

HYBRID POLYMERIC COMPOSITE FOR TRIBOLOGICAL APPLICATIONS

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NEHA SINGH

Abstract

Materials are used for manufacturing a wide range of dynamic machine and vehicle components. Depending on the operating conditions, those materials are subjected to heavy loading, rolling and sliding. These operating conditions lead to high friction, wear and erosion depending upon the different material combinations in contact. Thus, there is a requirement of tribological solutions to put a stop on direct contact between metals for friction control and protect them against unwanted wear. Lubricants used for Industrial applications to enhance tribological performances are complex sulphates and phosphates such as zinc dialkyldithiophosphate (ZDDP). These additives are environmentally harmful and have damaging effects on the steel components. Hence, to find alternate solutions for enhancing the tribological performances of the lubricants with sustainability, proper environmentally-friendly additives are also required. Polymers are visco-elastic/visco-plastic materials which can provide sufficiently high strength and with lower frictional stress at the interface. Polymeric coating and bulk present possible tribological solutions which enhance the wear life of mechanical components in sliding and rolling contacts, decrease the COF (coefficient of friction), reduce the frictional damage and save the maintenance cost of the component. Among polymers, thermoset epoxies are widely used for their high strength, stiffness and thermal stability if they are suitably made into composites. Similarly, epoxy-based composites provide low friction and low specific wear resistance with high wear life under dry and base oil lubricated conditions. Thus, the present work is focused on achieving improved mechanical properties with better tribological properties (friction < 0.1 , and Specific wear rate $< 10^{-7}$ mm³/Nm) for epoxy composites for bearing and other tribological applications. The present work is divided into five parts which also form the five main objectives of the work.

The first part involves tribological studies of epoxy composites as coatings. Here, epoxy with ultra-high molecular weight polyethylene (UHMWPE) and MoS₂ fillers was coated on to a bearing steel (SAE 52100). Frictional and wear properties of the coated samples in sliding contact were investigated on a pin-on-disc tribometer under a normal load of 10 N and a linear sliding speed of 1 m/s against a bearing steel ball. Bearing steel was selected as the counterface for its wide applications in tribological machine elements. The optimized coating composition (72 wt% Epoxy+7 wt% hardener+18 wt% UHMWPE+3 wt% MoS₂) showed highly improved tribological properties compared to those of pure epoxy and other epoxy-based composites. There was 75% reduction in the coefficient of friction (COF) in the dry interfacial condition (COF reduced from 0.2 to 0.05) over pure epoxy and 80% reduction with grease as the lubricant. The specific wear-rate of the composite was lower by five orders of magnitude over that of pure epoxy. Other mechanical properties such as hardness, tensile strength, and Young's modulus of the composite showed increments of 86%, 121%, and 43%, respectively, with respect to those of pure epoxy. 3 wt% of MoS₂ had drastic effects on improving compressive strength and reducing friction and wear of the composites. For dry sliding, initial abrasive and adhesive wear mechanisms led to transfer film formation on the steel counterface, and afterwards, the shearing was mainly within the transfer film reducing steady-state wear. For the grease-lubricated case, a thin and stable layer of grease helped in easy shearing, and the polymer transfer film formation was avoided.

The second part includes effects of liquid soaking on the epoxy/UHMWPE/MoS₂ composite on mechanical and tribological properties. Liquid absorption and tribological studies of epoxy-based composite with ultra-high molecular weight polyethylene (UHMWPE) and MoS₂, sliding against steel were conducted. Composites, as coating and as a bulk, were soaked in water, base oil, ionic liquid and lithium-based grease for different intervals of days or months. It was found that liquid weight% gain was more in polar liquids when compared to non-polar.

Coated composite soaked in grease for 10 days showed coefficient of friction of 0.08 with wear-life of more than 1 million cycles and wear rate of 1.7×10^{-8} mm³/Nm. Bulk polymer composite soaked in grease for 180 days provided the least coefficient of friction of 0.06 and specific wear rate of 2.60×10^{-7} mm³/Nm.

