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Characterization of thermally evaporated MoS_2 thin film coatings

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Abstract

Tool steel is extensively used in metal cutting and forming operations. Its surface properties such as wear resistance and hardness can be improved by various surface modification methods. Conventional thermo chemical surface modification methods like carburizing, nitriding and boroding will not be able to provide the intensive energy source necessary to modify surface structure without disturbing microstructure and properties of core. One of the promising methods to improve wear resistance is thermal evaporation of Molybdenum disulphide (MoS_2) powder on tool steel substrates. The metallographic study and chemical composition analysis of thermally evaporated MoS_2 coated thin films were conducted using scanning electron microscope integrated with energy dispersive x-ray spectroscopy. The samples were observed using the scanning electron microscope while the EDX detector was used to collect composition data from the sample. The adhesion of thin film with substrate is evaluated by conducting a film substrate adherence test. A low friction, wear resistant and well adherent thin film of molybdenum disulphide is successfully deposited on tool steel substrates by thermal evaporation technique and characterized.

Keywords: Tool steel; Thermal evaporation; MoS_2.

1. Introduction

Solid lubricant coatings can be classified in to hard and soft coatings. Hard coatings include oxides, carbides, nitrides, borides and low dehydrogenated diamond like carbon. Soft coatings include polymers, halides, graphite and molybdenum disulphide [1]. Thin films of low friction and wear resistant MoS_2 coatings can be deposited on steel

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substrates using different PVD techniques. MoS_2 has anisotropic crystal structure which consists of layer molybdenum atoms arranged in hexagonal array with each molybdenum atom surrounded by six sulfur atoms placed at corners of triangular prism. There is a strong covalent bonding within the MoS_2 sandwiches and weak vandervaal forces exist between the sandwiches. The weak bonding between layers of MoS_2 results in reduced shear strength and hence low friction in the sliding direction. It has been reported that film substrate adhesion strongly influences the wear life of the film, stronger the adhesion, longer the wear life [2].

Several Researchers produced MoS_2 films using sputtering process .RF sputtered MoS_2 coatings show low friction coefficients, better adhesion and low wear rates. DC Closed field unbalanced magnetron sputter ion plated MoS_2 films exhibit close compositional and micro structural control, good adherence and good uniformity [3]. However the cost of molybdenum target in sputtering is expensive. MoS_2 films can be produced by vaporizing the MoS_2 powder and deposit on substrates by thermal evaporation in a cost effective manner.

Xiying Ma et al found a simple method of modified thermal evaporation deposition and fabricated MoS_2 film of atomic thickness [4]. The morphology and structures were characterized by atomic force microscope. The AFM image has shown many MoS_2 slices of 50-200 nm in length and 5-6 nm thickness which is about ten layers of MoS_2. The sub layers have shown that there is large uniform continuous MoS_2 film with deep yellow color. It was observed that the film became amorphous with the increase of MoS_2 content in the films[5]. H.Wang et al proposed a new method of developing composite MoS_2 film by combining metal molybdenum with solid lubricant MoS_2.P.La et al observed that the microstructure of $MoSi_2$ coatings is related to the MoS_2 content in the coatings [6]

K.M. Garadkara et al fabricated MoS_2 thin films by chemical bath deposition technique and reported that films are polycrystalline in nature having hexagonal phase[7]. S. Voronin et al developed a method of depositing MoS_2 coatings by thermal diffusion synthesis as an alternative to the costlier PVD Coatings[8]. The thickness of the MoS_2 coatings was found to be in the range of 5 to 60 microns. The optical image of the MoS_2 coating surface showed the presence of near round sintered grains. The surface roughness of the coatings was found to be 3 to 5 microns. Da-Yung Wang et al developed a composite MoS_2-Ti film with increased wear life and micro hardness [9]. G.Weise et al reported that the MoS_2 films are found to be nanostructured and is strongly influenced by deposition process conditions [10]. In the present paper, MoS_2 thin films were deposited on high speed steel substrates by thermal evaporation. The coated samples were characterized by scan electron microscopy, EDX analysis, coating thickness measurement, and film substrate adherence test.

