Assessing the Electrical and Mechanical Performance of Wear-tested Au-ZnO Films

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-MOTIVATION-

Gold is the material of choice for **low resistance electrical contacts** for its long term **environmental stability** and **low resistivity.** However, cyclic contact loading cause mechanical wear over time and the high currents that are passed through these contacts lead to micro-welding, which adds additional deformation when the contacts break apart.

Increasing the hardness of nanocrystalline gold has been attempted using various methods (solid solution, oxide dispersion, nanolaminate to help increase the wear resistance of these films. Oxide dispersion strengthening shows greater promise for increasing hardness without greatly affecting conductivity [1].

- GOAL OF THIS STUDY -

Determine if addition of ZnO to Au thin films shows **increased wear resistance** with **no significant loss in electrical behavior**.

-FILM STRUCTURE-

Au-ZnO films were co-evaporated (Fig. 1) onto silicon substrates with a Ti/Pt adhesion layer at volume percentages of 0.1%, 0.5%, 1.0%, 2.0% ZnO. Energy Dispersive X-ray Spectroscopy (EDS) (Fig.2) shows that ZnO particles preferentially deposit at the grain boundaries (however some particles are still dispersed in the grains). Electron Backscatter Diffraction (EBSD) shows that the addition of ZnO particles decreases the (111) texture of the films (Fig. 3).

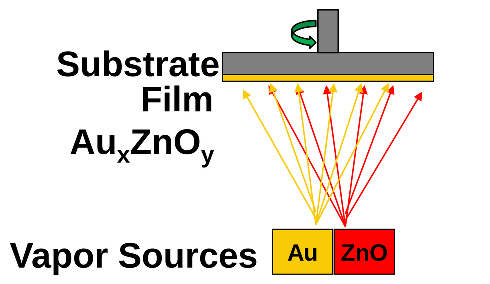


Fig. 1. Films were deposited using a co-evaporation technique.

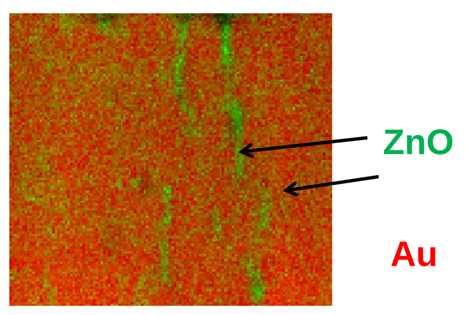


Fig. 2. EDS map of 5 vol% ZnO film, showing ZnO particles segregating mainly to grain boundaries [2].

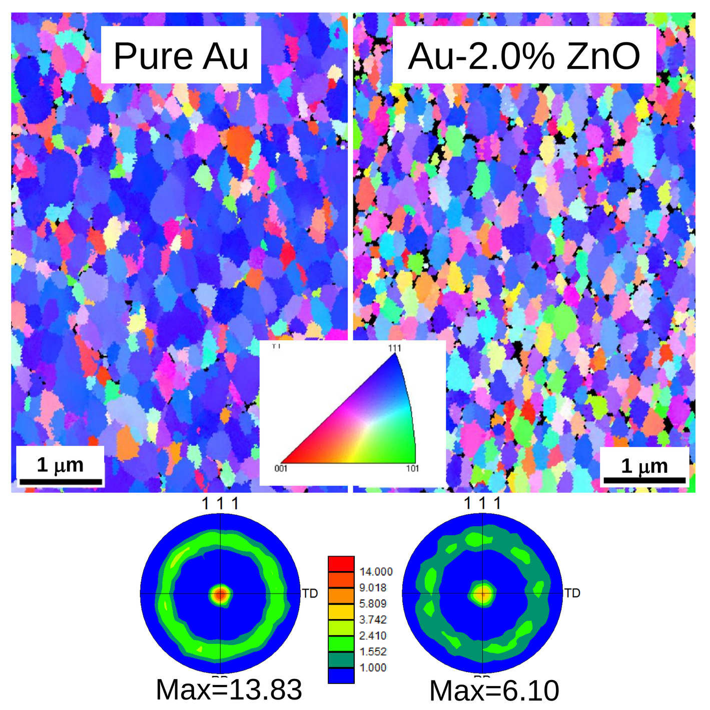
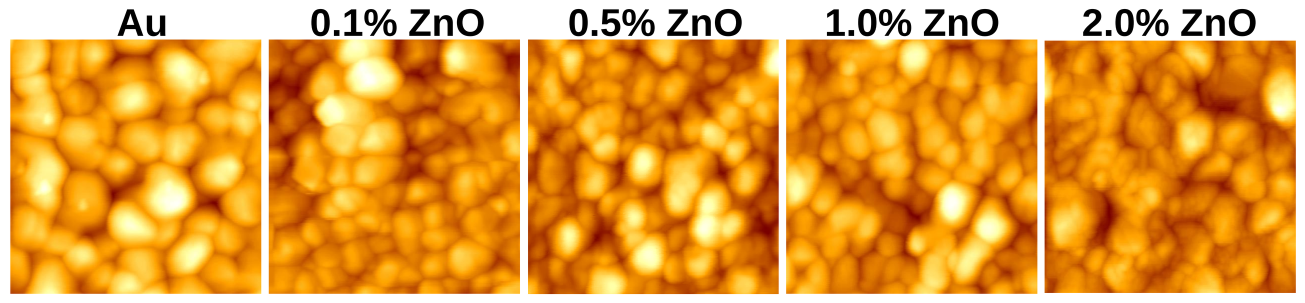