The third part of this thesis includes the study and comparison of the tribological, thermal and mechanical properties of epoxy-based composites with solid fillers (ultra-high molecular weight polyethylene and molybdenum disulphide), and different in-situ liquid lubricants in different volume proportions. Liquid lubricants used in this thesis are three types of silicon oils with different viscosities, base oil and polyalphaolefins (PAO). It was observed that the base oil-epoxy composite showed superior tribological performances among the three. The coefficient of friction of 0.1 and specific wear rate in the range of 10^{-7} mm³/Nm were obtained at an apparent contact pressure of 0.611 MPa. Transfer film formation with oil pockets on both the tribo-pair surfaces was identified as the main friction and wear reduction mechanisms.

The fourth part includes tribological performance studies of polymer bush bearings under severe contact conditions. Thermal, mechanical and tribological performances of epoxy-based composites, filled with UHMWPE, MoS₂ and Kevlar (aramid) fibres or base oil in different proportions, were investigated. Friction coefficient and wear resistance were examined by a block-on-cylinder tribotester in dry and wet sliding conditions under variable contact pressures, sliding velocities and temperatures. It was found that the composite with in-situ base oil possesses the lowest coefficient of friction (0.056) and highest wear resistance (specific wear rate of 5.77×10^{-6} mm³/Nm) at relatively high PV factor (contact pressure x sliding velocity) of 37 MPa-m/s when compared to other composites. Kevlar-filled composite provided a low wear rate at a very high PV factor of 52.5 MPa-m/s.

The last part of this thesis includes the applications of the prepared polymer bearings. For testing the prepared epoxy composite bearing, it was mounted on the front wheel bearing of a bicycle wheel and also installed in the DC motor used in a small sized drone. Before mounting the polymer bearing, conventional bearing was removed from the bicycle wheel and from the motor of the drone. It was found that with polymer composite bearing the bicycle and the drone motor were running smoothly.

The results of this study have demonstrated that epoxy-based composites with suitable fillers can provide sustainable and environmentally friendly tribological solutions to the traditional metal bearings within the given operating parametric limits.

शोध-निबंध सार

विभिन्न प्रकार की गतिशील मशीनों और वाहन घटकों के निर्माण के लिए पदार्थों का उपयोग किया जाता है। परिचालन स्थितियों के आधार पर, उन पदार्थों को भारी लोडिंग, रोलिंग और स्लाइडिंग के अधीन किया जाता है। इन परिचालन स्थितियों के कारण, विभिन्न सामग्री संयोजनों के आपसी संपर्क के आधार पर उच्च घर्षण, घिसाव और क्षरण होता है। इस आधार पर, घर्षण नियंत्रण के लिए धातुओं के बीच सीधे संपर्क पर रोक लगाने और उन्हें अवांछित टूट-फूट से बचाने के लिए ट्राइबोलॉजिकल (अभिघर्षिक) समाधान की आवश्यकता है। ट्राइबोलॉजिकल (अभिघर्षिक) निष्पादन को बढ़ाने के लिए औद्योगिक अनुप्रयोगों के लिए उपयोग किए जाने वाले स्नेहक जटिल सल्फेट्स और फॉस्फेट जैसे जेड-डी-डी-पी (ZDDP) हैं। ये एडिटिक्स पर्यावरण की दृष्टि से हानिकारक हैं और स्टील घटकों पर हानिकारक प्रभाव डालते हैं। इसलिए, वहनीयता की दृष्टि से स्नेहक के प्रदर्शन को बढ़ाने के लिए और वैकल्पिक समाधान खोजने के लिए, उचित पर्यावरण-अनुकूल एडिटिक्स की भी आवश्यकता होती है। पॉलिमर विस्को-इलास्टिक/विस्को-प्लास्टिक सामग्री हैं जो पर्याप्त उच्च शक्ति और इंटरफ़ेस पर कम घर्षण तनाव प्रदान कर सकते हैं। पॉलिमरिक कोटिंग और बल्क, संभावित ट्राइबोलॉजिकल समाधान प्रस्तुत करते हैं जो स्लाइडिंग और रोलिंग संपर्कों में यांत्रिक घटकों के घिसाव के प्रतिरोध क्षमता को बढ़ाते हैं, सीओ एफ (घर्षण का गुणांक) को कम करते हैं, घर्षण क्षति को कम करते हैं और घटक की रखरखाव लागत को बचाते हैं। पॉलिमर के बीच, थर्मोसेट एपॉक्सी का व्यापक रूप से उनकी उच्च शक्ति, कठोरता और थर्मल स्थिरता के लिए उपयोग किया जाता है, यदि उन्हें उपयुक्त रूप से कंपोजिट में बनाया जाता है। इसी