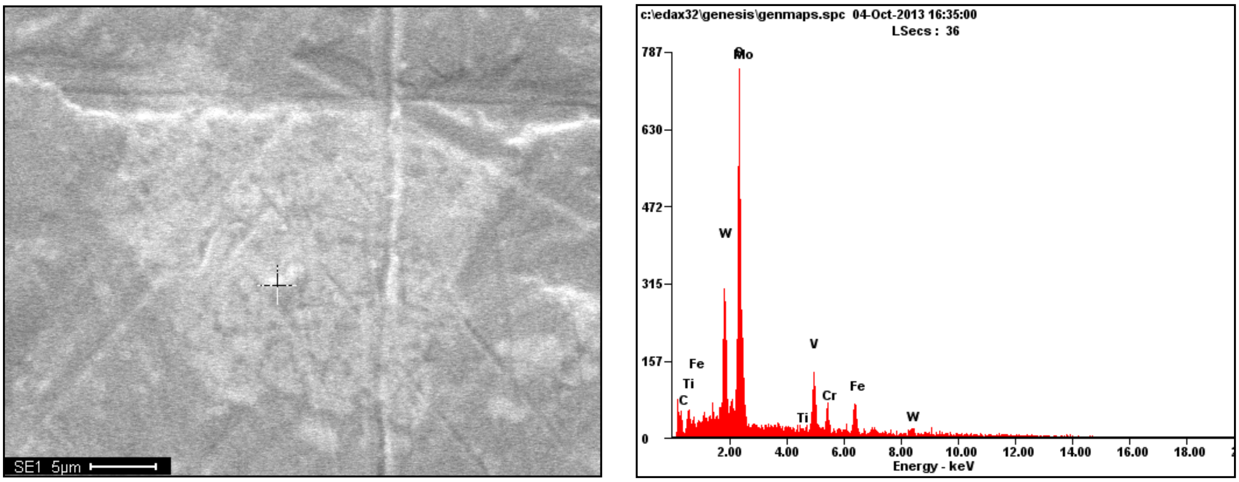
2. Experimental Procedure

The MoS_2 thin films were deposited on high speed steel surfaces by thermal evaporation deposition process. The deposition system consists of a chamber, a vacuum pumping system together with all electrical components necessary for coating process. The chamber is fabricated from stainless steel with a window for visual inspection of surface coating process. Before deposition, the chamber is evacuated by a Diffack pump and backed by a 250 litres/min double stage direct driven rotary vacuum pump. A boat type filament holder is fixed to low tension live electrode and a earth electrode. This resistance filament heater is used to evaporate the source material. The high speed steel substrates of 50 X 12 mm^2 and thickness 1mm in size were ground, polished using silicon carbide emery paper. The substrates were cleaned with acetone; ethanol and de ionized water, etched with natal solution and placed on the top of the chamber. The working pressure in the chamber was kept at $5X\ 10^{-6}$ Pa and substrate is heated to 160 ° C. In our experiment the source material pure MoS_2 powder of about 15 grams is placed on filament boat. The morphology of the coated samples were characterized by SEM and EDX Analysis. Film substrate adherence test is performed to evaluate bond strength of the film with substrate.

3. Results and Discussion

SEM Micrograph of MoS_2 thin film at 6000 X magnification is shown in Fig 1.The film shows distribution of grains randomly and reasonably covers well with the tool steel substrate. The increase of molybdenum concentration (31% by wt and 15% At%) is observed in this coating zone. However the sulfur concentration is detected as 8% by wt and 14% At%. This has revealed that MoS_2 is deposited on the tool steel substrates. The amount of Mo and S atoms formed in this study will be enough for formation of lamellar structure of MoS_2. The lamellar structure of MoS_2 will result in super low friction during friction, as confirmed in previous research study [11]. The substrate elements Fe, C, W were detected in the EDX spectrum of coatings and this has indicated that these elements had diffused in to the coating. Oxygen content was not detected in the EDX spectrum and this has indicated that porosity is almost absent in the coating. This is due to the low temperature nature of the thermal evaporation process.

EDX spectrum has shown evident peaks of W,Mo,C,Fe and V.A very small amount of impurity in of the form of Ti is detected in the EDX spectrum. The At% of carbon in this coating zone was 45.42% and this is due to carbide formation in the coating. This carbide formation at the surface will improve the wear resistance substrate. Fig 2 and 3 shows the SEM Image of MoS_2 thin film at 400 X and 100 X respectively.



KV 15.0 MAG 6000 TILT 0.0 MICRONSPERPIXY 0.093

Fig 1 SEM Micrograph of thermally evaporated MoS_2 coated tool steel substrates(b) EDX spectrum

