

OPTIMIZATION OF TRIBOLOGICAL PROPERTIES OF AN EPOXY HYBRID POLYMER COMPOSITE REINFORCED WITH ZrB_2 AND PTFE PARTICLES USING RESPONSE SURFACE METHODOLOGY FOR HIGH-TEMPERATURE TRIBOLOGICAL APPLICATIONS

OPTIMIZACIJA TRIBOLOŠKIH LASTNOSTI EPOKSIDNEGA HIBRIDNEGA POLIMERNEGA KOMPOZITA, OJAČANEGA S ZrB_2 IN PTFE DELCI IN UPORABO METODOLOGIJE POVRŠINSKEGA ODGOVORA ZA VISOKO-TEMPERATURNE TRIBOLOŠKE APLIKACIJE

Aswathi A. Narayanan*, R. S. Sudheesh

Department of Mechanical Engineering, APJ Abdul Kalam Technological University, Government Engineering College, Thrissur 680009, India

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Hybrid PTFE/epoxy composites are widely used as materials for self-lubricating spherical bearings, used in a high-temperature environment. In the present work, zirconium diboride (ZrB_2) particles are incorporated to enhance the high-temperature tribological properties of PTFE/epoxy composites. A pin-on-disc experiment is conducted with the aid of design of experiments (DOE) using the central composite design/response surface methodology (RSM). Under a load of 40 N and at a sliding speed of 1.25 m/s, optimum contents of 5.95 volume percent (ϕ %) of PTFE and 5.05 ϕ % of ZrB_2 yield an ultralow coefficient of friction (COF) in conjunction with a low wear rate of the composite. The addition of ultra-high-temperature ceramic ZrB_2 particles and solid lubricant PTFE is found to enhance the thermal conductivity and improve the heat transfer, thereby reducing the contact temperature. The optimum composition of the composite can reduce the wear rate and high local temperature due to friction, implying its potential use as a self-lubricating spherical-bearing liner material.

Keywords: zirconium diboride, composite, surface, bearing, liner

Hibridni kompoziti na osnovi politetrafluoretilena in epoksidnih smol (PTFE/epoksi) se pogosto uporabljajo kot materiali za samomazalne sferične ležaje v visokotemperaturnem okolju. V članku je predstavljena vgradnja cirkonij-diboridnih delcev (ZrB_2) v matrico iz PTFE/epoksi za izboljšanje triboloških lastnosti kompozitnega materiala pri povišanih temperaturah. S pomočjo dizajniranih eksperimentov (DOE) so bili izvedeni preizkusi obrabe materiala na vrtečem se disku s trnom (angl.: pin on disc experiment) in metodologija centralnega odgovora površine kompozita (RSM; angl.: central composite-response surface methodology). Ultra nizek koeficient trenja in zelo majhna obraba izdelanega kompozita sta bila dosežena pri optimalni vsebnosti PTFE 5,95 volumskih % (ϕ %) in 5,05 ϕ % ZrB_2 delcev za obremenitev trna 40 N ter hitrosti drsenja 1,25 m/s. Dodatek ultra visoko temperaturno obstojnih keramičnih delcev ZrB_2 v trdnem mazivu PTFE, je povzročilo izboljšanje toplotne prevodnosti kompozita in izboljšanje prenosa toplote, kar je znižalo tudi temperaturo na stičnih površinah. Uporaba optimalne sestave kompozita lahko zaradi trenja zmanjša obrabo materiala in zniža lokalno temperaturo, kar pomeni, da bi bil lahko ta material potencialno uporaben kot material za samomazalne sferične ležaje.

Ključne besede: cirkonijev diborid, kompozit, površina, samomazalni sferični ležaji

1 INTRODUCTION

Epoxy resin is a commonly used matrix phase for polymer-based composites due to its good elasticity, abrasive resistance and lightweight characteristics. Hybrid PTFE/epoxy composites are suitable for self-lubricating spherical plain bearings, used in aircraft landing gears, railway locomotives and shock-absorption systems.¹ The main disadvantage of this type of bearing is the wear of the bearing liner. PTFE fabric liner is extensively used as a self-lubricating liner of spherical plain bearings.² PTFE often leads to a decrease in the friction coefficient and wear rate through a reduction in the adhesion with the counter face or creation of a transfer film

with a low shear strength at the interface.³ Poor mechanical strength and excessive viscoelastic deformation of PTFE affect the service life of the bearing liner, as bearings are used in high-temperature environments. One of the present-day aims of the study of these composites is to enhance their high-temperature tribological properties.

The introduction of ceramic fillers, such as SiC, Al_2O_3 and Si_3N_4 with high wear resistance and thermal conductivity, is an effective method of improving the tribological and thermal performance of polymeric composites used under high-load conditions.⁴ Shen et al. investigated the tribological performance of epoxy composites filled with PTFE and SiO_2 particles. Tribo-tests confirmed that the composite exhibits a low COF and a low wear rate implying its potential use as a self-lubricating liner material.⁵ ZrB_2 is one of the most important

*Corresponding author's e-mail:
Aswathiii333@gmail.com (Aswathi A. Narayanan)

ultra-high-temperature ceramics with a high elastic modulus, high strength, excellent wear resistance and high thermal conductivity. Yang et al. incorporated ZrB₂ particles in a hybrid PTFE/nomex fabric/phenolic composite and observed an improvement in its high-temperature tribological properties.⁶ Yu et al. studied the influence of ZrB₂ on the tribological performance of epoxy composites.⁷

