

# **PERFORMANCE EVALUATION OF METAL DICHALCOGENIDES AND CENOSPHERE FILLED PHENOLIC FRICTION COMPOSITES**

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**INDIAN INSTITUTE OF TECHNOLOGY DELHI**

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# **PERFORMANCE EVALUATION OF METAL DICHALCOGENIDES AND CENOSPHERE FILLED PHENOLIC FRICTION COMPOSITES**

by

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**Centre for Polymer Science and Engineering**

**Submitted**

**in fulfilment of the requirements of the degree of Doctor of Philosophy**

**to the**



**INDIAN INSTITUTE OF TECHNOLOGY DELHI**

**SEPTEMBER 2016**

**Dedicated**  
**to**  
**My Parents**

## **CERTIFICATE**

This is to certify that the thesis entitled “**Performance evaluation of metal dichalcogenides and cenosphere filled phenolic friction composites**” being submitted by **Mr. Rakesh Kumar Kachhap**, to the Centre for Polymer Science and Engineering (CPSE), Indian Institute of Technology (IIT) Delhi, India, is worthy of consideration for the award of the degree of **Doctor of Philosophy** in Polymer Science and Engineering and is an authentic record of original bonafide research work carried out by him under my supervision and has fulfilled the requirements for the submission of this thesis.

The ideas, experimental work, results, analyses and conclusions reported in this thesis are original and have not been previously, to the best of my knowledge, submitted, in part or full, to any other University or Institute for the award of any degree or diploma.

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**Rakesh Kumar Kachhap**

## Abstract

The thesis comprehensively deals with fabrication and evaluation of the friction composites based on fly ash cenosphere in combination with metal dichalcogenides:  $WS_2$  and  $MoS_2$  as high temperature lubricants, in view of the reported potential of flyash cenosphere to be used as multi functional filler in friction composite materials. The composites have been systematically evaluated for their physical, thermal stability, compressibility, hardness and thermo-mechanical properties: the tribo performances of the composites were evaluated on a Krauss friction tester (ECE R-90) and on a Chase friction tester (SAE J661a) for the assessment of fade-recovery and load-speed-temperature sensitivity characteristics respectively. The wear surface morphology have been investigated by using SEM-EDAX, both for size-scale and elemental composition analysis of the friction contact patch formation and wear debris generation and compaction mechanism. The study revealed that the combination of low cenosphere (40–45 wt%) and high metal-dichalcogenides (10–5 wt%) may exhibit performance synergism. For the  $WS_2$  and  $MoS_2$  composition the optimum tribo performances were obtained in the presence of cenosphere: dichalcogenide ratio of 40–42.5:2.5–7.5. Interestingly, the use of simultaneous combination of  $WS_2$ : $MoS_2$  in the ratio of 1:1 has exhibited superior performance when  $WS_2$ : $MoS_2$  was kept at 5:5 with minimum sensitivity to load-speed-temperature variations vis-à-vis lower fade (13–24%) and stable recovery (115–130%). Furthermore, the lubricant combination usage in friction materials lead not only to faster friction decay; a phenomenon attributed to rapid friction film formation vis-à-vis easy rolling of the cenosphere at the braking interface, but also lead to minimal friction fluctuation. The performance analysis and optimization of composition with regard to various performance defining attributes was carried out following various empirical and optimization methodologies. Friction performance analysis was carried out following a linear additive polynomial expression where as wear was analyzed following a modified Rhee equation that

assumes wear as a function of fade, recovery and disc temperature rises. The analysis revealed friction and wear to be mostly temperature activated. Analysis of performance data using Multi Criteria Decision Making (MCDM) model revealed that “lubricant combination-cenosphere” based composites show superior performance: particularly in drag- braking mode (Fade-recovery tests on dynamometer). Grey Target Theory (GTT) assisted optimization of the compositional influence and overall performance ranking of the various “metal dichalcogenide-cenosphere” combinations could successfully establish that cenosphere-MoS<sub>2</sub> combination may lead to superior overall performance. GTT further demonstrated that incorporation of MoS<sub>2</sub> enhances friction-stability, fade-resistance and arrests disc-temperature rise whereas MoS<sub>2</sub>-WS<sub>2</sub> combination facilitates enhanced recovery with a high performance  $\mu$ . WS<sub>2</sub> proves to be useful when wear is the key concern in designing high-performance friction materials.