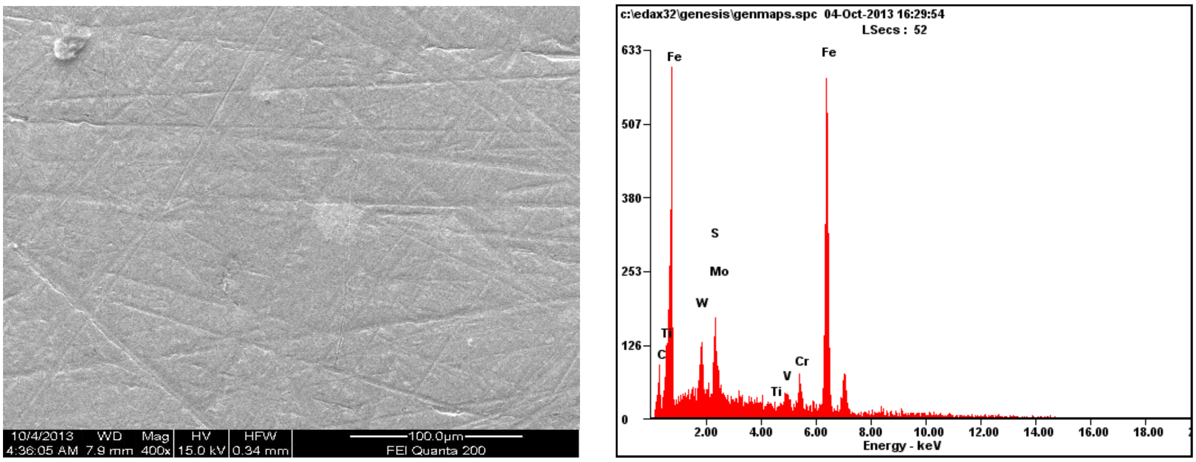


Fig 2 SEM Micrograph of thermally evaporated MoS coated tool steel substrates(b) EDX spectrum

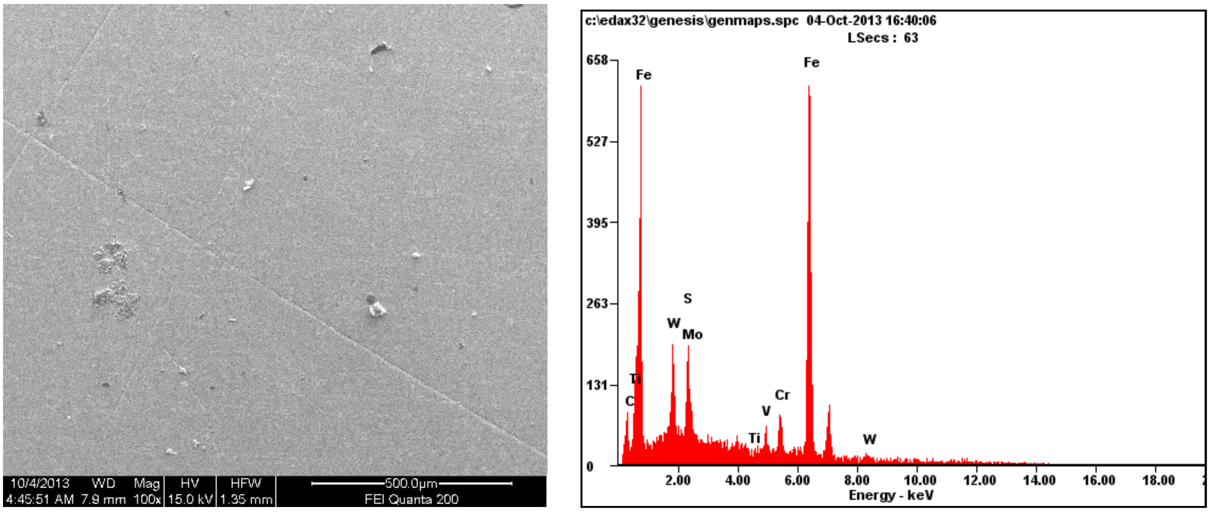


Fig 3 SEM Micrograph of thermally evaporated MoS coated tool steel substrates(b) EDX spectrum

Film substrate adherence test is conducted as in [2]. MoS_2 Coatings showed total failure at 53 N, with delamination starting at 32 N. The optical examination of the coatings after the scratch test has confirmed this result. The structure and microstructure of PVD coatings strongly depend on their thickness. An exact estimation of coating thickness can be performed by different techniques with different accuracies. The coating thickness was assessed in the present study using ellipsometer. The coating thickness of MoS_2 film was found to be 250 nm. Holmberg analyzed the various PVD and CVD techniques for lubricated coated surfaces and reported that thin MoS_2 coatings can provide low friction and excellent wear protection in dry sliding conditions[12]. The coating thickness achieved in this study will be able to provide low friction and good wear resistance.

4. Conclusions

MoS_2 thin films were deposited using a relatively simple thermal evaporation technique. Thermal evaporation is a promising technique for deposition of thin MoS_2 films and will be used to process coatings with compact microstructure, high micro hardness, low porosity and metallurgical bonding.

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