Fig. 3. EBSD orientation map showing change in amount of texturing due to addition of ZnO particles.

Atomic Force Microscopy (AFM) images of the as deposited films (below) show a reduced grain size with the addition of ZnO. This observation is confirmed using X-ray Diffraction (XRD) and point count measurements, shown in the table below.



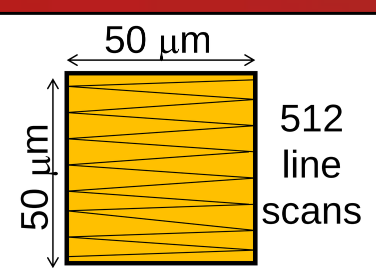
Grain size (nm)

Method	Au	0.1% ZnO	0.5% ZnO	1.0% ZnO	2.0% ZnO
XRD	97.2	97.2	71.4	86.7	78.0
Point Count	135	112	112	112	116

-WEAR TEST-

Wear test was conducted using Hystiron SPM scanning:

- 1 µm 90° conical diamond tip
- Loads from 25 µN to 400 µN
- 1, 2, 5 and 10 passes
- Sliding speed: 20 µm/s



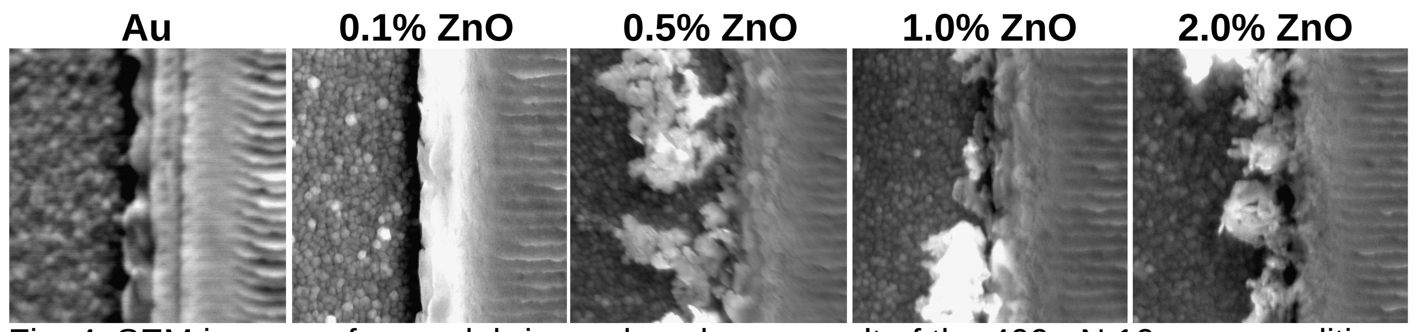


Fig. 4. SEM images of wear debris produced as a result of the 400 µN 10 pass condition. More plastic deformation (and less debris) is seen for the pure Au and 0.1% compositions.

Wear tests were conducted at 5 different loads and 4 number of passes to generate wear boxes at 20 different conditions. SEM images (Fig.4) of the highest load condition shows a reduction in the amount of plastic deformation as the ZnO is added to the film.

Ten different line scans of the 400 µN 10p wear box were averaged to determine the wear rate of the different films (right). The addition of ZnO to the Au films also decreases the wear rate of the films due to the increased hardness and roughness of the composite films.

