CONF-951155--1

LA-UR-95- 3925

Title: PROPERTIES AND CHALLENGES OF NANOLAYER COATINGS

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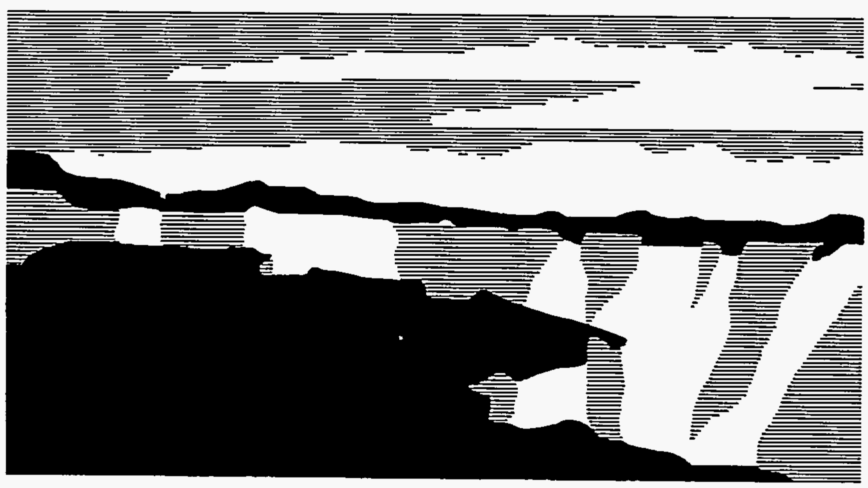
Submitted to: Nanostructured Materials and Coatings '95
November 14-16, '95
Atlanta, GA



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Properties and Challenges of Nanolayer Coatings

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The development of nanophase composites is at the forefront of materials research and may revolutionize the traditional materials design in various materials applications. Specifically, nanophase materials have great potential in structural, electrical, and magnetic applications. In most of these applications, the materials strength is of major concern. Over the past few years, the bulk of the efforts in nanophase structural materials research have been centered around equiaxed nanocrystalline materials. In general, the in-situ composites which were made by drawing two phase castings into fine wires with a massive reduction in area have yielded reproducible and systematic data. The powder processed materials are susceptible to contamination of surfaces, and the mechanical properties data from nanolayers is still very limited. Even though enhancement of the mechanical properties of nanoscale materials has been reported in a number of multilayer structures, the underlying strengthening mechanisms are still not clearly characterized. The lack of a fundamental understanding of how changes in microstructure and interfacial structure affecting the mechanical response can hinder advanced applications of nanophase materials. The objective of this research is to characterize the synthesis-structure-mechanical properties relationship in nanolayers with the central goals of gaining a fundamental understanding of nanophase materials design principles, and developing a comprehensive theory that will relate changes in structure to changes in mechanical behavior for nanolayer composites.

A systematic study has been conducted on several $MoSi_2$-based nanolayer coatings. Alternating layers with thickness ranging between 1 and 20 nm were prepared by sputtering technique. $MoSi_2$-SiC multilayers undergo crystallization, phase transformation and layer spheroidization when exposed to different annealing temperatures. The loss of layer structure also causes degradation in both hardness and modulus. It was first discovered by J-P. Hirvonen's group that nitrided $MoSi_2$ has a very high crystallization temperature (> 1000°C). Further experiments show that $MoSi_2N_x$ (x:3-4) can be used as a stable second phase reinforcement or diffusion barrier coatings. Stable nanolayered structures can be maintained in $MoSi_2$-$MoSi_2N_x$, Mo-$MoSi_2N_x$, and Mo-$MoSi_2$-$MoSi_2N_x$ even after 900°C annealing. The mechanical properties of the $MoSi_2$-based nanolayers depend strongly on the phase and morphology of the constituting layers. Significant increase in hardness and modulus is observed accompanying the crystallization process. In comparison with single or poly-crystals, the nanolayers have much higher hardness but lower modulus, which project higher toughness in the nanolayers. The wear properties are also influenced by the phase and layer thickness. Significant improvement in the wear resistance is observed with the decreasing layer thickness. Furthermore, studies show that single phase $MoSi_2N_x$ (x: 0-4.2) exhibits a wide range of hardness and modulus with varying nitrogen content and annealing conditions. These observations suggest the possibility of engineering $MoSi_2N_x$ to produce different material properties for different mechanical applications.