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## **List of abbreviations and symbols**

|          |                                                |
|----------|------------------------------------------------|
| Al-MMC   | Aluminum Metal Matrix Composite                |
| AES      | Auger Electron Spectroscopy                    |
| AHP      | Analytic Hierarchy Process                     |
| ANOVA    | Analysis of Variance                           |
| BEEP     | Brake Effectiveness Evaluation Procedure       |
| CM       | Cenosphere-to-Molybdenum disulphide ratio      |
| CMW      | Cenosphere-to-(Molybdenum+Tungsten disulphide) |
| CNBR     | Carboxy Nitrile Butadiene Rubber               |
| CNSL     | Cashew Nut-Shell Liquid                        |
| COF      | Coefficient of friction                        |
| CW       | Cenosphere-to-Tungsten disulphide ratio        |
| EDAX     | Energy Dispersive Analysis X-ray               |
| FAST     | Friction Assessment and Screening Test         |
| FIB      | Focus Ion Beam                                 |
| DMA      | Dynamic Mechanical Analysis                    |
| DSC      | Differential Scanning Calorimetry              |
| DTA      | Dynamic Thermo-Gravimetric Analysis            |
| DTG      | Dynamic Thermo-Gravimetric                     |
| DTR      | Disc Temperature Rise                          |
| DTV      | Disc Thickness Variation                       |
| ECE R-90 | Economic Commission of Europe Regulation-90    |
| FC       | Friction Composites                            |
| FESEM    | Field Emission Scanning Electron Microscopy    |
| GAIA     | Geometric Analysis for Interactive Assistance  |
| GAXRD    | Glancing Angle X-Ray Diffraction               |
| GRA      | Grey Regression Analysis                       |
| GST      | Grey System Theory                             |
| GTC      | Grey Target Coefficient                        |
| GTT      | Grey Target Theory                             |
| HB       | Moh's Hardness Value                           |
| MCA      | Movable Cellular Automata                      |

|                           |                                                                |
|---------------------------|----------------------------------------------------------------|
| MCDM                      | Multi-Criteria Decision Making                                 |
| NBR                       | Nitrile Butadiene Rubber                                       |
| OVAT                      | One-Variable-At-a -Time                                        |
| PA-6                      | Polyammide-6                                                   |
| PAN                       | Polyacrylonitrile                                              |
| PDA                       | Performance Defining Attributes                                |
| PF                        | Phenolic Resin                                                 |
| PVW                       | Pulse Velocity Wave                                            |
| SAE                       | Society of Automobile Engineering                              |
| SEM                       | Scanning Electronic Microscopy                                 |
| SBR                       | Styrene Butadiene Rubber                                       |
| TBA                       | Third Body Abrasion                                            |
| TEM                       | Transmission Electron Microscopy                               |
| TOPSIS                    | Technique for Order Preference by Similarity to Ideal Solution |
| VPR                       | Styrene Butadiene 2-vinyl pyridine rubber                      |
| $\tan \delta$             | Tangent delta                                                  |
| $T_{\max}$                | Maximum Temperature                                            |
| $\mu$                     | Coefficient of friction                                        |
| $\mu_F$                   | Coefficient of fade                                            |
| $\mu_{\max}$              | Maximum Coefficient of Friction                                |
| $\mu_{\min}$              | Minimum Coefficient of Friction                                |
| $\mu_{\max} - \mu_{\min}$ | Friction fluctuations/Friction undulations                     |
| $\mu - v$                 | Coefficient of friction -velocity                              |
| $\mu_P$                   | Coefficient of performance                                     |
| $\mu_R$                   | Coefficient of recovery                                        |
| $\mu_S$                   | Coefficient of static                                          |
| $\Delta\mu$               | Friction Fluctuations                                          |
| $E'$                      | Storage Modulus                                                |
| $E''$                     | Loss Modulus                                                   |
| P/M                       | Particle Material                                              |
| S/N                       | Signal to Noise                                                |