



FEM of cracking during nanoindentation and scratch testing in the coated systems

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Outline

- Introduction to IMR SAS/Divison of Ceramic & Non-metallic Materials
- Scientific lecture – modeling of cracking during nanoindentation and scratch testing
 - Tribological test vs. scratch test
 - Nanoindentation
 - Scratch testing

Institute of Materials Research of SAS (...since 1955)



Division of ceramic and non-metallic systems (2015-...)

Department of structural ceramics (1990-2015)



12 persons + 5 PhD. students

Publications and projects in VD02 in 2017

Main activities/fields of interest:

- Microstructure and **mechanical properties** of bulk monolithic structural ceramics
- (silicon nitride, alumina, silicon carbide...) and (nano)composite ceramics
- Reinforced ceramics for special applications (graphene platelets/CNT reinforced ceramics)
- Properties of ultrahigh temperature ceramics (ZrB₂ based composites)
- SPS of ceramics
- PVD of hard coatings
- Fibers by electrospinning

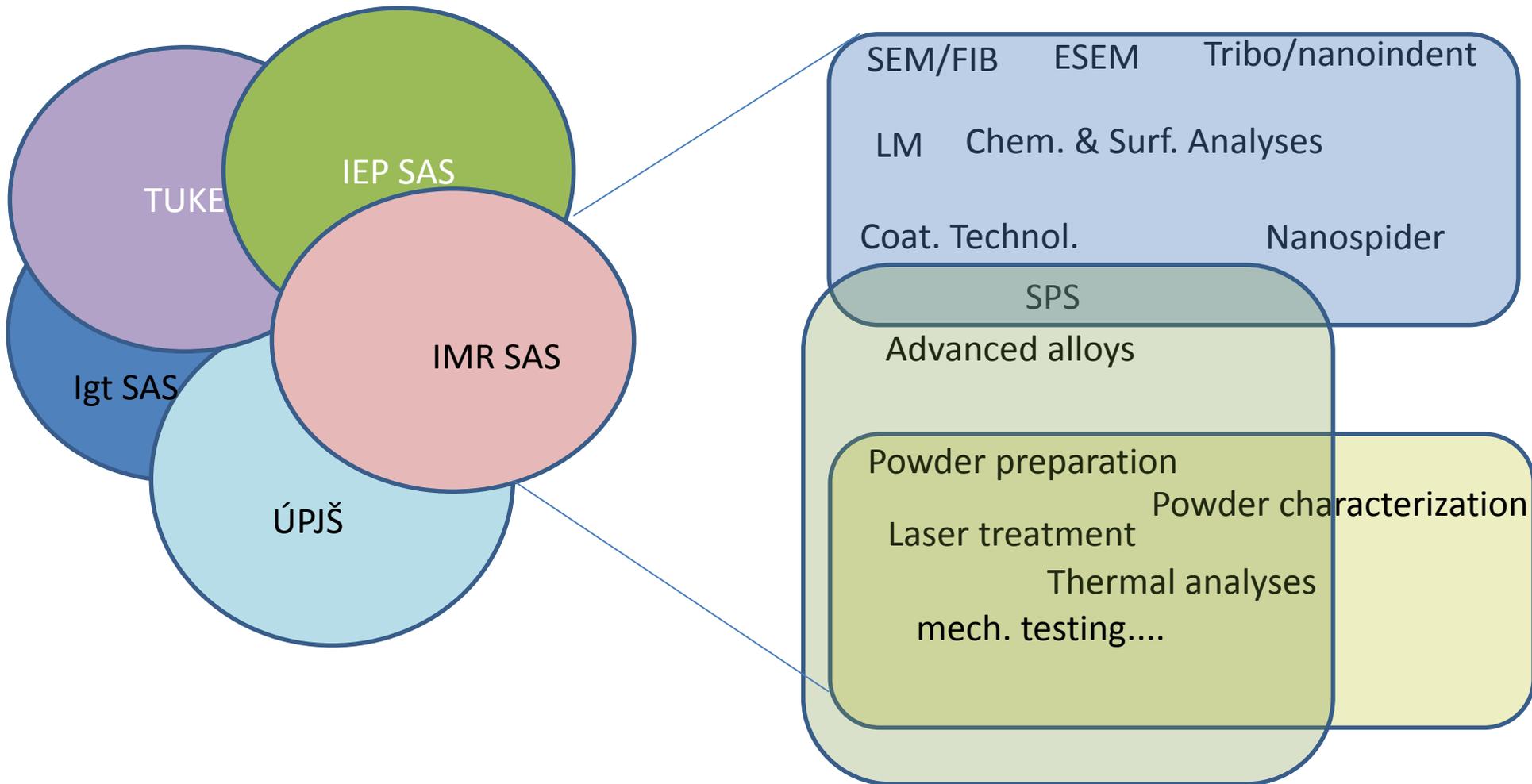
.....**nanindentation and tribological behavior** of different materials

Typ publikácie	Počet publikácie	Počet publikácie
ADCA-Zahr.karent.čas.impaktovaný	35	35
DAIb-Kvl.práce-doktorand.v angl.	3	3
Písomná časť PhD.slov.	1	1
AEDA-Ved.pr.v dom.recen.ved.zbor.konf.	8	17
ADEA-Zahr.nekarent.čas.impaktované	2	
ADEB-Zahr.nekarent.čas.neimpaktované	5	
ADFA-Dom.nekarent.čas.impaktované	1	
ADFB-Dom.nekarent.čas.neimpaktované	1	

APVV	6
VEGA	4
SF projects	0
M-ERA	3
COST	3
Bilateral	1
Total	17

PROMATECH

RESEARCH CENTRE OF ADVANCED MATERIALS AND TECHNOLOGIES



Technologies

- PVD coatings (DC/HiPIMS and HiTUS)
- Spark Plasma Sintering
- Nanofibers (electrospinning)
- Laser treatment
- Amorphous metals
- Microwave sintering

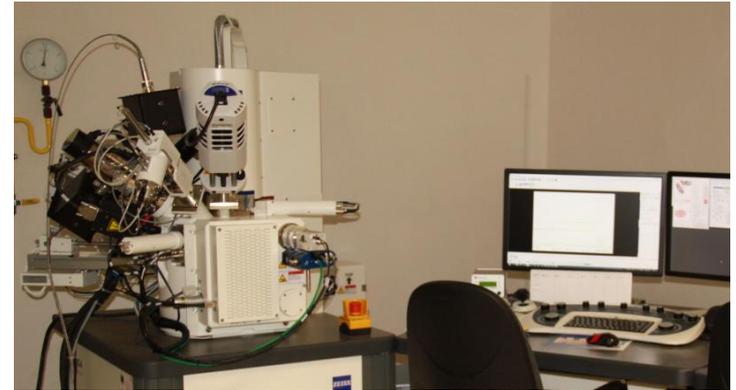


High resolution microscopy

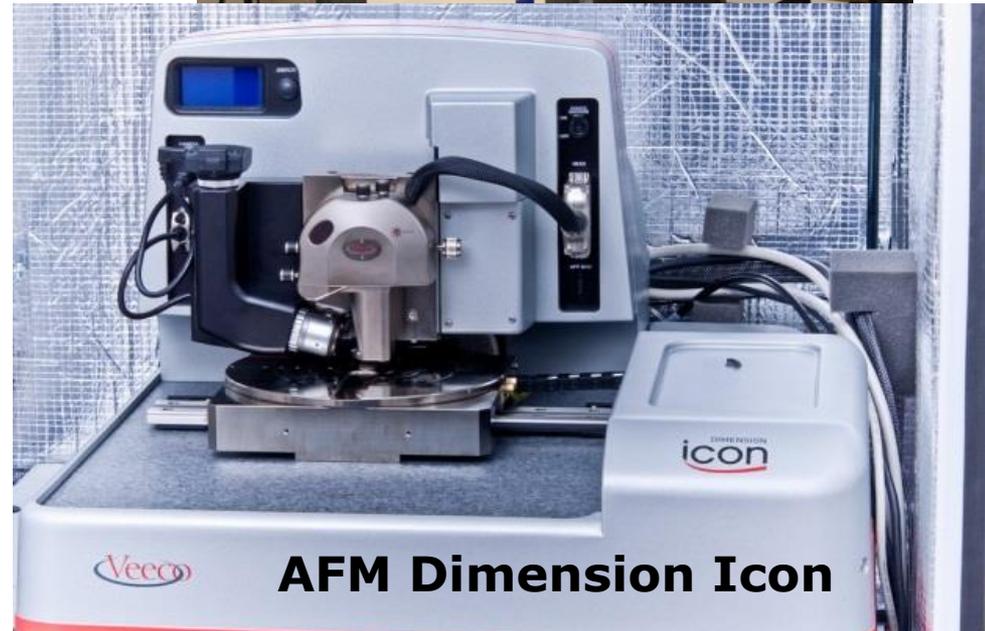
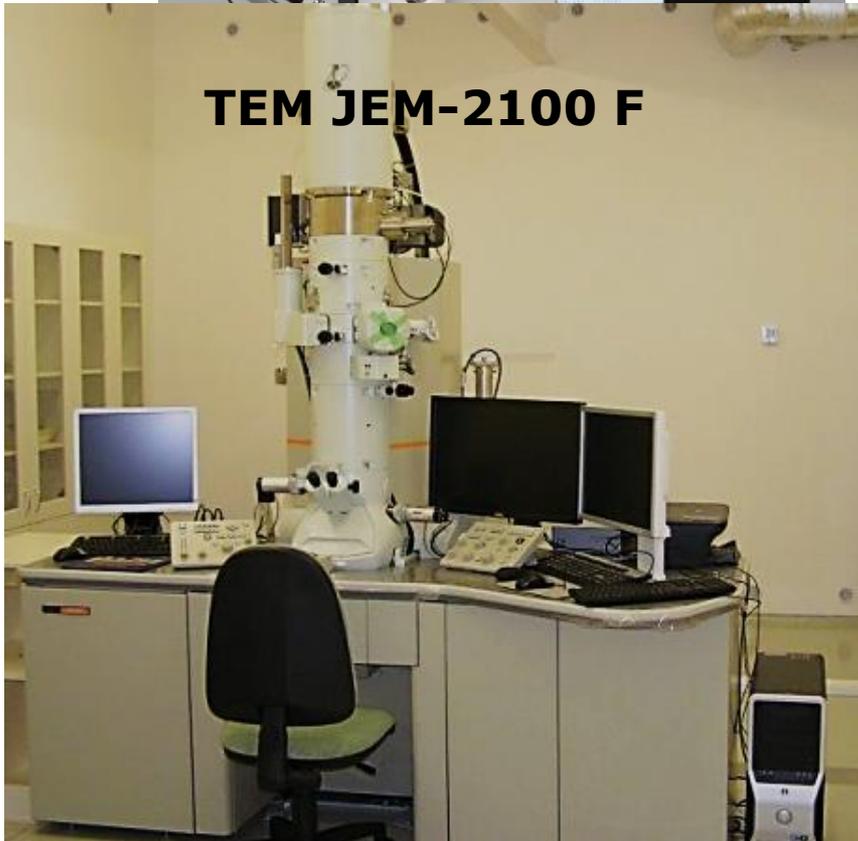
W filament eSEM/EDS, WDS



FIB/SEM DualBeam FESEM



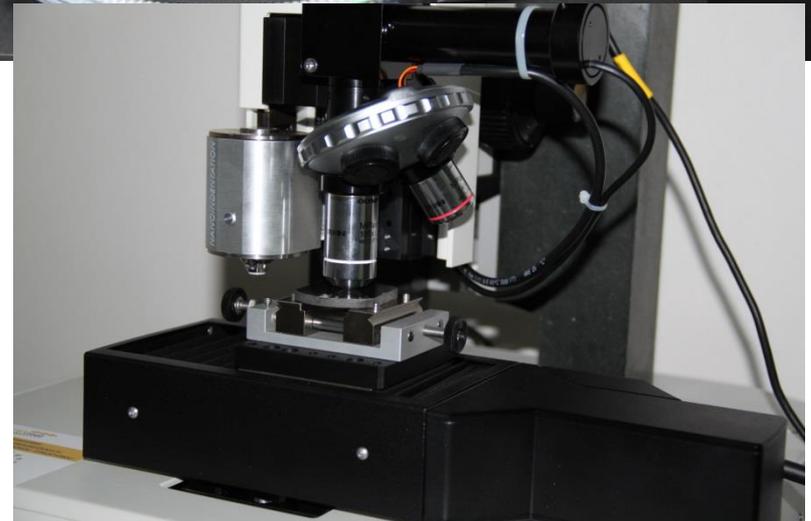
TEM JEM-2100 F



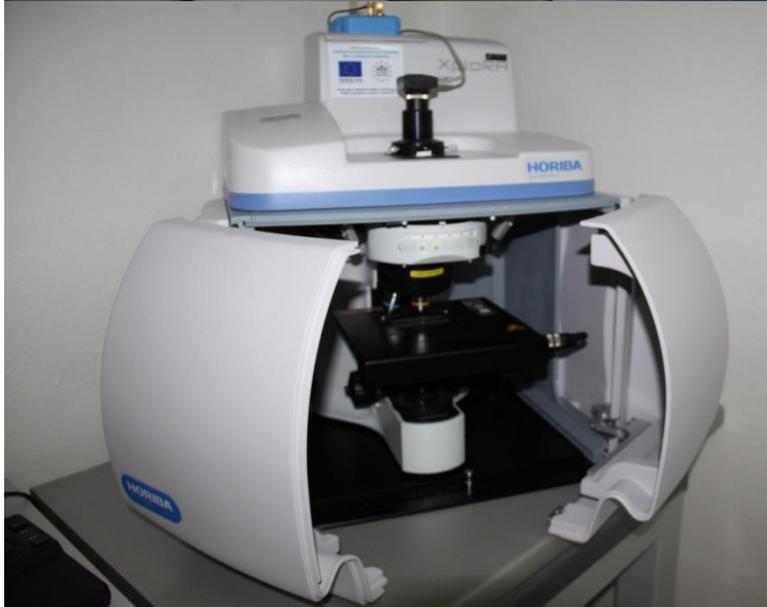
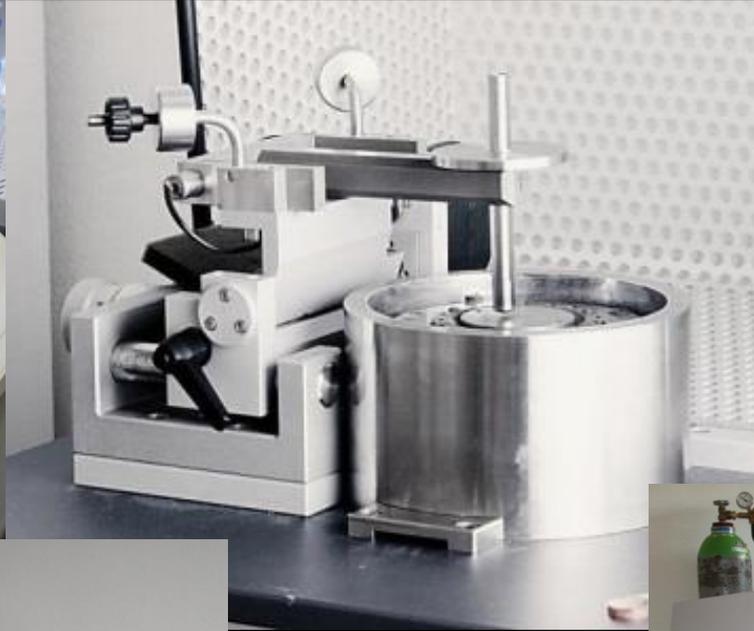
AFM Dimension Icon

