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Effect of humidity and temperature on the tribology of sputter-deposited ZnO and silver added ZnO (Ag-ZnO) films on titanium substrates

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KEYWORDS

Coefficient of friction
Sputter deposition
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ZnO thin films

ABSTRACT

The effect of humidity and temperature on the friction of sputter-deposited ZnO and silver added ZnO (Ag-ZnO) has been investigated. The variation of the mean coefficient of friction as a function of humidity and temperature was experimentally determined. The addition of silver improved the tribological performance of the sputter deposited films. For pure ZnO films, an increase in the coefficient of friction was observed with humidity, whereas, for silver added zinc oxide (Ag-ZnO), no humidity dependence was observed. As the temperature is increased, the coefficient of friction increased for pure sputter deposited ZnO film, but for Ag-ZnO films, a slight dip in the coefficient of friction occurred with increase in temperature. An SEM imaging, X-Ray, and EDS characterization of both films were conducted. It was found that silver addition significantly decreased the crystallite size and improved the overall performance of ZnO films. A scratch test to qualitatively understand the wear resistance of both the films were also conducted.

1.0 INTRODUCTION

Zinc oxide is one of the highly investigated materials because of its unique crystal structures. Its potential application in different fields has resulted in many investigations and literature publications on synthesis and characterization. Application of ZnO includes a large spectrum ranging from medical, cosmetics, electronics, chemical, metallurgical, catalytic and manufacturing (Jiang, Pi and Cai 2018). Around 60% of the global production of ZnO is used as a vulcanizing

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activator in the rubber industry (Mostoni et al. 2019). It is also used in the plastic industry as a stabilizing agent and catalyst (Devasahayam et al. 2019), as a pigment in the paint industry, and also as a photocatalyst for the degradation of dyes from industrial wastewater (Li et al. 2019). The wurtzite structure of ZnO has gained significant interest in the semiconductor industry as a naturally n-type semiconductor, piezoelectric ceramic, with a high direct band-gap (~3.37 eV) and high bond energy (~60 meV) (Manu, Gupta, and Jayatissa 2021). The piezoelectric property of ZnO is used in the manufacture of flexible pressure sensors and actuators (Samoei and Jayatissa 2020). Because of its high bandgap ZnO absorb radiations in the UV region, and is used in sun cream and other cosmetic products as a UV blocking agent (Thi and Lee 2017). The UV blocking and water repellent properties are also exploited in the research of self-cleaning textile materials that are also UV blocking. The nanocrystalline ZnO particles are antibacterial due to their size and their distinctive physiochemical properties. Antibacterial property is generally attributed to the large surface area to volume ratio. These antibacterial properties can be further enhanced by implanting foreign materials such as silver on ZnO nanostructures. The antibacterial property of ZnO has become a topic of interest among researchers in the pharmaceutical industry for application in medical creams and as a thin film coating on surgical instruments and prosthetics (Sirelkhatim et al. 2015).

Fabrication of ZnO nanoparticles is easier than other materials. A wide variety of ZnO nanometric structures such as nanorods, tubes, plates, helix, flower, snowflakes can be manufactured using different physical, metallurgical and chemical processes. Some of these methods include precipitation in water solution, hydrothermal synthesis, sol-gel process, precipitation from microemulsion and mechanochemical processes. It is possible to obtain products with control in size, shape and spatial structure (Kolodziejczak-Radzimska and Jesionowski 2014).

ZnO is a natural ceramic with a melting point of ~1970 ^{0}C (Yuan et al. 2014). The high thermal stability coupled with ease of manufacture of nanocrystalline structures makes it a suitable candidate for high-temperature applications. One of the potential applications of ZnO lies as a lubricious solid thin film for reducing friction at high temperatures. Presently most of the research on high-temperature solid lubrication focuses on using molybdenum disulfide, tungsten disulfide and adaptive lubricants (materials whose properties change with increasing temperature to maintain low friction) (Gong et al. 2020). However, these materials lose their tribological properties at temperatures above 700 ^{0}C due to oxidation. Hence lubricious oxide films can be used as a substitute for high-temperature applications. The high-temperature stability combined with its non-toxicity to humans makes it a suitable candidate for solid film lubrication for medical and aerospace applications(Olofinjana et al. 2017) (Voicu et al. 2020).

