

EXPLORING THE ANTISTICKING PROPERTIES OF SOLID LUBRICANT THIN FILMS IN TRANSFER MOLDING

Sam Zhang¹, Xianting Zeng², Zhenggui Tang², Ming Jen Tan¹

1. *School of Mechanical and Production Engineering, Nanyang Technological University, 50 Nanyang Avenue, Singapore 639789, msyzhang@ntu.edu.sg*

2. *Gintic Institute of Manufacturing Technology, 71 Nanyang Drive, Singapore 637085*

In the plastic molding industry, plastic parts like pager and handphone cases, plastic containers, etc. are formed in a mold by applying temperature and pressure. The transfer molding is the standard workhorse for the electronics industry. Although the transfer molding is widely used, it is far from being optimized. Mold sticking is a serious practical problem in this industry. A solution to the problem is to apply mold-releasing agents on the mold to act as a lubricant layer between the plastic and the mold. This easily results in stains and degraded surface finish. This paper investigates the effectiveness of solid thin films on reducing the adhesion between polymer and mold steel of different surface roughness. WS₂, MoS₂, and DLC coatings are deposited on test surfaces via unbalanced magnetron sputtering before polymer blocks are molded on and pulled apart using an Instron Machine. The force required to separate the plastic part and the mold steel is used as an indication of the stickiness. After the separation, the coating surface is also examined under microscope for stains and polymer residues. The coatings are characterized using Raman spectroscopy and contact angle measurements. Generally, the stickiness increases with initial surface roughness for all coatings. Initial test indicates that the DLC coating has the highest contact angle with water (100°) and the best anti-sticking properties among the samples tested, and could reduce the stickiness by 80% as compared to bare steel.

1 Introduction

Mold-sticking accounts for a significant part of the down time in the plastic molding industries. Yet, there is no satisfactory solid antisticking coating for the process, especially for application on the precision mold and parts, and neither is there a proper testing jig and/or procedure for antisticking effect evaluation.

Conventional releasing agents are solvent or aqueous solutions in aerosol, liquid or paste form, applied to mold surface by spraying or painting. The carrier vehicle (solvent or water) evaporates, leaving a thin film coating on the mold. Another type is paste and liquid waxes commonly used in polyester, vinyl ester and epoxy composites. It works well on highly porous mold surfaces but is labour intensive and time consuming because wax film builds up quickly and need frequent stripping. They may also migrate onto the mold surface, causing poor adherence or blisters in a surface finishing coat. Polyvinyl alcohol (PVA) is a film forming barrier release agent most widely used to polyester glass fiber molds. PVA has excellent resistance to vaporizing styrene from new and "green" molds. When the part releases, PVA sticks partly to the mold and partly to the part. It must, therefore, be water washed from both surfaces, after molding, and before continued use. Other release agents include silicones in aerosol form, polymeric release agent such

as fluoro-telomers and polydimethylsiloxanes, and vegetable derivatives, fluorotelomers, and polymeric blends, etc. [1] However, most mold release agents are not resistant to high temperatures and decompose in applications which results in contamination of the plastic part. All these mold release agents requires frequent spraying and cleaning thus is time-consuming and affects productivity.

Amorphous carbon (a-C or DLC), MoS₂ and WS₂ are good solid lubricating materials. Applying these materials to plastic molds to achieve good bonding strength between the coating and the mold surface and antisticking properties between the coated surface and the epoxy parts is the real challenge. Physical vapor deposition can be used to deposit hard, lubricious and smooth coatings, and is used in this paper to study the antisticking effect of a few coatings on mold steel with different surface patterning.

2 Experimental procedures

2.1 Testing blocks

Test sample blocks of different surface textures ranging from less than 0.1 micron to 2.0 microns in arithmetic roughness (R_a) were prepared from mold steel according to industrial practice. Each test block had one textured surface measuring 25 mm x 10 mm. The other dimension of the block was 25 mm in height. To facilitate pulling operation without slippage, a through hole of 5 mm in diameter was drilled in the block towards the end farther away from the textured surface. A small tapered steel block with a 5 mm through hole at the center was also prepared to be molded in the epoxy. Steel pins would go through these hole to facilitate pulling in determination of the stickiness.

2.2 Coating process

There are a number of techniques to deposit amorphous carbon [2], MoS₂ [3] and WS₂ [4, 5, 6] coatings. In this study, the unbalanced magnetron sputtering [7, 8, 9] was used to deposit DLC, MoS₂ and WS₂ coatings of about 1 μ m thickness to the textured surface of the testing block. In synthesis of diamond like carbon, graphite target was used, in synthesis of MoS₂ and WS₂, MoS₂ and WS₂ sputtering targets were used respectively. The background pressure in the sputtering chamber was pumped below 5×10^{-5} Torr before the sputtering took place. The substrates were placed in a rotary sample holder with the textured surface facing the rectangular (330 x 133 mm) targets (99.9% purity graphite or MoS₂ and WS₂). A standard radio frequency (13.56 MHz) bias power was applied to the substrate. To compensate the loss of sulfur during deposition, limited amount of H₂S gas was introduced as reactive gas [10].

