Machining and corrosion studies on HfC reinforced ZE41 magnesium matrix composites

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Abstract

In this paper, Hafnium Carbide (HfC) reinforced ZE41 Magnesium Matrix Composites (MMCs) were prepared by using stir casting method. Using three different reinforcement percentages of HfC such as 5%, 10% and 15% by wt., ZE41-HfC MMCs were prepared. The mechanical characteristics of ZE41-HfC MMCs were evaluated by subjecting them to tensile and surface micro-hardness studies. Using X-Ray diffraction (XRD) studies, chemical compounds formed in the interfacial layer between HfC & ZE41 Mg was observed. Using optical microscopy (OM) and scanning electron microscopy (SEM), the surface modifications in the composites due to HfC addition was studied. Using electron backscatter diffraction analysis (EBSD), the changes in particle grain sizes and orientation of ZE41-HfC MMCs were studied. Energy Dispersive Spectroscopy (EDS) analysis was used to identify the variations in elemental composition of the prepared ZE41-HfC MMCs. ZE41-HfC MMCs were subjected to drilling studies for identifying the variations in cutting forces. Using electrochemical studies, the corrosion resistance of ZE41-HfC MMCs was observed. SEM images of corroded ZE41-HfC MMCs revealed micro cracks and dense pits near HfC agglomerated region.

Introduction

The demand for low weight components in manufacturing and automotive sectors has led many researchers to develop composite materials [1]. Magnesium Matrix Composites (MMCs) are a favorable option, which are prepared using magnesium (Mg) as matrix material, reinforced with Carbides or Ceramics [2]. Mg and its alloys have many advantages as they exhibit high strength, low density [3], better malleability [4], good castability [5] and excellent machinability [6]. Different reinforcements are being used for preparing Mg based MMCs such as carbon nano-tubes (CNT) [7], boron-carbide (B4C) [8], titanium-carbide (TiC) [9]. Hafnium carbide (HfC), which is a refractory material exhibits excellent characteristics such as high hardness (1670 HV) [10], appreciably good elastic modulus (450–460 GPa) [11], very high melting point (3950 °C)[12], making it a preferable reinforcement material. There are many constraints and challenges while preparing MMCs with reinforcements such as intermetalics formation [13] and brittleness of the composites [4]. These issues occur due to differences in the mechanical and chemical characteristics of the matrix and the reinforcement material [14]. Machining characteristics of MMCs are important as they can be used for identifying its usability in manufacturing sector. Further studies in machining aspects of MMCs are needed [15]. The forces generated during drilling can be used to identify the machining aspects of MMCs [16]. MMCs subjected to corrosion analysis helps in predicting their surface resistance, in volatile environment [17]. Electrochemical corrosion analysis helps to identify the effectiveness of reinforcements in MMCs [18] and MMC processing methods in enhancing corrosion resistance [19]. In this manuscript, stir cast MMCs were prepared by using ZE41 Mg alloy as matrix material and hafnium carbide (HfC) as reinforcements. The prepared ZE41-HfC MMCs were subjected to mechanical, machining and corrosion studies.
Materials & methods

In this investigation, Magnesium Matrix Composites (MMCs) were prepared by using ZE41 Magnesium (Mg) as matrix material. ZE41 Mg material was purchased from M/S. Met India Alloys Pvt. Ltd, Charni Road, East Mumbai, India. The chemical composition of ZE41 Mg material was evaluated by using spark spectrometer. The chemical composition of ZE41 Mg was identified by igniting sparks at different regions and the elements are shown in Table 1.

Figure 1 shows the SEM image of as-received ZE41 Mg material. Annealed grain structure was observed in figure 1. Equiaxed \( \alpha \)-Mg was found to be uniformly distributed [20]. Presence of eutectic compounds was also found along grain boundaries [21].

For reinforcement in MMCs, Hafnium Carbide (HfC) was used in this investigation. (99.9% pure) HfC (chemical compound of hafnium with carbon) micro powder was procured from M/S. Nano Research Elements, New Delhi, India. As it was highly pure, it was directly used in the experiments without further refinement. SEM image of as-procured HfC is shown in figure 2. The grain structure of HfC was found to be fine and homogeneous [22].