During sliding, frictional heat and the resulting contact temperature determine the tribological performance of polymer composites. A low thermal conductivity usually leads to an accumulation of frictional heat, which degrades the organic ingredient. This affects the properties and functionality of the composites. A high thermal conductivity can improve the heat transfer. Hence, a low friction coefficient and a relatively high thermal conductivity are required to achieve good performance.⁸

Response surface methodology (RSM) is nowadays a widely used statistical tool for process optimization through a relatively small number of systematic experiments that can reduce time, cost and resources. It has always been a challenging task to optimize multi-response characteristics as it includes a prolonged computation like in traditional methods. In the last two or three decades, the use of design of experiment (DOE) has grown rapidly and has been adapted for many applications in different areas.⁹ Desirability function analysis (DFA) was introduced by Derringer and Suich (1980). DFA can solve a multi-response optimization problem by converting it into a single-response optimization problem, which enables us to reduce the computational work.¹⁰

In this work, an epoxy hybrid polymer composite reinforced with micro-sized ZrB₂ and PTFE particles was prepared to enhance the thermal and tribological properties. The RSM with a central composite face-centered design is used to study the effect of filler contents on the hardness, wear rate, and coefficient of friction. To the best of the authors' knowledge, there are no reported works on ZrB₂ filled PTFE reinforced polymer composites that could help us to improve the tribological and thermal performances, implying the significance of this work. The present work aims to achieve a material with a low friction coefficient and high thermal conductivity by reducing frictional heat accumulation – a viable alternative material for a self-lubricating spherical-bearing liner over a conventional material.

2 EXPERIMENTAL DETAILS

2.1 Material

Bisphenol-A-based epoxy resin (Lapox L12), a medium-viscous semi-solid polymer with a density of 1100 kg/m³ was used as the matrix and K-6 aliphatic polyamine was the curing agent. The reinforcement materials used here are zirconium diboride (ZrB₂) and polytetrafluoroethylene (PTFE). Properties of the reinforcements are given in Table 1.

Table 1: Properties of reinforcements

Material	Size (μm)	Density (kg/m ³)
ZrB ₂	1–3	6080
PTFE	10–12	2200

2.2 Preparation of the epoxy hybrid composite

Initially, reinforcements ZrB₂ and PTFE of required weights for each composition were preheated to 120 °C in a muffle furnace to remove the moisture. The reinforcements were added to the matrix and mixed continuously using an ultrasonic mixer for 30 min. One mL of curing agent was added and hand-stirred gently to avoid trapping bubbles and the mixture was then poured into molds. The specimens were allowed to cure in the molds for 24 h at room temperature. The molds were prepared as per the specimen standards for testing. The molded samples were then polished with silicon carbide abrasive papers up to the 1000 grade. The surface of the polished composite was rinsed with distilled water prior to tribo-tests.

2.3 Characterization

A tensile and compressive test of the composites was conducted using a computerized universal testing machine with a crosshead speed of 1 mm/min. Test samples were prepared as per ASTM E4 Standard. For each composition, five tests were conducted and the average value was taken. The hardness of the composites and the matrix polymer was measured using a Rockwell hardness tester. Cubes of 20 mm were used for the testing. Indentation was made using a steel ball indenter of 6.35 mm in diameter with a pre-load of 10 kg and an applied load of 60 kg for 5 s (the dwell time).

Tribological characterization of the epoxy hybrid composites and the matrix polymer was conducted using a pin-on-disc tribometer (DUCOM TR-20-CH 600). The cast composites were machined to a size of 7.5 mm in diameter and 30 mm in length (ASTM G99 standard). The pin-on-disc tribometer, with EN31 hardened to a 60-HRC disc with a diameter of 150 mm, was used for wear testing. The test parameters, namely the sliding speed, load, contact pressure and sliding distance were kept constant at 1.25 m/s, 40 N, 0.91 MPa and 750 m, respectively. The test included 3000 repetitions at 299 min⁻¹ and a track diameter of 80 mm. The study employed a Hot Disk TPS 500 thermal constants analyzer to evaluate the thermal conductivity of the composite. The contact temperature on the wear track was measured using a FLUKE thermal infrared imager. All the tests were performed at room temperature (22 ± 2) °C. The microstructure study of the composite was done with a ZEISS optical microscope (AXIO Vert. A1.0) and scanning electron microscope F50.

3 FIXING THE WORKING RANGE, CODING THE VARIABLES AND SETTING UP THE DOE MATRIX

The RSM approach was employed to study the impact of two independent variables on the hardness, wear rate and COF of the epoxy hybrid composites. The independent variables that were chosen were ZrB₂ φ/% (volume %) and PTFE φ/%, while the responses were the hardness, wear rate and COF. A large number of initial trial runs were carried out to achieve the required range and span of variables for the DOE. The trials were performed by varying one variable at a time and keeping the other variables constant. The samples produced in this way were visually inspected, and the lowest and highest values of the variable under study, which produced externally defect free samples, were identified and set as the range of that variable. Typical defect-free samples include 3–7 φ/% of ZrB₂ and 3–9 φ/% of PTFE. The actual and coded levels of the variables used for the experimental study are given in Table 2.

Table 2: Actual and coded levels of the variables

Variable (factor)	Symbol	Level		
		-1	0	1
ZrB ₂ (φ/%)	A	3	5	7
PTFE (φ/%)	B	3	6	9

The experiments were designed according to the face-centered design of the central composite method of RSM, providing relatively high-quality predictions over the entire design space.