प्रकार, एपॉक्सी-आधारित कंपोजिट सूखा और बेस-ऑयल स्नेहक वाली स्थितियों के तहत उच्च घिसाव वाले जीवन के साथ कम घर्षण और कम विशिष्ट घिसाव के प्रतिरोध प्रदान करते हैं। इस प्रकार, वर्तमान कार्य बेयरिंग और अन्य ट्राइबोलॉजिकल अनुप्रयोगों के लिए एपॉक्सी कंपोजिट के लिए बेहतर ट्राइबोलॉजिकल गुणों (घर्षण <0.1 , और विशिष्ट घिसाव की दर $<10^{-7}$ mm³/N-m) के साथ बेहतर यांत्रिक गुणों को प्राप्त करने पर केंद्रित है। वर्तमान कार्य को पाँच भागों में विभाजित किया गया है जो कार्य के पाँच मुख्य उद्देश्य भी हैं।

पहले भाग में कोटिंग्स के रूप में एपॉक्सी कंपोजिट का अध्ययन शामिल है। यहां, अल्ट्रा-हाई आणविक भार पॉलीथीन (UHMWPE) और MoS₂ फिल्स के साथ एपॉक्सी को एक बेयरिंग स्टील (SAE 52100) पर लेपित किया गया था। स्लाइडिंग संपर्क में एपॉक्सी कोटिंग्स नमूनों के घर्षण और घिसाव के गुणों की जांच एक पिन-ऑन-डिस्क ट्राइबोमीटर पर 10 N के सामान्य भार और एक असर वाली स्टील की गेंद के खिलाफ 1 m/s की रैखिक स्लाइडिंग गति के तहत की गई थी। ट्राइबोलॉजिकल मशीन तत्वों में इसके व्यापक अनुप्रयोगों के लिए बियरिंग स्टील को काउंटरफेस के रूप में चुना गया था। अनुकूलित कोटिंग संरचना (72 भार % एपॉक्सी + 7 भार % हार्डनर + 18 भार % UHMWPE + 3 भार % MoS₂) ने शुद्ध एपॉक्सी और अन्य एपॉक्सी-आधारित कंपोजिट की तुलना में अत्यधिक बेहतर ट्राइबोलॉजिकल गुण दिखाए। शुद्ध एपॉक्सी की तुलना में सूखे इंटरफेसियल स्थिति में घर्षण के गुणांक (COF) में 75% की कमी (COF 0.2 से 0.05 तक कम) और स्नेहक के रूप में ग्रीस के साथ 80% की कमी थी। कंपोजिट की विशिष्ट घिसाव-दर शुद्ध एपॉक्सी की तुलना में परिमाण के पांच गुना कम थी। अन्य यांत्रिक गुणों जैसे

