XPS ANALYSIS ON TRIBOFILM FORMATION STUDY FOR METAL SLIDING SURFACE

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DEDICATION

Special dedication to

My beloved mother and father;
Sarimah Binti Aliman and Md. Yasser Bin Sawijan,

My dear husband;
Fitri Norman

Supervisor;
Professor Ts. Dr. Zaidi Bin Embong

Co Supervisor;
Professor Dr. Erween Bin Abd Rahim

Thank you for all your support and encouragement during my master's degree studies.

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ABSTRACT

Mineral oils and Synthetic Ester (SE) are widely used as lubricants in manufacturing industry. However, the resources depletion and environmental pollution effects of these two lubricants occur and this research presents the significance of Modified Jatropha Oils (MJO) (vegetable oil) used as bio-based lubricants for machining lubrication. MJO was enhanced with Ammonium Ionic Liquid (AIL) and Phosponium Ionic Liquid (PIL) additives under selected nominal percentage weight; 1%, 10% for AIL and 1% for PIL. The difference in nominal percentage weight determined from the machining performance. This led to various results on thin film; also known as tribofilm where it is formed at the tool-chip contact region due to the friction occurred during sliding contact of two bodies in the presence of lubricants. Throughout the literature review, previous researches are aware regarding bio-based lubricants application to reduce pollution and formation of tribofilm built up by oxide layers at the tool-chip contact region. The mixtures of MJO, MJO+AIL1%, MJO+AIL10%, MJO+PIL1%, and SE samples undergone a physicochemical test under American Standard for Testing Materials (ASTM) D6751). Fourier Transform Infrared (FTIR) used to determine functional groups in the bio-based lubricant mixtures. X-ray Photoelectron Spectroscopy (XPS) was employed to study the characteristics of tribofilm formed and its composition in weight percentage. Physicochemical test and FTIR bandwidth vibration showed all lubricants satisfied the ASTM D6751 standard the fingerprints of alkane, alkene, and hydroxide from all samples tested. From the XPS findings, W4f curve represents the contribution of Tungsten. Narrow scan of W4f photoelectron peak showed no presence of WS_2 compound for the non-lubricated tool-chip contact region. However, the percentage concentration of the WS_2 compound has increased from the value of 9.27% for MJO+AIL 1% and 32.43% for MJO+AIL 10%. In conclusion, WO_2 is agreed as a sacrificial layer that its decrement correspond to the increment of WS_2 where the thermodynamic is the factor to the breaking of the chemical bonding, led to the chemisorption between W^{4+} ion from the cutting tool and the S^{2-} from the lubricants.

Therefore, this study proposed new oxide species namely as Tungsten Disulphide (WS2) developed after the machining process. It fits the criteria that tribofilm formed in the presence of lubricants via adsorption process.

ABSTRAK

Minyak mineral dann Ester Sintetik (SE) telah digunakan secara meluas dalam industry pembuatan. Tetapi, ini menyebabkan penghakisan terhadap sumbernya dan pencemaran udara yang berlaku sebagai kesan penggunaannya. Penyelidikan ini melibatkan Minyak Jatropha Terawat (MJO) yang dicampurkan bersama 1%, 10% Ammonium Ionic Liquid (AIL) dan 1% Phosphonium Ionic Liquid (PIL) bagi menambaik fungsi MJO untuk digunakan sebagai pelicir berasaskan bio dalam permesinan. Perbezaan peratusan IL menjurus kepada perbezaan kualiti permesinan. Ini menyumbang kepada kepelbagaian lapisan filem nipis atau dikenali sebagai tribofilm yang terhasil pada kawasan mata alat-cip yang mengalami geseran akibat pergeseran dua jasad tersebut dengan kehadiran pelincir. Kajian sebelum ini mengatakan pelicir berasaskan bio berpotensi mengurangkan pencemaran dan menyebabkan tribofilm terhasil dari binaan lapisan oksida pada kawasan mata alat-cip. Ujian fizikokemikal telah dijalankan bagi campuran sampel-sampel MJO, MJO+AIL1%, MJO+AIL10%, MJO+PIL1%, dan SE mengikut piawaian American Standard for Testing Materials (ASTM) D6751). Alat Fourier Transform Infrared (FTIR) juga telah digunakan bagi mengetahui functional group pada pelicir berasaskan bio. X-ray Photoelectron Spectroscopy (XPS) digunakan untuk mengetahui ciri-ciri tribofilm yang terhasil serta berat peratusan komposisi bagi kandungan kimia yang terdapat padanya. Ujian fizikokemikal dan getaran jalur FTIR menunjukkan kewujudan alkane, alkene, dan hydroxide. Analisa XPS menunjukkan lengkungan W4f mewakili elemen Tungsten. Imbasan sempit puncak fotoelektron W4f menunjukkan tiada kehadiran WS2 pada kawasan mata-alat cip yang digunakan tanpa pelincir. Walaubagaimanapun, kandungan WS2 bertambah menjadi 9.27% bagi MJO+AIL 1% dan 32.43% bagi MJO+AIL 10%. Kesimpulannya, WO2 telah dipersetujui sebagai lapisan penting kerana pengurangannya menjurus kepada kenaikan WS2 di mana factor termodinamik telah memecahkan ikatan kimia, mendorong kepada fenomena chemisorption di antara ion W4+ dari mata alat dan S2- dari sampel-sampel minyak pelincir. Oleh itu, kajian ini mencadangkan jenis

