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Tribology and sliding electrical contact resistance of e-beam hard Au: Effects of annealing

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ABSTRACT

Nanocomposite Au-ZnO thin films in the dilute oxide (<5.0 vol%) regime were synthesized by electron beam (e-beam) evaporation, as alternatives to electroplated Au hardened with Ni. Tribological measurements of e-beam hard Au were made while passing current through sliding contacts; electrical contact resistance (ECR) and friction data were simultaneously acquired during the test. The friction, wear and ECR behaviour were studied for the as-deposited film condition, and after annealing at 250 °C and 350 °C in air. The study revealed that the 250 °C annealed Au-2 vol% ZnO film exhibited the lowest, stable friction coefficient s ($\mu \sim 0.25$) and ECR (~ 35 mΩ) during sliding. Furthermore, the wear rate of this 250 °C annealed ZnO hardened Au nanocomposite film was an order of magnitude lower at 1.5×10^{-5} mm^3/N m than for a typical Ni hardened, electroplated Au film at 1.3×10^{-4} mm^3/N m. Cross-sectional transmission electron microscopy studies inside the wear surfaces revealed that the extremely stable, low friction coefficients and wear rate of annealed Au-2 vol% ZnO film was due to partial coverage of the wear surface with a ZnO tribofilm that reduced the adhesive contact contribution to wear with minimal impact on ECR. The potential implications of this study in the search for an environmentally friendly alternative to widely used electroplated hard Au are discussed.

1. Introduction

The necessity to provide efficient transfer of electrical current between moving parts remains an engineering challenge. With increasing implementation of electrical motors and generators and further increase in electrical contacts with the expanding electronics industry, the technological applications of sliding electrical contacts has become widespread. Sliding electrical contacts can be found in DC motor and generator current collector slip rings, printed circuit board edge connectors, data cable pin connectors and receivers, microelectromechanical systems (MEMS), circuit relays and switches, instrument signal transfer slip rings and many more applications. The main engineering challenges associated with these applications, especially long service life slip rings, is minimized wear and stable wear performance, acceptable friction to minimize mechanical energy losses, low and stable electrical contact resistance (ECR) to minimize Joule heating, and thermal stability of the sliding materials.

The use of gold in the electronics industry has reached over 300,000 kg per year in 2010 [1]. According to an evaluation by Goodman in 2002, when gold use in the electronics industry was 280,000 kg per year, the majority of this gold was used in electrical contacts, most frequently electroplated gold [2]. Gold is used in sliding electrical contacts due to its high conductivity and excellent corrosion and oxidation resistance. Although electroplated hard gold (gold alloyed with typically 1–2 wt% of cobalt or nickel) has produced an improvement over pure gold plating in friction and wear performance, it has reliability issues with ECR over time and at elevated temperatures [3]. Additionally, there are growing concerns with environmental hazards associated with the use and disposal of cyanide and arsenic baths used in plating chemistries. The reliability issue is related to an increase in ECR due to the formation of surface oxide films that can cause system failure in low voltage and high frequency applications, especially systems which have expected service lives on the order of years. Improvement of mechanical and tribological performance in hard Au has been realized by co-depositing with ZnO to synthesize ZnO-hardened Au nanocomposite films [4–7]. It has been determined that the room temperature sliding friction and ECR in the dilute oxide regime ($<$ 5 vol% ZnO) are lower than Ni hardened gold films [4]. However, it is unknown if annealing these films results in any improvements in the sliding electrical contact properties. As such, this paper presents results on friction, ECR and wear properties of as-deposited and annealed composite Au-ZnO films with

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comparisons made to as-deposited and annealed Ni hardened Au films. The sliding-induced structural and chemical changes of the worn surfaces and subsurfaces that control these properties will also be presented and discussed.

2. Experimental methods

2.1. Electron-beam film deposition

A Thermionics Vacuum Products Co. 10 kV Triad e-beam evaporation system was used for the deposition of both pure Au and composite Au-ZnO films. The electron beam is generated by a field emission source and magnetically deflected to the crucible to provide localized heating resulting in the sublimation of the film material. Once sublimated in a vacuum, the film material deposits on the target substrate in line-of-sight from the crucible. Prior to all film depositions the vacuum chamber was pumped to a base pressure of less than 1×10^{-6} Torr. The film material source to substrate distance was 305 mm and all depositions were conducted at room temperature. A rotating substrate mount was used to achieve compositional and thickness uniformity of the films. Prior to e-beam deposition, the Si substrates were first sputter coated with a nominally 300 nm thick Ti layer for adhesion followed by a nominally 300 nm thick Pt layer to act as a diffusion barrier. These layer thicknesses were determined by cross-sectional microscopy. For comparison purposes, electroplated hard Au films were obtained from a commercial plating company (Theta Plating Inc. Albuquerque, NM, USA). Nickel was used as the hardening element and plated per ASTM Type I specifications (ASTM B488-11 standard), also comprising of a 5 μm thick electroplated Ni diffusion barrier, deposited on Alloy 52 substrates (nominally a 50–50 wt% Fe-Ni alloy). Thickness of electroplated Ni hardened gold (Ni HG) film was 1.5 μm.