Ceramic materials are brittle in nature and the mode of wear for ceramics is by crack propagation (brittle fracture). The wear debris formed is particulate, which also assists in more wear (A.H Jayatissa et al. 2021). However, research has been conducted in the past to control the microstructure, stoichiometry, crystal orientation, crystal size of the ZnO thin films such that it promotes ductile fracture instead of crack propagation and failure(Karch, Birringer, and Gleiter 1987)(Chai et al. 2016)(Prasad and Zabinski 1997). One of the ways to achieve this was by using sub-stoichiometric ZnO coatings with oxygen vacancies. These vacancies can lead to the development of new crystallographic shear systems due to diffusion creep; the shear strength and friction coefficient can be controlled by controlling stoichiometry. The grain size of the ZnO films also helps in reducing friction. Nanocrystalline ZnO films can reduce friction due to their sub-micrometer-scale spherical nature, which changes the contact configuration from sliding to rolling. However, for these sub-micrometer-sized films, the anti-wear properties are poor due to low hardness. One of the ways to increase the hardness and hence the wear resistance is by adding other elements such as nitrogen and carbon. These particles squeeze into the grooves on the rubbing surfaces to reduce wear. Nitrogen has been used extensively as inclusion for decreasing the grain size.

The grain boundary strengthening by adding noble metals is also a widely accepted method to reduce diffusion and dislocation along the grain boundary. In this publication, the effect of silver addition on the friction coefficient of ZnO films on titanium substrate at various environmental conditions is investigated. Presently very few publications are available on solid film lubrication using titanium as the substrate(Manu, Schroeder, and Jayatissa 2020)(Manu and Jayatissa 2020). Because of the novel applications of titanium in different fields as a substitute for aluminum, it is vital to understand how titanium substrate effect the tribological properties of lubricious solid films. The effects of various levels of humidity and temperature on the friction coefficient of the pure sputter-deposited ZnO films and silver added zinc oxide (Ag-ZnO) films were experimentally determined. The different phases in the ZnO and AgZnO films were characterized using X-Ray diffraction (XRD). The microstructure was examined using a scanning electron microscope (SEM), and the atomic fractions of elements in the film were estimated by the EDS technique. A scratch test on ZnO and Ag-ZnO films was also conducted to qualitatively understand the wear resistance.

2.0 EXPERIMENTAL DETAILS

As already described, films were deposited on industrial-grade titanium by the sputter-deposition process. High purity silver and zinc targets were used for this process to reduce contaminations. The pin-on-disk tribology test was conducted at different humidity levels and temperatures and the test gave a real-time variation of friction coefficient as a function of time. The details of the deposition process and tribo-testing is described below.

2.1 Substrate Preparation and Film Deposition

The manufacturer's specification of the titanium substrate used for these experiments is shown in Table. 1. The titanium plate surface was mechanically polished using an automatic grinding machine. An aluminum oxide abrasive grinding tool was used for this operation. The ground samples were again polished using a liquid suspension containing 0.25 μm size silicon carbide nanoparticles, to get a smoother appearance. The polished plates were then cleaned for 5 mins in an ultrasonic cleaner, rinsed with distilled water, sprayed with ethanol, and dried under a heat gun.

The Ag-ZnO and ZnO films were coated on titanium substrate by using a radio frequency (13.56 MHz) co-reactive magnetron sputtering process. The high purity silver and zinc (99.9% purity) targets were bombarded with positive argon-ions, which displaced zinc ions (Kauzlarick 2003). A pressure of 125 mTorr (24×10^{-4} psi) was created using a multi-stage vacuum pump. The argon to oxygen ratio was controlled at 1:1 to influence the composition of the sputter-deposited films. The coatings were deposited at 250 W for 3 hrs. The substrates were maintained at 30 ^{0}C during the coating process. The average range of thickness of the deposited films was between 650-700 nm (Jayatissa et al. 2021).

Table 1: Chemical composition of titanium substrate plate.

Weight %	Ti	C	Fe	N	O	H
Titanium TA2 Grade	Balance	0.10	0.30	0.03	0.25	0.015

2.2 Measurement of Friction Coefficient

The tribology tests were conducted using a custom-made pin-on-disk tribometer. It consisted of a cantilever with a cylindrical pin having a spherical tip attached at the free end. The pin had the provision to allow the addition of different weights for experiments at different loads. Two strain gauges were attached to the lateral faces of the cantilever to measure the deflections. The test specimen was screwed to a rotating platform and rotated at a constant speed. Once the platform reached steady rotation, the pin, with normal weight, was placed carefully so that the initial interaction between the pin and the titanium plate was measured with the least amount of error. Due to friction, a lateral force was exerted on the cantilever, which deflected the strain gauges. The inner strain gauges underwent compression, whereas the outer one was subjected to tension. The piezoelectric property of these strain gauges gave rise to a differential voltage, which was collected via a data acquisition system and was interfaced with a computer running custom LabVIEW software. Real-time deflection and frictional force data were collected and saved for further analysis.