4 DEVELOPMENT OF EMPIRICAL MODELS AND OPTIMIZATION

Response surface regression was used to construct a complete quadratic mathematical equation for the hardness, wear rate and COF of the epoxy hybrid composites. Each model was subjected to an analysis of variance (ANOVA). The designs were matched with the quadratic polynomial Equation (1):

$$Y = b_0 + \sum_{i=1}^2 b_i X_i + \sum_{i=1}^2 b_{ii} X_i^2 + \sum_{j=2}^2 b_{ij} X_i X_j \quad (1)$$

where Y is the response variable (hardness, wear rate or coefficient of friction), b_0 is a constant, b_i is the linear coefficient, b_{ii} is the quadratic coefficient, b_{ij} is the interaction coefficient, and X_i and X_j are the dimensionless coded independent variables. Equation (2) is applied to convert the independent variables of the regression model into dimensionless coded variables:

$$X_i = \frac{x_i - x_0}{\Delta x_i} \quad (2)$$

where X_i and x_i are the coded and actual values of the independent variables, respectively; x_0 is the actual value of the independent variables at the central point; Δx_i is the step-change value of the low and high levels of x_i . Design-Expert 13, a statistical software from Stat-Ease, Inc., specifically dedicated to performing the design of experiments (DOE) for developing mathematical models and optimization processes, was used to generate the response surface plots and contour plots.

The optimization of multi-response characteristics was achieved using the desirability function approach, which is one of the widely used methods in industries. In accordance with its general methodology, a response is first converted into an individual desirability function (d_i) that varies from 0 to 1 (the lowest desirability to the highest desirability). The individual-desirability scores for the predicted values for each dependent variable (hardness, wear rate and coefficient of friction) are then combined into an overall desirability function, D , which can be calculated by using Equation (3).

$$D = \left(\sum_{i=1}^n d_i^{w_i} \right)^{1/\sum w_i} \quad (3)$$

where w_i is the response weight. A higher composite-desirability value implies better product quality. Therefore, on the basis of the composite desirability (D), the parameter effect and the optimum level for each controllable parameter can be estimated. Depending on whether a particular response y_i is to be maximized or minimized, different desirability functions (d_i) can be used.

For the maximization of hardness, the individual desirability index (d_i) for the corresponding responses is calculated using Equation (4)

$$d_i = \begin{cases} 0, & y_i \leq y_{\min} \\ \left(\frac{y_i - y_{\min}}{y_{\max} - y_{\min}} \right)^r, & y_{\min} \leq y_i \leq y_{\max}, r \geq 0 \\ 0, & y_i \geq y_{\max} \end{cases} \quad (4)$$

For the minimization of hardness and wear rate, the individual desirability index (d_i) for the corresponding responses is calculated using Equation (5)

$$d_i = \begin{cases} 1, & y_i \leq y_{\min} \\ \left(\frac{y_i - y_{\max}}{y_{\min} - y_{\max}} \right)^r, & y_{\min} \leq y_i \leq y_{\max}, r \geq 0 \\ 0, & y_i \geq y_{\max} \end{cases} \quad (5)$$

where y_i is the response value, y_{\min} is the minimum acceptable value for response i , y_{\max} is the maximum acceptable value for response i , and r is the weight used to determine the scale of desirability.

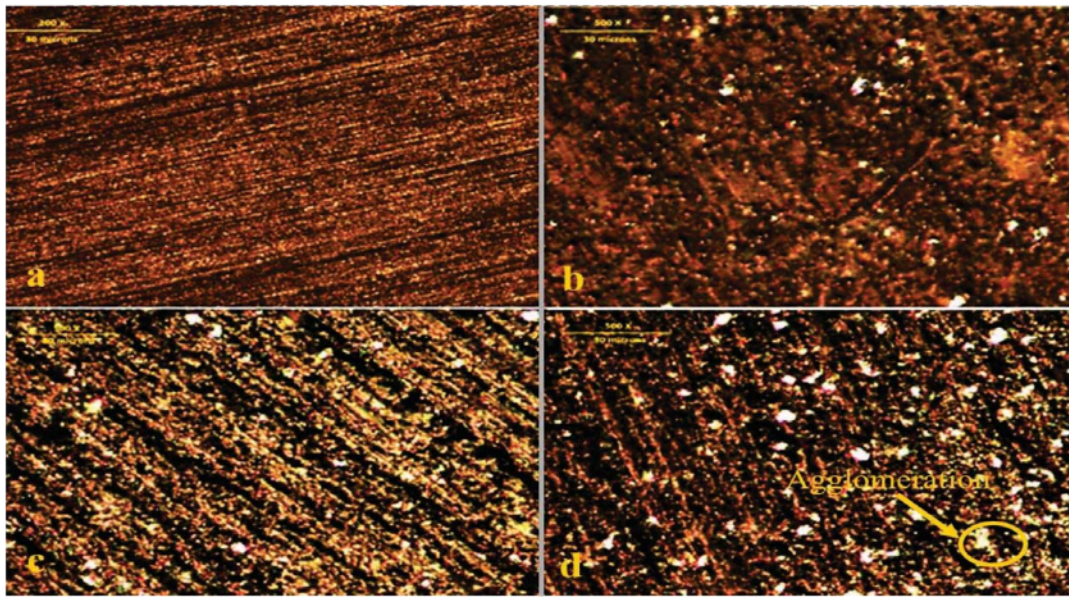


Figure 1: Optical images of composite materials before the wear test: a) bare epoxy, b) 3 φ1% PTFE + 3 φ1% ZrB₂, c) 6 φ1% PTFE + 5 φ1% ZrB₂, d) 9 φ1% PTFE + 7 φ1% ZrB₂

5 RESULTS AND DISCUSSION

5.1 Mechanical and metallographic properties of composites

Figure 1 shows optical images (500×) of composites of different compositions before carrying out the experiment. Reinforcement particles are clearly visible in the images. Particles are uniformly distributed in the composite and agglomeration is less than 10 %.