Nanohardness testing

Nanohardness testers G200 (Agilent) NHT CSM Instruments



Tribology



**Raman
GDOES**





FEM of cracking during nanoindentation and scratch testing in the coated systems

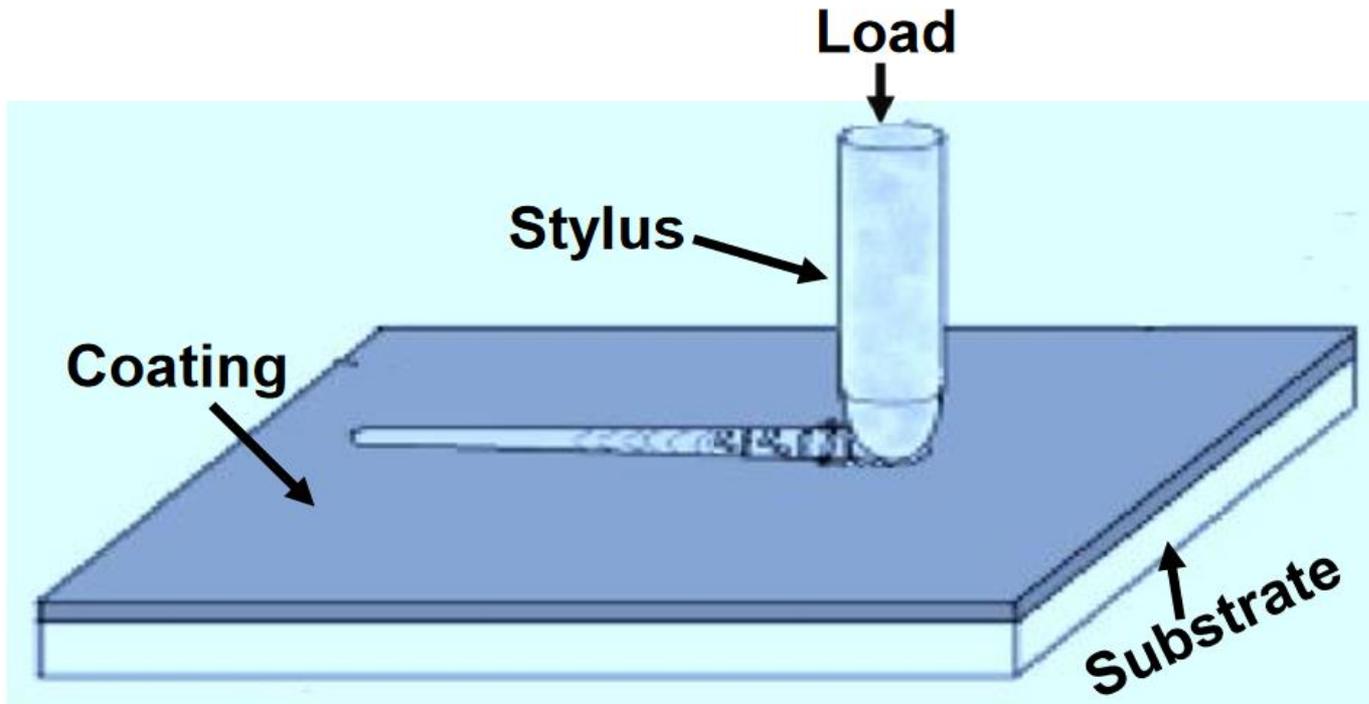
František Lofaj, Dušan Németh*

Institute of Materials Research of the Slovak Academy of Sciences
Košice, Slovakia

* (currently at: Robert Bosch, Ltd., České Budějovice, Czech Republic)

Introduction

Tribological test vs. scratch testing
(constant load/progressive load increase)



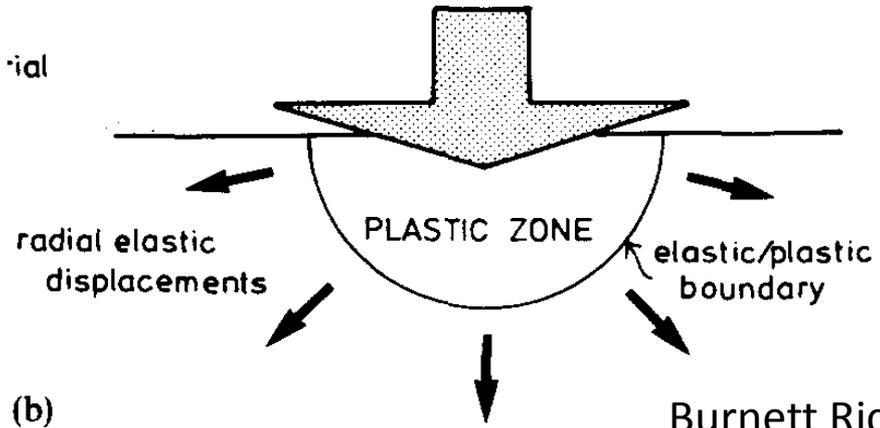
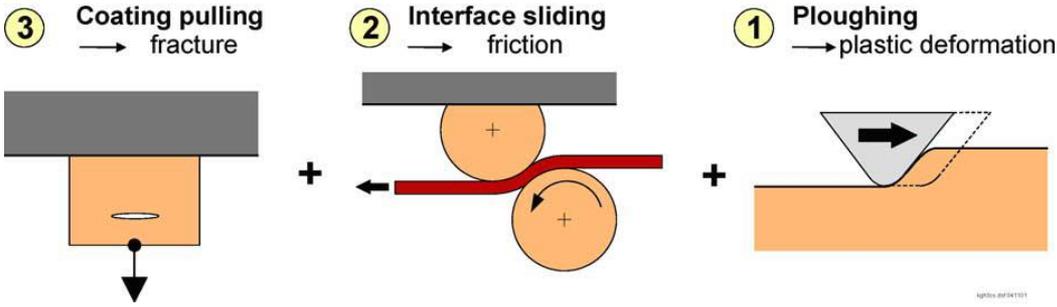
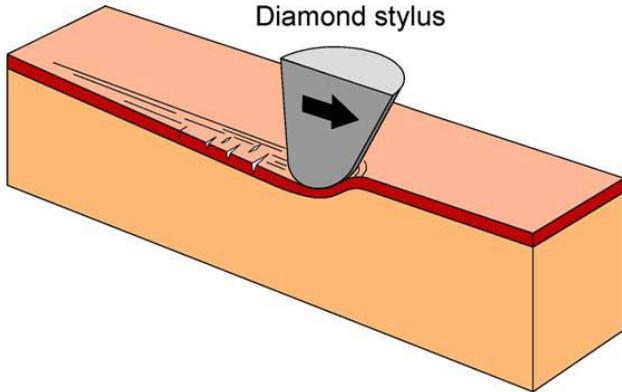
Kuiry, Bruker 2012

Scratch Direction →

Differences: ball diameters, higher maximum loads – stronger **deformation** of the substrate..

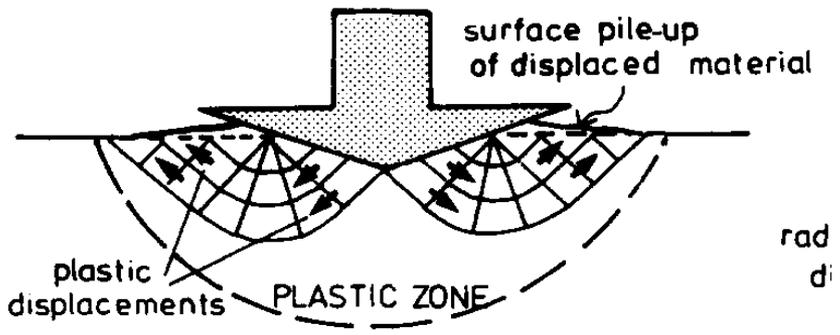
Basic processes during scratch test

Holmberg Trib. Int. 2005



(b)

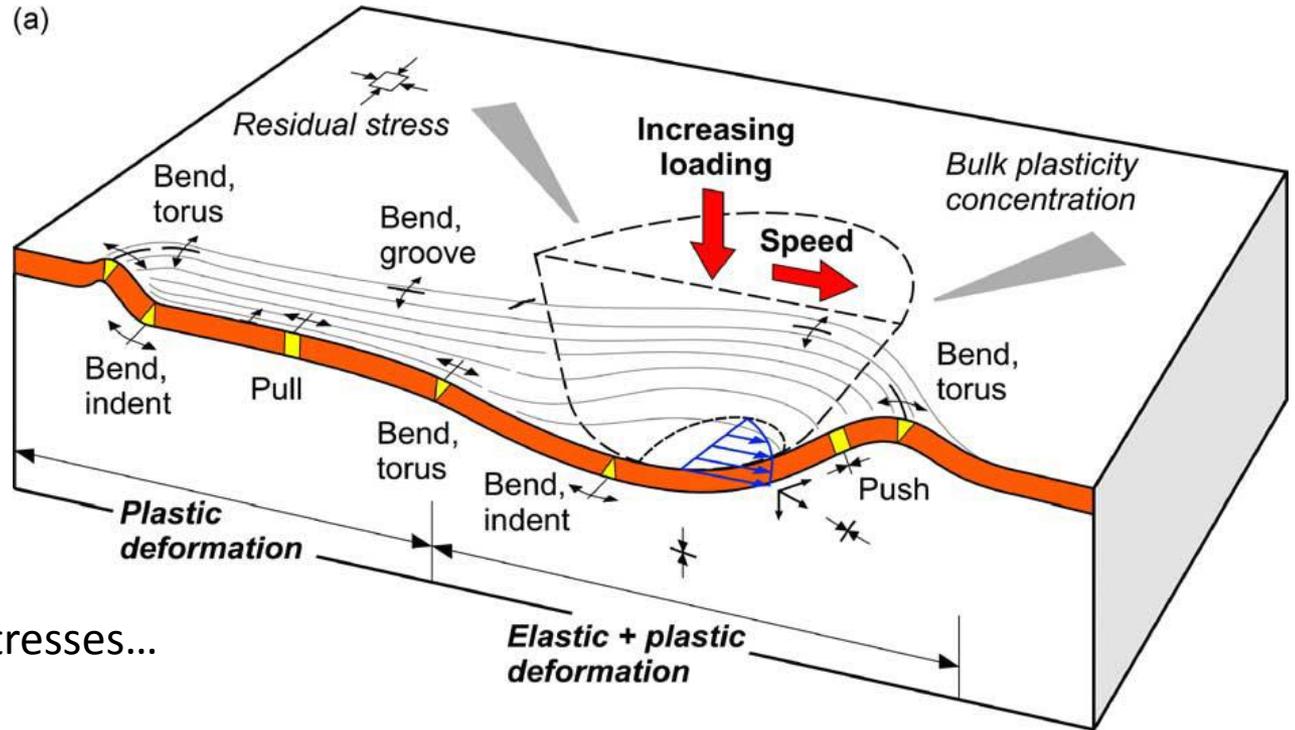
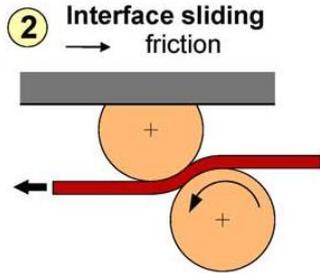
Burnett Rickerby TSF 1988



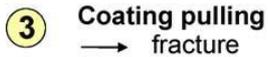
rad di

(b)

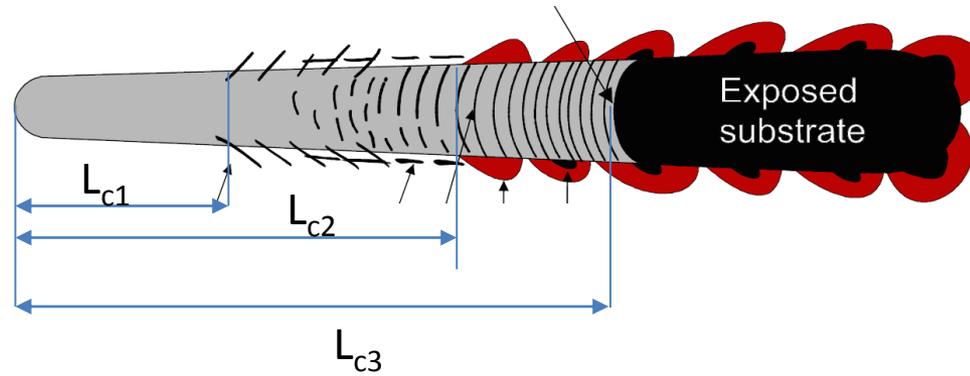
Effects of interface sliding and friction during scratch test



Tensile and compressive stresses...

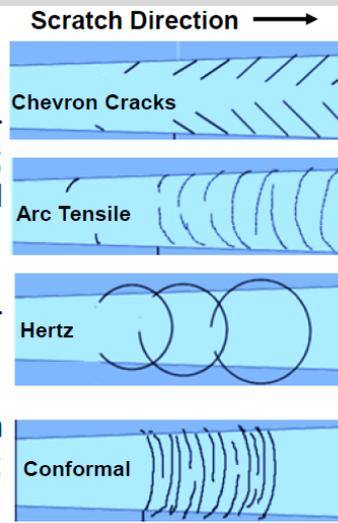


Holmberg SCT 2006



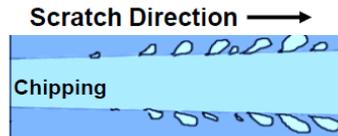
Through Thickness Cracking

- *Brittle Tensile Cracking*: Nested micro-cracks; open to the direction of scratch; straight and semi-circular; formed behind the stylus.
- *Hertz Cracking*: Series of nested micro-cracks within the scratch groove
- *Conformal Cracking*: micro-cracks form while coating try to conform to the groove; open away from the direction of scratch.



