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D. Ross Economy

Emilio Jiminez

Bobak Ranjbaran

Brad M. Schultz

Marian S. Kennedy

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# Effect of Sliding Contact on the Structure of Cu-X Nanolaminates



## **Introduction:**

Metallic nanolaminates consist of alternating nano-scale metallic layers and have increased resistance to dislocation flow due to high density of interfaces when compared to other composites.

They have potential as both freestanding high-strength foils<sup>1</sup> and wear-resistant coatings<sup>2</sup>. Their mechanical properties (strength) can be tailored by controlling the component layer thicknesses<sup>1,3,4</sup>.

**Research Hypothesis** 

It is hypothesized that as the dislocation density and residual stresses within metallic nanolaminates increase, their wear mechanisms will change.

#### Aims:

- Understand the effects of initial stress on mechanical behavior of model nanolaminate systems.
- Determine dislocation (and deformation zone) evolution of model nanolaminate systems as a result of dynamic loading.
- Elucidate residual stress evolution in nanolaminate films during thermal cycling.



Fig. 1 Concept map of major issues for stable and durable nanolaminate systems.

Cu/Ag and Cu/Nb were chosen as model systems due to their ability to form semi-coherent and incoherent interfaces<sup>1,3,4</sup>, this is due to their various crystal structures (Cu: FCC, Ag: FCC, Nb: BCC).

## **Experimental Methods:**

- **Fabrication of Cu/Nb and Cu/Ag:**
- Substrate: (100) P-type silicon ( $R_a < 2$  nm).
- Deposition rates: 4.0 nm/min (Cu), 2.8 nm/min (Ag), and 2.8 nm/min (Nb).
- Total coating thickness: 1 µm (1000 nm).
- Individual layer thickness: 20 or 100 nm.
- Kurt J. Lesker magnetron sputter deposition system (COMSET).

#### **Characterization of Sliding Deformation:**

- Linear reciprocating wear tests conducted using CETR UMT-2.
- Counterface: 440C SS 3/8" ball bearing.
- Table 1 shows parameters used for the scratch testing.
- Plastic deformation observation using non-contact profilometry (Wyko white light interferometer) and field emission scanning SEM (Hitachi S-4800).

**Table 1** Scratch test parameters used for this study.

Scratch Test Parameters			
Load	Velocity	Length	# of Passes
0.5 N	1 mm/sec	2 cm	20 Passes



Fig. 2 Linear reciprocating wear test of Cu/Nb nanolaminate and stainless steel counterface.

#### Hardness:

- Measured with a Hysitron Triboscope nanoindentation system
- Three-sided diamond Berkovich tip.
- Displacement-controlled indentation mode to maximum depths between 100 and 50 nm, to minimize effects of the underlying substrate.
- Oliver-Pharr indentation analysis used to determine mechanical properties.
- Hardness is defined by:  $H = P / A_{c}$  where P is maximum load and A<sub>c</sub> is contact area

#### Grain Size:

- Measured with a Digital Instruments Nanoscope IIIa atomic force microscope.
- Five areas (scan length: 750–1500 nm) measured.
- Grain size calculated using line intercept method.

D. R. Economy<sup>1</sup>, E. Jimenez<sup>2</sup>, B. Ranjbaran<sup>3</sup>, B. M. Schultz<sup>1</sup>, M. S. Kennedy<sup>1</sup> <sup>1</sup>Department of Materials Science and Engineering, Clemson University, Clemson, SC 29634 <sup>2</sup>Department of Engineering, James Madison University, Harrisonburg, VA 22807

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**Initial Properties of Nanolaminate Systems:** Hardness of nanolaminate systems behave similar to description by the confined layer slip model<sup>1,2</sup>. Decreased layer thickness shows elevated hardness (Fig. 3A). Initial nanolaminate structures were formed (Fig. 3B, 3C, 3D).



**Results:** 



Fig. 3 Hardness of nanolaminate systems increase with decreasing thickness (A), 20 nm nanolaminate geometry as seen in Cu/Nb TEM (B), initial XRD patterns for Cu/Nb (C), and Cu/Ag (D).

#### Scratch Damage of Cu/Ag Systems:

Upon observing morphology of the scratch path, a shift from plowing abrasion in the thicker system to cutting abrasion in the thinner system was noted. Additionally, a deeper scratch path on the 100 nm system shows that the thicker system underwent more damage due to the sliding contact. These results are consistent with those described by previous work<sup>2,5</sup>.



#### **Sliding Direction**

Fig. 4 Plan-view of sliding wear deformation of 100 nm (A) and 20 nm (B) Cu-Ag nanolaminates.

#### Sliding Friction of Cu/Ag Systems:

The coefficient of friction increases with the increase in bilayer thickness (Fig 6.), similar to trends described by previous researchers<sup>2</sup>.





Fig. 6 Coefficient of friction of Cu/Ag nanolaminates during scratch testing. Thicker system shows higher steady state friction level.

Fig. 7 Cross-sectional views of sliding wear deformation of 100 nm (A) and 20 nm Cu-Ag (B) nanolaminates.

#### Scratch Damage of Cu/Nb Systems:

When sliding contact was imposed on the 100 nm Cu/Nb systems, the films buckled due to compressive stress. Additionally, when viewed in cross-section, plastic deformation was observed. This deformation in the upper surface of the film was due to the scratching and not the delamination. Also, it was seen that the deformation was localized to the uppermost layers and did not permeate through the thickness of the film.



Fig. 8 Cross-sectional view of deformation in 100 nm Cu/Nb following sliding contact. Buckling and localized scratch damage can be seen.

# <sup>3</sup>School of Mechanical and Materials Engineering, Washington State University, Pullman, WA 99164





Fig. 5 Measured film profiles following scratch testing. Thicker system (100 nm) shows more deformation in scratch path.

#### **Sliding Direction**

## **Results (continued):**

**Microstructure Evolution of Cu/Nb Systems:** Sliding friction causes localized heating at the point of contact. In order to understand implications of that heating on long term stability, additional work has sought to examine microstructural evolution.



Fig. 9 Grain growth observed in 20 nm Cu/Nb systems in varying environments. Possible grain pinning due to impurities.

### **Discussion and Conclusions:**

- groups<sup>1,3,4</sup>.
- elsewhere<sup>2</sup>.
- Similar to trends reported elsewhere<sup>2</sup>.

- pinning of grain growth by impurities.

### **Future Work:**

- Further examination of friction progression in nanolaminates using scratch and wear testing.
- Cu/Nb.
- Use of FIB and TEM to examine dislocation evolution in individual layers due to deformation.

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Fig. 10 Non-uniform grain size observed by AFM in Cu/Nb system heated in vacuum.

The same relationship between layer thickness and hardness were observed as in other

Decreased friction observed in thinner layered Cu/Ag system. Similar to trends reported

Transition in abrasion morphology from plowing to cutting as layer thickness is decreased.

Friction and wear morphology behavior is likely due to hardness of film systems<sup>5</sup>.

Buckling and localized yielding observed along scratch path in Cu/Nb. Deformation due to scratch did not penetrate through depth of nanolaminate.

Effect of heating and aggressive species on microstructural development observed, possible

Non-uniform grain growth observed in system heated in vacuum.

Examination of Cu/Ni systems to complement Cu/Ag and

Correlation with theory in dislocation dynamic simulations. image taken from Misra<sup>1</sup>.



Fig. 11 Confined layer slip model,

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