oksida baharu yang dinamakan Tungsten Disulfida (WS2) telah berkembang selepas proses permesinan. Ianya merangkumi ciri-ciri tribofilm yang terhasil dengan kehadiran pelincir melalui proses adsorption.

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LIST OF SYMBOLS AND ABBREVIATIONS

θ	-	Angle/Diffraction angle
%	-	Percentage
α	-	Tool rake angle
°	-	Angle degree
˚C	-	Degree Celcius
d	-	Depth of cut
d	-	Lattice spacing
λ	-	Wavelegth
l	-	Litre
f	-	Feed
v_c	-	Cutting speed
w	-	Width of cut
AIL	-	Ammonium Ionic Liquid
AISI	-	American Iron and Steel Institute
ASTM	-	American Society for Testing and Materials
Au	-	Aurum/Gold
BE	-	Binding Energy
CJO	-	Crude Jatropha Oil
COF	-	Coefficient of Friction
CO**2**	-	Carbon Dioxide
EP	-	Extreme pressure
ESCA	-	Electron Spectroscopy for Chemical Analysis
FAME	-	Fatty Acid Methyl Esters
FAST	-	Faculty of Applied Sciences and Technology
F_C	-	Cutting force
Fe	-	Ferum/Iron
FFA	-	Free Fatty Acid

FKMP	-	Faculty of Mechanical Engineering andManufacturing
FP	-	Flash point
H	-	Hydrogen
H_3PO_4	-	Orthophosphoric acid
H_2SO_4	-	Sulphuric aid
IL	-	Ionic Liquid
ILs	-	Ionic Liquids
kN	-	kilonewton
KOH	-	Potassium Hydroxide
KV	-	Kinematic Viscosity
MJO	-	Modified Jatropha Oil
MoS_2	-	Molybdenum Disulphide
MQL	-	Minimum Quantity Lubrication
MWF	-	Metalworking Fluid
MWFs	-	Metalworking Fluids
N	-	Nitrogen
NaOH	-	Sodium Hydroxide
NC	-	Numerically controlled
O	-	Oxide
P	-	Phosphorus
PIL	-	Phosphonium Ionic Liquid
SE	-	Synthetic Ester
TAN	-	Total Acid Number
TMP	-	Trimethylolpropane
UHV	-	Ultra High Vacuum
VI	-	Viscosity Index
W	-	Tungsten
WC	-	Tungsen Carbide
WO	-	Tungsten Oxide
WO_2	-	Tungsten Dioxide
WO_3	-	Tungsten Trioxide
WS_2	-	Tungsten Disulphide
wt%	-	Weight percentage

XRD - X-ray Diffraction

XPS - X-ray Photoelectron Spectroscopy

ZDDP - Zinc dialkyldithiophosphates

m / min - meter per minute

mm / rev - millimeter per revolution/feed per revolution

C_2H_5OH - Ethanol

PREMACH - Precision Machining Research Center

$NaOCH_3$ - Sodium Methoxide

SEM-EDX - Scanning Electron Microscope-Energy Dispersive

MJO+AIL 1% - Modified Jatropha Oil blended with 1% of Ammonium Ionic Liquid additives