Cohesive cracks (in the coating) analogous to “frame” cracks formed due to local tensile stresses

Adhesive cracks in the coating



Chipping

Rounded regions of coating removal extending laterally from the edges of the scratch groove

Spallation

- *Buckling* : coating buckles ahead of the stylus tip; irregularly wide arc-shaped patches missing; opening away from scratch direction.
- *Wedging* : Caused by a delaminated region wedging ahead to separate the coating; regularly spaced annular-circular that extends beyond the edge of the groove.
- *Recovery*: regions of detached coating along one or both sides of the groove; produced by elastic recovery behind the stylus and plastic deformation in the substrate.
- *Gross Spallation* : Large detached regions; common in coating with low adhesion strength.

Scratch Direction →

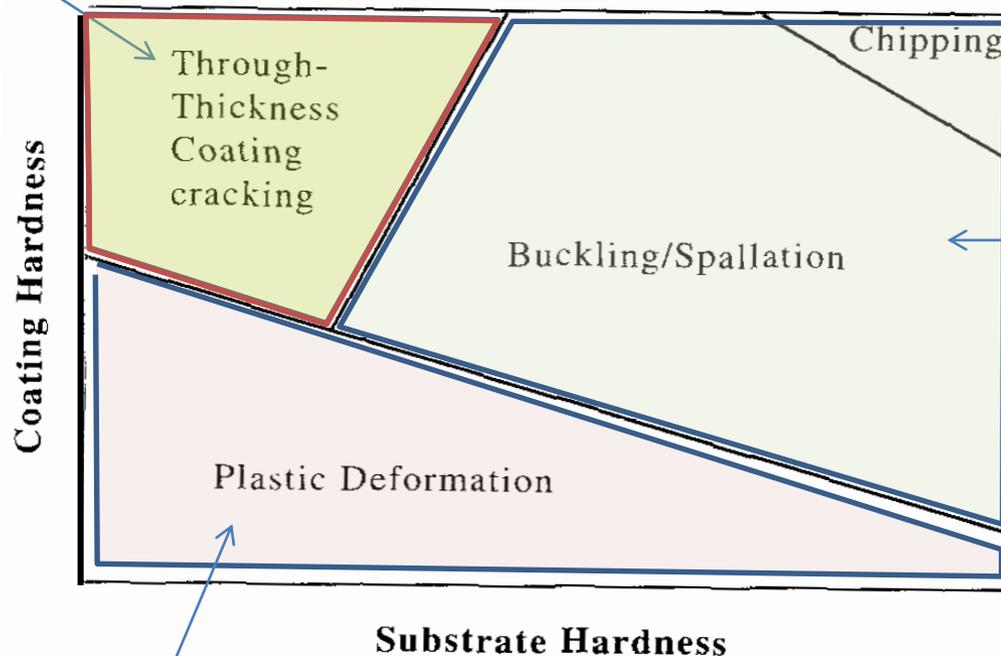


Adhesive cracks at the coating/substrate interface

Modelling of the scratch tests

Modelling ... in 2D; the first 3D models – in 2003 (Holmberg)

modelling of cohesive coatings - xFEM (+ FEM)



Bull Trib. Int. 1997

Modelling of adhesive cracks

modelling of plastic deformation - FEM

Nanoindentation = progressive load increase scratch test with zero velocity...

Objectives

Finite element modeling of the processes of:

- cohesive cracking in hard brittle W-C coating/steel substrate during nanoindentation and scratch testing
- attempt to get information about the strength and toughness of coatings.

Applicability: cracking in the transfer layer during tribological tests

Experimental procedure

Substrate: construction steel (12050) after hardening (860°C/30 min, quenching into oil, tempering 200°C/120 min); polished to $R_a \sim 10$ nm

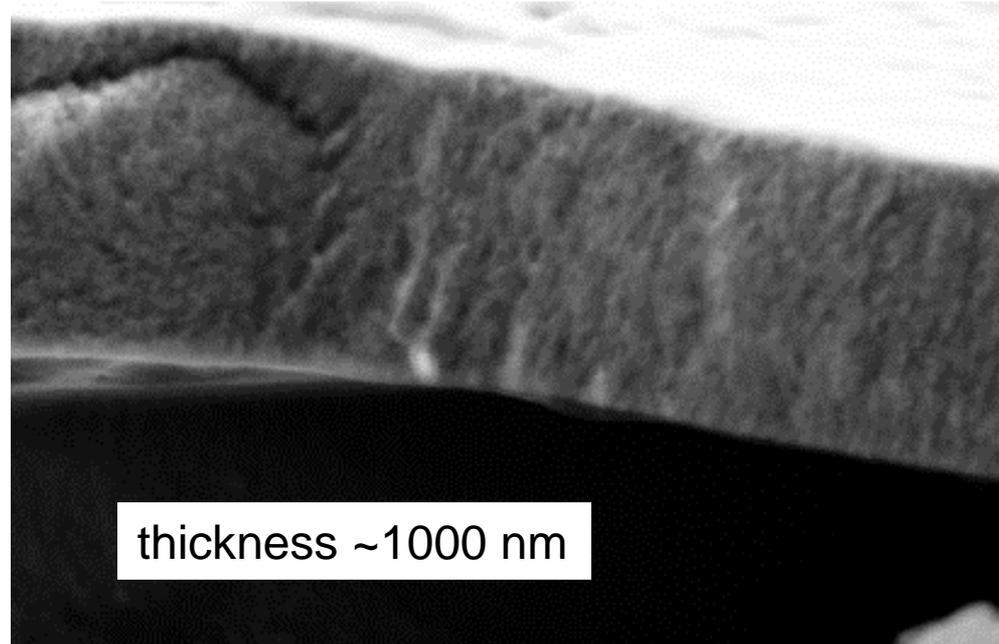
Deposition procedure

HiPIMS, 3 inch WC target, working pressure 0,5 Pa, 500W, $\alpha = 3\%$ (200 Hz, 150 us), 2 vol.% of C_2H_2 in Ar atmosphere, 34 min

W-C coating, $t = 991 \pm 35$ nm.



Cryofox Discovery 500, Polyteknik, Danmark



amorphous structure....

Experimental techniques

Nanoindentation

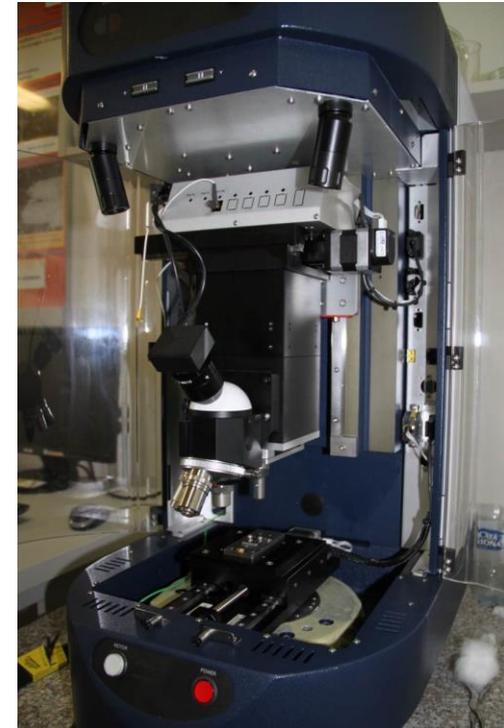
G200, Agilent Technologies



Berkovich indenter tip with $R = 1.15 \mu\text{m}$
CSM mode, $f = 45 \text{ Hz}$, amplitude 2 nm
constant depth of 1500 nm ;
constant strain rate 0.05 s^{-1} , $4 \times 4 = 16$ indents.

Scratch tester

Model UMT 2, Bruker, USA



Rockwell C diamond tip $R = 200 \mu\text{m}$; cone angle 120°
pre-load 2 N ; linearly increased up to 100 N
scratch length 7 mm
indenter velocity 0.014 mm/s

Results

Multiple circular „frame“ cracks in hard W-C coating/steel substrate during indentation

