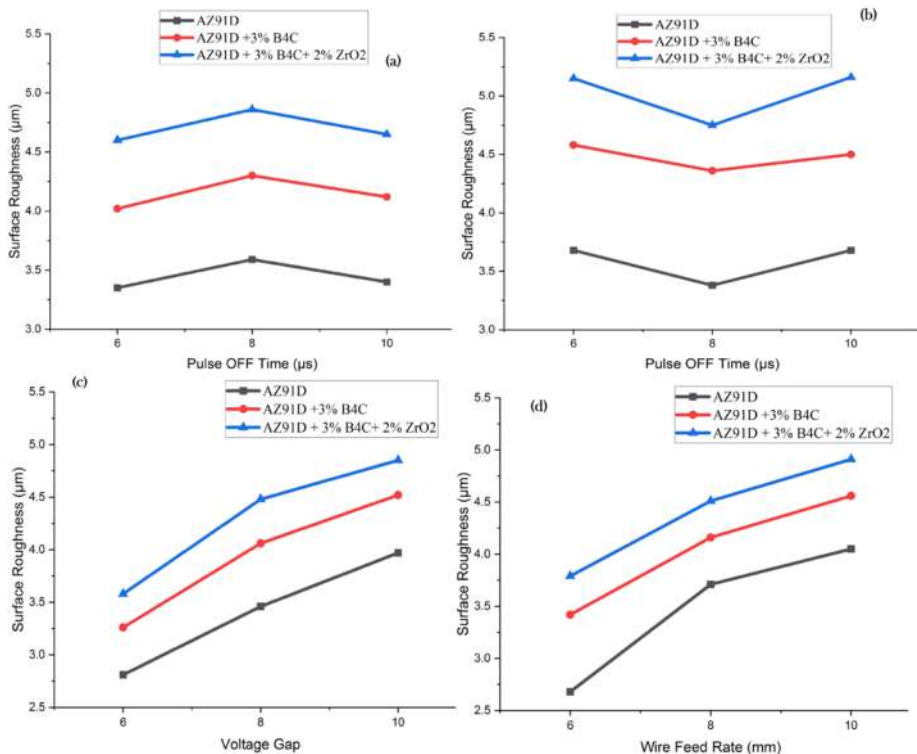


Fig. 10. Graphs of the Reaction to Surface Roughness

Upon examination of figures 10a-d, it becomes evident that the surface roughness has an average value of $1.31\mu\text{m}$. This is accomplished by using an adequate quantity of AZ91D in connection with 0, 2, and 3%, weight of B4C and ZrO₂. The results showed that the surface roughness is significantly improved. The obtained for these values are 1.02, 1.12, and $1.38\mu\text{m}$ respectively. An increasing weight % with strengthening exists may be seen throughout the process of metal matrix composites (MMCs) when it is possible to conduct experiments as shown in figures 10a through 10d. The inclusion of hard particles in MMCs as a reinforcement are shown to be directly responsible for the surface roughness increasing over time. This is discovered as a direct result of the presence of hard particles. Because of the spark that are formed when the tool and the work components came into touch with one another during a wire electric discharge machining variations, this becomes in addition showed its surface roughness lowered as the pulse-on time is raised. The surface roughness will grow in a manner that is proportionate to the increase in the pulse-off

time. There is the potential to manage ejections by increasing the pulse-on time. The pulse through gives the deepest point on the work components concentration, which in turn causes a rise in material that is both higher and more concentrated[33].

4. Conclusions

Stir casting was used to create AZ91D composites reinforced with B4C and ZrO₂ in this study. The composites that were made had a mixed microstructure that included reinforced particles as well as areas of solid solution and β -phase (intermetallics).

- The ductility was preserved while the hardness and tensile strength were enhanced by the addition of B4C and ZrO₂.
- Wear tests performed under dry sliding circumstances utilising a pin-on-disc machine revealed that the composites exhibited reduced frictional force and wear rate due to the reinforcement, which enhanced their mechanical behaviour.
- The hybrid composite containing ZrO₂ and B4C performed better than the base alloy and single phase composite. The findings highlight the potential benefits of using ZrO₂ and B4C as reinforcements in AZ91D to create composites with excellent performance.
- The pulse-off time, in conjunction with the voltage gap that exists between the tool and the workpieces, is the fundamental factors that must be considered while machining the MRR trail by wire feed. The bare minimum value that is need for this procedure is this one.
- Furthermore, in addition to the ZrO₂ and B4C reinforcements that are included into AZ91D, the rate at which material is being withdrawn is gradually decreasing.
- Due to the fact that wire electrical discharge machining (WEDM) has the capability to machine composite materials that are difficult to cut, the percentage of reinforcements in AZ91D increases as the surface roughness of the value increases. On the other hand, the percentage of reinforcements in MMCs stays constant regardless of the process rate of material removal.

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