MJO+AIL 10% - Modified Jatropha Oil blended with 10% of Ammonium Ionic Liquid additives

MJO+PIL 1% - Modified Jatropha Oil blended with 1% of Phosphonium Ionic Liquid additives

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i. Mona Alis Md Yasser, Zaidi Embong, Erween Abd Rahim, Amiril Sahab Abdul Sani, Kamaruddin Kamdani. "Study of ionic liquid (AIL and PIL) viscosity and its functional groups through heat treatment on cutting tool surface using Fourier-Transform Infrared Spectroscopy" *Trans Tech Publications Ltd. Materials Science Forum, (2020) 981,* 98–103.

CHAPTER 1

INTRODUCTION

1.1 Background of the study

Metalworking fluids (MWFs) are important in cutting process as it provides cooling and lubrication to the cutting zone where direct contact occurs as the tool-chip and tool-workpiece interfaces (Abdul Sani, 2018; Astakhov & Davim, 2008). This is to enhance the surface finishing including the residual stress, cold work, surface roughness, and surface transformations that have potential in affecting the fatigue and stress corrosion (Astakhov & Davim, 2010). The type of lubricants is to be considered for cutting applications. The use of petroleum-based lubricant products has been applied abroad. These petroleum derivatives lubricants were determined to have good additives for antioxidant, antiwear, and viscosity improver, however, it is toxic and harmful to human health, wildlife, and environment (Samion, 2017). A number of publications regarding the use of bio-based oil as lubricant stated that vegetable oil such palm oil and jatropha oil offer the advantage in renewability and high biodegradability characteristics (Samion, 2017; Abdul Sani, 2018). Therefore, additives are required in improving the existing properties of the base oil and the machining performance to minimize the wear and friction (Samion, 2017).

It is good to note that there is a direct correlation between manufacturing procedure and the surface integrity where usually the last machining process is assumed of being accountable for rupture. Aside from the type of lubricants, the lubrication techniques play a negligible role in the cutting process. There are a few techniques in delivering the lubricants at the contact region; dry, flood, cryogenic, and Minimum Quantity Lubrication (MQL) techniques (Lawal *et al*., 2012). One of the previous publications proposed that MQL gives a better surface finishing and prolong

the tool life when applied with vegetable oil due to its ability to reduce the cutting forces (Astakhov & Davim, 2010). This is supported by another study involving palm oil use as machining lubricant that reported MQL technique as more efficient in improving the formation of lubrication film on the interfaces of tool-chip and tool-workpiece (Rahim & Sasahara, 2017).

Tribology is a study on the interactive surface between two bodies that experiencing relative motion with the presence of lubrication and produce friction and wear as the consequences. Tribofilm is the interfacial layer that formed as the consequences of sliding contact, friction and wear, with the presence of lubricants. Tribofilm is also known with other names such as tribolayer, third body, self-lubricating, and transfer film where the definition might vary (Skiöld Nyberg H. 2014). The use of tribofilm term is more preferred for this research. This study was performed to investigate the tribofilm formation throughout the works with the applications of Modified Jatropha Oil as bio-based lubricant blended with 1%, 10% of Ammonium Ionic Liquid and 5% of Phosphonium Ionic Liquid additives delivered by Minimum Quantity Lubrication (MQL) technique for orthogonal machining.

1.2 Problem statement

It is explained that the applications of MWFs in machining process are necessary in order to provide cooling and lubrication where several techniques are applicable in delivering the lubricant at the cutting tool-workpiece contact region. Therefore, Davim (2008) has proposed that Minimum Quantity Lubrication (MQL) technique is able to provide a better lubrication to the contact region compared to other techniques such as dry cooling, cryogenic cooling, and flood cooling technique. V. P. Astakhov and Davim (2008) added that the lubrication through MQL technique is able to deliver a minimum quantity of lubricant evenly to the interfaces since it is sprayed in the mixture form of air and oil known as an aerosol with small quantity of lubricant supply. It is possible that the simple diffusion to occur between the aerosol with the air surrounding during the supply of lubricant. Hence, changes in environment and air composition might lead to health issue. Although there is the fact that mineral oil composition offers good additives such as antioxidant, antiwear,

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