2.3 Coating Characterization

After coating, Raman spectra were obtained with a Raman imaging microscope (Rennishaw Model 127) using a He-Ne laser beam of 632.8 nm as the excitation source. The surface contact angle between the coating and deionized water was measured using a Rame-Hart goniometer for samples of smooth surface ($R_a < 0.1 \mu$ m) --- for rougher surfaces, measurement of contact angle was not possible and inaccurate because the coating followed the contour of the wavy surface. The coating thickness was characterized using a Laser Stylus Profilometer (Rank Taylor Hobson Form Talysurf Series).

2.4 Molding and pulling

After coating, an epoxy block was molded onto the testing block on the textured and coated surface. A special transfer mold was fabricated for this purpose. The molding was done at a manually operated Lauffer transfer-molding machine (Lauffer Pressen Model VSKO 135) at a pressure of 1400psi. After molding, the epoxy block was cured at 175°C together with the steel block for two hours before jiggging onto Instron machine to measure the force required to pull the epoxy block away from the steel block. The maximum force (or tensile stress) required to pull the epoxy block away was taken as the measurement of stickiness.

3 Results and discussion

3.1 Raman spectroscopy

The Raman spectrum for DLC coating was typical [11]. Raman peaks of WS₂ coating were illustrated in Figure 1. Also plotted in the figure were the Raman peaks from powder-sprayed WS₂ coating as comparison and WS₂ powders as reference. Even though the sputtered coating demonstrated a slight peak shift, it was seen from the plot that the Raman curves of the WS₂ coatings from either deposition method had identical peak positions compared to those of the WS₂ powders. The powders had the least background, the sputtered coating displayed much higher background signifying the existence of amorphous component. A slight change in the relative peak intensity was also experienced in the sputtered coating.

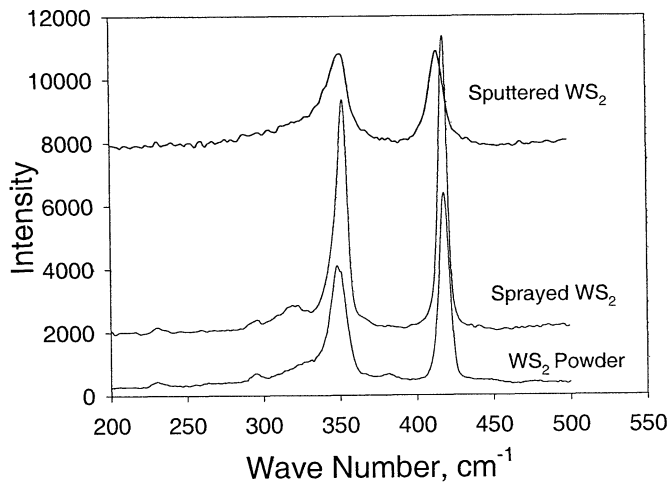


Figure 1. Raman peaks of WS₂ coatings prepared by powder-spraying method and magnetron sputtering. Raman curve from WS₂ powders was also inserted as a reference. Though sputtered coating exhibited a slight shift, basically identical peak positions were observed with the sputtered coating displaying much higher background.

3.2 Contact Angle

On a smooth sample before coating, the contact angle of distilled water with the steel was 66° as measured by laser goniometer. After deposition, the contact angle increases up to 85° for MoS₂, 93° for WS₂, and 98° for DLC coated surface. The maximum tensile stress required to pull the epoxy block away was taken as the measurement of stickiness. Before the coating, the stickiness was as high as 14 MPa for the roughest surface. After DLC coating, it reduced to about 2 MPa, a reduction of more than 80%. The stickiness data for the coated samples were plotted in Figure 2 against steel surface roughness before coating. A few observations were noted in Figure 2: (1). Though there was no general tendency of stickiness going up or down with pre-coating surface roughness, MoS₂ coating exhibited much larger stickiness than WS₂ and DLC coating. (2). Among DLC and WS₂ coating, there was slight increase in stickiness with surface roughness. (3). In the case of MoS₂ coating, the stickiness went up first and then reduced with roughness for reasons still not understood. The result was visually confirmed in the post-separation photograph of the steel surfaces as shown in Figure 3. (4). Among the three coatings, DLC-coated surface had the best anti-sticking property. WS₂ ranked quite close to DLC. Microscopically, the steel surface was clean after the separation in the case of WS₂ and DLC coating. The poor performance of the MoS₂ coatings may be owing to the high humidity environment of the experiment. As pointed out by Pritchard et al. [12], the coefficient of friction could increase from about 0.1 to 0.5 as relative humidity changes from 0 to 40%. Co-sputtering of molybdenum disulphide and polytetrafluoroethane (PTFE) [13], nickel [14], gold [15] and more recently titanium, et al. [16,17] improves the lubrication properties of molybdenum disulphide considerably. However, possible chemical reaction between MoS₂ and epoxy compound may be another source of stickiness. Details studies have to be done to ascertain this.