For this work, the pin-on-disk tribology test was conducted to plot the variation of coefficient of friction with time on the sputter-deposited ZnO and Ag-ZnO films. The mean coefficient of friction was calculated at humidity ranging from 10 to 80% RH and plate temperature from 25 to 60 ^{0}C. The temperature tests were conducted at a relative humidity of 10% RH. The films coated on titanium substrates were heated using an infrared heater and the temperature was determined using an optical thermometer. The experiments were conducted at a load of 1.3 N and 100 rpm speed. For all the test run, dry sliding without the use of a liquid lubricant was carried out. The effect of humidity and temperature on the coefficient of friction was investigated. The films were characterized by using SEM, XRD and EDS methods.

3.0 RESULTS AND DISCUSSION

This section focuses on the results of film characterization and tribology tests along with a short discussion on the observed behaviors. The films were characterized using X-Ray diffraction, scanning electron microscopy and energy dispersive spectroscopy to get insight on the lattice parameters, surface morphology, grain size and relative concentrations of element and silver dopants.

3.1 Film Characterization

3.1.1 X-Ray Diffraction (XRD) Characterization

Figure 1 shows the X-ray diffraction peaks of ZnO and Ag-ZnO from 2θ values of 30^0 to 38^0 with ZnO diffraction planes and corresponding angles as insert. The silver content in the Ag-ZnO films were ~ 6 wt.% and no significant peaks were observed in the XRD. The peak intensity is slightly shifted to the left compared with standard values for both ZnO and Ag-ZnO samples. Since sputter deposited ZnO films are inherently substoichiometric in nature with oxygen vaccancies, this leftward shift is expected due to increase in lattice constants value imparted by oxygen vaccancies. Siver addition increased the crystallinity of the films, which was evident from increase in peak intensities. Also, a more leftward shift was observed for Ag-ZnO sample compared with pure ZnO. This shows silver addition further increased the interplanar distance and silver may have been deposited in the ZnO lattice as substitutional or interstitial point defect. From XRD peaks, a cystallite size of 8.6 nm for Ag-ZnO and 14.4 nm for ZnO were determined along (002) by FWHM method. This confirms that silver addition causes significant microstructural changes and decreases the crystallite size of sputter deposited ZnO.

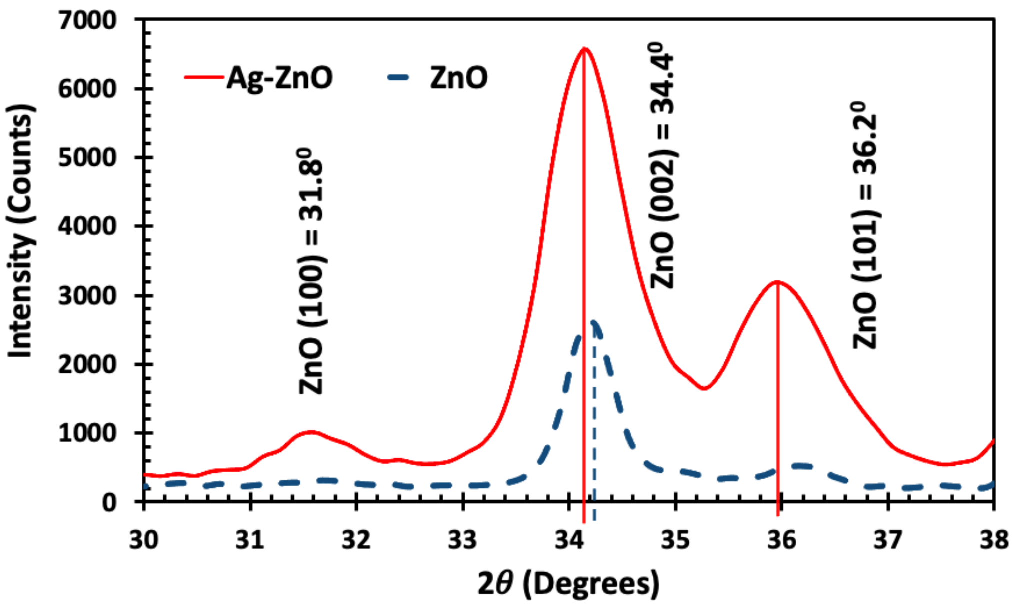

Figure 1: XRD graph of Ag-ZnO and ZnO sputter deposited films.

