Identifying toughening concepts in CrN/AlN multilayer coatings

Yinxia Zhang, Matthias Bartosik[a], Steffen Brinckmann[b], Subin Lee, Christoph Kirchlechner

[a] Department of Materials Science, Montanuniversität Leoben, Leoben, A-8700, Austria
[b] Microstructure and Properties of Materials (IEK-2), Forschungszentrum Jülich, 52425, Jülich, Germany

Motivation Hard coatings are used to protect component surfaces. Critical components can suffer failure due to an insufficient toughness of the coating. One promising way to toughen is utilizing AlN phase transformation from cubic to hexagonal under CrN/AlN multilayer coatings, which requires additional energy during crack propagation.

Objective Enhance the toughness of the CrN/AlN coating by promoting phase transformation at crack tips. Determine the optimal AlN layer thickness.

Methodology

1) Sample preparation

Thin film synthesis: Different period coatings, e.g. 2/2 4/2 nm CrN/AlN, monolithic CrN, and AlN coatings are synthesized on silicon and steel substrate by collaborating with Dr. Bartosik.

Cantilevers preparation: Removed the substrate with KOH etching, milled cantilevers with a focused ion beam, 3 nA and 300 pA at 30kV for coarse milling and 100 pA for fine milling. Pre-notches were fabricated with 5 pA current.

- Single cantilever with a bridge notch

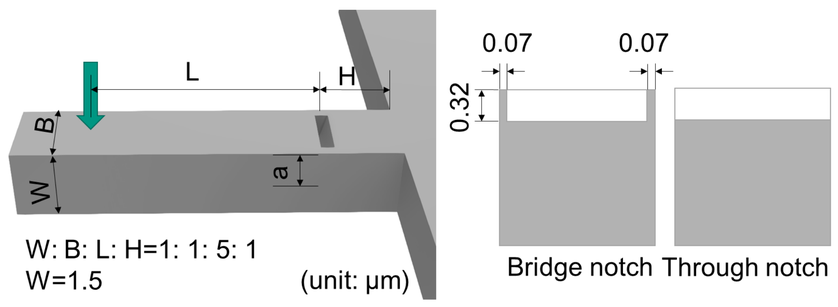


Fig. 1. Geometry of bridge pre-notch cantilever

Bridge notch

- “easy” to mill
- Low scatter in obtained data
- natural sharp crack initiates

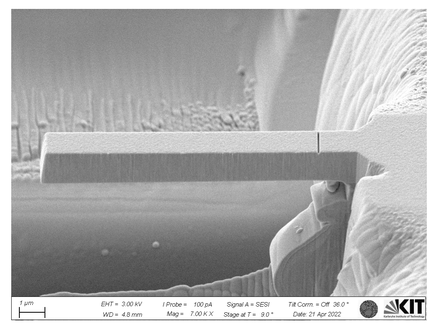


Fig. 2. CrN cantilever

2) Mechanical testing

Determine the toughness with the *in situ* SEM indenter bending fracture tests. A Hysitron PI 89 PicoIndenter equipped with a 10 μm wide diamond wedge used in displacement controlled mode, at 5 nm/s.

Results

- Optimization of cantilever geometry using CrN monolithic coating

- Pre-notch calibration

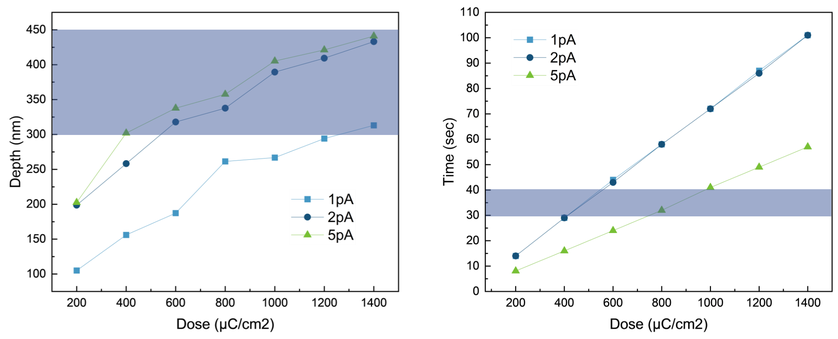


Fig. 3. The notch depth and milling time at different milling current at 30 kV

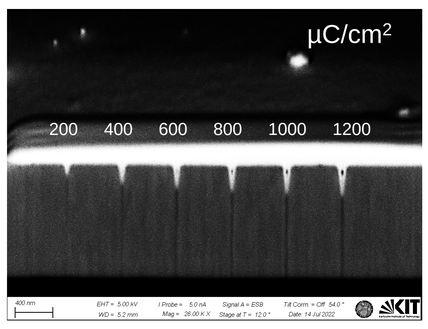
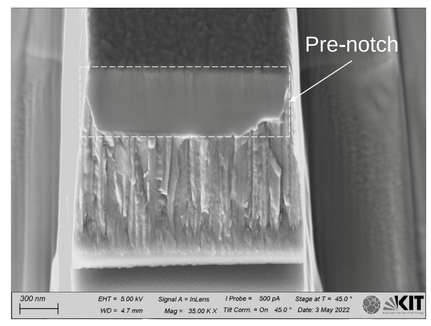


Fig. 4. Notch depth with different dose @30kV, 5 pA

- Check geometry



Good notch:

- straight
- symmetric
- Sharp
- Bridge＜100 nm

Fig. 5. Cross section of CrN cantilever fracture

There is a balance between short time and sharp beam, milling total time should be lower than 40 seconds, and notch depth should between 300nm and 450nm (a=0.2W~0.3W). So the optimal dose for CrN is 800 μC/cm² at 5 pA , notch depth is 320 nm, and total time is about 30 seconds.

Next steps

- Establishing protocols for testing hard coating cantilevers.
- Quantification of fracture toughness of multi-layered coatings.
- Post-mortem analysis of the cantilevers to investigate phase transformation from cubic to hexagonal using XRD or TEM.
- Microtensile testing of multi-layered coatings to generates cracks over the bulk sample so that the phase transformation can be detected by XRD.

Yinxia Zhang
Email: yinxia.zhang@kit.edu
Institute for Applied Materials
Mechanics of Materials and Interfaces (IAM-MMI)

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