Wear Rate (μN^2)	
Au	7.5±0.8
0.1% ZnO	5.5±0.6
0.5% ZnO	4.8±0.6
1.0% ZnO	2.9±0.6
2.0% ZnO	1.5±0.7

-EFFECTS ON HARDNESS-

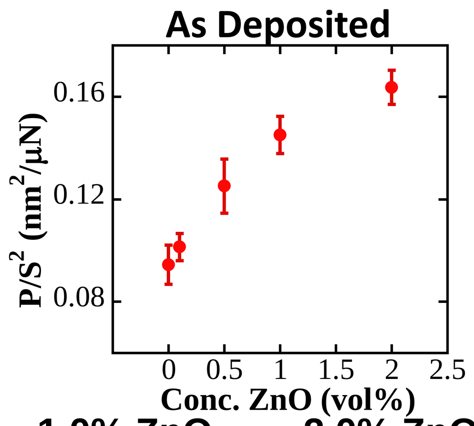
Nanoindentation was performed using a Hysitron TI 900 TriboIndentor with the load function described below. All conditions were indented to less than 15% of the film thickness (~150nm) to avoid substrate effects. A 30 second hold was applied at the peak load to reduce the creep effect. The P/S2 method for hardness correction was used to reduce the effect from pile-uo and changes in roughness.

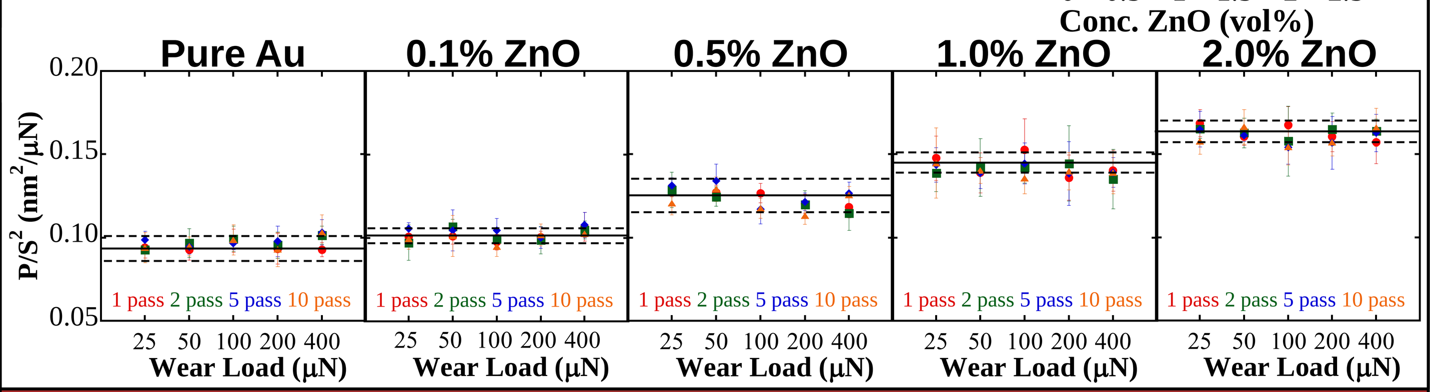
LOAD CONTROLLED INDENTATION:
- Peak Load- 1250 µN
- Loading Rate- 200 µN/s
- Hold Segment- 30s

Corrected hardness values for **roughness** and **pile-up** effects on contact area approximation

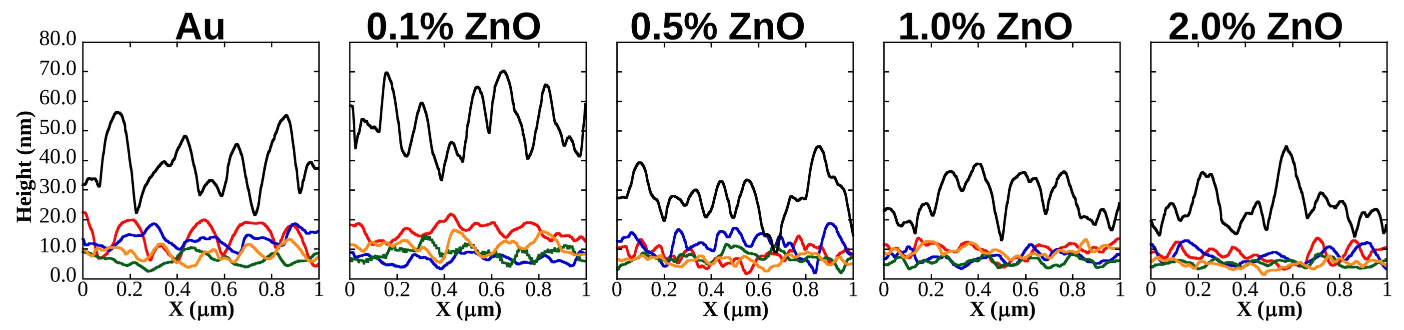
$$H \propto \frac{P}{S^2}$$

(nm2/µN)





