ROMA TRE
UNIVERSITÀ DEGLI STUDI

CENTRO DI RIFERIMENTO INSTM
" Laboratorio di Ingegneria dei Trattamenti Superficiali "

Innovative method for residual stress analysis via FIB relaxation

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EFUG

EFUG2013

30th September 2013, Arcachon, France

MECHANICAL PERFORMANCE OF THIN FILMS

- The knowledge of **elastic properties (E, G, v)** and **residual stress (σ_R)** of thin film is a key issue for the assesment of in-service mechanical behavior of thin films.

Hardness and stiffness (***H, E***)

Indentation resistance

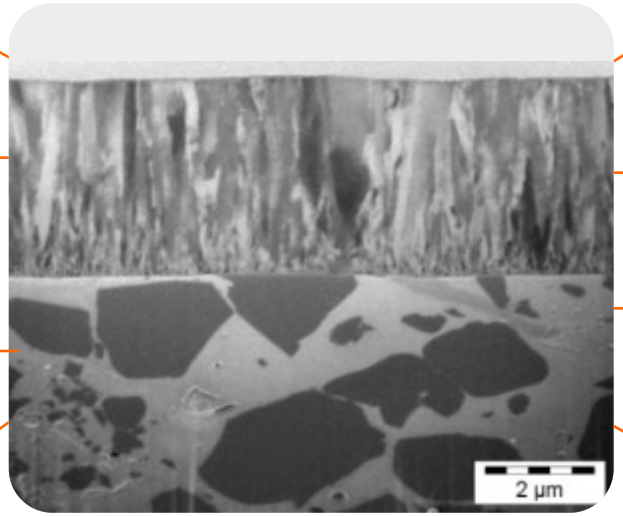
$$\frac{1}{E^*} \approx \frac{(1-\nu^2)}{E}$$

Shear modulus

$$G = \frac{E}{2(1+\nu)}$$

Toughness

$$K_C \propto \left(\frac{E}{H}\right)^n, \boldsymbol{\sigma_R}$$



Load bearing capacity (***H³/E²***)

Adhesion (***E, H, σ_R***)

Wear resistance (***H/E, σ_R***)

Thermal shock resistance

$$R_{TS} = \frac{\lambda R_m}{\alpha E}(1-\nu)$$

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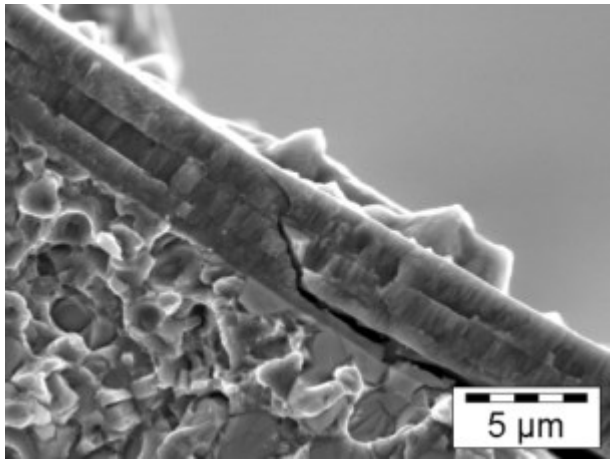
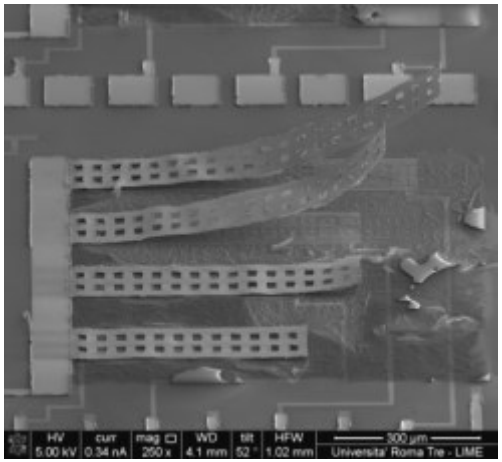
Introduction and Motivation

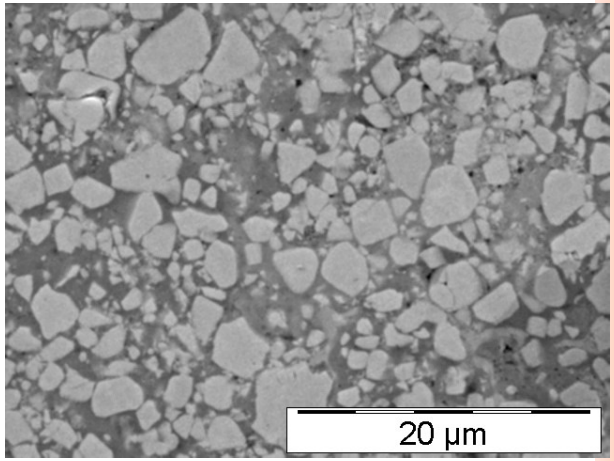
- **Residual stress** (RS) play a crucial role in determining the deformation behaviour and performance of engineered components and materials;
- Many advanced materials and devices require stress measurement with **sub-micrometer** 3D spatial resolution.
- Existing high resolution methods (i.e. using synchrotron XRD) are very expensive, time consuming and not suitable for **amorphous materials**