ZE41–HfC MMCs were prepared by using stir casting method [23]. According to the required proportions, appropriate quantity of ZE41 material & HfC powder was mixed in a steel crucible and heated up to 700 °C. The
mixture was heated till it turned semisolid. Then, the contents were stirred at 300 rpm for 20 min, to uniformly distribute the HfC powder in ZE41 matrix. Further, the temperature was increased to 950 °C and maintained at that temperature for 30 min. Then, the temperature was slowly decreased to 750 °C and stirred at 250 rpm for 10 min to completely disperse HfC in ZE41 Mg. Then, the molten mixture was poured into cylindrical moulds of length 300 mm and 30 mm diameter. To eliminate directional solidification, the moulds were pre-heated to a temperature of 450 °C. For minimizing oxidation and fire, the casting experiments were performed in argon atmosphere. Three sets of ZE41-HfC MMCs were prepared by varying the reinforcement percentage HfC at 5%, 10% & 15% by wt. For identifying the effect of HfC reinforcement, one sample was cast without adding HfC (0% HfC by wt.%). The prepared ZE41-HfC MMCs have been designated as shown in table 2.

Using electro-pneumatically controlled universal testing machine (Make—INSTRON, 30 kN capacity), tensile tests on ZE41-HfC MMCs were conducted. As per ASTM E 08 standards, tensile test samples were prepared with 25 mm gauge length, 6.25 mm width & 2.5 mm thickness. ZE41-HfC MMCs were subjected to tensile testing with cross head speed of 1 mm min⁻¹. Using vickers micro-hardness testing equipment (Make—QATM), surface micro-hardness of ZE41-HfC MMCs were evaluated. Micro-hardness experiments were conducted as per ASTM E 384 standards, using 20 kgf test load & 15 s dwell time. Using X-Ray Diffraction (XRD) studies, the compounds formed due to high temperature oxidation and the interaction between ZE41 & HfC was observed. XRD studies were conducted by using RIGAKU XRD equipment. Using copper target and with a step size of 0.002, scanning was done from 20° to 80° two theta.

For conducting microscopic studies on ZE41-HfC MMCs, standard metallographic procedures were followed. The surface of ZE41-HfC MMC specimens was polished by using different grades of emery sheets and using ultrasonic cleaning, the impurities in the surface was removed. Then, ZE41-HfC samples were subjected to electro-polishing at 15 V, at 25 °C, using A2C electrolyte. Microscopic evaluation of ZE41-HfC MMCs were done using Olympus-BX51 M optical microscope. Using Leads India make Scanning Electron Microscope, the surface modification in ZE41-HfC MMCs due to increase in reinforcement percentage was studied.

Using Carl Zeiss make Electron Backscatter Diffraction (EBSD) equipment, surface grain orientation and grain size of ZE41-HfC MMCs were evaluated. With a scanning region of 600 × 400 μm² & step size of 1 μm, EBSD studies were done. TSL OIM software was used for evaluating EBSD grain boundary maps. The average grain sizes of ZE41-BM, ZE41-05HfC, ZE41-10HfC & ZE41-15HfC composites were recorded. Elemental composition of ZE41 MMCs with different HfC reinforcements was studied using Energy Dispersive Spectroscopy (EDS). Drilling experiments were performed on ZE41-HfC MMCs for studying its machining properties.

Figure 3. Tensile test curves of ZE41-BM & ZE41-HfC MMCs.

| S NO | ZE41 Mg (wt.%) | HfC (wt. %) | Designation |
|-----|--------------|-----------|------------|
| 1   | 100          | 0         | ZE41-BM    |
| 2   | 95           | 5         | ZE41-05HfC |
| 3   | 90           | 10        | ZE41-10HfC |
| 4   | 85           | 15        | ZE41-15HfC |

Table 2. Prepared ZE41 Mg composites and their designation.
characteristics. Experiments were performed using 8 mm diameter twist drill, which was fixed in heavy type Vertical Milling Machine (Make—PHILLIPS). Using two different speeds (such 200 rpm & 400 rpm) and two different feed rates such as (20 mm min$^{-1}$ and 40 mm min$^{-1}$), drilling operations were performed. Dynamoseter (Make—Magtrol) was fixed in the bed of Vertical Milling CNC and the ZE41-HfC MMC samples were placed over that. Force measurement was started a few milliseconds before the tool came in contact with the surface of ZE41-HfC MMCs. Recording was done till stabilization of the cutting forces.