-WEAR TOPOGRAPHY-



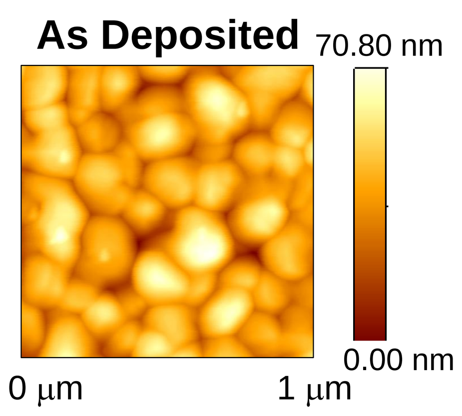
Average Roughness (nm)

Wear Condition		Au	0.1% ZnO	0.5% ZnO	1.0% ZnO	2.0% ZnO
As Deposited		8.35	10.77	8.60	6.73	6.16
50 µN	1 pass	3.66	3.94	2.00	2.25	3.10
	2 pass	2.05	1.66	3.00	1.45	2.64
	5 pass	2.26	3.15	1.47	1.69	1.33
	10 pass	2.16	2.45	1.31	1.07	1.09

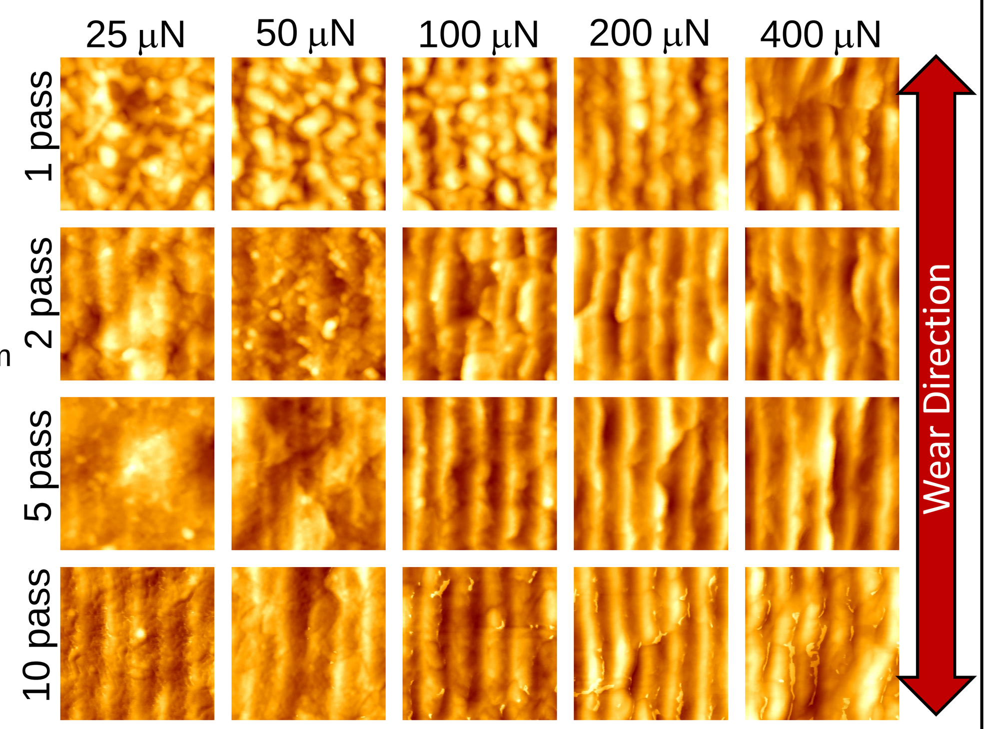
The average roughness values determined from 1 µm AFM scans showing a general trend of decreasing roughness with increasing ZnO concentration and number of passes at a single load condition.