200 mN

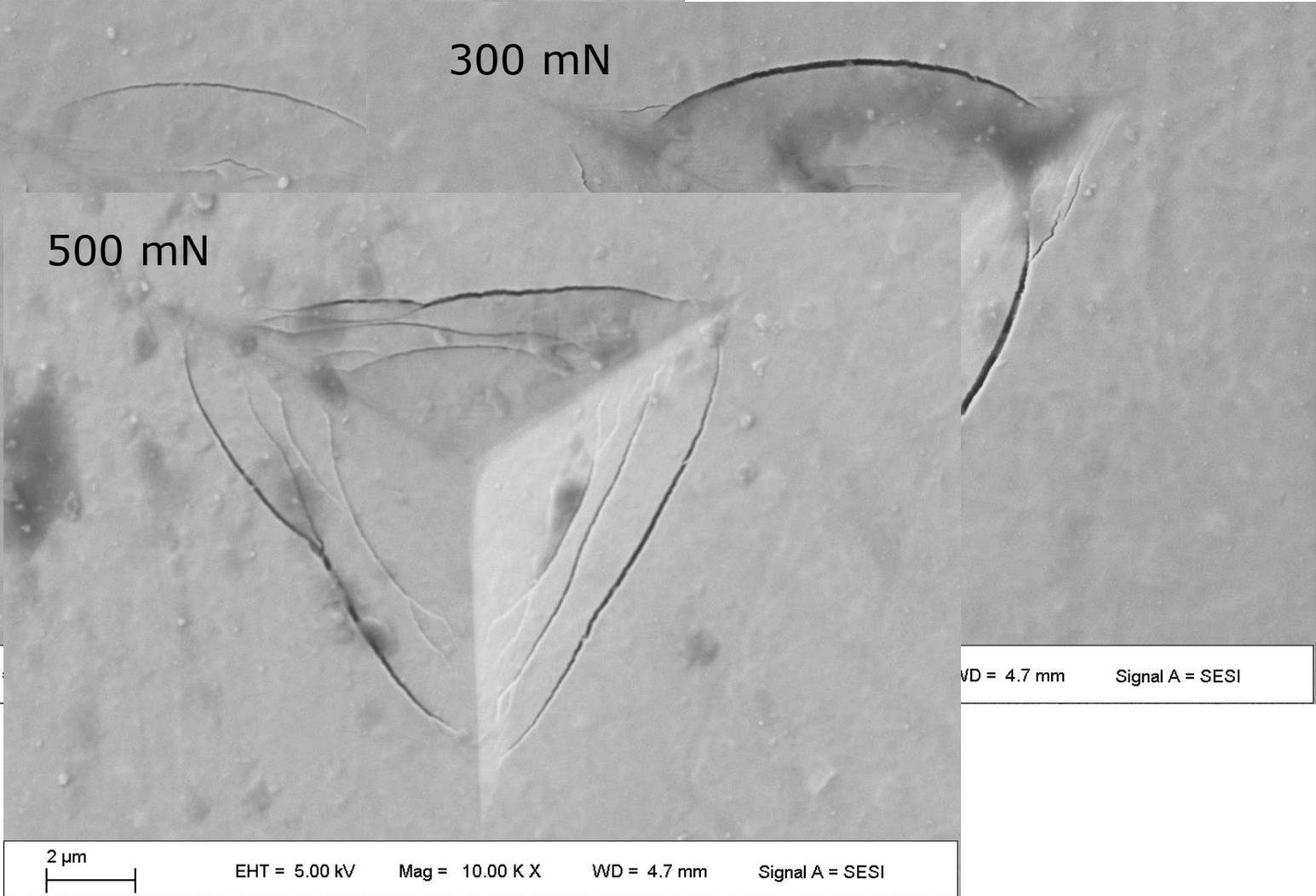
300 mN

500 mN

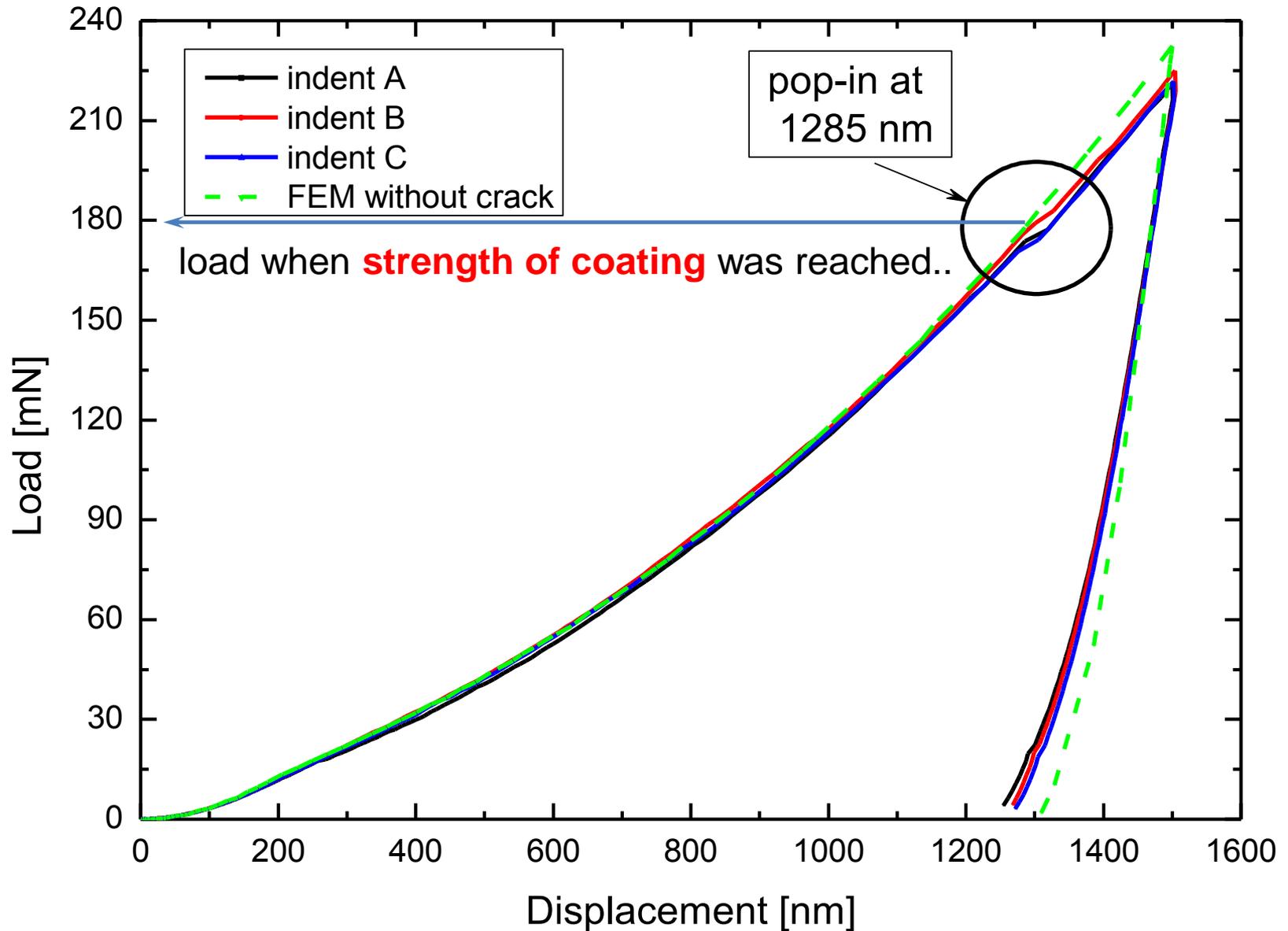
2 μ m
EHT = 5.00 kV

WD = 4.7 mm
Signal A = SESI

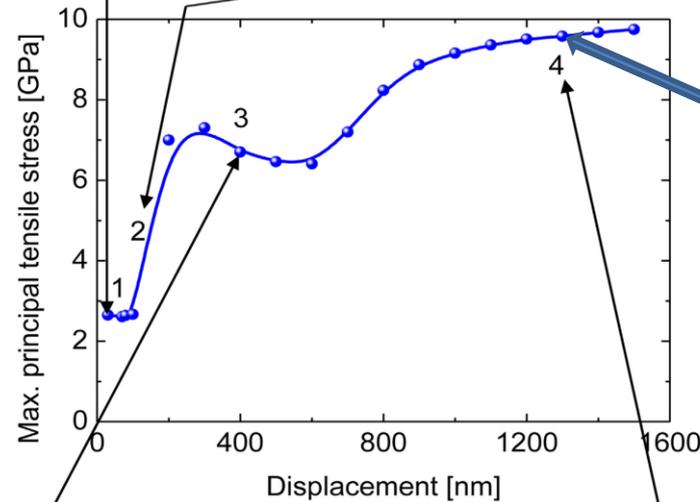
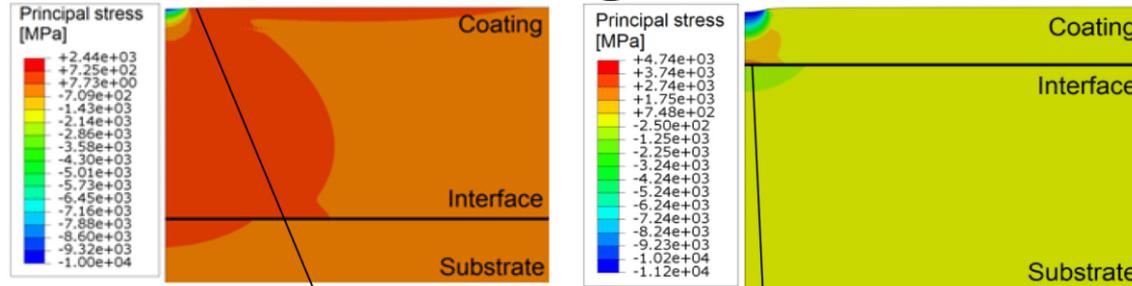
2 μ m
EHT = 5.00 kV
Mag = 10.00 K X
WD = 4.7 mm
Signal A = SESI



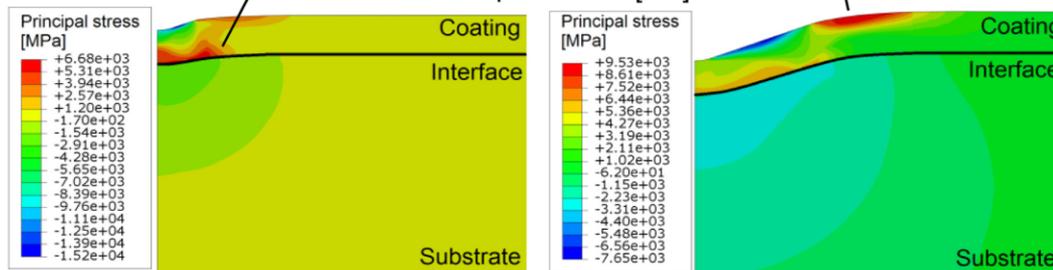
Cracking during indentation



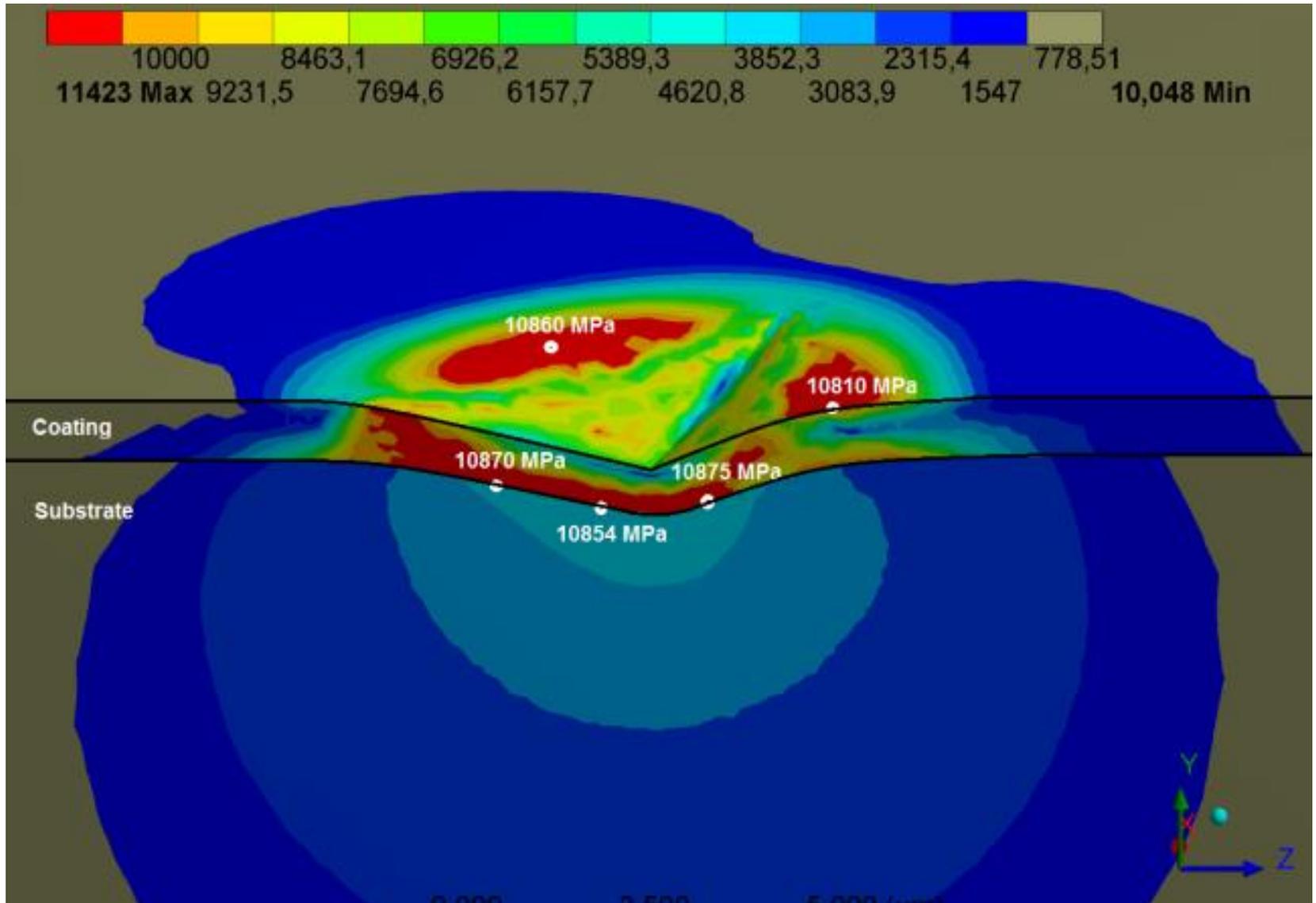
Evolution of principal tensile stresses and plastic deformation during nanoindentation



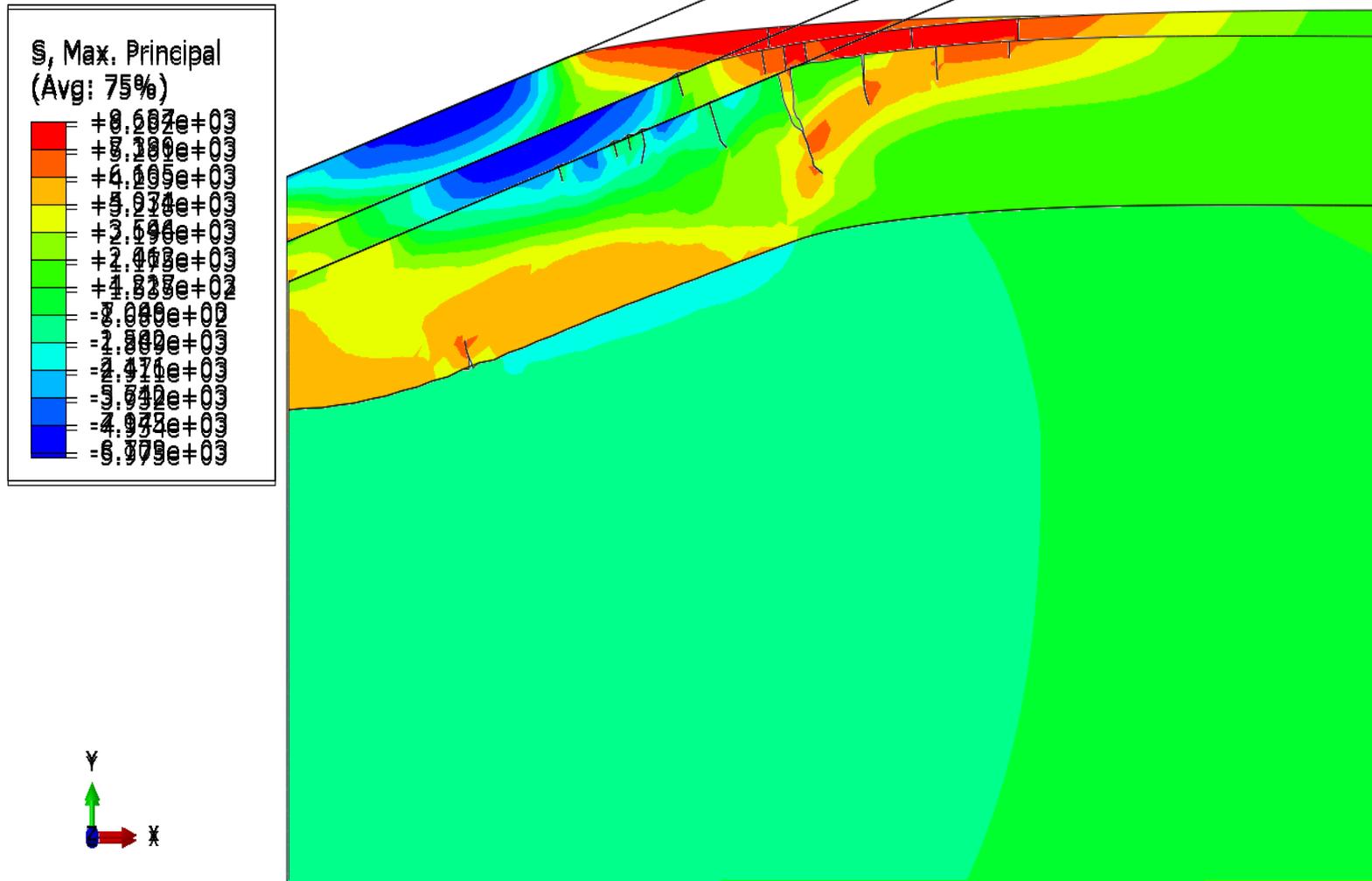
Strength of the coating..
...cohesive cracking..
controlled by substrate
deformation