Using electrochemical testing equipment, corrosion tests were performed on ZE41-HfC MMCs. The corrosion test samples of ZE41-HfC MMCs were cleaned with ethanol before placing them in corrosion testing equipment. From previous literatures and investigations\cite{24–27} pertaining corrosion studies on reinforced metal matrix composites, 1 cm$^2$ surface area was chosen for investigation. For benchmarking and to identify the corrosion potential per cm$^2$ of the prepared ZE41-HfC MMCs, a surface area of 1 cm$^2$ was chosen for conducting potentiodynamic electro chemical corrosion testing experiments. Using rubber holders, 1 cm$^2$ surface of ZE41-HfC MMCs were subjected to corrosion. Using 3.5% conc. NaCl solution as electrolyte, graphite rod as counter electrode and saturated calomel electrode (SCE) as reference, the corrosion tests were conducted. After establishing an open circuit potential for 30 min, at scanning rate of 1 mV s$^{-1}$, corrosion experiments were performed. Potentio-dynamic polarization curves (PDP) were developed using Tafel exploration. The corroded surfaces were studied using SEM analysis for evaluation of the corroded surfaces.

**Results & discussion**

The tensile test curves of ZE41-BM & ZE41-HfC MMCs are shown in figure 3. The surface micro-hardness of ZE41-BM & ZE41-HfC MMCs are shown in figure 4. The tensile test results & surface micro-hardness values of ZE41-BM & ZE41-HfC MMCs are shown in table 3. Compared to ZE41-BM, ZE41-05HfC exhibited 39.2% increase in yield strength (YS), 63.8% increase in ultimate tensile strength (UTS), 16.34% increase in surface hardness & 3.5% decrease in elongation till fracture. Upon increasing the reinforcement % HfC in ZE41-HfC MMCs, elongation % till fracture decreased and surface micro hardness increased. For ZE41-10HfC, YS and UTS increased up to 64 MPa and 227 MPa respectively. On increasing the reinforcement percentage of HfC

| Composite | Yield Strength (YS) (MPa) | Ultimate Tensile Strength (UTS) (MPa) | Elongation % | Microhardness (HV) |
|-----------|---------------------------|---------------------------------------|--------------|--------------------|
| ZE41-BM   | 28 ± 2                    | 108 ± 2                               | 12.1 ± 0.2   | 55 ± 3.2           |
| ZE41-05HfC| 39 ± 3                    | 177 ± 3                               | 08.6 ± 0.3   | 64 ± 0.71          |
| ZE41-10HfC| 64 ± 2                    | 227 ± 3                               | 05.7 ± 0.6   | 69 ± 0.58          |
| ZE41-15HfC| 59 ± 3                    | 194 ± 4                               | 04.3 ± 0.5   | 82 ± 1.12          |

Figure 4. Surface microhardness of ZE41-BM & ZE41-HfC MMCs.

Table 3. Tensile characteristics of ZE41-HfC composites.
above 10%, excessive HfC in the matrix increased the brittleness & reduced the ductility of ZE41-15HfC. Compared with ZE41-10HfC, YS & UTS of ZE41-15HfC decreased by 7.81% & 14.53%.

The XRD spectrum of ZE41 HfC composites is shown in figure 5. The XRD graph of HfC powder is shown in figure 5(a), indicating the presence of HfC and C. The XRD spectrum of ZE41 Mg is shown in figure 5(b), indicating Mg and T phase particles. Figure 5(c) indicates the XRD spectrum of ZE41-05HfC MMC. Apart from Mg & HfC, presence of Mg2C3 was observed in figure 5(c) due to interaction between ZE41 & HfC at high temperature [28]. The XRD spectrum of ZE41-10HfC is shown in figure 5(d). Presence of C, Mg2C3, HfC & HfO2 was found. 10% by wt. reinforcement of HfC in ZE41 Mg resulted in oxidation of Hf during stir casting [29]. Figure 5(e) indicates the XRD spectrum of ZE41-15HfC. Apart from Mg & HfC, presence of Mg2C3, C, HfO2 was observed. 10% HfC reinforcement in ZE41 Mg resulted in creation of voids at certain regions, due to spatial inconsistency between HfC and ZE41 Mg. Increasing HfC reinforcement up to 15% in ZE41-HfC MMCs resulted in formation of voids which serve as crack initiation sites. The maximum temperature during stir casting was around 950 °C. At temperatures lower than 1000 °C, the kinetics of oxide formation depends upon diffusion of gases through the pores [30]. In HfC based systems, thermally induced stresses increase void fraction and oxidation rate. Oxide cracking at elevated temperature in HfC systems reduces the capability of oxides to form a protective coating [31]. Oxide formation begins upon substitution of one-fourth of carbon sites in hafnium carbide lattice [32]. During stir casting, ZE41-10HfC and ZE41-15HfC reacted with O2 present in the stir casting atmosphere to form HfO2 [33]. The oxidation reaction is indicated as follows

\[
HfC + 2O_2 \rightarrow HfO_2 + CO_2.
\]  