कठोरता, तन्यता ताकत और समग्र के यंग मापांक में शुद्ध एपॉक्सी के संबंध में क्रमशः 86%, 121% और 43% की वृद्धि देखी गई। MoS₂ के 3 भार % का संपीडन शक्ति में सुधार और कंपोजिट के घर्षण और घिसाव को कम करने पर कठोर प्रभाव पड़ा। ड्राई स्लाइडिंग के लिए, प्रारंभिक अपघर्षक और आसंजक घिसाव तंत्र ने स्टील काउंटरफेस पर ट्रांसफर फिल्म के निर्माण का काम किया, और बाद में, शियर मुख्य रूप से ट्रांसफर फिल्म के भीतर स्थिर-अवस्था के घिसाव को कम करने के लिए हुई। ग्रीस- स्नेहक वाले मामले के लिए, ग्रीस की एक पतली और स्थिर परत ने आसानी से शियर में मदद की, और पॉलिमर ट्रांसफर फिल्म का निर्माण नहीं हुआ।

दूसरे भाग में यांत्रिक और ट्राइबोलॉजिकल गुणों पर एपॉक्सी/UHMWPE/MoS₂ मिश्रित पर तरल अवशोषण के प्रभाव शामिल हैं। अल्ट्रा-उच्च आणविक भार पॉलीथीन (UHMWPE) और MoS₂ के साथ एपॉक्सी-आधारित मिश्रित के तरल अवशोषण और ट्राइबोलॉजिकल अध्ययन, स्टील के विरुद्ध स्लाइडिंग आयोजित किए गए थे। कंपोजिट, कोटिंग के रूप में और बल्क के रूप में, दिनों या महीनों के विभिन्न अंतरालों के लिए पानी, बेस-ऑयल, आयनिक तरल और लिथियम-आधारित ग्रीस में भिगोए गए थे। यह पाया गया कि गैर-ध्रुवीय की तुलना में ध्रुवीय तरल पदार्थों में तरल भार % की वृद्धि अधिक थी। 10 दिनों तक ग्रीस में अवशोषित किये गए कोटिंग्स कंपोजिट ने 1 मिलियन चक्र से अधिक के घिसाव के जीवन और $1.7 \times 10^{-8} \text{ mm}^3/\text{N-m}$ की घिसाव की दर के साथ 0.08 के घर्षण गुणांक को दिखाया। 180 दिनों तक ग्रीस में अवशोषित किये गए बल्क पॉलिमर कंपोजिट ने घर्षण का न्यूनतम गुणांक 0.06 और विशिष्ट घिसाव दर $2.60 \times 10^{-7} \text{ mm}^3/\text{N-m}$ प्रदान किया।

इस शोध-निबंध के तीसरे भाग में ठोस भराव (UHMWPE और MoS₂) के साथ एपॉक्सी-आधारित कंपोजिट के ट्राइबोलॉजिकल, थर्मल और मैकेनिकल गुणों का अध्ययन और तुलना शामिल है, और अलग-अलग मात्रा अनुपात में अलग-अलग इन-सीटू तरल स्नेहक हैं। इस थीसिस में उपयोग किए जाने वाले तरल स्नेहक विभिन्न विस्कोसिटी वाले तीन प्रकार के सिलिकॉन तेल, बेस ऑयल और पॉलीअल्फाओलेफ़िन (PAO) हैं। यह देखा गया कि बेस-ऑयल-एपॉक्सी कंपोजिट ने तीनों के बीच बेहतर प्रदर्शन दिखाया। घर्षण का गुणांक 0.1 और विशिष्ट घिसाव दर 10^{-7} mm³/N-m की सीमा में 0.611 MPa के स्पष्ट संपर्क दबाव पर प्राप्त किया गया था। दोनों ट्राइबो-जोड़ी सतहों पर तेल की पॉकेट्स के साथ स्थानांतरण फिल्म निर्माण को मुख्य घर्षण और घिसाव कम करने वाले तंत्र के रूप में पहचाना गया था।