Principal stress distribution in coating/substrate at 200 mN load

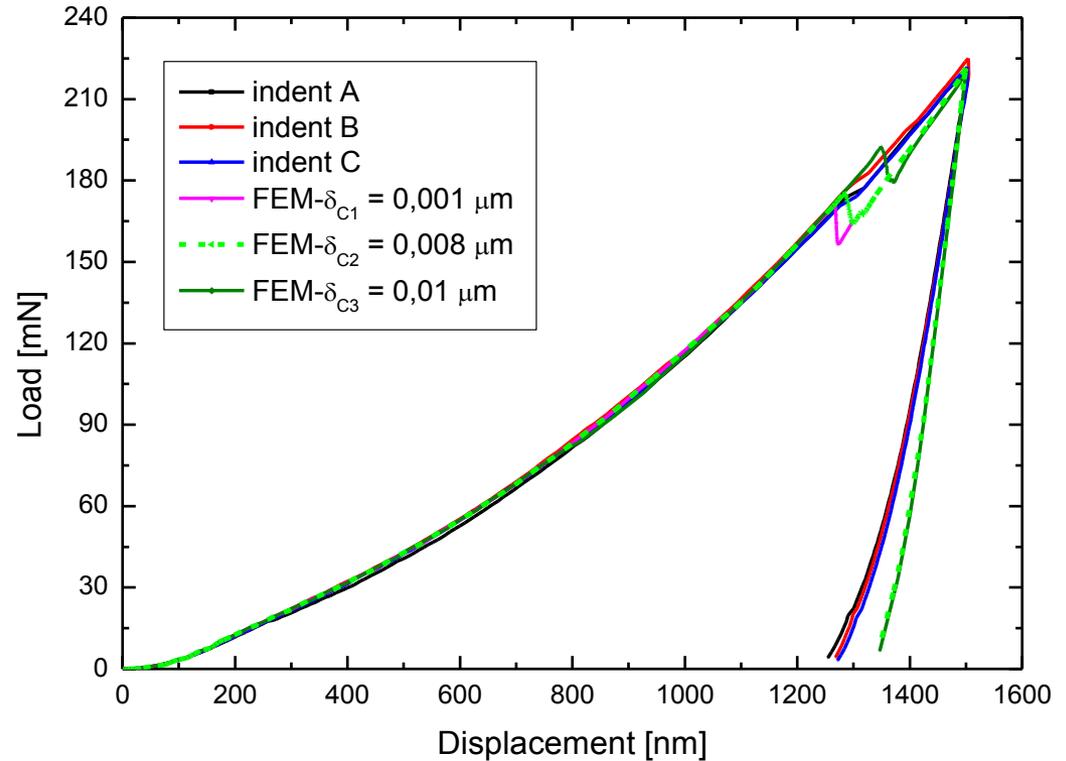
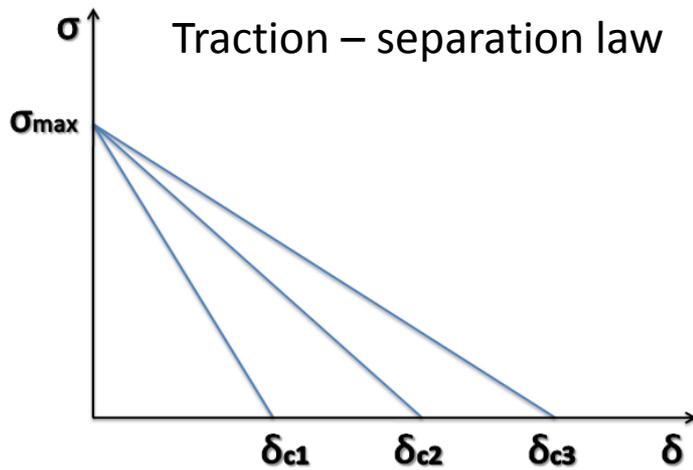


xFEM multiple crack evolution



infinite bonding between the coating and substrate

Fracture toughness of W-C coating on steel from XFEM

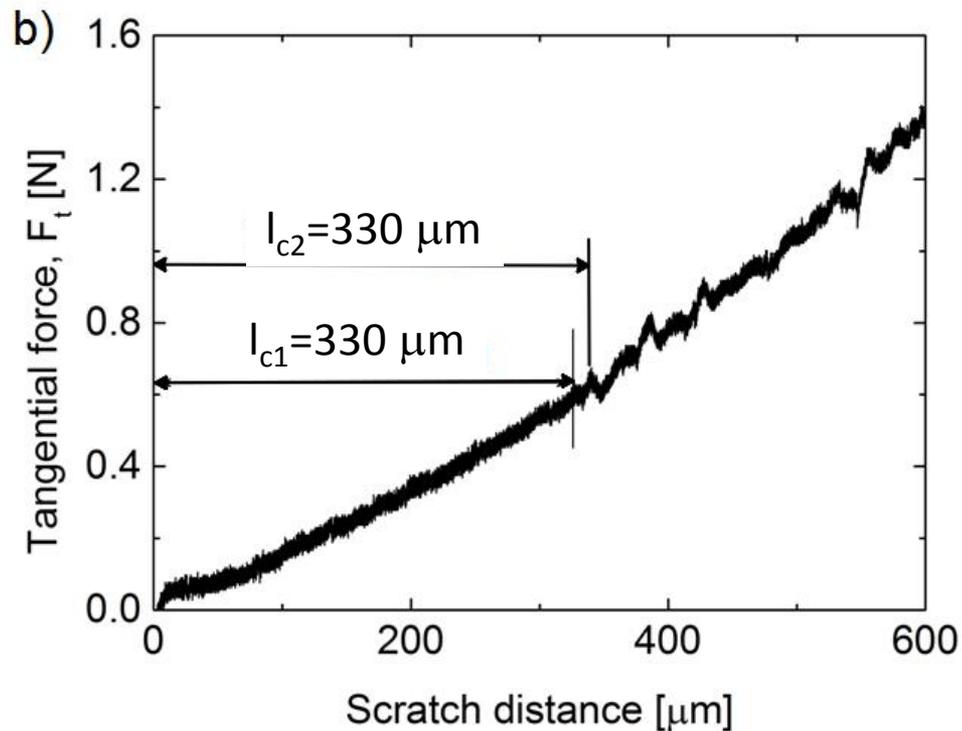
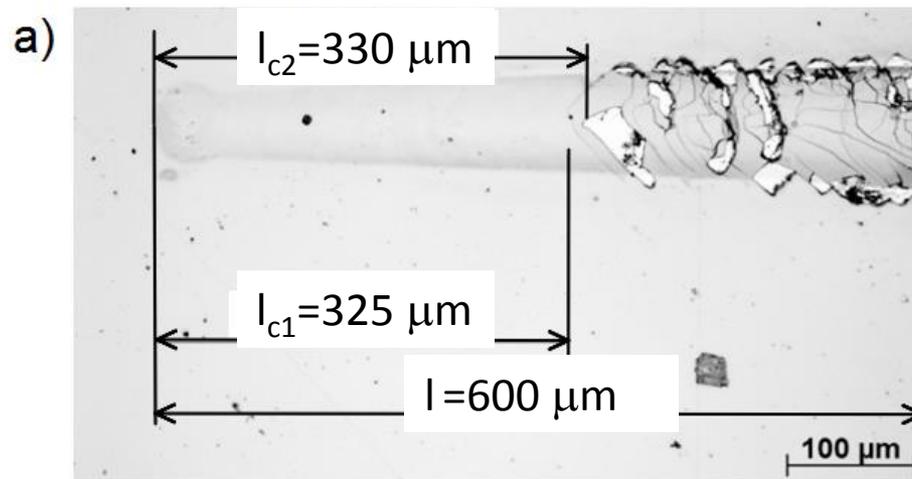


W-C coating fracture toughness

	σ_{\max} [GPa]	G_c [Gpa. μm]	K_{Ic} [MPa. $\text{m}^{1/2}$]	L_{cz} [μm]
$\delta_{c1} = 0,001 \mu\text{m}$	9,535	0,0047675	1,246	0,006828
$\delta_{c2} = 0,008 \mu\text{m}$	9,535	0,03814	3,524	0,054623
$\delta_{c3} = 0,01 \mu\text{m}$	9,535	0,047675	3,939	0,068279

Scratch testing

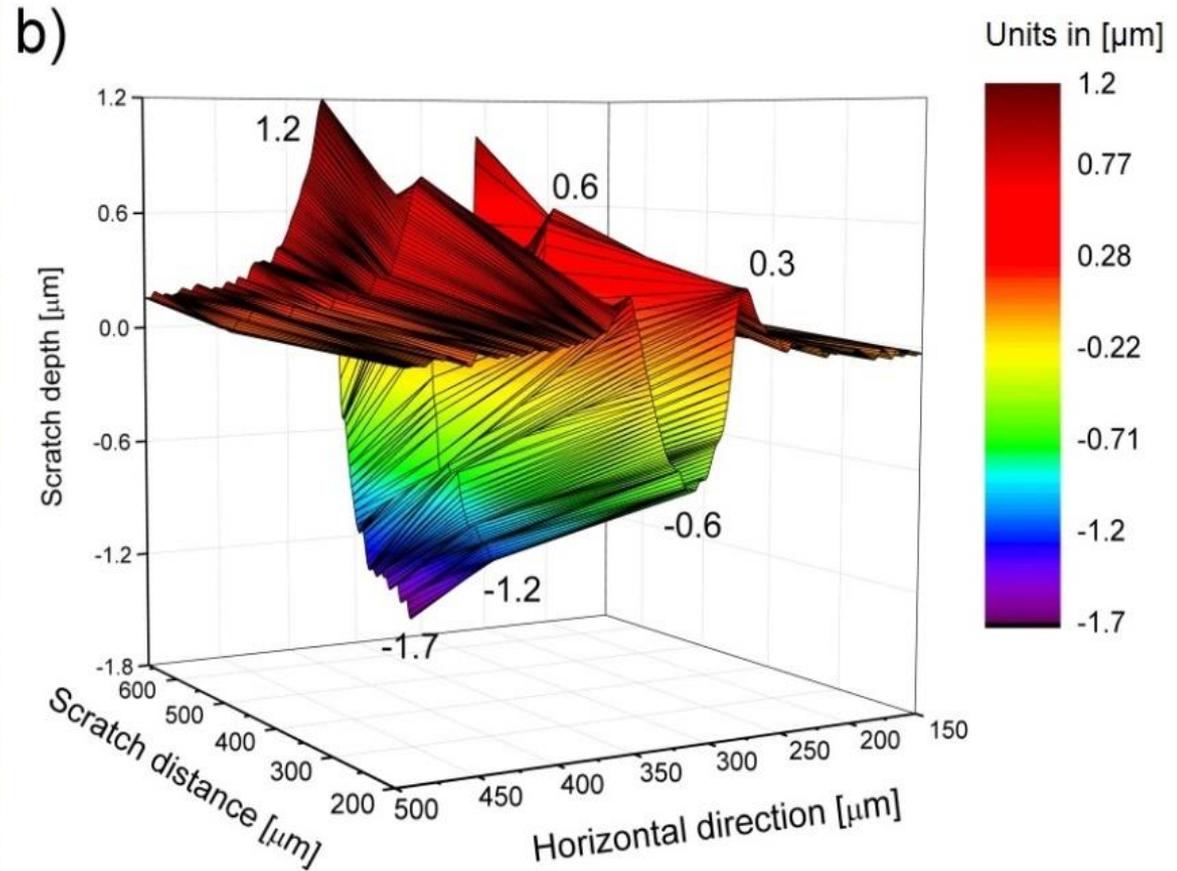
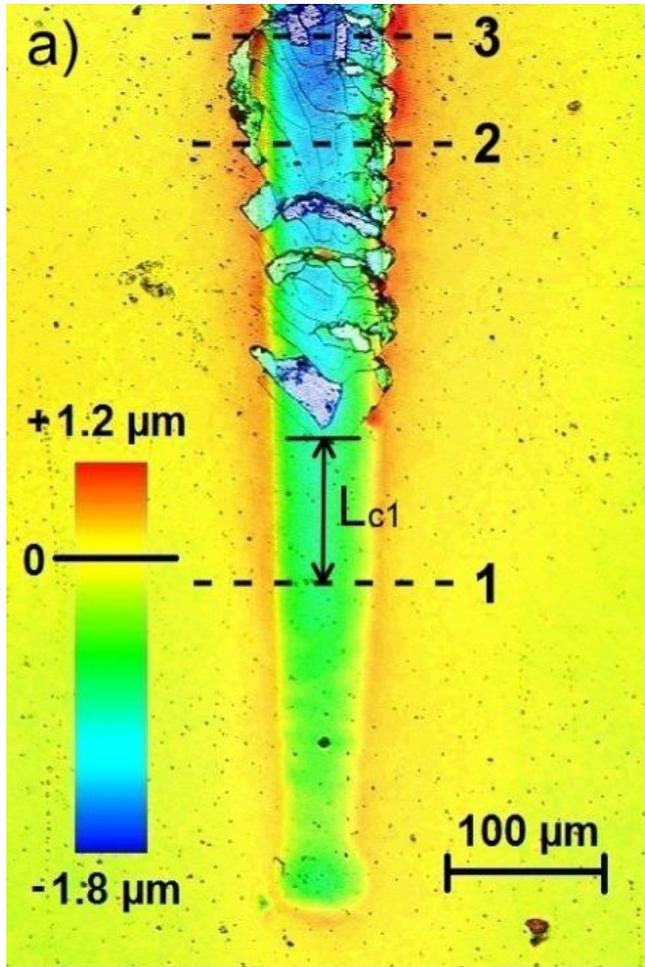
Cohesive cracking during scratch testing