(1)

Optical microscopic (OM) images of ZE41-BM & ZE41-HfC composites are shown in figure 6. Stirring and heating of ZE41-BM material resulted in annealing of Mg grains, which is shown in figure 6(a). Solidification of the stirred ZE41 material resulted in transformation of its gross morphology [34]. Figure 6(b) indicates the OM image of ZE41-05HfC, in which HfC was found to be distributed as agglomerated layers. Non-uniform distribution of HfC in ZE41 Mg matrix was observed. Figure 6(c) indicates the OM image of ZE41-10HfC. As Mg has hcp crystal structure; its interaction with HfC resulted in deformation twinning, observed as short and straight lines [35].
OM image of ZE41-15HfC is shown in figure 6(d). Deformation of HfC was marginally higher. Dispersion of HfC was more due to its two-dimensional structure. During solidification, formation of interdendritic HfC particles occurred [36]. They interacted with pre-eutectic blocky HfC particles which were formed prior to solidification [37]. While preparing ZE41–HfC MMCs in stir casting furnace, at high temperature stirring, pre-eutectic blocky HfC particles were pushed to the periphery. Higher HfC content and difference in the interconnected carbide network [38] of interdendritic HfC and pre-eutectic blocky HfC particles resulted in deformation (ZE41-15HfC).

Micro structural investigations on ZE41–HfC composites, using Scanning Electron Microscopy (SEM) is shown in figure 7. SEM image of ZE41–BM is shown in figure 7(a). Grain distribution was observed to be fine, with annealed Mg particles. SEM image of ZE41-05HfC is shown in figure 7(b). Homogeneous distribution of reduced HfC particles was observed. Dislocation results in formation of dimples in the surface [40]. Figure 7(c) shows SEM image of ZE41-10HfC MMC. Agglomeration of HfC particles was observed in ZE41-10HfC, on increasing HfC substitution up to 10% by weight. Density of ZE41–HfC MMCs varied upon adding HfC. This resulted in deformation of Mg matrix structure. SEM image of ZE41-15HfC indicated porosity (figure 7(d)). Porosity of ZE41–15HfC was greater than those Mg ZE41 MMCs with 5% and 10% reinforcements.

EBSD grain boundary maps of ZE41–HfC MMCs is shown in figure 8. During stir casting, ZE41 Mg with HfC was heated to elevated temperatures and stirred before it was poured in moulds for solidification. Post processing of stir cast ZE41 Mg with HfC composite samples was not done. Hence EBSD results indicate the surface of MMCs with various concentrations of HfC reinforcements. EBSD map of ZE41–BM indicates fine grained structure (figure 8(a)). Elevated temperatures during stir casting process and strain created in reinforcement particles while solidification induced dynamic recrystallization [41].

EBSD map of ZE41-05HfC MMC samples indicates nucleation (figure 8(b)). As the stir casting temperature was between 700 °C and 900 °C, interaction between ZE41 Mg and HfC particles resulted in particle simulated nucleation. This caused grain refinement in ZE41-05HfC MMCs. EBSD map of ZE41-10HfC is shown in figure 8(c). Grain orientation was found to be random [42]. EBSD maps of ZE41-15HfC are shown in figure 8(d). Basal plane slips of Mg alloys (having hcp structure) with HfC (having cfc cubic crystal system) was found to be high.
The Grain size distribution graphs of ZE41-HfC composites, corresponding to their EBSD grain boundary maps is shown in figure 9. Average grain size of ZE41-BM was found to be 29.3 \( \mu \text{m} \) (annealed without addition of reinforcements) (figure 9(a)). Upon reinforcing HfC up to 5\% by wt., increased densification of reinforcements resulted in enlargement of grains. For ZE41-05-HfC, an average grain size of 47.4 \( \mu \text{m} \) was observed (figure 9(b)). 10\% reinforcement of HfC further increased the average grain size of ZE41-15HfC to 58.7 \( \mu \text{m} \) (figure 9(c)). Increasing the reinforcement of HfC to 15\% by wt., in ZE 41 Mg composites, the average grain size increased to 76.4 \( \mu \text{m} \) (figure 9(d)). Compaction between Mg & reinforcement particles resulted in increase of average grain size. Higher distribution of reinforcement particles induces dynamic recrystallization [43]. The fraction of low angle boundaries in ZE41-15HfC was observed to be more than those observed in ZE41-10HfC grain boundary maps. Hence, the effect of dynamic recrystallization was more pronounced in ZE41-10HfC than ZE41-15HfC.