Figure 2 shows the mechanical properties of epoxy hybrid composites. The tensile strength and compressive strength are high compared to the bare epoxy matrix due to the presence of the ZrB₂ particles. Results indicate that the composites are mechanically and metallographically sound for further tribological studies.

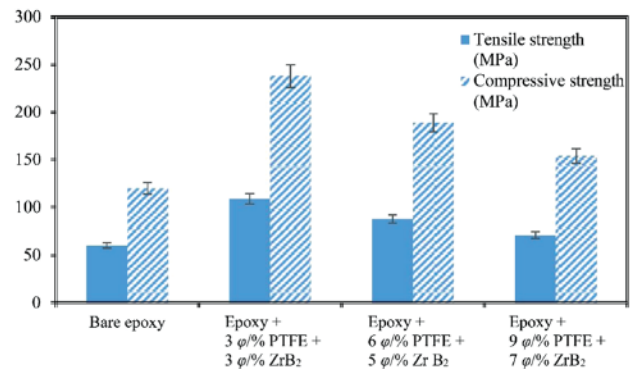


Figure 2: Mechanical characterization of composites

Table 3: Design layout and the corresponding responses

Run order	Coded values of the variables		Actual values of the variables		Response 1 Hardness (HRB)	Response 2 COF	Response 3 Wear rate × 10 ⁻⁴ (mm ³ /N·m)
	A	B	A: ZrB ₂ (φ1%)	B: PTFE (φ1%)			
1	0	-1	5	3	80.33	0.307	6.69
2	1	-1	7	3	63.67	0.363	7.03
3	0	0	5	6	79.33	0.18	3.34
4	-1	-1	3	3	70	0.332	10.55
5	0	0	5	6	79.5	0.183	3.38
6	0	1	5	9	68	0.229	5.02
7	-1	0	3	6	65.33	0.26	8.37
8	1	0	7	6	60	0.21	5.86
9	0	0	5	6	79.8	0.185	3.41
10	-1	1	3	9	57.67	0.290	13.39
11	0	0	5	6	79.23	0.189	3.32
12	1	1	7	9	59.33	0.256	7.36
13	0	0	5	6	79.65	0.188	3.39

5.2 Design of experiments

Design of experiments (DOE) is a systematic method for a statistical examination of the influence of various input variables on the output or response of a process. The parameters used for the experimentation and their corresponding responses in this study are given in Table 3. Design Exert 13 statistical software was used for the random generation of the order of the experiment.

5.3 RSM model, test for adequacy of the model and validation of the model

Equations (6), (7) and (8) are the empirical relations for the hardness, wear rate and COF for the epoxy hybrid composites model as functions of the independent variables of φ % of ZrB₂ (A) and φ % PTFE (B) in coded units after the backward-elimination process ($p < 0.05$ is significant). In the case of hardness and coefficient of friction, all the terms are significant when considering a p-value lower than 0.05. Hence, the raw and refined equations are the same. In the case of wear rate, all the terms except for quadratic term B have a significant influence on the wear rate.

$$\text{Hardness} = 78.77 - 1.67 A - 4.83 B + 2.00 AB - 14.26 A^2 - 2.76 B^2 \tag{6}$$

$$\text{Wear rate} = 0.0003 - 0.0002A - 0.0001AB + 0.0003A^2 + 0.0002B^2 \tag{7}$$

$$\text{COF} = 0.1861 - 0.0087A - 0.0376B - 0.0163AB + 0.0460A^2 + 0.0795B^2 \tag{8}$$

The analysis of variance and fit summary of the models obtained is shown in Tables 4, 5 and 6. In the case of hardness, wear and coefficient of friction models, the F-value is 47.24, 30.42, and 71.89, respectively, implying that the models are significant. The F-value is the ratio of the model mean square and residual mean square. Since the probability values (p-values) are below 0.05, the terms in the model have a significant influence on the responses. In the case of hardness, wear and coefficient of friction, the predicted R² is in reasonable agreement with the adjusted R² as the difference between them is less than 0.2. An adequate precision ratio greater than 4 is desirable. Therefore, the models that were generated were significant.

Table 4: Analysis of variance for hardness

Source	Sum of squares	Degree of freedom	Mean square	F-value	p-value
Model	951.15	5	190.23	47.24	< 0.0001
Residual	26.73	7	3.82		

Standard deviation = 2.01 R² = 0.9712
 Mean = 70.91 Adjusted R² = 0.9507
 Adequate precision = 17.7934 Predicted R² = 0.7764

Table 5: Analysis of variance for wear rate

Source	Sum of squares	Degree of freedom	Mean square	F-value	p-value
Model	1.140 × 10 ⁻⁶	5	2.280 × 10 ⁻⁷	30.42	0.0001
Residual	5.247 × 10 ⁻⁸	7	7.495 × 10 ⁻⁹		

Standard deviation = 0.0001 R² = 0.9560
 Mean = 0.0006 Adjusted R² = 0.9246
 Adequate precision = 15.4798 Predicted R² = 0.7533

Table 6: Analysis of variance for coefficient of friction

Source	Sum of squares	Degree of freedom	Mean square	F-value	p-value
Model	0.0463	5	0.0093	71.89	< 0.0001
Residual	0.0009	7	0.0001		

Standard deviation = 0.0114 R² = 0.9809
 Mean = 0.2441 Adjusted R² = 0.9673
 Adequate precision = 22.1489 Predicted R² = 0.8188

The accuracy of a model is ensured by validating the model using the responses obtained by conducting conformity trials at randomly selected factor setting. A total of five trials with factor combinations, except for that used as part of DOE, were conducted, and the hardness, wear and coefficient of friction of the joints were determined using the standard procedure. The details of the factor setting, actual and predicted values of the response and percentage of error are shown in Table 7. The experimental results agree well with the predicted values, with an average error of less than ± 1 %.