3.1.2 Scanning Electron Microscopy (SEM) Characterization

The SEM images of Ag-ZnO and pure ZnO is shown in Figure 2. The microstructure of ZnO was more defined compared with Ag-ZnO. Figure 3 is the enlarged surface morphology of pure ZnO at X90,000 magnification. Even at a higher magnification no significant grain distinction was observed for Ag-ZnO sample. Since the crystallite size calculated from XRD data showed a very low grain size (8.6 nm), the grain boundary distinctions could be observed for Ag-ZnO sample at much higher magnifications.

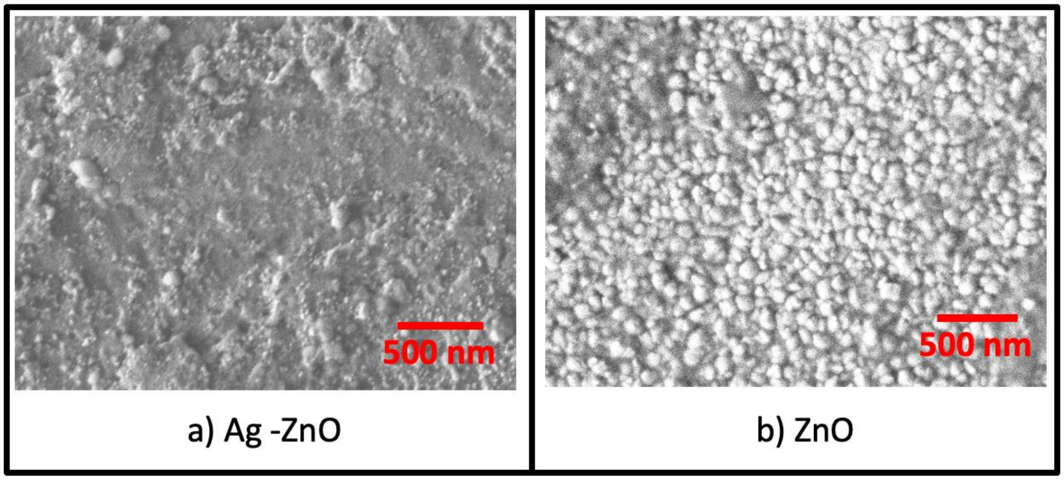

Figure 2: SEM images of the microstructure of sputter deposited a) Ag-ZnO and b) Pure ZnO (2kV).

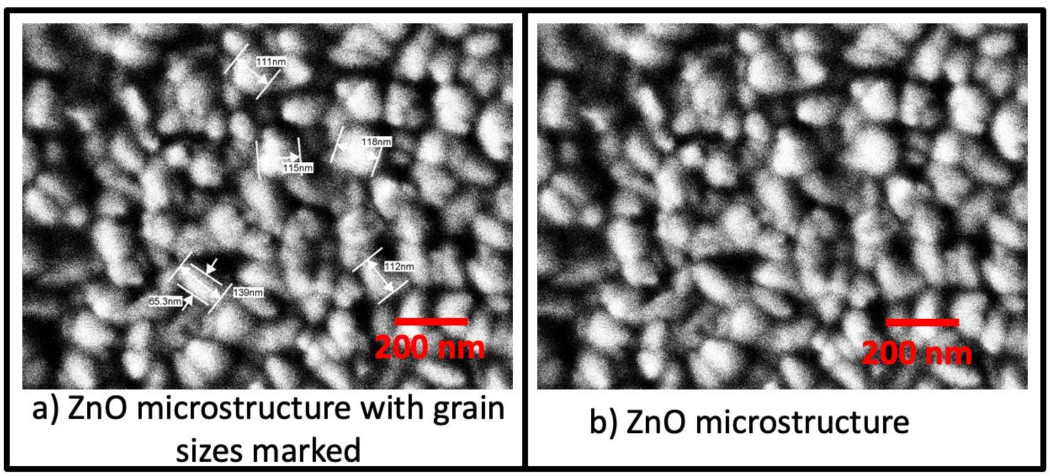

Figure 3: SEM images of the microstructure of sputter deposited Pure ZnO at X90,000; a) with grain sizes marked b) same picture without microstructure marked.

3.1.3 EDS Characterization

The EDS spectrum of ZnO and Ag-ZnO sputter deposited films is shown in Figure 4. The proportion of silver in the sample was about ~6 wt.%, which corresponded to ~2 at.%. A higher atomic concentration of oxygen was observed on ZnO films, this may be from the oxidation of titanium substrate, which required lower energy for oxidation.