For finding the internal chemistry of HfC reinforced ZE41 composites, they were subjected to Energy Dispersive Spectroscopy (EDS) analysis. EDS analysis of ZE41-HfC composites is shown in figures 10(a)–(d). Figure 10(a) indicates the EDS spectrum of ZE41-BM. Apart from Mg and Zn, rare earth metals such as Nd & Gd was found. Figure 10(b) shows the EDS spectrum of ZE41-05HfC. Presence of Hf (2.13\%), C (1.973\%) & traces of O was found, apart from Mg, Nd & Gd. EDS spectrum of ZE41-10 HfC is shown in figure 10(c). Increase in Hf reinforcement from 5\% to 10\% by wt., caused increase in Hf from 2.13\% to 4.08\% & C from 1.973\% to 3.86\%. EDS spectrum of ZE41- 15HfC is shown in figure 10(d). On increasing HfC quantity from 10\% to 15\% in ZE41 MMCs, further increase in Hf from 4.08\% to 6.32\% & C from 3.86\% to 5.75\% was observed.

The cutting force profiles during drilling experiments on ZE41 Mg reinforced with HfC at different wt. percentages (0\%, 5\%, 10\%, 15\%) is been shown in figures 11(a)–(d). Figure 11(a) indicates the cutting force versus time profile of ZE41- HfC composites at 200 rpm drilling tool speed and feed rate of 20 mm min\(^{-1}\). Relatively higher cutting force was exhibited throughout. Upon contact with the drill tool and specimen, exponential increase in cutting forces was observed for a few seconds and then it was stabilized. ZE41-05HfC & ZE41-10HfC exhibited lower cutting forces. Cutting forces increased upon drilling ZE41-15HfC due to excessive reinforcements. Figure 11(b) indicates cutting force profiles of ZE41 Mg-HfC composites at 200 rpm drilling speed and 40 mm min\(^{-1}\) feed rate. For all four combinations, the fluctuations in cutting forces were lower than the experiments which were conducted with 20 mm min\(^{-1}\) feed rate. Slow feed rate increased

![SEM micrographs of ZE41-HfC MMCs.](image)
vibration distortion, which increased the cutting force fluctuations to a greater extent. Figure 11(c) indicates the cutting force graphs at 450 rpm drilling speed and 20 mm min⁻¹ feed rate. Figure 11(d) shows the cutting force graphs at 450 rpm drilling speed and 40 mm min⁻¹ feed rate. Substantial reduction in cutting forces was observed throughout the drilling time, on adding HfC, as they acted as solid lubricant. On increasing the reinforcements beyond 10% by wt., excessive HfC particles created thermo-mechanical stir, thereby increasing the cutting forces. During machining, the dislocation density generated at the interface between reinforcements and matrix material plays an important role in reducing cutting forces [44].

Reduction in cutting forces was observed due to thermal mismatch induced due dislocation densities between HfC reinforcement and Mg matrix [45]. HfC reinforcements acted as solid lubricant during machining, resulting in cutting force mechanism. Similar reduction in cutting forces was observed in metal matrix composites upon using SiC & RHA [46], TiC [47], TiB₂ [48]. Potentio dynamic polarization (PDP) curves developed from the electrochemical corrosion experiments is shown in figure 12. The pitting corrosion parameters recorded for all the four samples of ZE41 Mg MMCs is shown in table 4.

Corrosion resistance increased on incorporating HfC reinforcements. 5% by wt. HfC reinforcement made the corrosion potential E_corr (V) to shift towards positive, from −2.75 (V) to −2.30 (V). Till 10% by wt. reinforcement of HfC, corrosion resistance increased and the shift was positive (−2.07 V). ZE41-15HfC exhibited negative shift of E_corr value (−2020 V). Corrosion resistance reduced on increasing HfC beyond 10% by wt.