5.4 Effect of the filler content on hardness, wear rate and coefficient of friction

The response surface of hardness obtained from ANOVA is shown in Figure 3. It depicts the relationship

Table 7: Experimental validation of the developed model

Trial no.	Coded values of the factor		Actual values of the factor		Actual values			Predicted values			% Error		
	A	B	A	B	Hardness (HRB)	Wear rate × 10 ⁻⁴ (mm ³ /N·m)	COF	Hardness (HRB)	Wear rate × 10 ⁻⁴ (mm ³ /N·m)	COF	Hardness	Wear rate	COF
1	0	-1	6	3	71	5.6	0.33	75.44	5.3	0.32	-5.88	5.66	3.1
2	0	0	6	7.5	74	3.1	0.18	71.76	3	0.19	3.12	3.33	-5.2
3	0	1	6	9	65	4.4	0.34	67.78	4.25	0.33	-4.1	3.5	-3
4	-1	0	3	7.5	64	8.5	0.26	62.08	9	0.25	3.09	-5.5	4
5	1	0	7	7.5	62	3.8	0.27	60.74	4	0.26	2.07	-5	3.8
Average % error										-0.34	0.39	0.54	

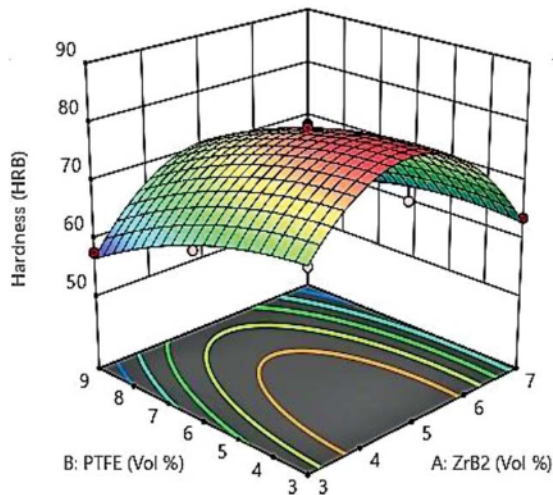


Figure 3: Response surface of hardness

between the hardness, ϕ % of PTFE and ϕ % of ZrB₂. An increase in the ϕ % of ZrB₂ and PTFE particles resulted in an increase in the hardness, exceeding the bare epoxy polymer matrix hardness of 53.7 HRB. The incorporation of hard ceramic particles improves the hardness of the composites. By adding reinforcement particles to the matrix, the surface area of reinforcements increases. The presence of hard ceramic particles on the surface provides high resistance to plastic deformation, leading to an increase in the hardness of the composite. When considering individual reinforcement particles, the hardness increases with the addition of ZrB₂ of up to 5 ϕ % of particles, while a further addition of the reinforcement reduces it. A reduction in the hardness can be due to the agglomeration of the filler particles in the composite, observed through the optical microscope. In the case of the PTFE reinforcement particles, their addition decreases the hardness. PTFE, being a softer material compared to the hard ceramic ZrB₂ particle, acts as a solid lubricant in the composite.

