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Characterizing the interface toughness in functional material systems

Eloho Okotete¹, Steffen Brinckmann², Subin Lee¹, Christoph Kirchlechner¹

1 – Institute of Applied Materials, KIT Germany

2 – Forschungszentrum Jülich, Germany



Functional properties and interfaces



Loss of battery capacity electrolyte

El-Amiri et al. IRSEC (2017)

Loss of electrical functionality

Lead

Ju et al. NDT & E Intl. 34 (2001)

Measuring interface fracture in coatings

Peel off test



https://www.impact-solutions.co.uk/peel-adhesion-test/



Roshanghias et al. Surf. Coat. Technol. 259 (2014)

- Easy and quick measurement
- Huge experimental scatter
- Geometry and size dependent results
- · Intrinsic material properties cannot be extracted
- Mechanisms based understanding and design not possible

Quantitative measurements



Gopalan et al. Mat. Des. 224 (2022)



Liu et al. Appl. Phys. Lett. 102 (2013)

Comparisons of existing geometries



Geometry that is artefact free and has high success rate HELMHOLTZ

Focus of present work

- Simple geometry
- Stable crack growth
- Sharp pre-notch / No FIB damage
- High success rate
- Applicable to thin films



- Finite element 2D calculations (Abaqus)
- In-situ SEM experiments with PI89 nanoindenter

Validation of the geometry by FEM



Experimental validation

Material system (Materials Chemistry, RWTH Aachen University, Aachen, Germany)

- Film (Hf-Nb-Ta-Zr)C
- Substrate Si

Selective etching of Si substrate by KOH



FIB milled Cantilever



Experimental validation



Existing vs New geometries

Geometries	Crack Growth	Fabrication	Notch	Success Rate
2 <u>µ</u> m	Unstable	~ 3 hours	Poor	5/5
1 μm	Stable	Complex	???	5/10
	Stable	~ 2 hours	Tolerant	18/22

Conclusion and Outlook

We have now been able to measure the fracture properties of individual interfaces in functional material systems.

- Classical problems of focused ion beam (FIB) based fracture mechanics, i.e. blunt notch, Ga⁺ damage, are not present due to stable crack growth.
- Our tool can be used for all material systems where the interface toughness is limiting reliability.
- We proved that for the interface of multi metal carbides (high entropy carbides). This can also be used for other multilayered systems.

In the future

11 • Measure fracture properties of functional interfaces at higher temperatures (up to 500°C), MHOLTZ

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Thank you for your attention