The corrosion tested ZE41-HfC MMCs were subjected to SEM evaluation. SEM images of corroded ZE41-HfC MMCs are shown in figure 13. Figure 13(a) shows SEM image of corrosion tested ZE41-BM. Corroded Mg regions were observed. SEM image of corroded ZE41-05HfC MMC is shown in figure 13(b). Occurrence of corrosion pits near HfC particles was observed. SEM image of corroded ZE41-10HfC MMC is shown in figure 13(c). Microcracks [49] were observed near HfC agglomerated regions. SEM image of corrosion tested ZE41-15HfC has been shown in figure 13(d). Relatively denser pits near HfC cultures were observed. These micro cracks act as crack initiation regions, which reduced the quality of the surfaces. Large and deep corrosion pits were observed in ZE41 Mg BM sample. HfC reinforcement interfered with pits and flakes formation. Bonding between matrix and reinforcements inhibits corrosion [50]. Increase in HfC reinforcements to 5% and 10% by wt., microstructure of the composites were altered. Interaction between HfC and ZE41 Mg inhibited corrosion propagation [51]. Inert and non-reactive nature of reinforcement enhanced corrosion resistance [52].
This caused limiting current tendency. Upon increasing HfC reinforcement to 15% by wt., excessive HfC in Mg matrix enhanced susceptibility of the composite surface to corrosion. It created pits and micro blisters during corrosion tests [53].

Strengthening mechanisms of HfC in Mg matrix has to be studied for predicting the strength aspects of HfC reinforced ZE41 Mg MMCs. For metal matrix composites, researchers have proposed different mechanisms to identify variations in yield strength of the composites to reinforcements [54–57].

In this study, strengthening mechanisms in ZE41 Mg-HfC MMCs have been related with four factors such as coefficients of thermal expansion mismatch strengthening, load transfer strengthening, Orowan strengthening & Hall Petch mechanism. As observed in average grain size distribution histograms, an increase in average grain size was observed upon increasing HfC concentration in ZE41 Mg matrix.

Addition of HfC influenced the mechanical properties of the prepared ZE41-HfC composites. Increase in HfC % in ZE41-HfC MMCs increased surface hardness. Till 10% HfC reinforcement, tensile properties of ZE41-HfC increased. Upon increasing HfC reinforcement to 15%, tensile properties reduced. The contribution to composite strength due to variations in grain size was calculated using Hall-Petch equation as follows

\[
\Delta \sigma_{\text{Hall}} = \frac{k_{\text{Hall}}}{D^{1/2}}
\]

In the above equation, \(\Delta \sigma_{\text{Hall}}\) is the Hall-Petch composite strength due to variations in grain size, \(k_{\text{Hall}}\) is Hall-Petch constant (\(\approx 0.067\) MPa m\(^{1/2}\)) [58]) and D is the mean grain size of the Matrix. Depending of the number of slip systems in Mg matrix, \(k_{\text{Hall}}\) value varies. \(k_{\text{Hall}}\) for hexagonal closed-packed structure is more than body-centered cubic structure metals and face-centred cubic structure metals. As Mg exhibits hcp crystal structure, the strength of its composites depends on its grain size [39].
Figure 10. EDS evaluation of ZE41-HfC Magnesium Matrix Composites.

(a) ZE41-BM

(b) ZE41-0.5HfC

(c) ZE41-1HfC

(d) ZE41-15HfC
Orowan looping is important in identifying the restricted movement of dislocations in the reinforced MMCs on incorporating reinforcement particles [60]. For calculating Orowan strengthening ($\Delta\sigma_{\text{Orowan}}$), assuming spherical HfC reinforcement particles and uniform distribution throughout the Mg matrix, the following equation was used [61, 62].