4 Summary

This paper directly measured the stickiness between the epoxy molding compound and WS₂, MoS₂ and DLC coatings deposited via unbalanced magnetron sputtering process on patterned mold steel sample blocks. Though the sputtered WS₂ coating exhibited a slight shift and high background, basically identical peak positions were observed with that of the WS₂ powder. The WS₂ and DLC coatings studied had similar antisticking properties with DLC being slightly better than WS₂. That was in agreement with the contact angle results. The stickiness could be reduced up to more than 80%. The MoS₂ coatings exhibited unexpectedly large stickiness with the epoxy compound. Moisture sensitivity was suspected one of the reasons. It may be also true that unexpected chemical reaction had taken place between the coating and the epoxy compound. Further studies have to be carried out to ascertain the cause. It was concluded that with proper engineering, DLC and/or WS₂ coatings could be good candidates for antisticking coating in epoxy molding applications.

Acknowledgement

The authors are grateful to E. Kuah for patterned samples, to C.G. Lim for part of the data collection, and to A. Yeo and H. Xie of Gintic for their assistance in Instron and microscopic work.

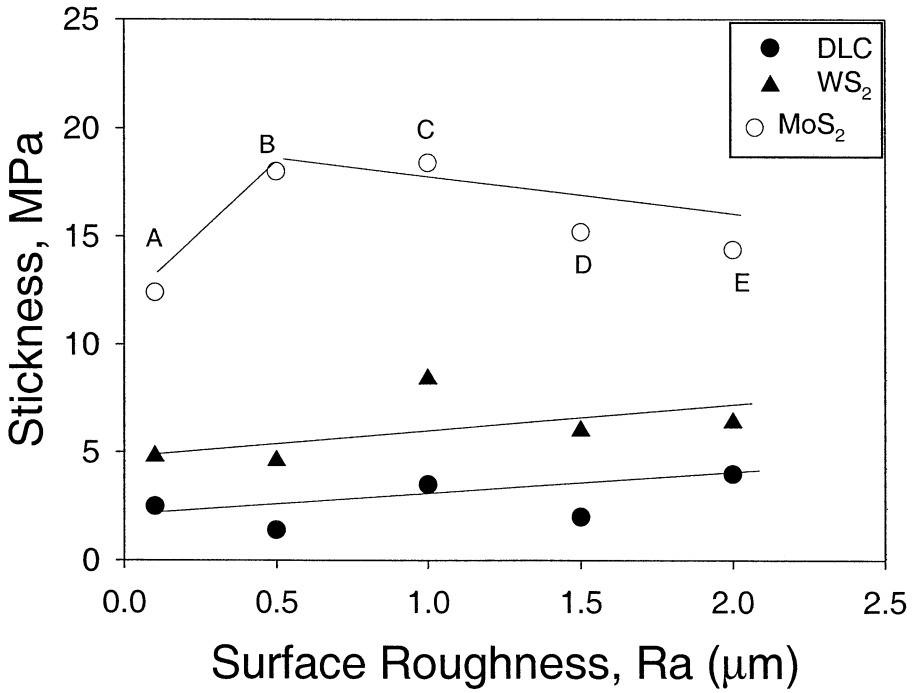


Figure 2. The stickiness for different coating systems against pre-coating surface roughness: MoS₂ coating exhibited much larger stickiness than WS₂ and DLC coating.

