

Indentation-induced delamination and adhesion work evaluation at elevated temperature in industrial semiconductors

Speaker: PJ Wei 24 April, 2023



#### **Outlines**

- Basic Mechanisms of Adhesion Measurements and Correlation between Results Collected by Different Techniques
- Applications of thin film adhesion measurement at Elevated
   Temperature



## **Basic Mechanisms and Methodology Correlation**



#### Interface Bonding Issues of Adhesion in Thin Film Industries

xSol<sup>®</sup> - Heating, Cooling, Humidity Heating: 800°C • Cooling: -120°C • Humidity: 10-75% RH





https://www.researchgate.net



### Overview of Adhesion Metrology Techniques Measurements for Interfacial Bonding



- special sample preparation
- Low spatial resolution
- Ex-situ measurement



#### Nano indenter's Advantages:

- Direct measurements
   Nanoindentation or nanoscratch tests can directly
   performed on product surface.
   No sample preparations are needed.
- 2. High spatial resolution Nanoindentations or nanoscratches are at sub-micron or micron scale.





#### Scratch-Induced Cohesion and Adhesion on Low-k Film





#### Scratch-Induced Cohesion and Adhesion of Low-k Film









### Indentation-Induced Cohesion and Adhesion on Hard Coating



#### In-situ Nano-Indentation in SEM



### Indentation-Induced Cohesion and Adhesion Check of Post- SPM Imaging





#### **Correlation of Critical Load Results**





Sample		Scratch	Method	Indentation Method		
		Ave (uN)	Std (uN)	Ave (uN)	Std (uN)	
С		696.2	38.7	7231.2	573.5	
D	)	792.5	42.2	7702.0	715.7	
A	L.	433.3	19.9	4173.5	419.4	
В	B 510.7		22.9	5249.3	464.3	





#### High Speed Indentation with Pop-in Signal



#### How it works:

- Approach routine makes contact with the sample
- Electrostatic actuation to perform experiment and withdraw
- Between indents, piezo is moved to next position

#### TI-980: up to 6 indents/s





### Indentation-Induced Delamination Using XPM



Post-SPM image after XPM



Cube corner tip Applied load: 9mN Array: 5x5 @ 30s Spacing: 15um



## **Challenges at Elevated Temperature**



#### **XPM Indentation at Elevated Temperature**



xSol heating on TI980



XPM at high temperature



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#### XPM Results at Different Temperature Levels Low-k Films on Silicon

#### Sample 2



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### Comparison of Critical Load Results at All Temperature Levels Low-k Films on Silicon



Wafer 2	20°C	100°C	200°C	300°C	
ave (uN)	431	393	371	348	
std (uN)	45	31	29	47	
CV	10.4%	7.9%	7.8%	13.5%	

Wafer 6	20°C	100°C	200°C	300°C	
ave (uN)	349	319	308	286	
std (uN)	15	21	20	18	
CV	4.3%	6.6%	6.5%	6.3%	

### High Load Indentations at Elevated Temperature Polymer Film on Copper



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### Adhesion Work Calculation at Temperature Levels Polymer Film on Copper





Post Optical image

	V	H (GPa)	T (°C)	E (GPa)	t (nm)	L <sub>crit</sub> (uN)	a (um)	r (um)	G (J/m <sup>2</sup> )
$(1/r) \cdot (1-\nu)^2$	0.3	0.49	35	6.02	10000	115218.2	30.3	11.5	6.81
	0.3	0.39	150	4.69	10000	84091.5	40.7	16.0	6.04
	0.3	0.11	215	1.59	10000	68276.1	41.9	17.4	1.68

L.G. Rosenfeld, et al., Journal of Applied Physics 1990, 67(7), 3291

Poisson ratio is assumed to be 0.3 at all temperature levels.

 $\mathbf{G} = \frac{0.627 \cdot H^2 \cdot t \cdot (1 - \nu^2)}{-\tau} \cdot \mathbf{G}$ 

## Adhesion Work Calculation at Temperature Levels Polymer Film on Copper











#### Take Away

- special sample preparation
- Low spatial resolution
- Ex-situ measurement







pressure blister test



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laser blister test

fatigue friction test











# Thank you!

PJ Wei Pal-Jen.Wei@bruker.com

Innovation with Integrity