CHARACTERIZATION AND EVALUATION OF THERMAL PROPERTIES OF A NANO LUBRICANT BY THE ADDITION OF MOS2 NANOPARTICLES

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Abstract
A wet chemical synthesis method has been developed for the production of molybdenum disulfide Nano powders. The synthesis as carried out in an aqueous solution, by forming Mo: citrate complexes that react with ammonium sulfide in order to achieve MoS2 Nano particles. The characterization has been carried out using Scanning Electron Microscopy (SEM) and Diffract meter (XRD). Particle sieve analysis also used to determine mean size of the nano particles. It has been demonstrated that the citric acid content affected not only the formation of the Mo: citrate complexes but also the morphology of the final products, through a template action during the nucleation and growth of the particles. The dynamic viscosity and flash & fire points of the lubricant is increased about 30% and 7% respectively.
Index terms: Molybdenum disulfide (MoS_2), X--Ray Diffraction, Scanning Electron Microscopy

I. INTRODUCTION

The property of reducing friction is known as lubricity(Slipperiness). Any material with particle size is less than 100 nanometers, is defined as Nano particle. When the material is made into nano particles, reactivity increases. Smaller the particle size, higher the surface area. Nano particles have a very high surface area to volume ratio. Due to this a higher percentage of atoms (in nano particles) can interact with other matter. Therefore surface area (measured in square meters per gram) is most important unit of measure for a Nano lubricant. Higher the surface area, higher will be the lubricity.

A. Fluid Lubricant

Fluid lubricants are used in almost every field of human technological activity, they reduce frictional resistance, protect the contacting surfaces of engines against wear, remove wear debris, reduce heat and contribute to cooling, improve fuel economy and improve emissions. The liquid lubricants usually used are mineral oils and synthetic oils. The mineral oils are most commonly used because of their cheapness and stability.

B. Solid Lubricant

Graphite and Molybdenum disulfide (MoS_2) are the predominant materials used as solid lubricants. The MoS_2 nano powder is used as a lubricant by mixing it with the commercial lubricants because of the following reasons:

- The rolling friction is comparatively less than sliding friction.
- Low starting and running friction except at very high speeds
- Ability to withstand momentary shock loads
- Mostly used in rolling contact bearings.

Table 1 shows the Physical and Technical properties of Molybdenum disulfide (MoS_2).

Property	Molybdenum disulfide
Color	Blue-silver Gray

Appearance	Crystalline solid
Melting point	1185^0C
Boiling point	450^0C
Density	$5060Kg.m^3$
Molecular weight	160.08
Thermal stability in air	Coefficient Of Friction <0.1 @316^0C Coefficient Of Friction 0.1 to 0.5 @594^0C
Load bearing ability	2,50,000psi
Lubrication temperature range	Ambient : -185^0C to 350^0C Vaccum : -185^0C to 1100^0C
Chemical durability	Inert substance, Non-toxic
Magnetism	Non-Magnetic

Table 1: Physical And Technical Properties

II. LITERATURE REVIEW

The study of nano particles for the lubricant applications is necessary to obtain greater efficiency in the engines and other machine parts where the friction is the main criteria. In almost all the machine elements friction and wear are the main properties which are taken into consideration. So improving the properties of the lubricants is done by using nano particles where the reactivity of nano particles increases as the size of nano particles reduces to nano scale.

Previous Works

L. Rapoport, Y. Feldman:The main goal of the present work was to study the tribological properties of IF-WS_2 particles in comparison to 2H-WS_2 and MoS_2 platelets over a wide range of loads and sliding velocities. The average size of the particles of IF-WS_2 particles was found to be 120nm,while that of 2H-WS_2 and $MoS_2$was 0.5μm and 4μm. chemical reactivity of the powders were also verified by heating in ambient atmosphere.

Wang Hai-doua, XuBin-shia:To develop a new way to produce the molybdenum disulfide (MoS_2) solid lubrication film, the following two-step chemical reaction technique was attempted. Firstly, a Mo film was prepared by multi-arc ion plating technique, and secondly the Mo film was sulfurized by a low temperature ion sulfuration technique to obtain the MoS_2 solid lubrication film. This MoS_2film was a composite film consisted of MoS_2 and Mo. The lubricant MoS_2 is dominant in the surface and metal Mo is dominant in the deep layer. It is an ideal frictional surface. The tribological properties showed that the solid lubrication MoS_2film possessed an excellent anti-friction property.

The inorganic solid lubricant molybdenum di sulfide (MoS_2) is a kind of solid lubricant, which has been applied extensively for a long time. Its crystalline microstructures, tribological properties and anti-friction mechanisms have been studied .There are lots of techniques for preparing a MoS_2film, such as magnetron sputtering a MoS_2film, such as magnetron sputtering [4,5], ion beam assisted deposition anode oxidation combined with heat treatment, chemical reaction and high temperature annealing as well as sol-gel method.