AFM images of the full wear matrix of pure gold and 2.0%ZnO films are shown below. The pure gold film shows more deviation from the as deposited structure at than the composite film, even the lowest loads.

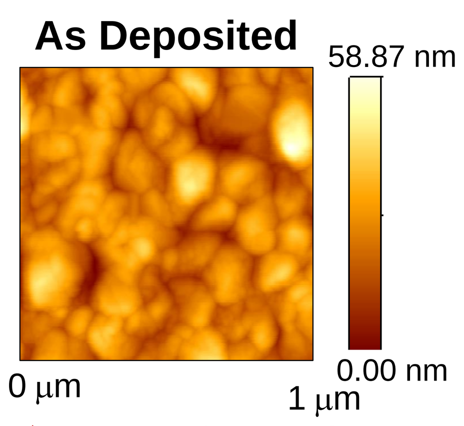
-Pure Au-



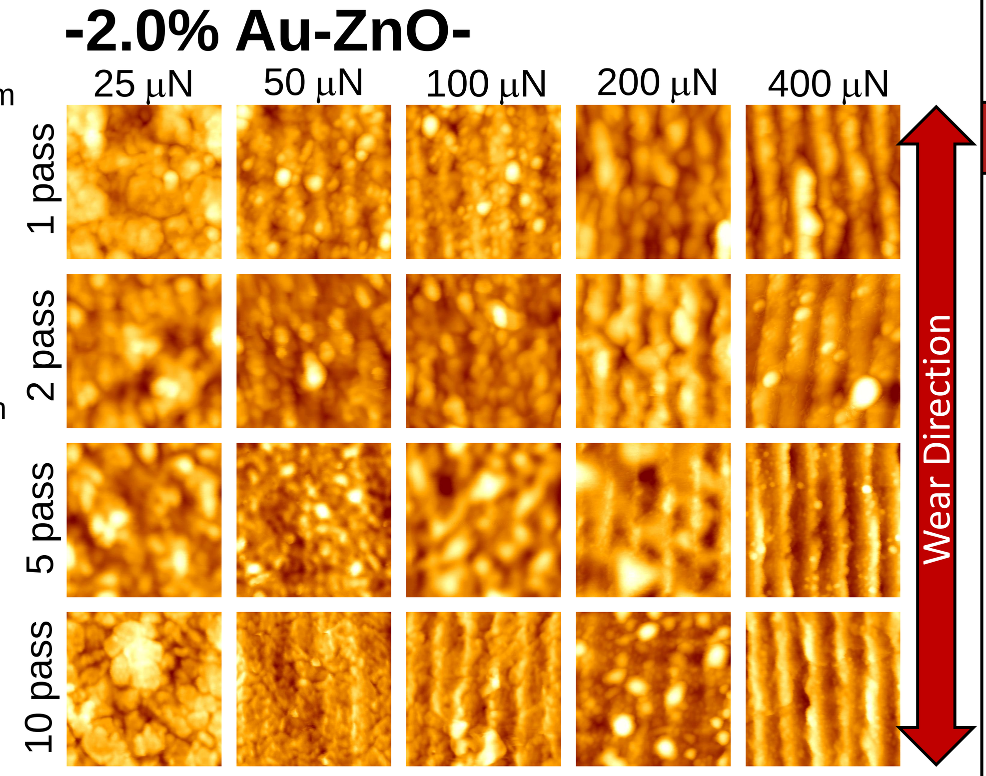
- Evidence of grain coalescence starting at lowest wear condition.
- Obvious wear track formation at low loads