$$L_{c1} = 4.64 \text{ N}$$

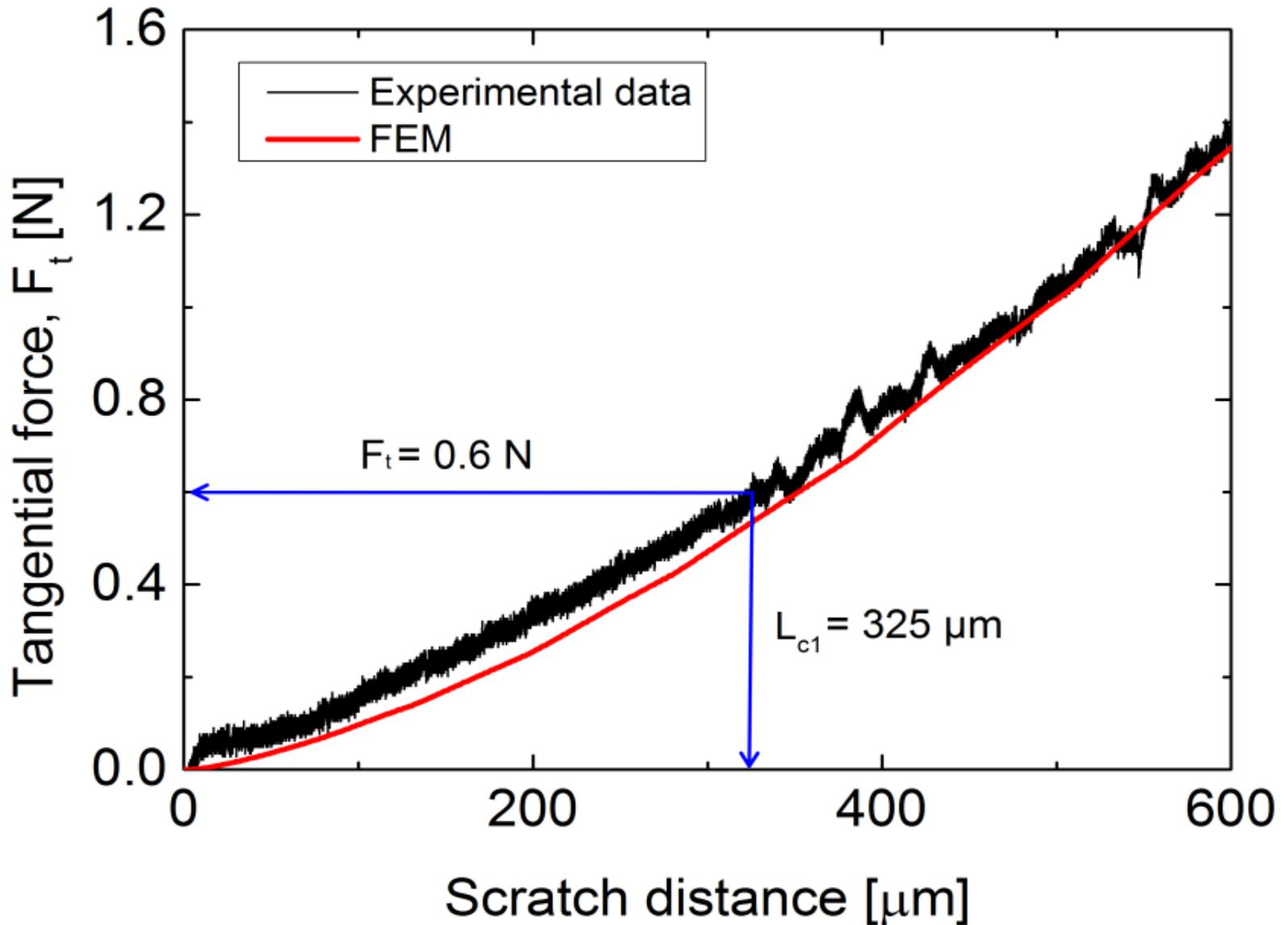
$$L_{c2} = 4.71 \text{ N}$$

Scratch topography

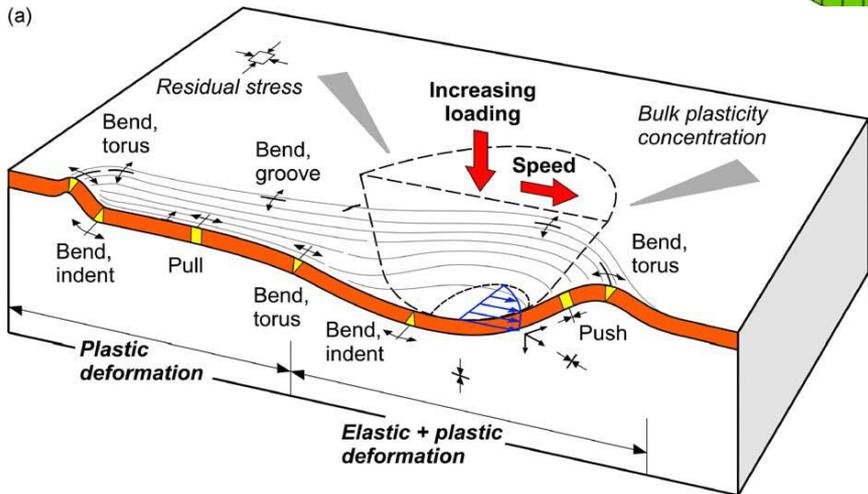
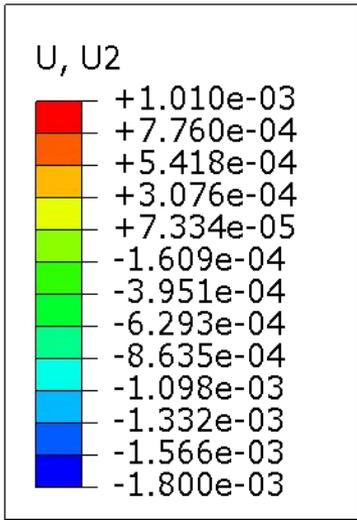
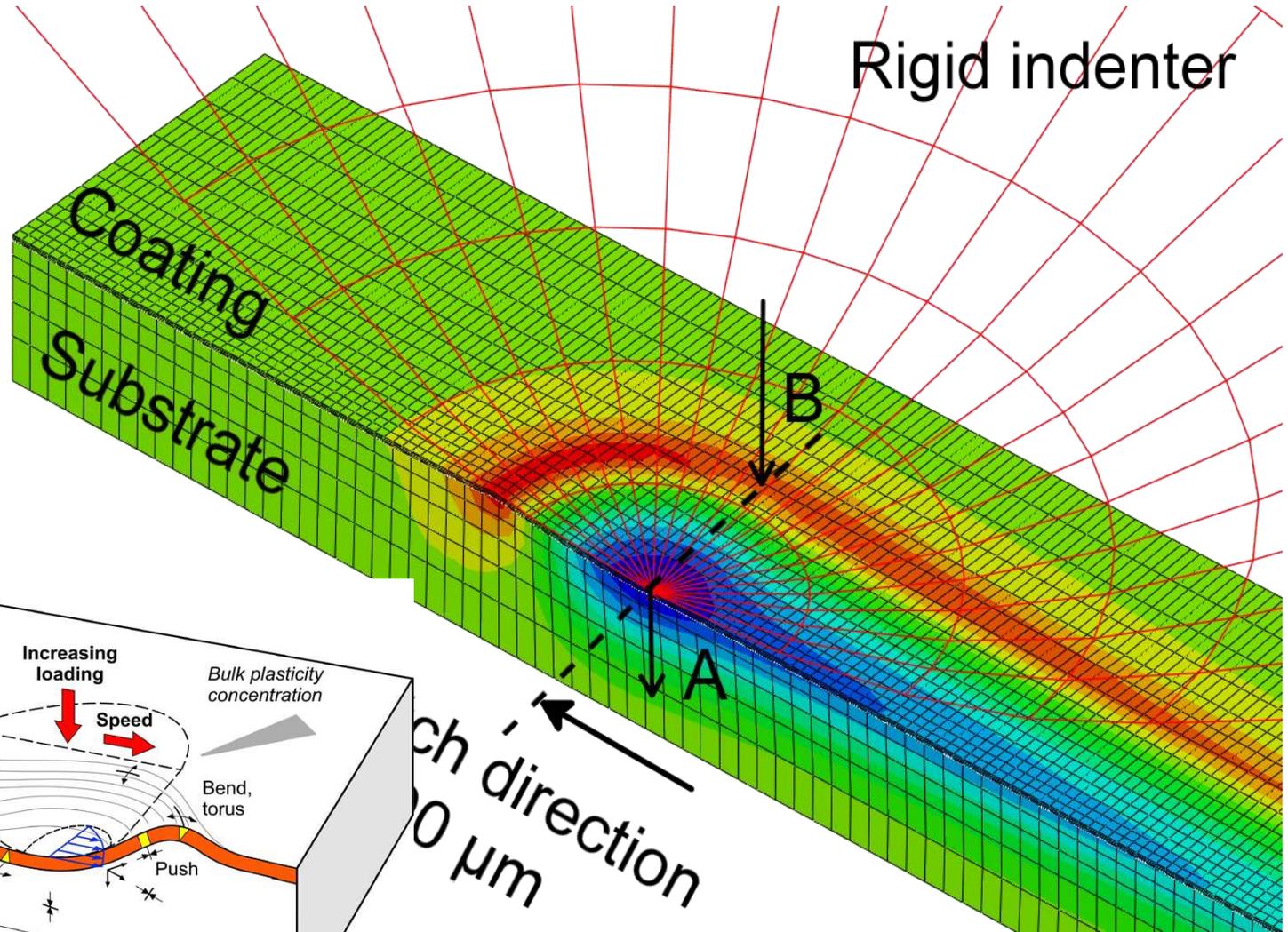