The pure e-beam Au films, nominally 2 μm thick, were deposited by evaporation of high purity Au pellets (99.999% purity) sourced from Materion Advanced Chemicals. The resultant Au film average root-mean-square (RMS) surface roughness was determined by scanning white light interferometry (SWLI) to be ~3 nm. The composite Au-ZnO films, nominally 2 μm thick, of varying composition were also deposited onto Si substrates sputter coated with Ti/Pt layers. The e-beam deposition source materials were pure Au pellets, mentioned above, and ZnO tablets (99.9% purity) from Materion Advanced Chemicals. The deposition rates were varied between 0.00 and 2.00 nm/sec to achieve the desired volume fraction of ZnO in the film in the dilute oxide (<5.0 vol%) regime. The resultant film average RMS surface roughness of composite Au-ZnO films was determined by SWLI to be ~4 nm. Prior to tribological testing, some of the pure Au and composite Au-ZnO films were ex situ annealed in an open air furnace to either 250 °C or 350 °C for 24 h dwell time.

2.2. Sliding electrical contact testing

A custom built linear tribometer equipped with an Agilent Technologies B2911A digital source/meter, photograph and accompanying schematic, shown in Fig. 1, was used to conduct all simultaneous friction and ECR measurements. The tribometer independently measures applied normal force and lateral force through independent double leaf flexures, which constrain the displacement to in plane linear motion, coupled to load cells. The load cells were independently calibrated using dead weights and calibrated scales throughout the expected experimental loads, thus incorporating the leaf flexure spring rate into the calibration. Continuous ECR measurements are made by operating the source/meter in voltage-regulated remote sensing mode and measuring the voltage drop and current using a four-point probe bridged across the film surface and counterface pin. Both the substrate of the film and the counterface pin are electrically isolated from the rest of the rig by custom fixtures machined from polycarbonate and polyether ether ketone, respectively.

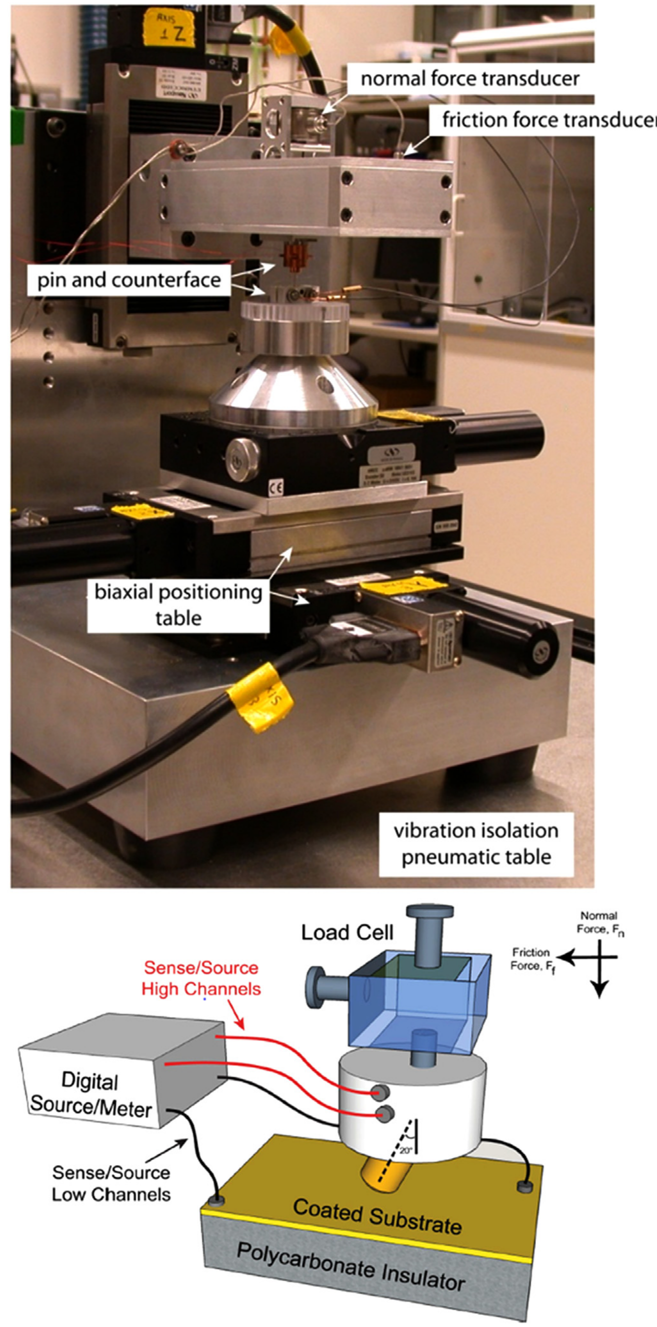

Fig. 1. Photograph and accompanying schematic of custom-built linear ECR tribometer.

All tribological friction ECR tests were conducted in unidirectional sliding motion by translating the bottom fixture by the 2 mm track length while recording data then lifting the upper load cell assembly and translating the bottom fixture to the start position and repeating. A normal force of 100 mN and a linear speed of 1 mm/s were used for a duration of 100 sliding cycles. Prior to each sliding ECR experiment, a static normal force of 100 mN was applied and a voltage set to produce approximately 100 mA of direct current through the ECR circuit. All tests were conducted at room temperature in lab air (15–30% relative humidity). The