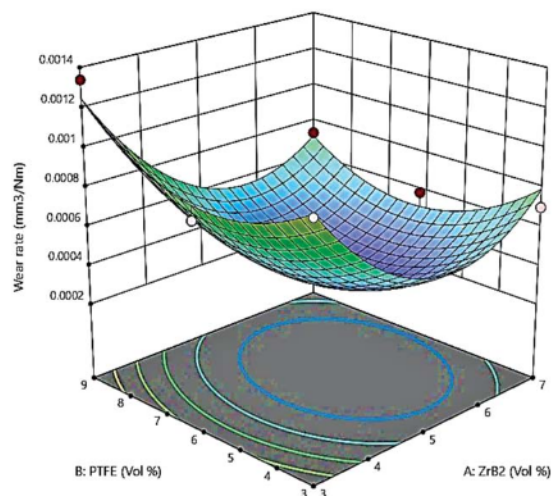


Figure 4: Response surface of wear rate

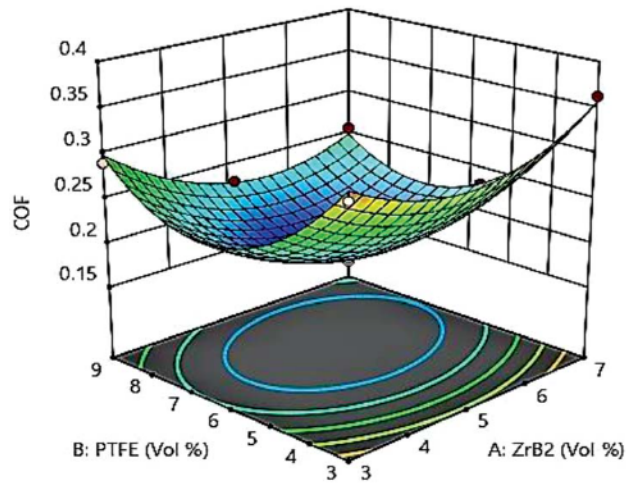


Figure 5: Response surface of coefficient of friction

The response surface of wear rate obtained from ANOVA is presented in Figure 4. It depicts the relationship between the wear rate, ϕ % of PTFE and ϕ % of ZrB₂. The wear decreases considerably with the addition of fillers, below the wear rate of the bare epoxy polymer matrix of $35 \times 10^{-4} \text{ mm}^3/\text{N}\cdot\text{m}$. The hard ceramic ZrB₂ particles act as protuberances over the surface. These protuberances tend to protect the matrix from the uniform contact with the counter-facing steel disc, thereby reducing the contact area between the test surface and the rotating disc, resulting in reduced wear. PTFE acts as a lubricant, thereby reducing the friction and the resultant wear. The wear decreases with the addition of fillers of up to 5 ϕ %. Any further addition of the filler results in an increase in the wear due to the agglomeration of the fillers. The surface plot of coefficient of friction obtained from the ANOVA of the hybrid polymer composite is shown in Figure 5. It depicts the relationship between the coefficient of friction, ϕ % of PTFE and ϕ % of ZrB₂. The coefficient of friction decreases considerably with the addition of both ZrB₂ and PTFE in compar-

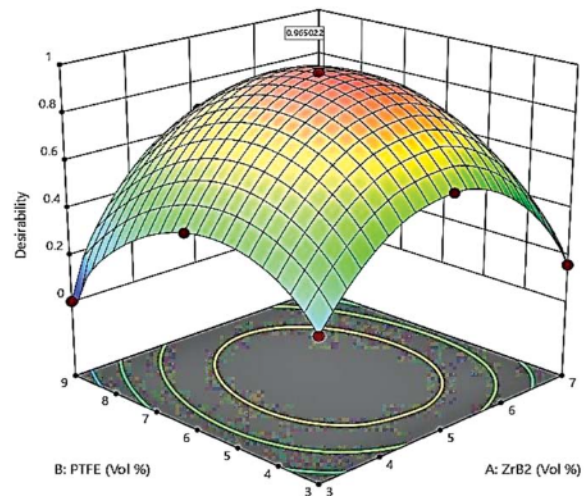


Figure 6: Surface plot of desirability

ison with the bare epoxy polymer matrix COF of 0.39. The protuberances on the surfaces of the composites help to reduce the contact area between the specimen surface and the counterface disc, aiding the reduction of friction. PTFE provides a lubricating effect between the contact regions, also aiding the reduction of friction.

5.5 Experimental validation of the optimum results

The optimum ϕ /% levels obtained using ANOVA are 5.05 ϕ /% of ZrB₂ and 5.95 ϕ /% of PTFE, with a 0.96 desirability. Figure 6 represents the surface plot of desirability for the optimum percentage of reinforcement particles at the maximum hardness and minimum coefficient of friction and wear rate. To validate the results experimentally, the composites were prepared as per the optimum condition. The hardness test and wear tests were carried out on the prepared composites under a load of 40 N and sliding speed 1.25 m/s. The results of the experiment were compared with the predicted results and error characteristics are given in Table 8. From these results, it can be observed that the model is in good agreement with the predicted results. By comparing the response variables of the epoxy hybrid composite with the base epoxy, the hardness increases by 46.7 %, while wear and COF decrease by 90.6 % and 52.2 %, respectively.

Table 8: Experimental validation of the optimum results

Exp. no	Hardness (HRB)	Wear rate $\times 10^{-4}$ (mm ³ /N·m)	COF
1	77.9	3.2	0.185
2	78.1	3.4	.0182
3	78.3	3.6	0.186
4	78.2	3.3	0.181
5	78.5	3.5	0.183
Average	78.2	3.4	0.1835
Predicted	78.8	3.3	0.1865
% Error	-0.767	3.03	-1.61

5.6 Thermal conductivity and contact temperature of the composites

The Hot Disk TPS 500 thermal constants analyzer accurately measures the thermal conductivity, thermal diffusivity and specific heat capacity for a wide range of materials. The measured values of the thermal conductivity are given in Table 9. The thermal conductivity increases with the increase in the ZrB₂ content. The thermal conductivity of the bare epoxy matrix is 0.2 W/m·K. At the optimum composition, the thermal conductivity is improved by 50 %, to a value of 0.3 W/m·K. PTFE is a self-lubricating material that helps to reduce the heat accumulation due to friction. ZrB₂ is an ultra-high-temperature ceramic with high thermal conductivity, thereby providing high thermal conductivity to the composite. The improved thermal conductivity can provide better heat transfer and reduce the contact temperature on the surface of the material, resulting in a reduction in the wear. As the surface temperature at the point of contact during experimentation could not be measured accurately, the surface temperature of the wear track was measured as soon as the experiment was stopped.

Table 9: Measured thermal conductivity (W/m·K) for different compositions

ZrB ₂ (ϕ /%)	PTFE (ϕ /%)		
	3	6	9
3	0.2781	0.28	0.282
5	0.2961	0.2983	0.3005
7	0.3113	0.3137	0.3162

For the base epoxy, the contact temperature developed on the surface is 141.6 °C. The contact temperature corresponding to the epoxy hybrid composite with the optimum composition is 72.8 °C, which is a reduction by 48.7 %. This is due to the presence of the particles of ultra-high-temperature ceramic ZrB₂ and frictional modifier PTFE.