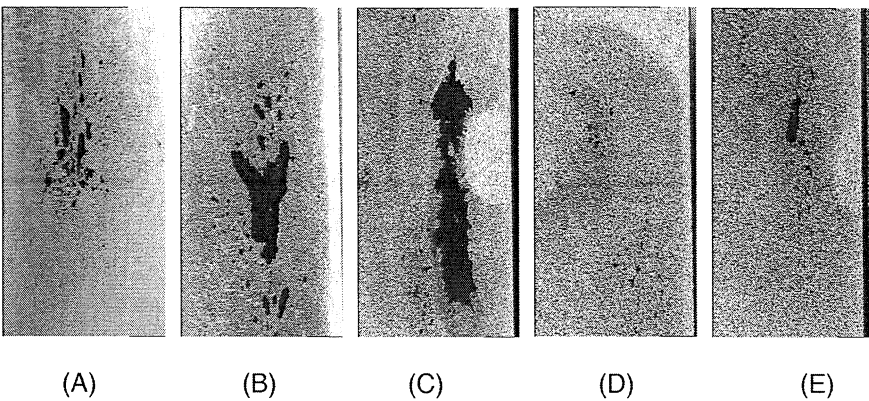


Figure 3. Post-separation photographs of the steel surfaces coated with MoS₂. The sample numbers correspond to the data points in Figure 2. The change in stickiness was confirmed by the change in amount of epoxy stuck on the sample surface. The width of the sample was 10 mm.

References

1. Modern Plastics Encyclopedea, New York, McGraw Hill 1991
2. S. Zhang, B. Wang and J. Y. Tang: 'Processing and characterization of diamond-like carbon films', *Surface Engineering*, 13(4)1997 pp. 303-309.
3. A. R. Lansdown (editor), *Molybdenum Disulphide Lubrication*, Elsevier, 1999
4. K. Ellmer, c. Stock, K. Diesner and I. Sieber, Deposition of C-orientatd tungsten disulfide (WS₂) films by reactive DC magnetron sputtering from a W-target in Ar/H₂S, *Journal of Crystal Growth*, 182 (1997) 389-393.
5. A. K. Rai, R. S. Bhattacharya, J. S. Zabinski, K. Miyoshi, Acomparision of the wear life of as-deposited and ion-irradiated WS₂ coatings, *Surface and Coatings Technology* 92 (1997) 120-128.
6. J. Sobota and g. Sorensen, Ion bombardment of solid lubricating nanoparticle coatings, *Nuclear Instruments and Methods in Phycis research B* 127/128 (1997) 945-948.
7. S. Zhang, M.J. Tan, P. Hing, H. Xie, H.L. Wong , W.L. Ng: Nitrogenated Carbon Layer on Magnetic Recording Disks. *Journal of Materials Processing Technology*, Vol. 89-90, 1999 pp.556-560..
8. S. Zhang and H. Xie: Improving Adhesion of Amorphous Carbon on Cemented Carbide Through Plasma Cleaning, *Surface and Coatings Technology*, (113)1-2 (1999) pp. 120-125.
9. Zhang, S., Hsieh, J. H., Tan, B. H., Hing, P., Teer, D. G.: Characterization and tribological behaviors of MoS₂ coatings deposited by unbalanced magnetron sputtering, *Journal of Materials Processing Technology*, 1997, 55- 62
10. Jacob A. Obeng, Glenn L. Schrader, Reactive sputtering of molybdenum sulfide thin films, *Surface and coatings technology*, 68/69 (1994) 422-426.
11. Sam Zhang, Tan Ming Jen, Xianting Zeng, Hong Xie, Peter Hing: Raman and PEELS studies of magnetron sputtered a-C, *International Journal of Modern Physics B*, 14 (2&3) 2000 pp. 268-273.
12. Pritchard, C. and Midgley, J. W., The effect of humidity on the friction and life of unbonded molybdenum disulphide films, *Wear*, 13, 39, (1969). (Figure 7.1 in *Molybdenum Disulphide Lubrication*, Ed. A. R. Lansdown, Elsevier, 1999)
13. Niederhauser, P., Maillat, M., and Hintermann, H. E., Moisture resistant MoS₂-Based composite lubricant films, *Proc. 1st Europ. Symp. On space Mechanisms and Tribology*, Neuchatel, Switzerland, (12-14 Oct., 1983).
14. Stupp, B., Performance of conventionally sputtered MoS₂ versus co-sputtered MoS₂ and nickel, *Proc. 3rd Intl. Conf. On Solid Lubrication*, Denver, Colorado, (7-10 Aug. 1984).
15. Roberts, E. W. and Price, W. B., Advances in molybdenum disulfide film technology for space applications, *Proc. 6th European Space Mechanisms and Tribology symp. , Zurich, Switzerland*, (4-6 Oct., 1995).
16. V C Fox, N Renevier, D G Teer, J Hampshire, The Structure of Tribologically Improved MoS₂/Metal Composite Coatings And Their Industrial Applications, 6th International Conference on Plasma Surface Engineering, 14-18 September 1998, Garmisch-Partenkirchen.
17. N M Renevier, N Lobiondo, V C Fox, J Hampshire, D G Teer, Performance of MoS₂/metal composite coatings used for dry machining and other applications, *Surface and coatings technology* 123, (2000) 84-91