Properties and Challenges of Nanolayer Coatings

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Research sponsored by DOE BES/AEP and DPRP Program.

OUTLINE

1. **Background**
2. **Synthesis of Coatings**
3. **Fundamental Properties**
4. **Corrosion, Oxidation and Wear Resistance**
5. **Potential Applications and Challenges**
6. **Conclusions**

NANOLAYER COATINGS

PROPERTIES:

- Potential Improvement in Mechanical Properties Predicted
  Hall-Petch Model   Koeheler Model

- Higher Fracture/Yield Strength in Metal Multilayers
  Improved Wear Resistance in Metal Multilayers

CHALLENGES:

- High Temperature Structural Stability of Nanolayers
  Grain Boundaries/Interfaces as Fast Diffusion Paths

- Optimum Design Principles of Nanolayer Composites
  Tailor Properties By Control of Structure

OBJECTIVE

- TO CHARACTERIZE THE SYNTHESIS - STRUCTURE - PROPERTIES RELATIONSHIP IN NANOLAYERS.

- TO EXAMINE THE HIGH TEMPERATURE STRUCTURE STABILITY OF NANOLAYER COATINGS AND ITS INFLUENCE ON THE MECHANICAL PROPERTIES.

Examples: Cu/Nb
$MoSi_2$-SiC, $MoSi_2$-$MoSi_2N_x$, $MoSi_2$-$MoSi_2N_x$-Mo

MATERIALS PREPARATION

Sputter Deposition

Single Phase: $MoSi_2$, SiC, $MoSi_2N_x$ (x: ~0-4)
Cu, Nb

Multilayers: $MoSi_2$-SiC, Mo-$MoSi_2$, $MoSi_2$-$MoSi_2N_x$,
Mo-$MoSi_2N_x$, Mo-$MoSi_2Ni_x$-$MoSi_2$
Cu/Nb

Total Film Thickness: ~1 μm, Layer Thickness: 1-100 nm

Si {100} Substrate

Heat Treatment

Annealing in a Vacuum of 10^{-8} torr

500°C, 600°C, 700°C, 800°C, AND 900°C For 1 Hour

900°C for 2, 3,4, 8 and 16 Hour

COATINGS CHARACTERIZATION

- **Hardness and Modulus Measurement**
  - Nanoindentation: Nanoindenter™
- **Wear Experiment**
  - Room Temperature, 30% Humidity
  - 63 g on Si_3N_4 Pin, 1000 revolution (1/sec)
- **Microstructural Characterization**
  - Cross-Sectional TEM
  - HRTEM: Philips CM30ST
    JEOL 3000F
- **Corrosion and Oxidation Characterization**
- **Fracture Toughness Evaluation**

SUMMARY AND CONCLUSIONS

1. Improvement in Mechanical Properties (e.g. Hardness, Wear Resistance) Was Observed in Nanolayer Coatings with Decreasing Layer Thickness.

2. Cu/Nb Nanolayers Exhibit a Strong Texture Relationship. Changes in Defect Structure, Interfacial Structure and Constituent Layer Phases Were Observed with Decreasing Layer Thickness.

3. The Hardness and Modulus of $MoSi_2$-Based Nanolayers are Dependent on Phase and Geometry.

4. $MoSi_2N_x$ is non-reactive with $MoSi_2$ and Mo, making it a Stable Second Phase Reinforcement and Diffusion Barrier.

5. The Hardness and Modulus of Single Phase $MoSi_2N_x$ can be Tailored by Controlling the Nitrogen Content and Heat Treatment to Produce Different Materials Properties for Different Mechanical Applications.