Strong deformation zones along the scratch

Scratch test - Experiment vs. FEM

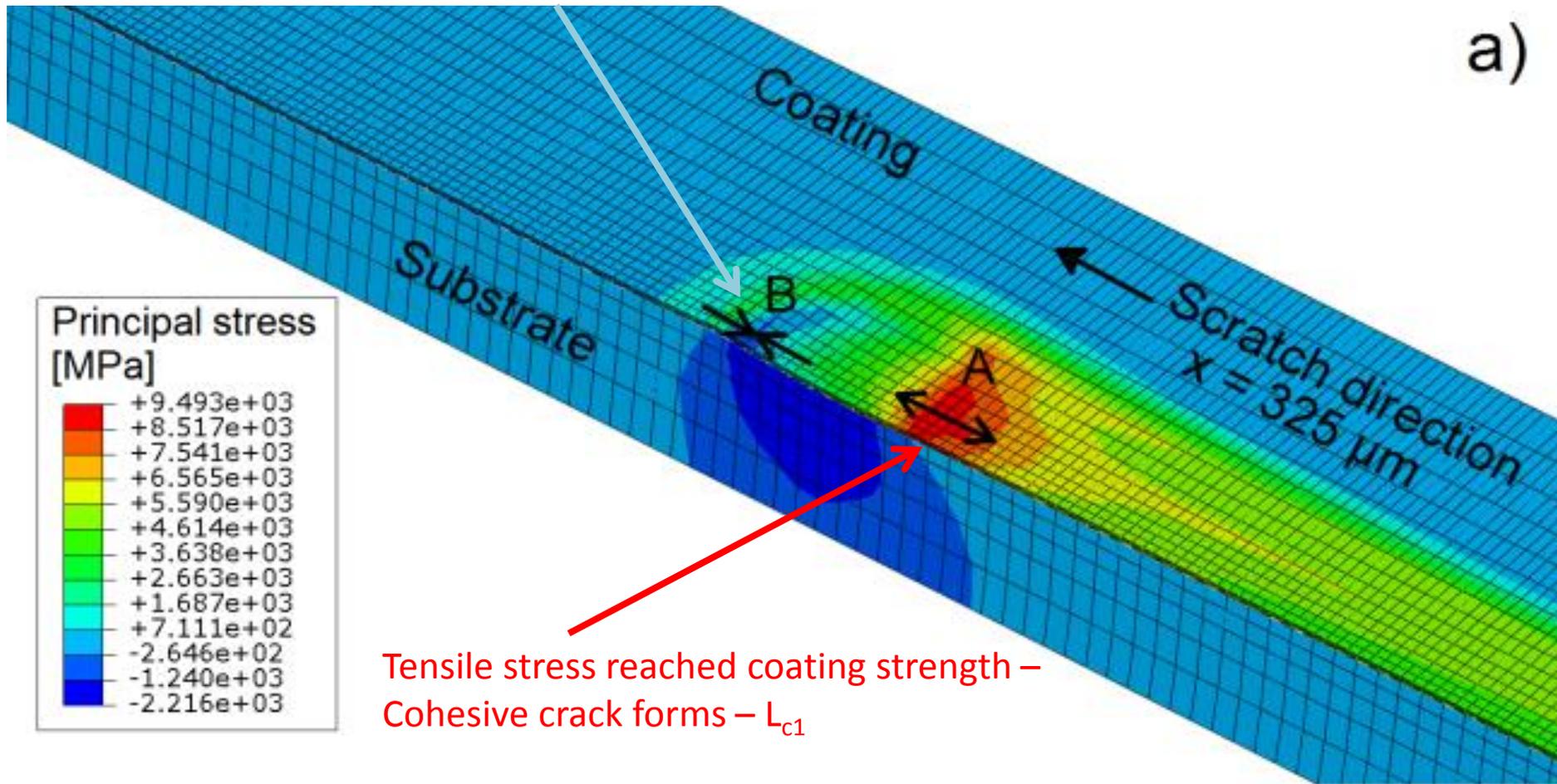


Vertical displacement along the scratch path

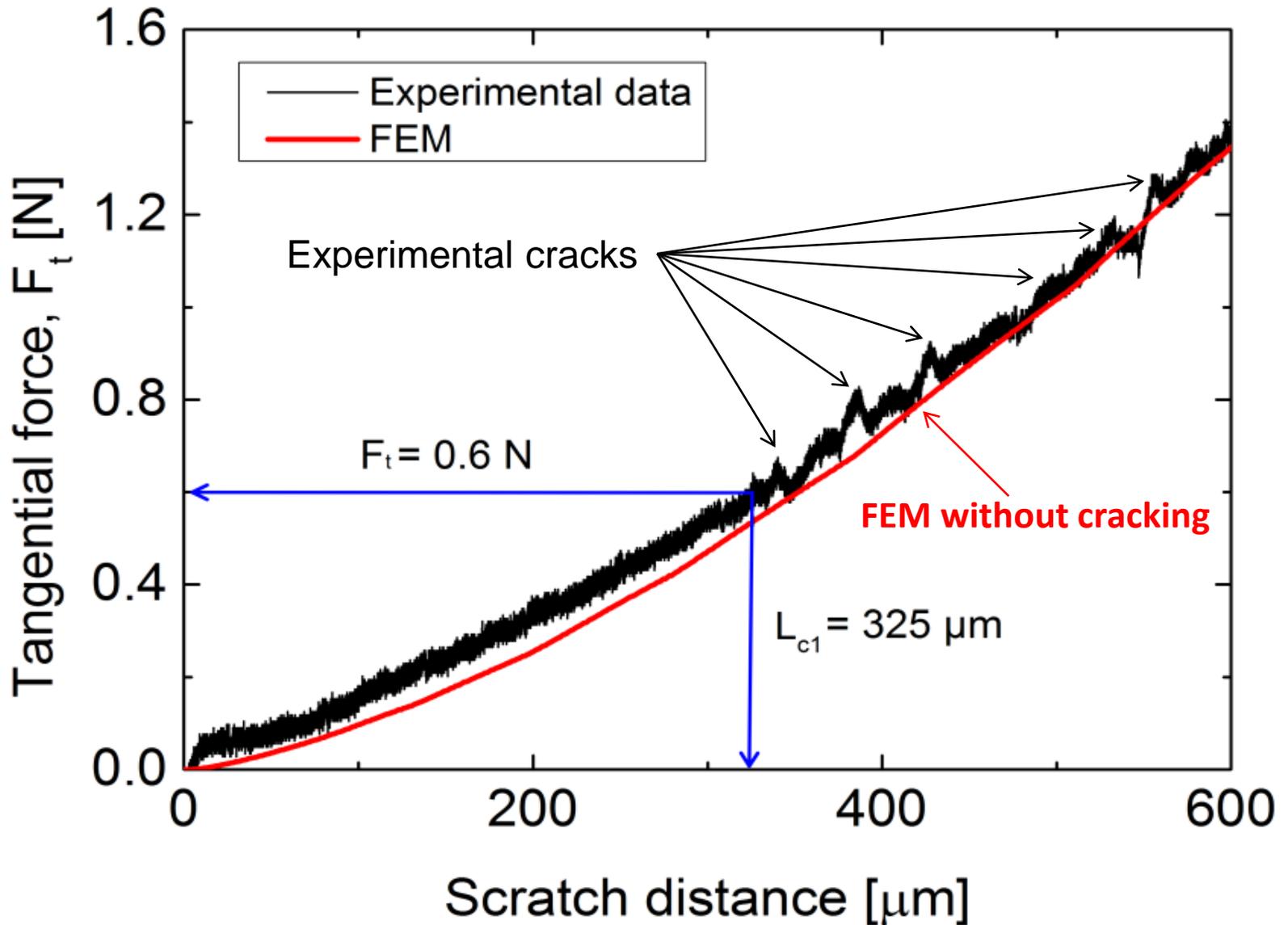


The distribution of principal stresses along the scratch path at L_{c1}

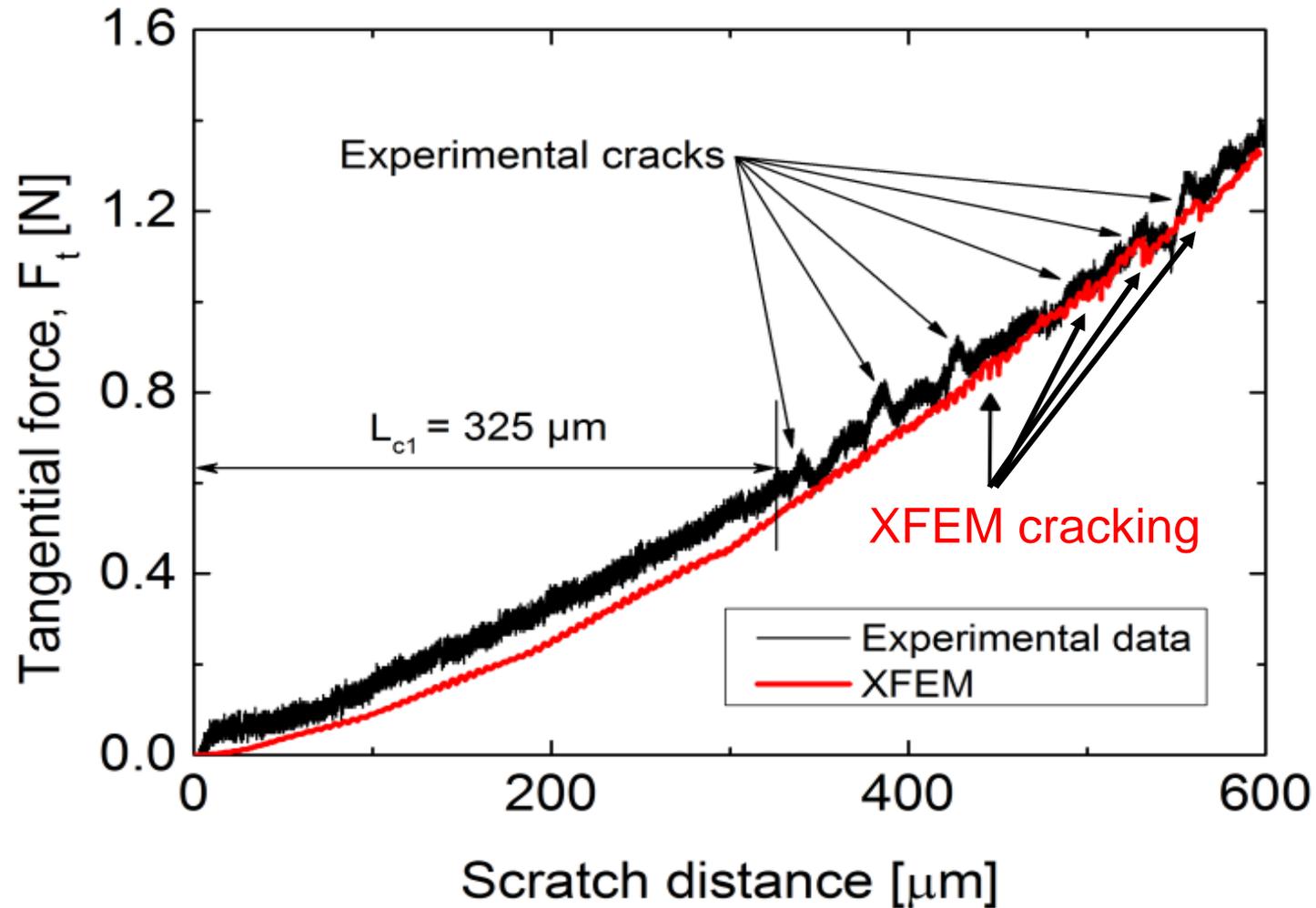
High compressive stresses ...spalling/buckling (CZM??)



Scratch test - Experiment vs. FEM

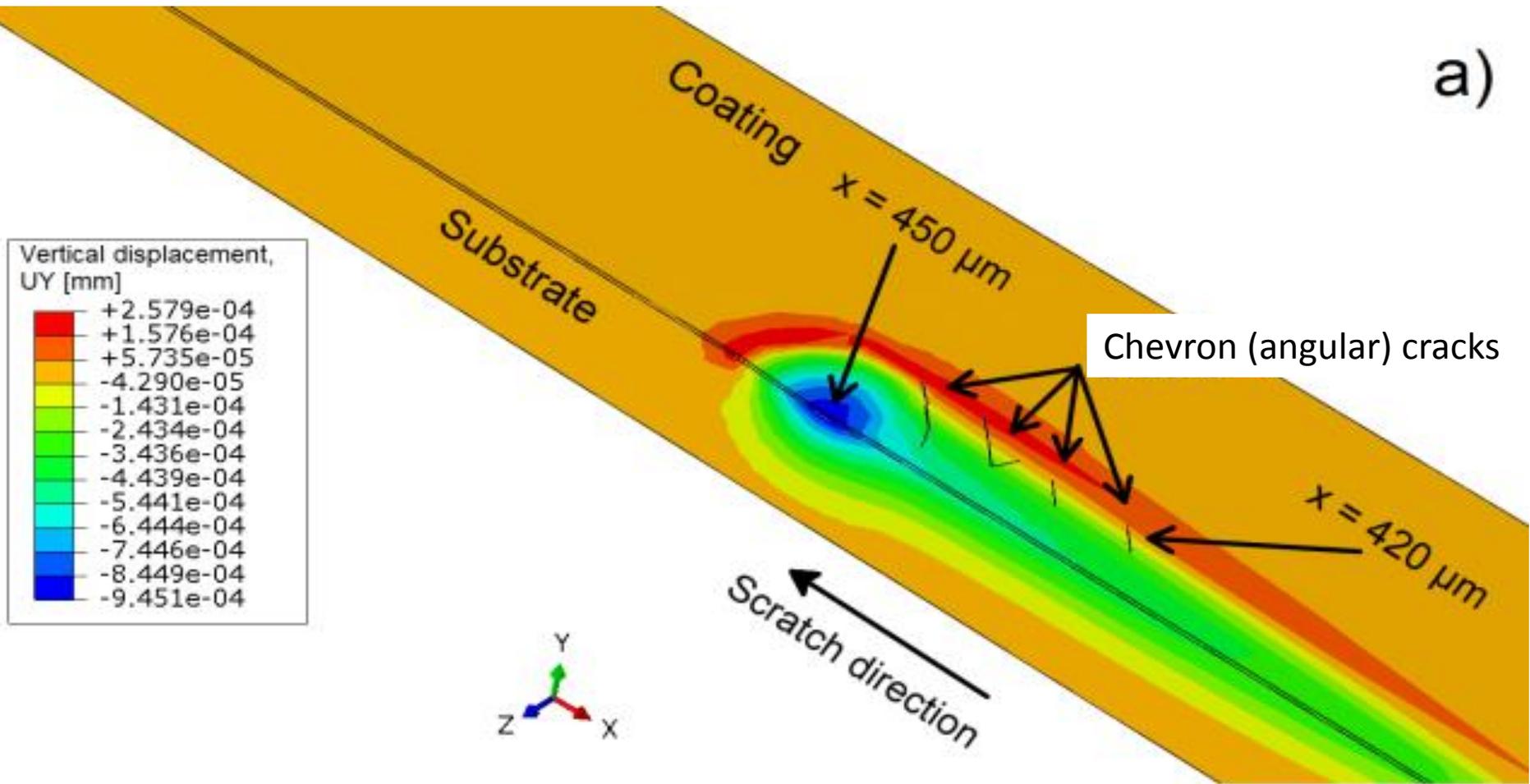


Scratch test - Experiment vs. XFEM

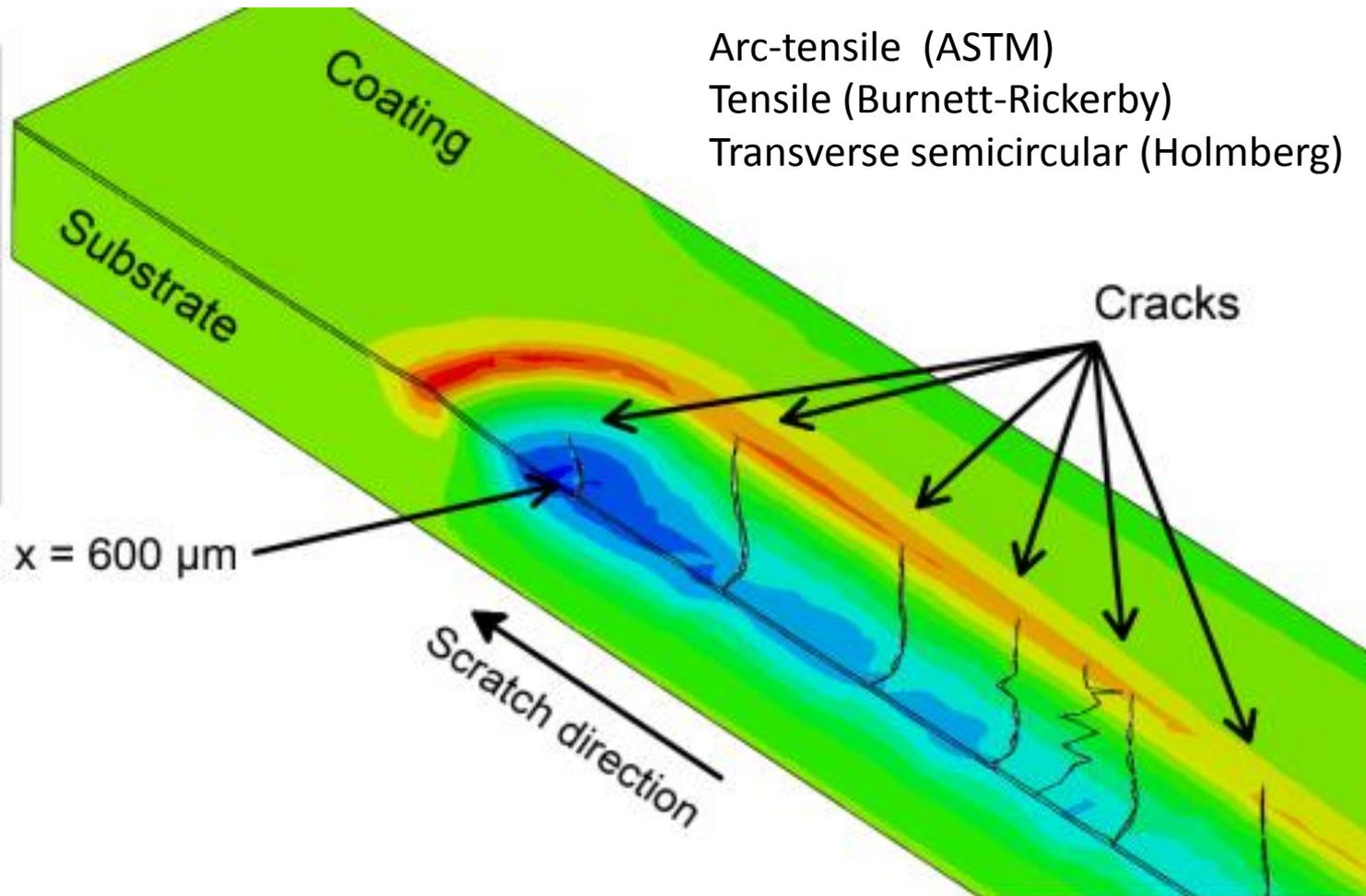
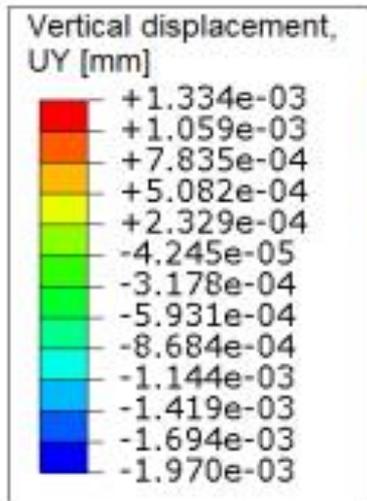


The difference between XFEM and experimentfriction omitted; coating strength variation.