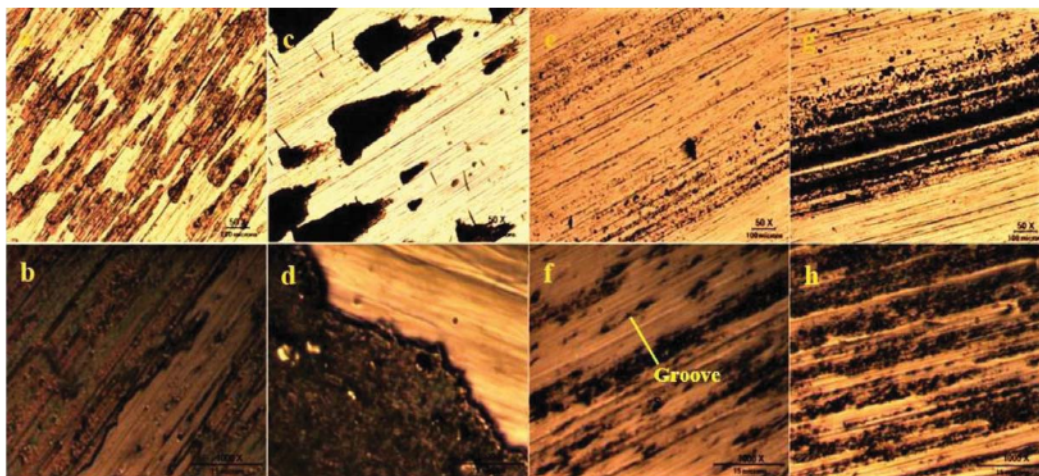


Figure 7: Optical microscope images of the counterface: a and b) bare epoxy, c and d) 3 ϕ /% PTFE + 3 ϕ /% ZrB₂, e and f) 6 ϕ /% PTFE + 5 ϕ /% ZrB₂, g and h) 9 ϕ /% PTFE + 7 ϕ /% ZrB₂ under 40 N and at a sliding speed of 1.25 m/s

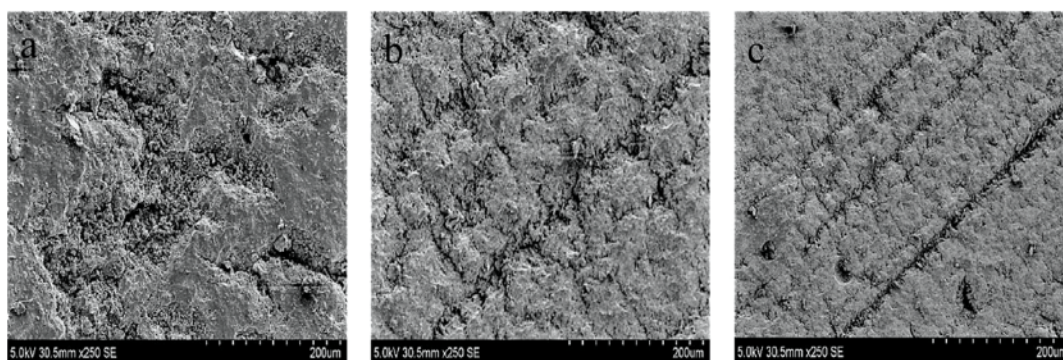


Figure 8: SEM images of worn surfaces of the epoxy composites: a) 3 ϕ 1% PTFE + 3 ϕ 1% ZrB₂, b) 6 ϕ 1% PTFE + 5 ϕ 1% ZrB₂, c) 9 ϕ 1% PTFE + 7 ϕ 1% ZrB₂ under 40 N and at a sliding speed of 1.25 m/s

5.7 Worn surface

The worn surface of the epoxy hybrid composite was also examined after the wear test. **Figures 7b, 7d, 7f, 7h** are magnified images (1000 \times) of **Figures 7a, 7c, 7e, 7g**, respectively. They reveal that the wear mechanism changes from adhesive wear to abrasive wear as the filler content increases. **Figures 7a** and **7b** show optical images of the counter part of bare epoxy. The adhered material shown in the figure is due to the adhesion of the epoxy polymer material onto the counter face. Adhesion is also visible on the epoxy composite with 3 ϕ 1% ZrB₂ and 3 ϕ 1% PTFE (**Figures 7c** and **7d**), but the quantity of the adhered material is lower than that of bare epoxy. As the volume fractions of the composite increase to 6 ϕ 1% PTFE and 5 ϕ 1% ZrB₂, small grooves are visible with the higher amounts of the fillers (**Figures 7e** and **7f**). The wear mechanism for the composite with higher volume fractions of the fillers is mainly abrasive wear. The change in the nature of wear from adhesive wear to abrasive wear is due to the presence of hard ZrB₂ particles.

Figure 8 shows SEM images of the worn surfaces of the epoxy composites with various fractions of reinforcement particles. It can be seen in the figure that the epoxy resin with 3 ϕ 1% PTFE + 3 ϕ 1% ZrB₂ is peeled off the surface, indicating that adhesive wear is the dominant wear mechanism. During sliding, the epoxy with low fractions of the fillers can soften and get deformed and further peeled off the block resin due to the temperature rise caused by friction, forming pits as well as ploughed spaces on the surface. During the friction process, the asperities on the surface of epoxy resin are deformed, sticking to the counterpart surface due to the load and the adhesiveness of epoxy resin.

As the counterpart rotates, the asperities of the epoxy resin are pulled out, causing the adhesive wear of the epoxy resin. With the introduction of ZrB₂ particles, the exposed ZrB₂ particles can play the role of asperities on the surface of an epoxy composite. Due to the high elastic modulus and hardness of the ZrB₂ ceramic, the deformation of the asperities on the epoxy composites is reduced. This contributes to the improvement in wear resistance and reduction in adhesive wear of epoxy hybrid compos-

ites. As the volume fractions of the fillers in a composite increase to 6 ϕ 1% PTFE and 5 ϕ 1% ZrB₂, small grooves become visible (**Figure 8b**), indicating the abrasive-wear mechanism. With higher volume fractions of the fillers, the higher hardness and thermal conductivity of ZrB₂ reduce the softening of the composite due to the frictional-heat accumulation with the improved heat transfer. Thereby, after sliding, narrow grooves are clearly visible, as the ones from **Figure 8c**. This also reduces the material damage due to adhesive wear.

