CONDUCTIVE ATOMIC FORCE MICROSCOPIC (C-AFM) STUDIES OF Au/MoS₂ NANOCOMPOSITE FILMS

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ABSTRACT

Au/MoS₂ nanocomposite films with high Au concentrations (75 to 90 at%), recently developed at The Aerospace Corp., have shown properties that are promising for use in sliding electrical contacts, such as slip rings and relays. For such applications, it is critical to maintain low contact resistance while maintaining low friction with controlled wear (i.e. removal and transfer of material). In this report, we present results from conductive atomic force microscopic (c-AFM) investigations of Au/MoS₂ nanocomposite structures and their dynamic material transfer phenomena under a sliding contact, which are both important in understanding the friction, wear and conducting mechanisms of the films. We have performed c-AFM to obtain topography, friction and current images simultaneously. Remarkable morphological changes were observed in a series of current images which initially showed distinct nanoscale metallic (Au) and semiconducting (MoS₂) phases that were relatively well dispersed, but repeated contact sliding in the same area resulted in gradual disappearance of the metallic phase and reduction of the overall friction. These results reveal that MoS₂ is transferred across the surface to provide lubrication while Au particles at or near the surface provide electrical conductivity. The c-AFM results provide real-time and real-space visualization of the lubrication mechanism occurring inside a nanoscale sliding contact.

INTRODUCTION

Co-sputtered nanocomposite Au/MoS₂ solid lubricant films are known to provide superior tribological properties compared to the respective pure materials. Our previous work showed that the Au:MoS₂ ratio has a major influence on film performance in different Hertzian contact stress regimes [1]. For low contact stress applications, such as sliding electrical contacts (e.g., slip rings, relays, and connectors), we are concentrating on films with relatively high Au concentrations (75 to 90 at%), since they demonstrated lower friction and greater endurance than films with lower Au content in ball-on-disk tribometry [1]. In addition, these Au-rich films are expected to provide low resistivity since Au is an excellent electrical conductor. However, the electrical properties of these films have not yet been studied. Here, we have employed conductive atomic force microscopy (c-AFM) to investigate nanometer-scale structure and material transfer of Au/MoS₂ nanocomposite films during sliding to understand friction, wear, and electrical behavior. The material transfer mechanism studied here may be broadly applicable to films with a wider range of metal concentrations.

EXPERIMENTAL DETAILS

The details of the film preparation have been described elsewhere [1]. Briefly, the sample films were grown in a custom sputter-deposition vacuum chamber, which is fitted with Au and MoS₂ rf-sputtering sources. The Au/MoS₂ films were grown on Si wafers mounted in the center of a table that rotated during deposition. Argon was used as the sputtering gas. Composition analysis of the films was conducted using Auger electron spectroscopy (AES) as well as Rutherford Backscattering Spectrometry (RBS).

The sample films were then analyzed in an Omicron UHV VT AFM system under a 3x10⁻¹⁰ Torr vacuum at room temperature. The samples were probed in a contact mode while a bias voltage was applied to the conducting AFM probe; the substrate was grounded through the sample stage. The normal and lateral deflection of the cantilever were monitored as well as the electrical current at the tip/sample interface in order to obtain topography, friction and current images simultaneously. The images were obtained at a small constant bias voltage (0.012V). In addition, specific locations within the image were selected to perform point spectroscopy, where the voltage was swept from −0.5 V to +0.5 V to obtain current-voltage (I-V) plots. The probe was made of silicon and coated with a platinum film to establish electrical conductivity (MikroMasch USA). During imaging, the probe was in contact with the sample surface with a constant repulsive force of about 2 nN in order to minimize tip/sample damage.
RESULTS AND DISCUSSION

In order to examine the structure of the nanocomposite, we performed c-AFM on films containing 75% and 95% Au to obtain topography, friction and current images over a small area simultaneously. Shown in Fig. 1A is the current image (100 nm x 100 nm) of the sample with 75% Au at a constant bias of 0.012V. Brighter color denotes higher conductivity, and thus two distinct phases of contrasting conductivity are observed. As a result, it is straightforward to visualize that Au and MoS2 particles are separated into distinct phases. Furthermore, I-V spectroscopy obtained in the dark and bright regions show that they are semi-conducting and metallic respectively (Fig. 1B), validating that they are likely phase-separated MoS2 and Au. The current image in Fig. 1A indicates that the sizes of the individual particles are roughly 10 to 30 nm, which agrees with the topography image (not shown). The observed metallic Au particles at or near the surface are likely a key in providing electrical conductivity, while lubrication is mostly provided by MoS2 on the surface.

Next, we investigated the morphological transformations of the two phases under a dynamic sliding contact to elucidate the lubrication mechanism. Contact imaging was performed continuously, which resulted in a series of sequential frames of topography, friction, and current images. Figure 2 shows frames selected from a series of 25 current images obtained sequentially on the 75% Au sample. These images illustrate a clear trend in which the metallic regions gradually reduce in size and disappear. This trend of decreasing conduction in the probed area suggests that MoS2 is transferred across the surface to cover the Au phases, assisted by the sliding contact. Consequently, the MoS2 transfer results in friction reduction. The average friction plotted against the frame numbers show that there is a clear trend of decreasing friction when the film is subjected to a continued contact sliding (Fig. 3A). The average frictional value was determined for each of the 25 images, from corresponding friction images that were obtained simultaneously. It shows that friction decreased rapidly for the first 5 frames after which it reaches a plateau. This is similar to the run-in process typically occurring during conventional ball-on-disk testing of nanocomposite Au/MoS2 solid lubricant films. It should be noted that the friction data in Figs. 3A and 3B were obtained with cantilevers with different force constants, thus quantitative comparisons of the friction values between the two plots are not meaningful. We focus our discussion on the qualitative trend of the friction reduction in each plot.

Next we performed identical experiments on a sample with lower MoS2 concentration (5% MoS2/95% Au); this sample exhibited a lower endurance than 75% Au sample in ball-on-disk wear tests [1]. Shown in Fig. 3B is the resulting plot of average friction, which shows a decreasing trend in friction similar to Fig. 3A. However, the rate of friction decrease appears to differ qualitatively. Unlike the 75% Au sample, where the friction reduction saturated after about 5 frames, the 95% Au sample shows a continual decrease throughout the 25 frames. The rapid saturation of friction reduction on the first sample can be attributed to the abundance of MoS2 material that could effectively cover the entire imaged area. On the second sample, there is a smaller amount of MoS2 available to cover the entire area that was probed. Comparing the last frame (25th frame) from each imaging series, the sample with 95% Au shows that a substantial fraction of metallic regions remained uncovered with MoS2, while on the sample with 75% Au virtually no bare metallic phases have remained at the end of the experiment (images not shown here).

These results indicate that contact sliding assists the formation of a thin MoS2 transfer film, which then is responsible for the observed friction reduction and decreased conductivity. This agrees with Auger Nanoprobe analysis that revealed the presence of a virtually pure MoS2 film after tribometric run-in, on top of the remainder of the Au/MoS2 film. The c-AFM results presented here provide real-time and real-space studies of the processes occurring inside a nanoscale sliding contact, providing actual visualization of the lubrication mechanism that is difficult to achieve by other techniques.

FIGURES

Figure 1. Current image (100 nm x 100 nm) and I-V spectroscopy that show phase-separated Au and MoS2.

Figure 2. Selected image frames (1, 7, and 25) from a series of 25 consecutive current images on the sample with 75% Au. Each image is 600 nm x 600 nm.

Figure 3. Average friction vs. image frame number for the samples with (A) 75% and (B) 95% Au.

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REFERENCE