चौथे भाग में गंभीर संपर्क स्थितियों के तहत पॉलिमर बुश बियरिंग्स के अभिघर्षकिये प्रदर्शन (tribological performance) का अध्ययन शामिल है। विभिन्न अनुपातों में UHMWPE, MoS₂ और केवलार (एरामिड) फाइबर या बेस-ऑयल से भरे एपॉक्सी-आधारित कंपोजिट के थर्मल, मैकेनिकल और ट्राइबोलॉजिकल प्रदर्शन की जांच की गई। घर्षण गुणांक और घिसाव के प्रतिरोध की जांच ब्लॉक-ऑन-सिलेंडर ट्राइबोटेस्टर द्वारा परिवर्तनीय संपर्क दबाव, स्लाइडिंग वेग और तापमान के तहत सूखी और गीली स्लाइडिंग स्थितियों में की गई थी। यह पाया गया कि इन-सीटू मिनरल बेस-ऑयल वाले मिश्रण में घर्षण का न्यूनतम गुणांक (0.056) और अपेक्षाकृत उच्च PV कारक (संपर्क दबाव x स्लाइडिंग वेग) पर उच्चतम घिसाव का प्रतिरोध (5.77×10^{-6} mm³/N-m की विशिष्ट घिसाव की दर) होता है।) अन्य कंपोजिट की

तुलना में 37 MPa-m/s। केवलार-भरे कंपोजिट ने 52.5 MPa-m/s के बहुत उच्च PV कारक पर कम घिसाव की दर प्रदान की।

इस शोध-निबंध के अंतिम भाग में पॉलिमर बियरिंग्स के अनुप्रयोग शामिल हैं। एपॉक्सी कम्पोजिट बियरिंग के परीक्षण के लिए इसे साइकिल के अगले पहिये के बियरिंग पर लगाया गया और छोटे आकार के ड्रोन में इस्तेमाल होने वाली डी. सी. मोटर में भी लगाया गया। पॉलिमर बियरिंग लगाने से पहले, साइकिल के पहिये और ड्रोन की मोटर से पारंपरिक स्टील बियरिंग हटा दी गई थी। यह पाया गया कि पॉलिमर कंपोजिट बियरिंग के साथ साइकिल और ड्रोन मोटर सुचारू रूप से चल रहे थे।

अतः, इस अध्ययन के परिणामों से पता चला है कि उपयुक्त फिलर्स के साथ एपॉक्सी-आधारित कंपोजिट दिए गए ऑपरेटिंग पैरामीट्रिक सीमाओं के भीतर पारंपरिक धातु बियरिंगों के लिए टिकाऊ और पर्यावरण के अनुकूल ट्राइबोलॉजिकल समाधान प्रदान कर सकते हैं।

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List of Abbreviations:

μ – Coefficient of friction

$\tan\theta$ - Tangential angle of interlocking asperities

F- Frictional force

F_a - Interfacial component

F_p - Ploughing component of friction

τ - shear stress

τ_0 - initial shear stress

α - constant

P - contact pressure

DLC - Diamond like carbon

TiAlN- Titanium aluminium nitride

CrAlN- Chromium aluminium nitride

ZrC- Zirconium carbide

ZrN- Zirconium nitride

PTFE - Polytetrafluoroethylene

PEEK - Polyetheretherketone

PAEK- Polyaryletherketone

PAO- Polyalphaolefins

UHMWPE - Ultra high molecular weight polyethylene

MoS₂ – Molybdenum disulphide

SU-8 - Epoxy

Ti - Titanium

Cr – Chromium

Sb₂O₃ – Antimony oxide

HMW-HDPE – High density Polyethylene

LixMoS₂ - Lithium intercalated molybdenum di-sulphide

WPU - Waterborne polyurethane

PA - Polyamide

MoO₃ - Molybdenum trioxide

SiO₂ – Silicon dioxide

ODA - Octadecylamine

PPS - Polyphenylene sulfide

POM - Polyoxymethylene

WS₂ - Tungsten disulphide

Rpm – Revolution per minute

PDMS - Polydimethylsiloxane

PIB - Porous thermosetting benzimidazole-containing aromatic polyimide

SEM - Scanning Electron Microscopy

EDX - Energy Dispersive Spectroscopy

TGA - Thermo-Gravimetric analysis

DMA- Dynamic Mechanical analyser

HSS - High Speed Steel

RF - Radio frequency

Wt% - Weight percentage

Vol% - Volume percentage