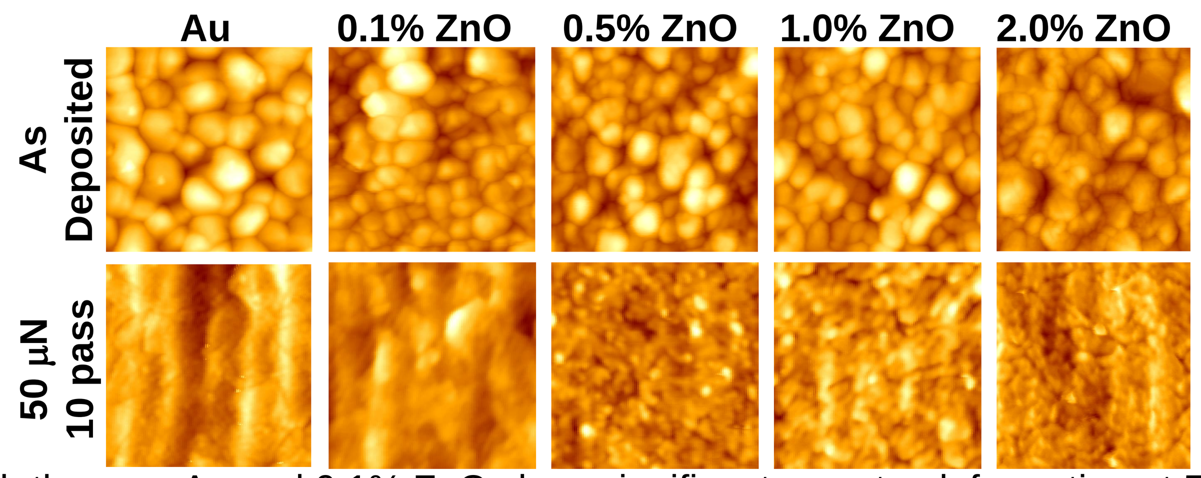
-2.0% Au-ZnO-



- Less evidence of wear tracks at low load conditions
- Some grain refinement in middle load conditions



-WEAR EFFECT-

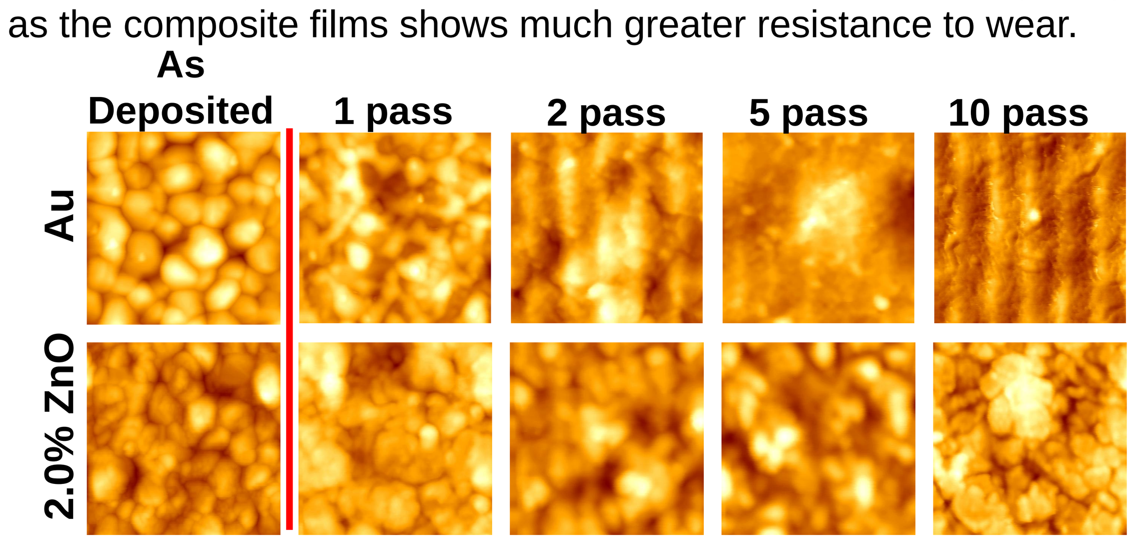


- Both the pure Au and 0.1% ZnO show significant wear track formation at 50 µN 10 pass condition.
- 0.5%, 1.0% and 2.0% films show likely grain refinement.

Point Count Grain Size Measurements (nm)

Wear Condition	Au	0.1% ZnO	0.5% ZnO	1.0% ZnO	2.0% ZnO
As Deposited	135	112	112	112	116
50 µN 10 pass	N/A	N/A	76	85	79

Since these films would mostly be used in low load conditions, the 25 µN wear condition is highlighted below for pure Au and the 2.0%ZnO film. The response of the two films are very different, with obvious deformation occurring the pure Au film where as the composite films shows much greater resistance to wear.



- Pure Au starts showing grain coalescence at lowest wear condition, followed by wear track formation in consecutive passes.
- 2.0% film shows little to no change in structure due to different wear conditions.