Thin coatings



MEMS/NEMS devices



Superalloys, multiphase ceramics and composites

Introduction and motivation

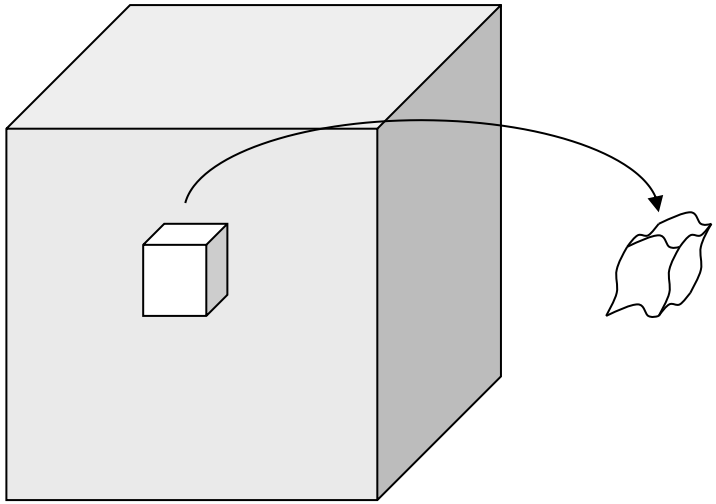
- Research needs
  - Conventional characterisation techniques are not completely exhaustive for describing all microstructural aspects which necessarily determine mechanical behaviour of nanostructured materials and coatings

- Production needs
  - New developed procedures should also be downgradable to in-line quality control processes, or at least cost-saving with respect to traditional high resolution characterisation techniques.

Furthermore, there is still needs of standards and standardization procedures at both levels

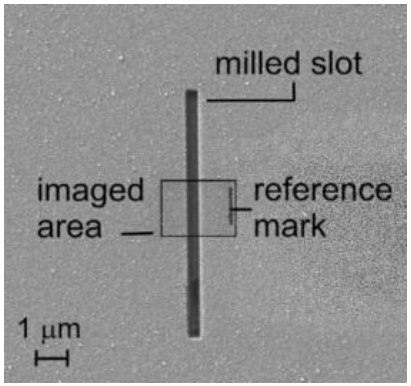
Material removal method for RS analysis

- Ideally, the most relaxed structure is a small volume which is completely removed from its original place;
- This volume will distort from its original shape leading to a completely stress free structure;
- Residual stress can be calculated from the measured strain tensor by usig appropriate constituive models.



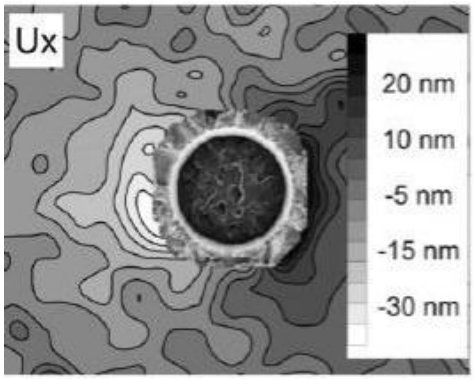
FIB-DIC RS MEASUREMENT TECHNIQUES

- Focused ion beam (**FIB**) techniques have been proposed as a **material removal method** for residual stress analysis at the micron scale.
- Digital image correlation (**DIC**) techniques are used for strain mapping after FIB milling
- **FEM** modeling is usually adopted for residual stress calculation from measured strains.