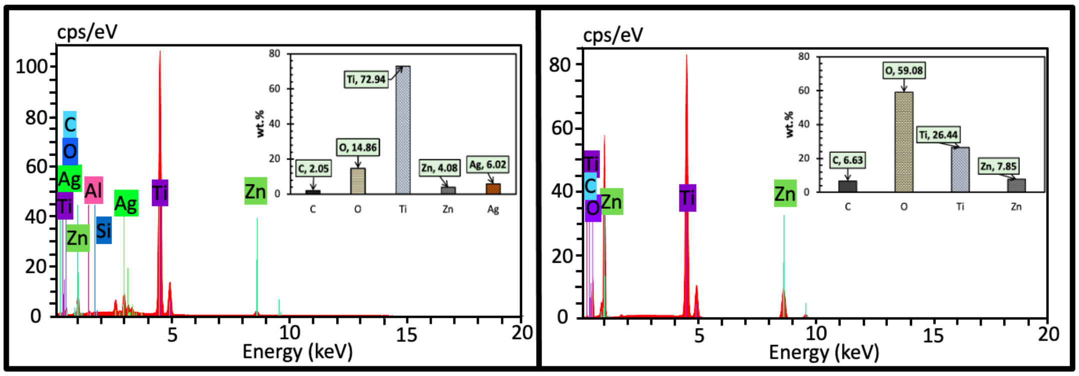

Figure 4: EDS spectrum of Ag-ZnO a), ZnO b). Weight percentage (wt.%) of each element is shown in the bar graph as the insert.

3.2 Results of Pin on Disk Test

3.2.1 Humidity Test

The variation of the mean coefficient of friction as a function of relative humidity is shown in Figure 5. At very low humidity levels the coefficient of friction of pure ZnO film was minimum (~0.1) and it increased up to a relative humidity of 40% (~0.22 at 40% RH). Beyond this, the coefficient of friction remained almost constant. On the other hand, silver addition on ZnO (Ag-ZnO) showed decrease in the friction coefficient of the film. The coefficient of friction remained almost constant with humidity with a very slight increase of ~0.02 from 10% RH to 33% RH (~0.106 at 10% and ~0.128 at 33% RH). Figure 6 and Figure 7 show the variation of friction coefficient with time during the pin-on-disk tribometer test for both the films at 10% RH (low humidity) and 50% RH (high humidity), respectively. At 10% RH, the variation of coefficient of friction for ZnO and Ag-ZnO was almost the same with the pure ZnO films having a larger dispersion compared with Ag-ZnO. Silver addition significantly improved the tribological performance of sputter-deposited ZnO films at high humidity conditions.

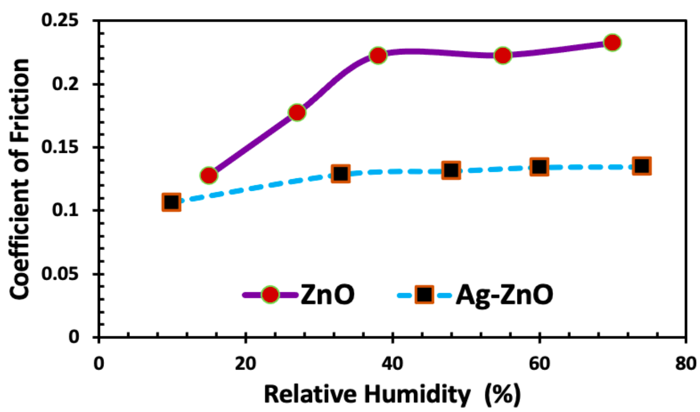

Figure 5: Variation of mean coefficient of friction for sputter deposited ZnO and Ag- ZnO films as a function of relative humidity at plate temperature of 25 ^{0}C (the markers represent the humidity at which the tests were conducted).