(a) Tool speed 250 rpm & feed rate 20 mm/min
(b) Tool speed 250 rpm & feed rate 40 mm/min
(c) Tool speed 450 rpm & feed rate 20 mm/min
(d) Tool speed 450 rpm & feed rate 40 mm/min

**Figure 11.** Cutting force versus time profile for ZE41-HfC MMCs at various drilling parameters.

**Figure 12.** PDP curves from electrochemical corrosion tests conducted on ZE41-HfC MMCs.
In the above equation, $T$ is Taylor factor ($2.4$ [63]), $S$ denotes shear modulus ($17$ GPa [64]), $b$ denotes burgers vector ($0.612$ nm [65]), $m$ is Poisson ratio ($0.28$ [66]), $d_r$ is the diameter of HfC particles and $f_r$ is the volume fraction of HfC particles. Orowan mechanism indicates strengthening when reinforcement particles exist within the grain interiors [67]. Occurrence of HfC reinforcements along grain boundaries attribute to the deviation between the theoretical $\Delta \sigma_{\text{Orowan}}$ and actual yield strength of ZE41-HfC MMCs.

Variations in load transfer ability of ZE41 Mg composites was observed on increasing HfC reinforcements. Volume fraction of reinforcement particles affects load transfer mechanism, thereby modifying the strength of composites [68]. Contribution of reinforcement particle to strengthening ($\Delta \sigma_{\text{load}}$) by load transfer mechanism was estimated using the following equation [69]

$$\Delta \sigma_{\text{load}} = 0.5f_r \sigma_{\text{ym}}$$

In the above equation $\sigma_{\text{ym}}$ indicates the yield strength of ZE41 Mg matrix ($28$ MPa). For spherical reinforcements, effect of load transfer reinforcement is lower than orowan strengthening mechanisms [70]. The wide difference between elastic modulus and thermal expansion coefficients of ZE41 Mg matrix and HfC reinforcements is responsible for generation of geometrically necessary dislocations (GND) in the matrix [71].

### Table 4. Electrochemical pitting test parameters recorded upon testing ZE41-HfC MMCs.

| Specimens   | Corrosion potential, $E_{\text{corr}}$ (V) | Corrosion current, $I_{\text{corr}}$ (A cm$^{-2}$) |
|-------------|------------------------------------------|---------------------------------------------------|
| ZE41-BM     | $-2.76 \pm 0.031$                       | $1.51 \pm 1.10 \times 10^{-4}$                   |
| ZE41-05HfC  | $-2.30 \pm 0.021$                       | $1.23 \pm 0.71 \times 10^{-4}$                   |
| ZE41-10HfC  | $-2.07 \pm 0.063$                       | $3.50 \pm 0.17 \times 10^{-3}$                   |
| ZE41-15HfC  | $-2.20 \pm 0.043$                       | $2.90 \pm 0.32 \times 10^{-3}$                   |

In the above equation, $\Delta \sigma_{\text{Orowan}}$ is given by

$$\Delta \sigma_{\text{Orowan}} = \frac{0.81 T S b \ln(d_r / b)}{2\pi \sqrt{1 - \frac{1}{2} \frac{d_r \left(3\pi - d_r \sqrt{2f_r - d_r}\right)}{d_r \sqrt{2f_r - d_r}}}}$$

Figure 13. SEM images of corrosion tested ZE41-HfC MMCs.
The distorted geometrical features of the HfC particles act as stress transferring medium [72], inhibiting crack formation.

Delay in yield deformation associated with yield strength of the composites [73] occurred upon increasing the reinforcement percentage of HfC till 10%, by wt. Beyond 10% reinforcement, bigger particles induce modifications in composite strength due to elastic modulus mismatch to a greater extent than smaller reinforcements [70]. It attributes to the increase in yield strength of ZE41-HfC composites upon increase in grain particle size till 10% HfC reinforcement. Due to mismatch in coefficient of thermal expansion between ZE41 Mg (26 × 10^{-6} K^{-1} [74]) matrix and HfC (6.6 × 10^{-6} K^{-1} [75]) reinforcements, dislocations generate upon cooling the composites.

Increase in yield stress due to mismatch between the coefficient of thermal expansion of ZE41 Mg and HfC (\(\Delta \sigma_{CTE}\)) was calculated from the following equation [76]

\[
\Delta \sigma_{CTE} = \alpha Sh \left( \frac{12 \Delta T}{b \left( \frac{\Delta C}{d_r} \right)} \right)^{1/2}
\]

\(\alpha\) indicates constant (1.25 [77]). Difference between the testing and processing temperature is indicated as \(\Delta T\). \(\Delta C\) indicates the difference in coefficient of thermal expansion between ZE41 Mg and HfC. The theoretical yield strength of the ZE41-HfC composites (\(\sigma_{theoretical}\)) was estimated using the following equation

\[
\sigma_{theoretical} = \sigma_{ym} + \Delta \sigma_{Hall} + \Delta \sigma_{Orowan} + \Delta \sigma_{Load} + \Delta \sigma_{CTE}
\]

The contribution of all the four mechanisms for varying the yield strength is shown in figure 14 for various HfC reinforcement percentages. The same sized HfC reinforcements were used for preparing the three composites.