-ELECTRICAL PROPERTIES-

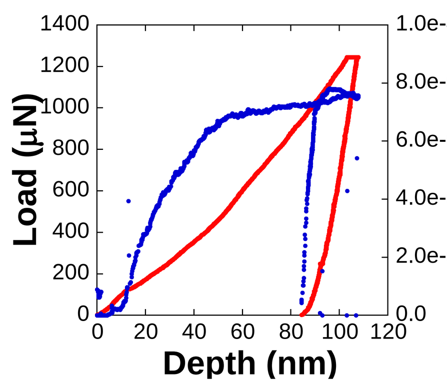


Fig. 5. Typical load-depth-current profile for nanoECR .

NanoECR

NanoECR is a technique developed to investigate the electrical response of a material. A voltage is applied through the boron doped diamond tip, through the sample and into the conducting stage.

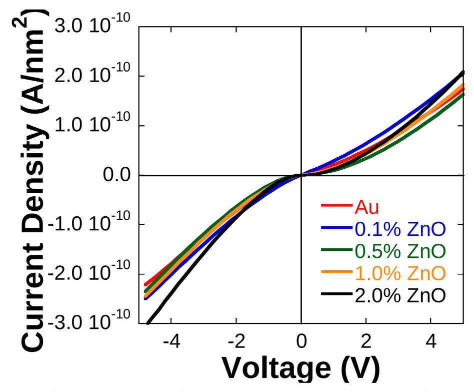
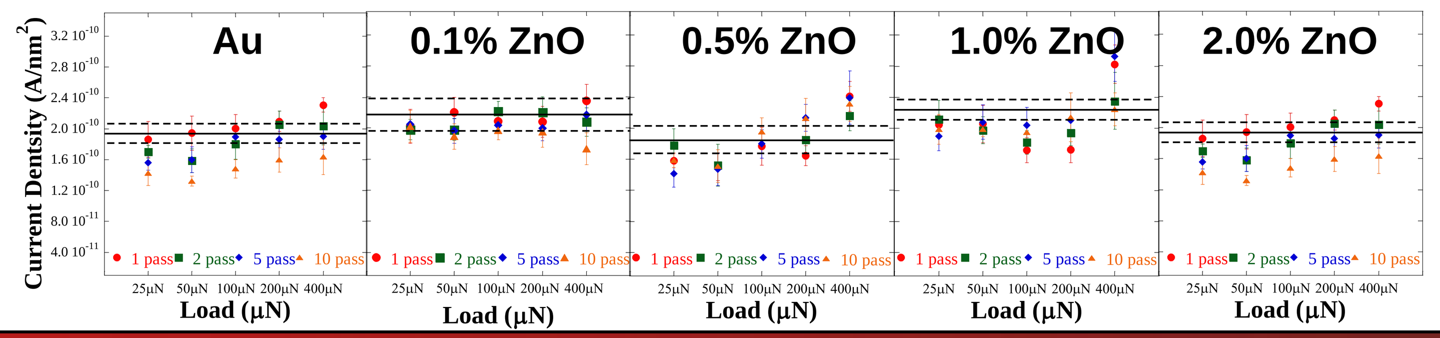


Fig. 6. Voltage sweeps of all as deposited films.

A typical load-depth-current profile from a nanoECR test is shown for the 2.0% ZnO film (Fig. 5). Voltage sweeps were conducted on the as deposited films (Fig. 6), showing similar I-V characteristics for all compositions. The deviation from pure ohmic behavior is due to the doped diamond tip, not the film itself. The current density of the films was calculated from the hold segment of the indent, with the results shown below. There is no significant change in current density as ZnO concentration increases up to 2.0 vol% or as a result of the different wear conditions. Small deviations in the values are not significant since they can be attributed to small miscalculations in the contact area due to changes in the roughness values.



-CONCLUSIONS-

- **SLIGHT DECREASE IN GRAIN SIZE** with addition of ZnO particles.
- **REDUCTION IN (111) TEXTURE** of as deposited films with ZnO particle addition
- **INCREASED HARDNESS** with increasing concentration of ZnO particles.
- **Different WEAR** conditions have a **NEGLIGIBLE EFFECT** on the hardness of the films suggesting little dislocation storage.
- Increasing **WEAR RESISTANCE** and possible **GRAIN REFINEMENT** as the concentration of ZnO increases above 0.1% ZnO.
- **NO SIGNIFICANT ELECTRICAL DEGREDATION** is seen due to ZnO concentrations (less than 2.0 vol%) or any of the wear conditions used in this study.

[1]
[2] Somuri Prasad, Ron Goeke, Nicolas Argibay, Paul Kotula, Joe Michael, ICMCTF *April 23-27, 2012*

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