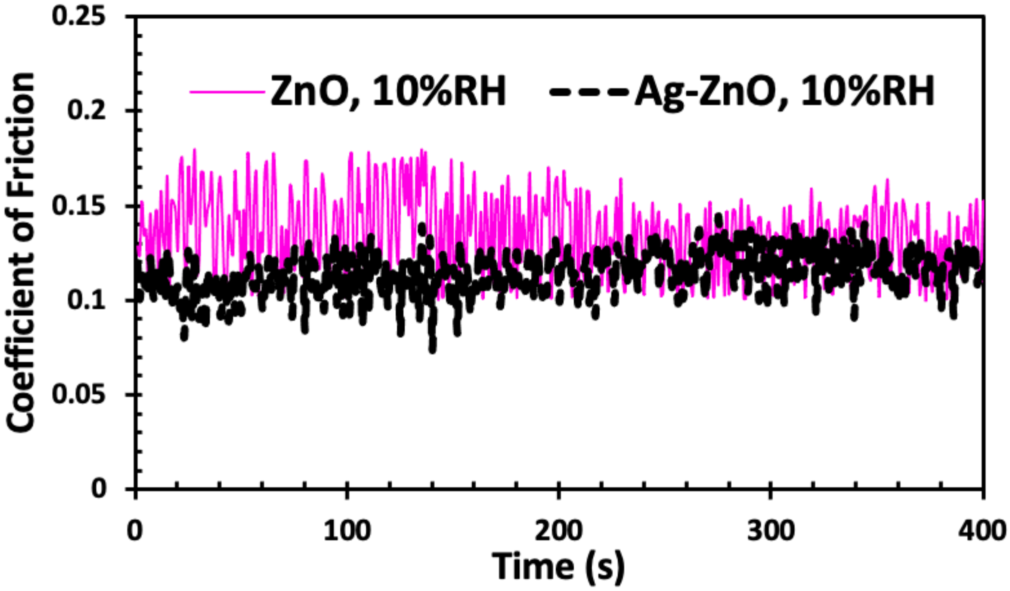

Figure 6: Variation of coefficient of friction with time for sputter deposited ZnO and Ag- ZnO films at 10% RH and plate temperature of 25 ^{0}C.

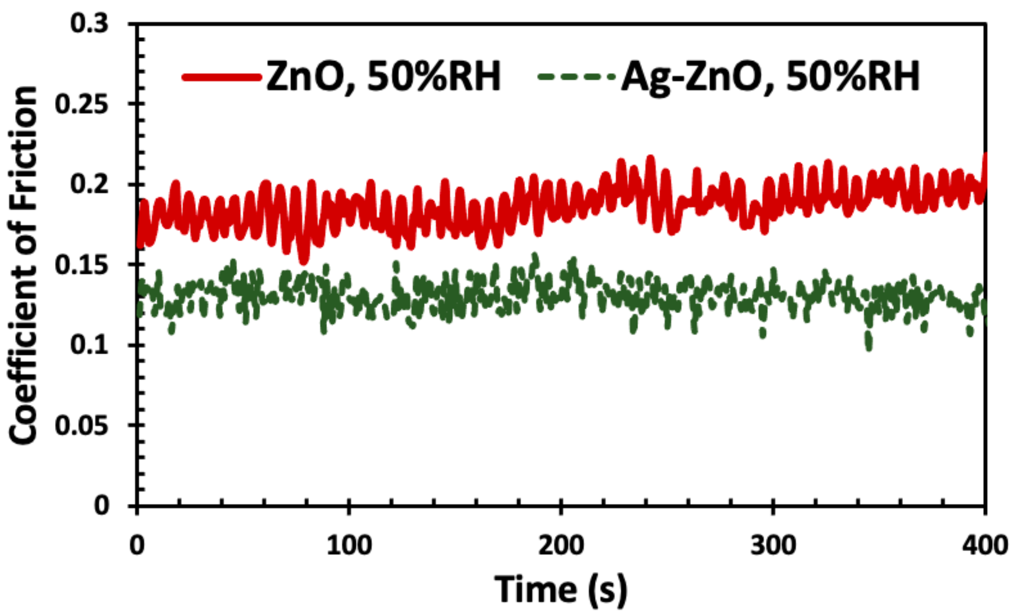

Figure 7: Variation of coefficient of friction with time for sputter deposited ZnO and Ag- ZnO films at 50% RH and plate temperature of 25 ^{0}C.

3.2.1.1 Discussion on Effect of Humidity

Water adsorption changes the tribological properties at the interface. Water films in the order of 5-10 molecular diameters thickness act as an induced solid with critical shear stress. This increases the surface energy at the interface and hence the friction(Yoshizawa and Israelachvili 1994). The presence of Van Der Waals forces, inter and intra hydrogen bonding between water molecules and the film interfacial molecules (termination layer molecules) is responsible for this increase in friction coefficient (Santos et al. 2021). For pure ZnO films, the variation of friction coefficient with humidity may be explained due to the solid-like behavior of water molecules due to Van Der Waals forces and hydrogen bonding.