The above techniques are useful, but obviously have the disadvantages, such as inaccurate atomic ratio between sulfur and molybdenum and low deposition efficiency, or oxidation after high temperature annealing, or poor bonding strength with substrate to develop the preparation technique for MoS_2 film, combined the advantages of high deposition efficiency in multi-arc ion plating, and the simplicity and low cost of low temperature ion sulfuration .

III.METHODOLOGY

The author adopted at two-step chemical reaction technique. Firstly, the multi-arc ion plating has been employed to prepare a layer of hard molybdenum (Mo) film on the substrate, and then used the low temperature ion sulfuration to sulfurize the Mo film, finally obtained a solid lubrication MoS_2 film in situ. In the present paper, the characterization and tribological properties of the MoS_2 film was studied in detail.

A. Synthesis Of Molybdenum Disulfide (MoS_2)

Wet Chemical Synthesis

Wet chemical methods is the common (slangy) name for a group of methods used for producing Nano- and ultra-dispersed inorganic powders from aqueous and non-aqueous solutions. The term "wet chemical methods" emerged in contrast to conventional and solid-state synthesis methods of compounds and materials widely used also in ceramics manufacturing. Today the term refers to a group of methods of powder and material production using liquid phase at one of the process stages. The main differences between wet chemistry products and similar products of solid-phase synthesis are much smaller grains (crystallites) and, usually, lower temperature and shorter duration of phase formation.

The synthesis of the nano structured MoS_2 powders was carried out by means of a new modified version by optimizing the ratio of reactants that are used in the synthesis process of Molybdenum disulfide nanoparticles.

B. Reactants Used

The reactants were ammonium hepta molybdate tetrahydrate $(NH_4)_6Mo_7O_{24}4H_2O$, citric acid $C_6H_8O_7$ and ammonium sulfide $(NH_4)_2S_2$.

C. Experimental procedure

STEP 1: Preparation Of Aqueous Solution

(a) An aqueous solution was prepared in a 250ml beaker by dissolving suitable amounts of ammonium molybdate and citric acid in distilled water in the following MO: citrate molar ratios of 1:1, 1:2 and 1:4.
(b) The amount of citric acid as reducing agent is optimized with the aim to obtain MoS_2 nano particles with controlled particles size.
(c) The quantity of citric acid employed in the synthesis was considered as the significant parameter and it was varied accordingly for optimization.
(d) The solution was kept at 90^0C on a magnetic stirrer with hot plate and is allowed to stir the ingredients in a beaker.

STEP 2: Mixing Of Sulfide Reagent

(a) After the complete dissolution of the reactants, 3.75 ml of a 20 wt% solution of ammonium sulfide in water was added drop-by-drop.
(b) The ammonium sulfide is added according to the ratio in which the reactants Mo and Citrate are mixed.
(c) The solution is allowed to stir till all the reactants are completely mixed.
(d) The solution changes from clear to dark red, and finally to **black.**

STEP 3: Hot Air Oven

(a) After settling the black liquid solution in the beaker, it is covered with aluminum foil with holes and is kept in Hot air over
(b) The temperature is set to 250^0C for about 2hrs till the liquid is completely evaporated in the beaker.
(c) After the precipitate is completely evaporated in the beaker, it is taken out and is crushed to fine powder.

STEP 4: Calcination In Muffle Furnace

(a) The fine powder obtained after crushing is taken into the silicon crucible and is further crushed.
(b) Then it is introduced into the Muffle furnace**.** The temperature is set to 900^0C.
(c) The furnace gets heated up slowly and reaches to the setup temperature.
(d) Now the crucible is kept for 2hrs in the furnace after reaching this temperature.
(e) The obtained powder is crushed and collected in the vials.
(f) This obtained sample is now sent for characterization.

IV.CHARACTERIZATION OF MoS_2 NANOPOWDER

Most properties of nano particles are size-dependent. In fact, the novel properties of nano particles do not prevail until the size has been reduced to the nanometer scale.Figusre 1 shows the nano lubricant sample.Figure 1 shows the nano lubricant sample.

Figure1: Mixure of Nano Powder in Lubricant.

A. Nano-Materials Structure Analysis By X-Ray Diffraction (XRD)

The analysis of crystalline materials, semi-crystalline materials and amorphous materials to nanometer scale are possible with X-Ray Diffraction. Crystallite dimensions and size are fundamental characteristics of nano materials and microstructure (nanostructure), and are useful for understanding and controlling bulk properties. Small crystallite size results in broadened diffraction patterns. Analysis of peak shapes give information about crystallite size and other aspects of microstructure, particularly lattice distortions due to composition or micro-strain variations, and faulting.

Figure 2 shows the XRD patterns of $MoS2$ nano particles.