5.8 Comparison of obtained tribological properties with those of other commercial liner materials

To study the performance of the composite to be used as the self-lubricating spherical-bearing liner material, a comparison was made including the other polymer-based commercial bearing materials.⁵ The epoxy/PTFE/ ZrB₂ hybrid composite was tested at a load of 40 N and sliding speed of 20 mm/s. At a low speed and load, the significant temperature rise due to friction was decreased. Hence, the wear rate and coefficient of friction obtained were lower than those of the other commercial materials as shown in **Table 10**.

Table 10: Comparison of tribological properties with those of other commercial liner materials

Composites	Wear $\times 10^{-7}$ (mm ³ /N·m)	Coefficient of friction
Epoxy/PTFE/ZrB ₂ composite	10	0.065
TX liner	700	0.08
Vyncolit X620-1	110	0.2

6 CONCLUSIONS

The tribological and thermal properties of an epoxy hybrid polymer composite reinforced with ZrB₂ and PTFE particles are studied in this work. The experimental and optimization results show that a maximum hardness of 78.8 HRB, minimum wear rate and COF of 3.3×10^{-4} mm³/Nm and 0.186, respectively, were observed to correspond to optimized values of 5.05 ϕ 1% of ZrB₂ and 5.95 ϕ 1% of PTFE. Ultra-high-temperature ceramic ZrB₂

particles act as protuberances over the surface. These protuberances tend to protect the matrix from a uniform contact. The self-lubricating property of PTFE provides a reduced wear rate and coefficient of friction. An optical microscope and SEM images of the worn surfaces of the epoxy hybrid composites reveal that the wear mechanism changes from adhesive wear to abrasive wear as the filler content increases. A higher volume fraction of the filler, higher hardness and thermal conductivity of ZrB_2 reduce the softening of the hybrid composite due to the frictional-heat accumulation with the improved heat transfer. The thermal conductivity of the composite is also enhanced by 50 % due to the addition of fillers. This also contributes to the improvement of the wear resistance and reduction of adhesive wear of epoxy hybrid composites.

A reduction in the coefficient of friction, and an increased heat transfer due to improved thermal conductivity reduce the heat accumulation during the process. The presence of the reinforcement particles ensures that the contact temperature of the optimum composition is reduced by 48.7 %. Due to these effects, this composite can ensure an increased service life for self-lubricating spherical plain bearing liner over a conventional material.

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7 REFERENCES

- ¹ J. K. Lancaster, Composite self-lubricating bearing materials proceedings of the institution of mechanical engineers, 182 (1967), 33–54, doi:10.1243/PIME_PROC_1967_182_009_02
- ² Y. Yang, C. Ma, S. Huang, L. Jing, H. Pang, Effects of mechanical properties of kevlar/PTFE fabric-reinforced self-lubricating liners on performance of self-lubricating spherical plain bearings, Applied Mechanics and Materials, 29 (2010), 197–202, doi:10.4028/www.scientific.net/AMM.29-32.197
- ³ D. Amrishraj, T. Senthilvelan, Dry Sliding wear behavior of ABS Composites reinforced with nano Zirconia and PTFE, Materials Today: Proceedings, 5 (2018) 2, 7068–7077, doi:10.1016/j.matpr.2017.11.371
- ⁴ K. Friedrich, Z. Zhang, A. K. Schlarb, Effects of various fillers on the sliding wear of polymer composites, Composites Science and Technology, 65 (2005) 15, 2329–2343, doi:10.1016/j.compscitech.2005.05.028
- ⁵ J. T. Shen, M. Top, Y. T. Pei, J. De Hosson, Wear and friction performance of PTFE filled epoxy composites with a high concentration of SiO_2 particles, Wear, 322 (2015), 171–180, doi:10.1016/j.wear.2014.11.015
- ⁶ M. Yang, J. Yuan, X. Menc, Z. Zhang, F. Guo, W. Liu, Effect of ZrB_2 particles incorporation on high-temperature tribological properties of hybrid PTFE/Nomex fabric/phenolic composite, Tribology International, 99 (2016), 289–29, doi:10.1016/j.triboint.2016.03.033
- ⁷ Y. Wu, Z. Yu, X. Liu, Tribological performance of in-situ epoxy composites filled with micro-sized ZrB_2 particles, Composites Part B, (2017), doi:10.1016/j.compositesb.2017.05.027
- ⁸ Y. Wu, Y. He, Z. Yu, X. Liu, Thermal conductive behavior of zirconium boride coated by nanoalumina with different mass proportions in epoxy composites, Progress in Natural Science: Materials International, 28 (2018), 345–353, doi:10.1016/j.pnsc.2018.04.012
- ⁹ B. P. Chang, H. M. Akil, R. B. Nasir, A. Khan, Optimization on wear performance of UHMWPE composites using response surface methodology, Tribology International, 88 (2015), 252–262, doi:10.1016/j.triboint.2015.03.028
- ¹⁰ K. R. Kumar, V. S. Sreebalaji, Desirability based multiobjective optimisation of abrasive wear and frictional behaviour of aluminium (Al/3.25Cu/8.5Si)/fly ash composites, Tribology - Materials, Surfaces & Interface, 9 (2015) 3, 128–136, doi:10.1179/1751584X15Y.0000000012