The adsorption of water molecules on the surface of silver added ZnO has been extensively investigated for improving the photocatalytic property of ZnO. From these studies, it was confirmed that the adsorption of water molecules on ZnO is through photon transfer and it depends on the ZnO termination layer (Qin et al. 2019)(Kaewmaraya et al. 2012)(Quaranta, Behler, and Hellström 2019). The addition of noble metals such as silver further improves the catalytic activity (Lu et al. 2015). Silver arrests the motion of electrons in the conduction band (electron trapping). This facilitates the adsorption of hydroxide ions (OH^-) due to its negative charge and increases the hydroxide ion concentration on the surface. The increase in hydroxide ion concentration on the surface weakens and breaks the hydrogen bonds due to repulsive forces between the hydroxides. Also, silver tends to self-assemble on the grain boundaries thus further arresting the motion of dislocations (grain boundary strengthening). From the XRD graph it was evident that Ag-ZnO films showed a lower crystallite size (8.6 nm for Ag-ZnO and 14.4 nm for ZnO). Grain boundary strengthening and smaller crystallite size increases the film hardness, whereas electron trapping decreases the surface energy; thus, providing better tribo-performance for Ag-ZnO films

3.2.2 Effect of Temperature on Sputter Deposited ZnO and Ag-ZnO Thin Films

To understand whether heating the coated plate effects the coefficient of friction, the pin-on-disk tribometer test was conducted at different plate temperatures on both pure ZnO and Ag-ZnO sputter deposited films. For silver added ZnO (Ag-ZnO) films, the coefficient of friction remained almost constant with temperature whereas, for pure ZnO films, the friction coefficient increased. Figure 8 shows the variation mean coefficient of friction with temperature. The real-time variation of friction coefficient during the tribometer test at a film temperature of 60 ^{0}C and 10% RH is also shown (Figure 9). Pure ZnO films have a mean coefficient of friction of 0.23 whereas for Ag-ZnO films the mean friction coefficient is only 0.13 at 60 ^{0}C.

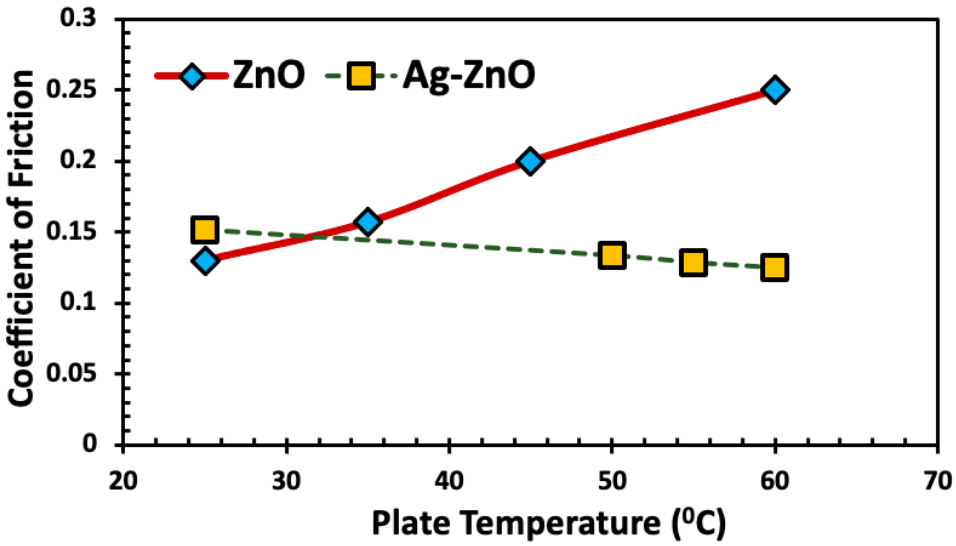

Figure 8: Variation of mean coefficient of friction for sputter deposited ZnO and Ag- ZnO films as a function of plate temperature at 10% RH (the markers represent the temperature at which the tests were conducted).