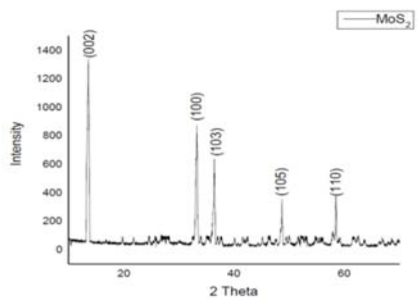

Figure2: XRD patterns of the MoS2 nanosized particles

B. Particle Size Analysis

Particle size analysis (PSA), particle size measurement, or simply particle sizing is the collective name of the technical procedures, laboratoryechniques which determines the size range, and/or the average, or mean size of the particles in a powder or liquid sample.studying the graph obtained from particle size analyzer as shown in Fig.3, the mean diameter of the nano particles obtained is found to be **63.6 nm.** The cumulative percentages are obtained. The mean particle size for the first 10% of the sample is found to be 21.2nm, the mean particle size for 50% of the sample is obtained as 44.5nm, the mean particle size for 90% of sample is 137nm. The mean of the final sample is found to be 63.6nm.Figure 3 shows the mean particle size analysis.

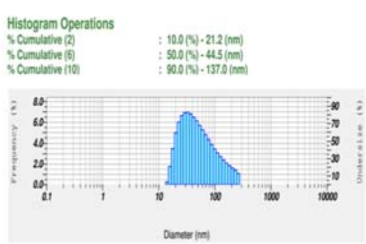

Figure3 : Mean particle size analysis

V. VISCOSITY MEASUREMENT

A. Formula used to determine Viscosity

Thus the absolute viscosity of the given liquid is found as follows:

$$\acute{\eta}2 = \frac{\acute{\eta}1 \times \rho2 \times T2}{\rho1 \times T1}$$

$\acute{\eta}_1$– Absolute viscosity of water.

ρ_1– Density of water.

T_1-Time of flow of water.

$\acute{\eta}_2$–Absolute viscosity of liquid.

ρ_2–Density of liquid.

T_2– Time of flow of liquid.

VI. FLASH AND FIRE POINTS MEASUREMENT

A. Flashpoint

With this test, the sample is confined in a closed container into which the pilot flame is periodically introduced. Additionally, the lubricant is agitated during the heating period and the lowest temperature at which a flash appears is recorded. As with the COC method, a considerable amount of fluid and time is needed to perform the test. However, fully automated instruments are available from various suppliers. In measuring fuel dilution, one advantage the Pensky-Marten apparatus has over the COC method is improved sensitivity to lower concentrations of fuel dilution

B. Fire Point

The fire point of a fuel is the temperature at which it will continue to burn for at least 5 seconds after ignition by an open flame. At the flash point, at lower temperature, a substance will ignite briefly, but vapor might not be produced at a rate to sustain the fire. Most tables of material properties will only list material flash points, but in general the fire points can be assumed to be about 10 °C higher than the flash points.

Neither the flash point nor the fire point is dependent on the temperature of the ignition source, which is much higher.

Table 2 shows the comparison of flash and fire points of lubricants with and without the addition of nano particles.

Lubricant(MoS2)	Flash Point (^0C)	Fire Point (^0C)
Without Nano Particles	156	168

With Nano Particles	170	182

Table 2: Comparison Of Flash Point And Fire Points of Lubricants

VII. RESULTS AND DISCUSSIONS

The results obtained above clearly shows that the tribological properties of the lubricants are improved by introducing Nano sized powders into the lubricant oils. The properties that are effected by addition of nanoparticles into the lubricants are viscosity, wear of the machine element, friction between the rubbing surfaces, flash point and fire point.

The dynamic viscosity of the lubricant is increased from 85Pa-sec to 111 Pa-sec which is a good sign for the commercial lubricants available in the market. The oil that is used for mixing the Nano powder is MOTOROL 20w40 2T oil. This is one of the finest commercial lubricants which are available in more quantity now-a-days. The viscosity, flash point and fire point are improved for this lubricant.

VIII.CONCLUSIONS

A wet chemical synthesis method has been developed for the production of molybdenum disulfide Nano powders. The final MoS_2Nano powders were highly amorphous and softly agglomerated with a mean particle size of about 63.3 nm.The results obtained by proper mixing of Nano sized MoS_2 in customized lubricants enhanced the basic properties like viscosity, fire & flash points that facilitates the engine to communicate the environment around it to dissipate the excess heat that gets accumulated on the walls of the cylinder, which in turn helps the engine to increase its life time by reducing the stress damages that are caused by the continuous exposure with high temperatures, which disturbs the grain distributions of the metallurgical alignments in the materials of the cylinder.Therefore we conclude that the friction can be reduced by introducing the Nano sized MoS_2 particles into the lubricants.

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