QUANTIFICATION OF INTERFACE STRENGTH OF A THIN FILM USING A NEW MICROCANTILEVER GEOMETRY

Eloho Okotete[1]*, Subin Lee[1], Steffen Brinckmann[2], and Christoph Kirchlechner[1]

[1]Institute for Applied Materials, Karlsruhe Institute of Technology, Karlsruhe, Germany, [2] Forschungszentrum Jülich, Jülich, Germany
** Presenting Author email: eloho.okotete@kit.edu*

Abstract
Interfaces failure occurs not only in structural materials but also in functional material systems including systems for energy conversion and storage. Such failures lead to degradation of mechanical and functional properties, such as battery capacity or electrical conductivity. In bulk scale, there are various experimental methods to investigate the interface strength and its failure mechanisms, for instance, peeling test, superlayer test, or indentation test. One of the disadvantages of these approaches is that it can be applied only to relatively thick coatings [1,2]. Small-scale mechanical testing is a powerful tool for studying interface properties because it can quantify micro- and nanometer sized thin films, and individual interfaces of interest can be tested by isolating them using focused ion beam (FIB). Single and double cantilever beams have been used to investigate fracture/delamination properties of single interfaces [3,4], however, these methods are prone experimental imperfections arising from testing geometries.

In this talk, we propose a new *in situ* scanning electron microscope (SEM) microcantilever design which provides reliable and quantitative interface toughness. In addition, the optimized geometry can promote a pre-notch (or crack) to propagate in a stable manner, which is important to generate a natural crack front without FIB-induced damage/artifacts.

1. Methodology
As a first step finite element calculations were used to model and optimize the new geometry and the output from the optimized geometry was used as a guide to model the first experiments. Our first experiments were conducted on 3 μm thick (Hf-Nb-Ta-Zr)C coatings deposited on silicon substrates. Sample pieces were cut from the wafer and etched in 30 wt.% KOH at 80ºC for 1 hour to get a free standing film. Subsequently, the etched sample was machined into microcantilevers according to the geometries from the FEM calculations using the FIB. The last step of machining geometry was introducing notches into the interface of the microcantilevers by FIB. Microfracture tests were conducted *in situ* on the micro samples and the data analysed and compared with FEM results.

2. Results
Figure 1a shows the new micro cantilever-based geometry where the cantilevers are loaded perpendicular to the interface plane in the presence of pre-crack lying on the. Contour integral was used to extract the driving force for crack growth finite element calculations. The first results from these calculations showed that the cantilever becomes compliant at longer crack length suggesting that the geometry is intrinsically stable regardless of the material system used. The first experimental results also agreed with the FEM results as the geometry was observed to be intrinsically stable and there was no sign of unstable crack growth during the testing. This is evident from crack growth at steady force increase, starting at the focus ion beam milled pre-notch and slowly growing along the thin-film substrate interface. Post mortem images were used to obtain the length of propagating crack at intervals and this was compared to the crack length obtained from unloading stiffness. These data helps us to determine the interface properties of the thin film studied.

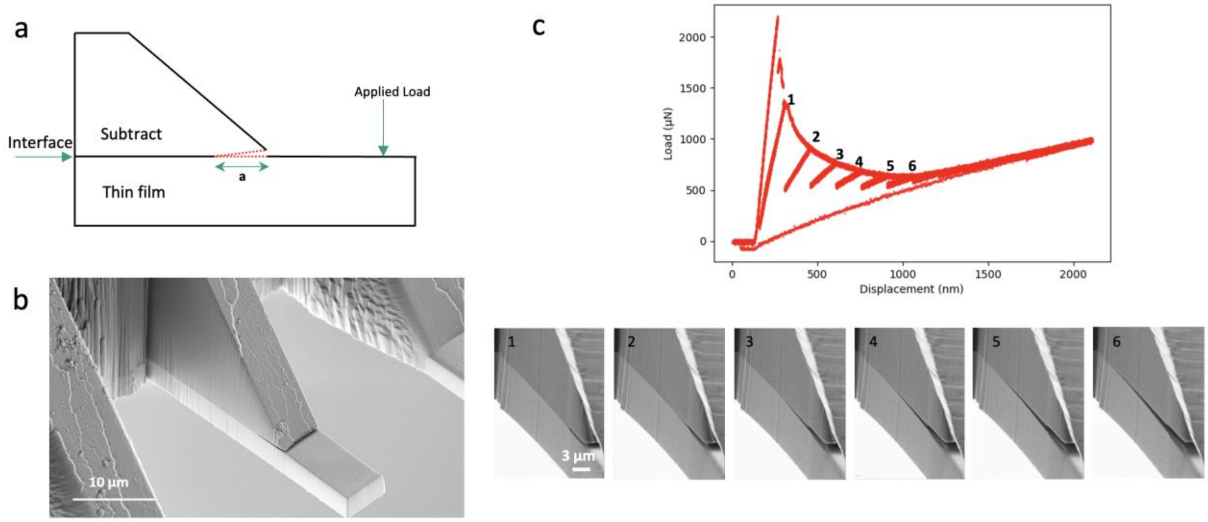

Fig. 1 – a) sketch of new geometry; b) SEM image of the FIB machined sample; c) results from *in situ* testing.

Acknowledgements

The authors acknowledge funding by the Robert Bosch Stiftungsprofessur for nanostructured functional materials.

References

[1] Z. Chen, K. Zhou, X. Lu, Y. C. Lam, "A review on the mechanical methods for evaluating coating adhesion", Acta Mechanica 225 (2014) 431 – 452.

[2] J. Chen, S.J. Bull, "Approaches to investigate delamination and interfacial toughness in coated systems: an overview", Journal of Physics D: Applied Physics 44 (2011) 034001.

[3] K. Matoy, T. Detzel, M. Müller, C. Motz, G. Dehm, "Interface properties of thin films studied by using micro-cantilever deflection technique", Surface & Coatings Technology 204 (2009) 878 – 881.

[4] G. Sernicola, T. Giovannini, P. Patel, J. R. Kermode, D. S. Balint, T. B. Britton, F. Giuliani, " In situ stable crack growth at the micron scale", Nature Communications 8 (2017) 108.