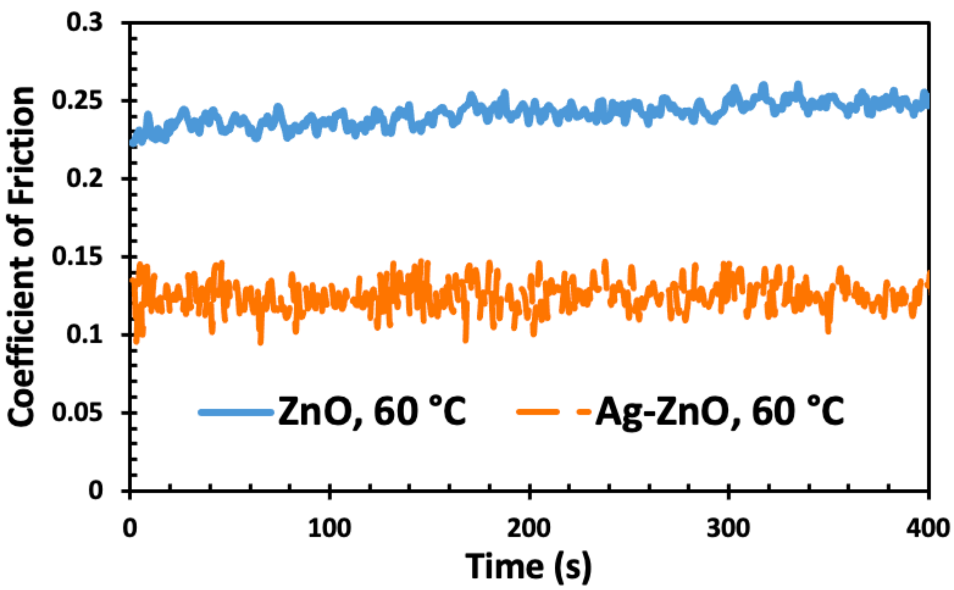

Figure 9: Variation of coefficient of friction with time for sputter deposited ZnO and Ag- ZnO films at 60 ^{0}C plate temperature and 10% RH.

3.3.1 Discussion on Effect of Film Temperature

Silver addition changes the microstructure and grain size of the film, which was evident from the XRD and SEM data. Also, silver added ZnO (Ag-ZnO) showed conductivity 3 orders of magnitude more than that of pure ZnO. At this concentration of silver, the crystallinity and preferential growth of ZnO films in (002), which is known for low surface energy happened. The decrease in coefficient of friction for Ag-ZnO sample with increase in plate temperature may be credited to desorption of adsorbed gases on the surface with temperature increase, low energy (002) termination layer and the increase in heat conductivity of ZnO due to silver addition.

3.4 Scratch Test on Ag-ZnO and ZnO Sputter Deposited Films

Figure 10 shows the SEM images of the wear tracks on the Ag-ZnO and ZnO samples after the scratch test using an indenter of 30μm flat tip with a load of 5N. The wear scar analysis showed a deformed grain boundary on ZnO films, but the AgZnO showed a much smoother wear track. However, the surface roughness of the wear scar was difficult to be measured. The small grain size and grain boundary strengthening by silver in Ag-ZnO sample can be responsible for this increased abrasion resistance.

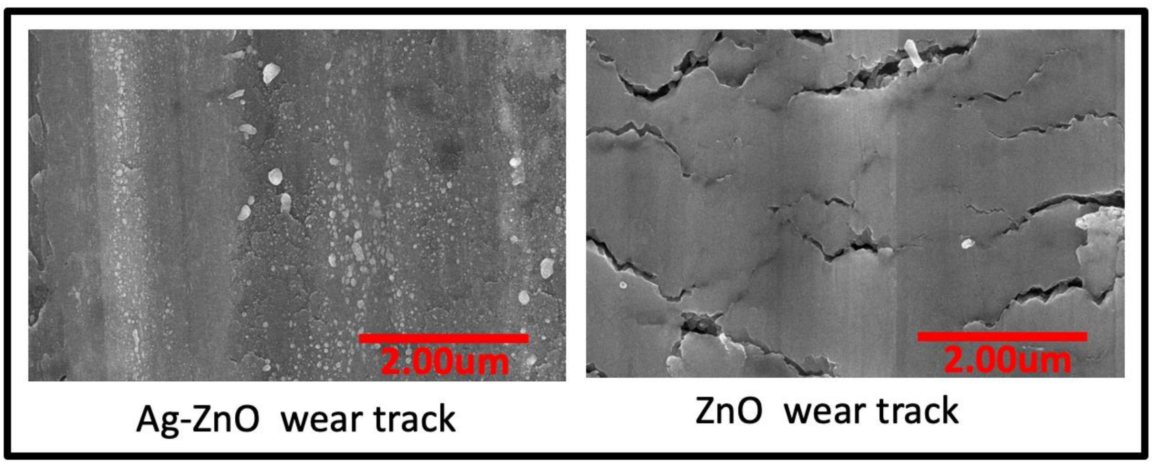

Figure 10: SEM images of the wear tracks after scratch test for Ag-ZnO and pure ZnO films.

CONCLUSION

The effect of humidity and temperature on the coefficient of friction of sputter-deposited ZnO and silver added ZnO was experimentally determined. Silver addition (Ag-ZnO) improved the tribological performance of the ZnO film. The coefficient of friction values increased for pure ZnO films with humidity and temperature whereas very little effect on coefficient of friction was observed for Ag-ZnO sputtered films with humidity and temperature. The XRD, SEM and EDS characterization Ag-ZnO films showed addition of silver in small concentration facilitated the grain growth in (002) direction, which has the lowest surface energy, which also masked the highly reactive edge sites from exposure to atmospheric gases and decreased the grain size. This reduced the coefficient of friction values for Ag-ZnO films (decreased coefficient of friction). This study showed addition of small amount of silver reduced the friction response of sputter-deposited ZnO films.

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