PERFORMANCE EVALUATION OF METAL DICHALCOGENIDES AND CENOSPHERE FILLED PHENOLIC FRICTION COMPOSITES

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by

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Submitted

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

to the

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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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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_2 combination may lead to superior overall performance. GTT further demonstrated that incorporation of MoS_2 enhances friction-stability, fade-resistance and arrests disc-temperature rise whereas MoS_2-WS_2 combination facilitates enhanced recovery with a high performance μ. WS_2 proves to be useful when wear is the key concern in designing high-performance friction materials.

Contents

List of Figures

List of Tables

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	Polyacryolnitrile
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
μ	Coefficient of friction
μ_F	Coefficient of fade
μ_{max}	Maximum Coefficient of Friction
μ_{min}	Minimum Coefficient of Friction
$\mu_{max}-\mu_{min}$	Friction fluctuations/Friction undulations
μ-ν	Coefficient of friction -velocity
μ_P	Coefficient of performance
μ_R	Coefficient of recovery
μ_S	Coefficient of static
$\Delta\mu$	Friction Fluctuations
E'	Storage Modulus
E''	Loss Modulus
P/M	Particle Material
S/N	Signal to Noise