Even though, orowan strengthening was less significant in micron sized reinforcements [78], enhanced interparticle spacing between HfC particles inhibit crack formation in interphase region. It increased yield strength of composites even though grain size increased till 10% HfC reinforcement. 15% HfC in Mg matrix resulted in high interaction with dislocations. As resistance of HfC reinforcement particles reduced in passing of

![Figure 14. Contribution of each mechanism to the improvement of yield strength for different HfC reinforcements and corresponding experimental and theoretical yield strength of ZE41-HfC composites.](image-url)
dislocations [79], yield strength reduced. Increase in theoretical yield strength of ZE41-HfC composites due to load transfer mechanism was observed. Contribution due to load transfer mechanism was proportional to the volume fraction of HfC reinforcements.

Contribution due to coefficient of thermal expansion strengthening mechanism reduced upon increasing the reinforcement percentage of HfC. Inconsistency in strength variations due to mismatch in coefficient of thermal expansion of matrix and reinforcements was observed [80]. Increase in internal stresses occurs upon increasing the size of reinforcements [63]. At same particle size, when volume fraction increases, addition of reinforcement micro particles aids in formation of dislocations. Micron sized reinforcements induce critical misfit strain thereby generating dislocations. [81].

Hence, the contribution of coefficient of thermal expansion strengthening mechanism reduced on increasing the volume fraction.

Contribution of each mechanism to the improvement of yield strength for different HfC reinforcements and corresponding experimental and theoretical yield strength of ZE41-HfC composites is shown in figure 14. As it was assumed that the HfC reinforcement distribution was homogeneous (in orowan mechanism), difference between theoretical and experimental yield strength was observed. This difference is attributed to deviations in homogeneous distribution of HfC particles in Mg matrix during solidification of the composites. Presence of HfC micro particles in grain interiors, along boundaries, partly homogenous distribution and reinforcement particle size induces variations in the predicted and actual yield strength of the ZE41-HfC composites [82, 83].

Conclusions

Hence, in this paper, HfC reinforced ZE41 Magnesium Matrix Composites were prepared, subjected to mechanical, machining and corrosion studies and the following conclusion were drawn.

i. Tensile strength increased from 108 MPa (UTS of ZE41-BM) to 227 MPa (UTS of ZE41-10HfC), upon increasing HfC reinforcement to 10% by wt. Beyond 10% HfC reinforcement, UTS of ZE41-15HfC reduced to 194 MPa. Surface micro-hardness was found to increase from 55 HV (ZE41-BM) to 82 HV (ZE41-15HfC), upon increasing HfC reinforcement.

ii. XRD analysis of ZE41-HfC indicates Mg, T phase particles, HfC, Mg2C3, C & HfO2. OM images of ZE41-HfC indicates annealed of Mg, agglomerated HfC in Mg matrix, HfC twinning. SEM analysis revealed homogeneous distribution of HfC in ZE41 Mg. Dislocation line between Mg and HfC particles were found with HfC agglomeration.

iii. EBSD grain boundary maps indicated fine grains due to dynamic recrystallization, particle simulated nucleation with basal plane slips. Average particle grain size increased (29.3 μm to 76.4 μm) upon increasing HfC reinforcement.

iv. EDS analysis showed Mg, Zn, Hf, C & rare earth metals. Hf increased from 2.13% to 6.32% & C increased from 1.973% to 5.75% upon increasing HfC reinforcement from 5 to 15%. Cutting force graphs showed appreciable reduction in cutting forces upon reinforcing HfC till 10%. Beyond 10% reinforcement of HfC, cutting forces increased.

v. Potentio-dynamic polarization (PDP) curves indicated enhanced corrosion resistance of ZE41-HfC MMCs till 10% by wt. addition of reinforcements (−2.76 V for ZE41-BM to −2.07 V for ZE41-10HfC). Beyond 10% reinforcement of HfC by wt. %, excessive disorientations decreased corrosion resistance.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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