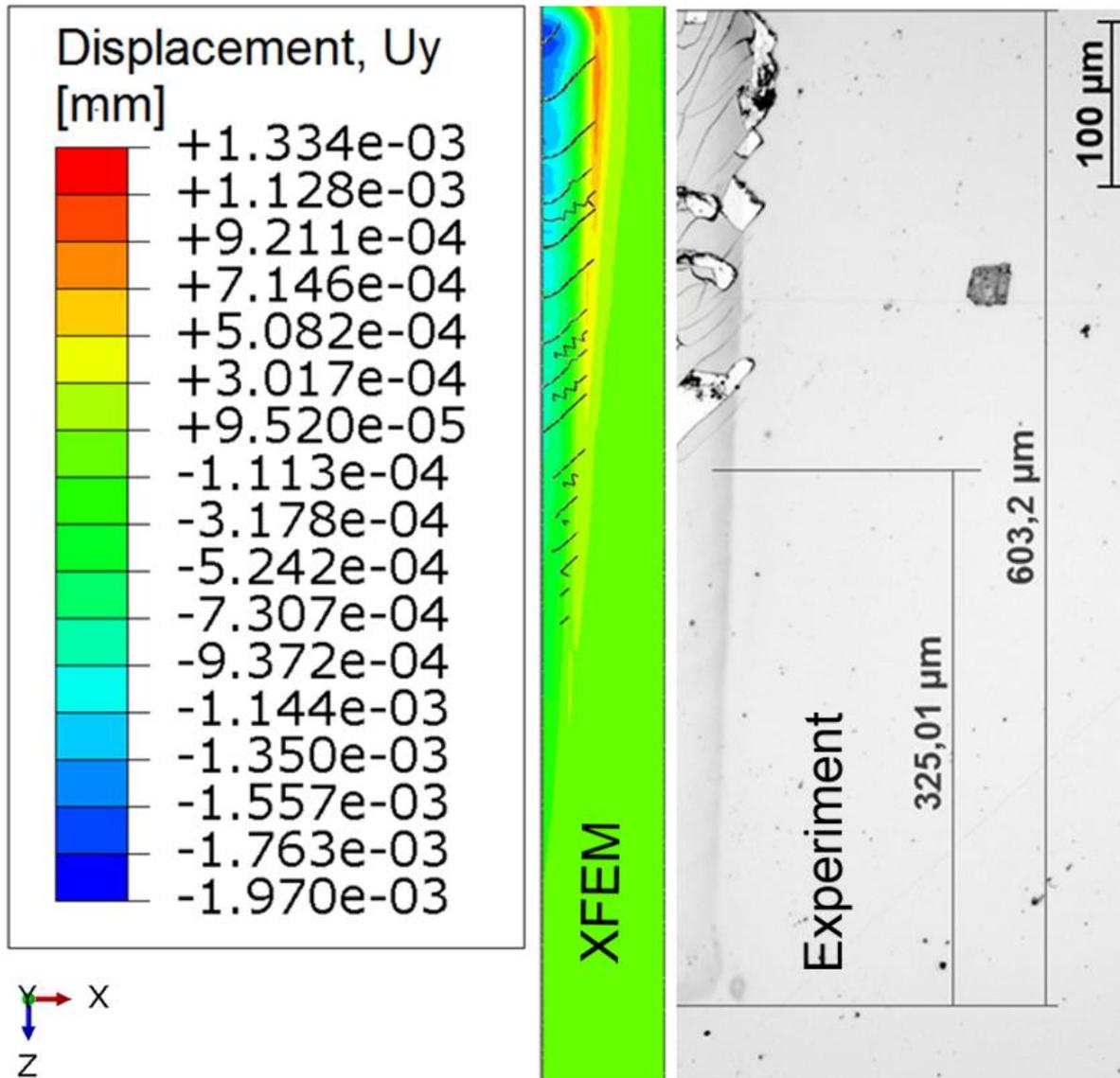
XFEM development of Chevron cracks in W-C coating during scratch test



XFEM evolution of Chevron cracks in W-C coating into arc-tensile cracks during scratch test



Scratch test - Experiment vs. FEM*



Fracture toughness of the W-C coating

$$K_{IC} = \sqrt{\frac{G_c E}{1 - \nu^2}}$$

G_c – fracture energy, strength and δ_c identical to those from nanoindentation

$$K_{1c} = 3.5 \text{ MPa.m}^{1/2}$$

(* Coating strength intentionally reduced to 7 GPa)

Conclusions

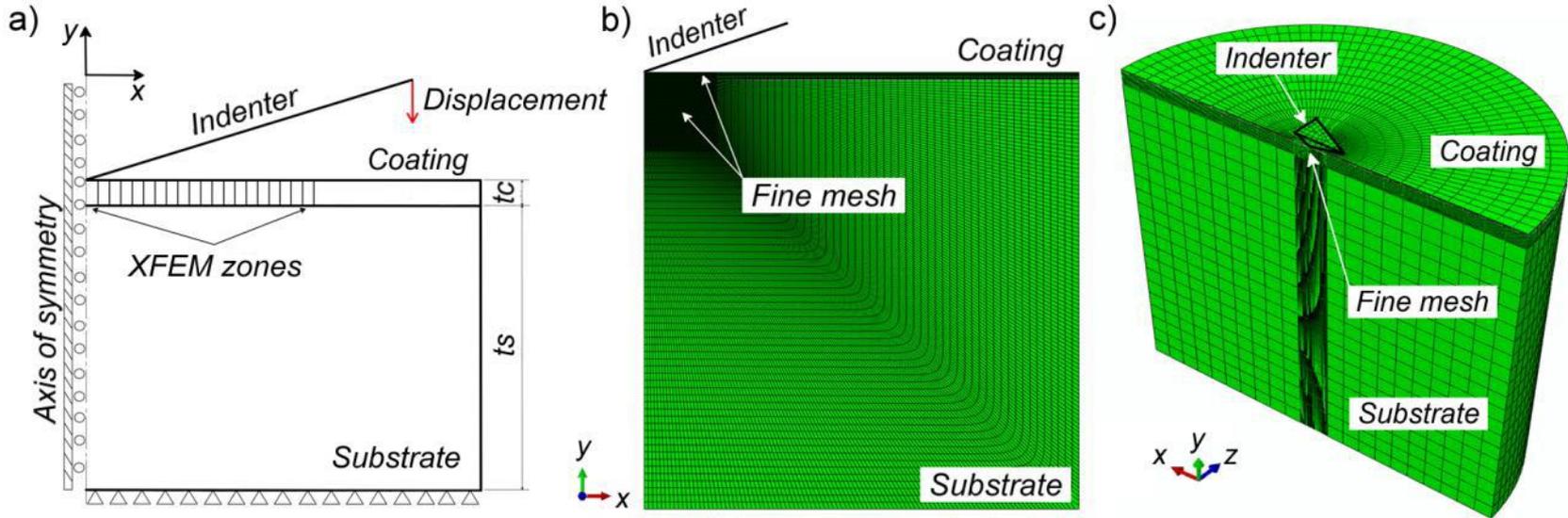
Current work on the W-C coating/steel system successfully modelled:

- Cohesive cracks originating from the top coating surface in the sink-in zone and coating/substrate interface, during nanoindentation.
- Chevron cracks evolved into arc-tensile cracks during scratch testing.
- The formation of these cracks is controlled by the plastic deformation of the substrate.

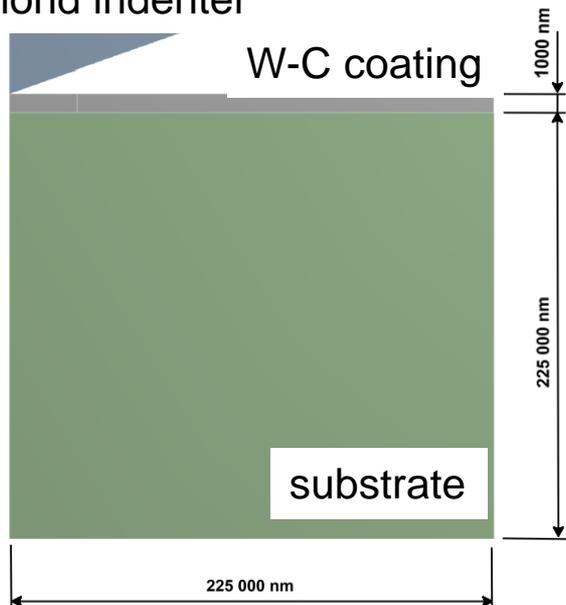
Nanoindentation and/or scratch testing in combination with XFEM can be used for the determination of the fracture toughness of the brittle hard coatings on softer substrates.

Early stages of wear in coated systems can be understand....

FEM parameters (+ xFEM)



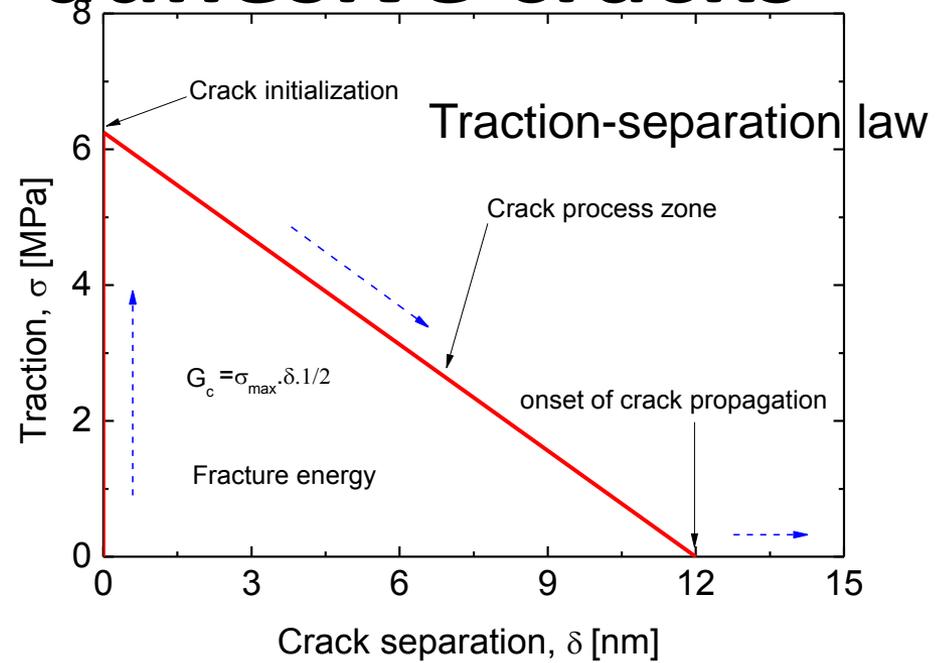
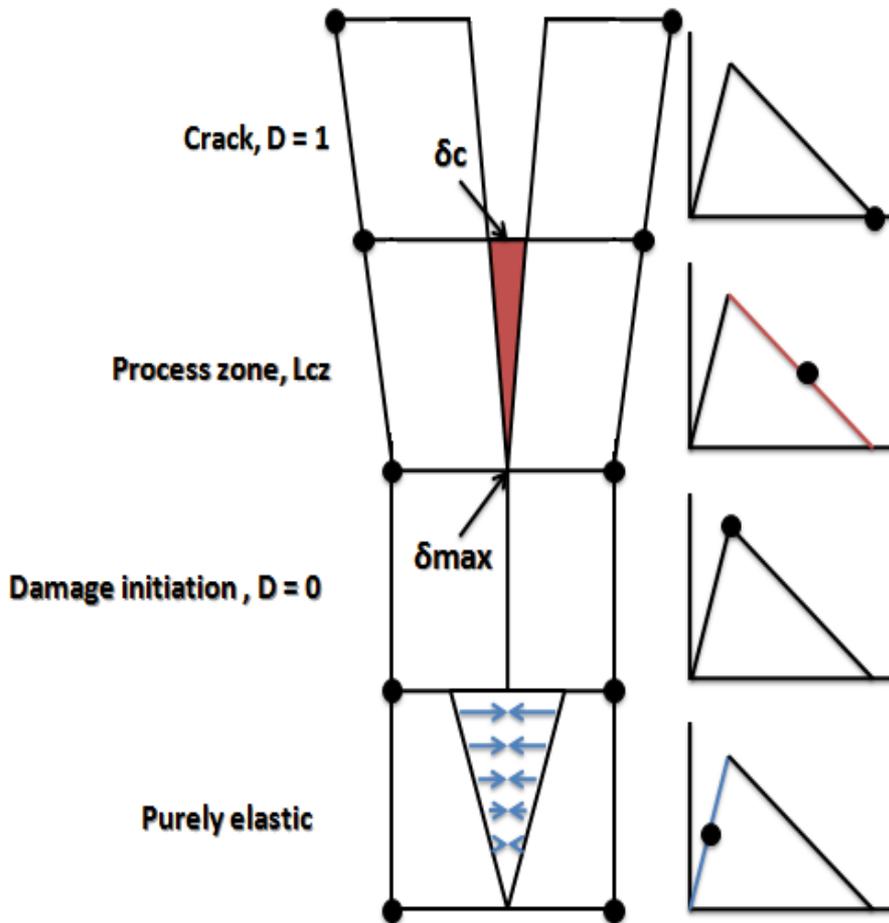
diamond indenter



mesh refinement close to the indent

	Material parameters		
	Young's modulus E [GPa]	Yield stress Rp [GPa]	Poisson's ratio ν
Indenter	1141	-	0,07
W-C coating	250	9	0,28
Steel substrate	210	1,1	0,30

Extended FEM for adhesive cracks



Work of adhesion (or the fracture energy):

$$G_c = \frac{1}{2} \sigma_{max} \delta_c = \frac{K_{IC}^2}{E} (1 - \nu^2)$$

Fracture toughness:

$$K_{IC} = \sqrt{\frac{G_c E}{1 - \nu^2}}$$

Cohesive process zone:

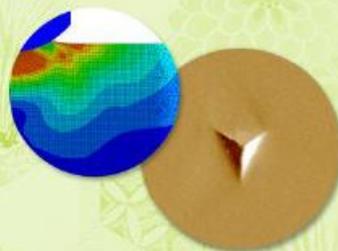
$$L_{cz} \approx 0.2 \frac{E \delta_c}{(1 - \nu^2) \sigma_{max}} = 0.4 \left(\frac{K_{IC}}{\sigma_{max}} \right)^2$$

IIW6 6th International Indentation Workshop

July 1
2018

July 6
2018

Sapporo, Japan



TOYO Corporation
Quest for Precision

Keynote Speakers

- **Prof. Munawar Chaudhri**, University of Cambridge, UK
- **Prof. Karsten Durst**, Technical University Darmstadt, Germany
- **Prof. Frantisek Lofaj**, Slovak Academy of Sciences, Slovakia
- **Prof. Andrew Minor**, University of California, Berkeley, USA
- **Dr. Warren Oliver**, Nanomechanics, Inc., USA

Topics

- Analysis of deformation and fracture during indentation
- Modeling and simulation of mechanical behavior in small scale
- Extracting mechanical properties and multi scale modeling
- Advanced nanomechanical characterization including in-situ probing
- New developments and improvements in mechanical testing
- Instrumented indentation under various environmental conditions
- Application to broad area in inorganic and organic materials

Important Date

Abstract submission due date: Dec. 21. 2017.
Early registration due date: Feb. 28. 2018.



Registration page opened!!

by Feb. 28. 2018. after Mar.1st. 2018

Regular	60,000-	70,000-
Student	40,000-	50,000-
Accompanying	30,000-	40,000-

(JPY : Lunch, Excursion, Banquet all included)

poster award

We will have a **poster session** especially for young scientists and students **with poster award** for them!

Workshop Chair

Prof. Takahito Ohmura,
National Institute for Material Science, Japan

Venue

Hokkaido University Conference Hall
Kita-8 Nishi-5, Kita-ku-Kita, Sapporo,
Hokkaido 060-0808 Japan

Sponsors needed!!