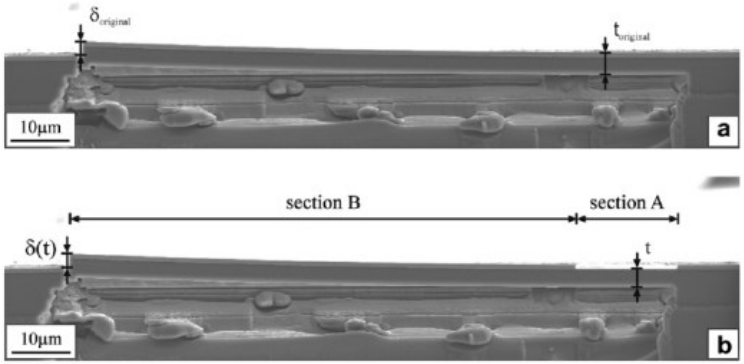
Slot-milling method

Thin Solid Films 443 (1-2), pp. 71-77



Hole drilling method

Nanotechnology 17, Number 20 , 2006



Micro-cantilever method

Acta Materialia 57 (6), 2010

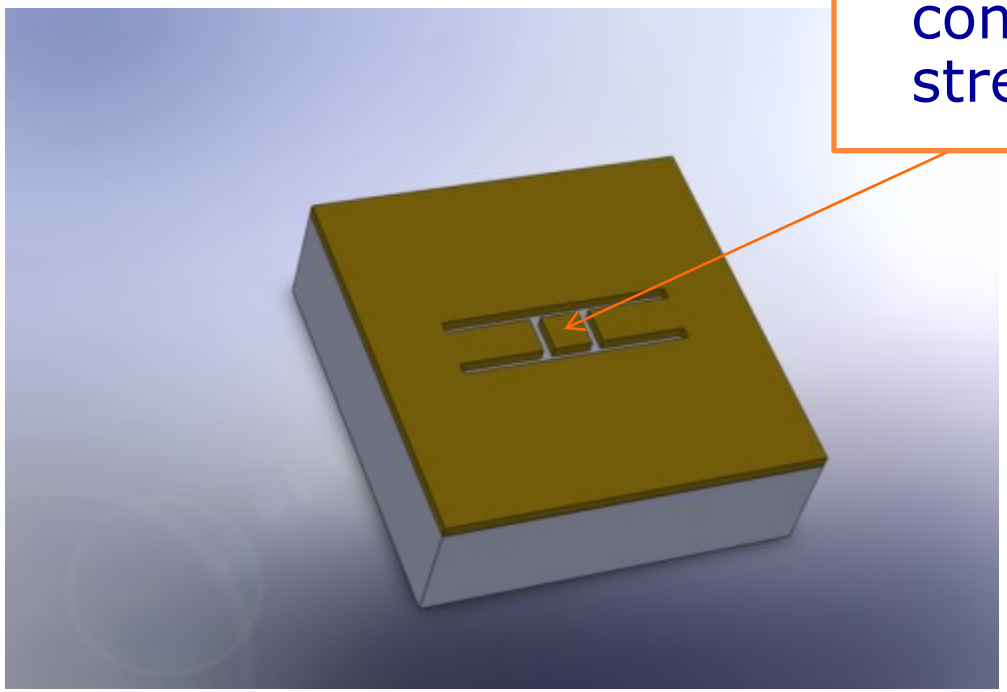
GAP OF KNOWLEDGE

- The adopted geometries are **not** optimized for the high resolution stress analysis on coatings and MEMS structures because
  - The maximum amount of relaxation strain is often located near the **FIB-damaged** area (hole and slot)
  - Adopted geometries involve relaxation **strain gradients** in proximity of the milled trench (hole and slot)
  - The analysis of non-equibiaxial and non-uniform stress fields is not straightforward (slot and cantilever)

Improved milling geometry

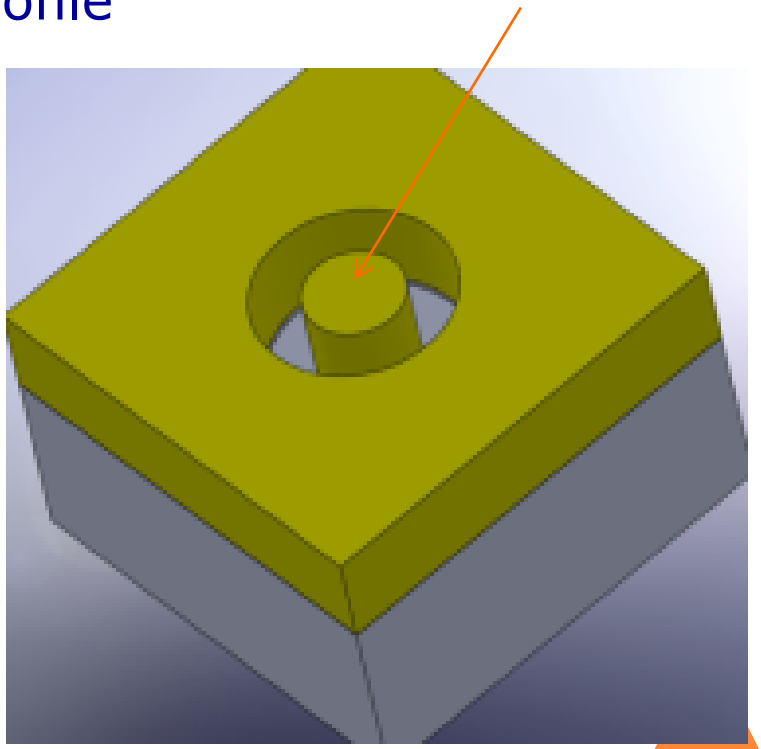
- Starting from the slot geometry, You can think to get a more relaxed structure by milling a stress relieved cube, which is only attached to the substrate;
- This is the geometry which better reproduce a completely stress relieved volume, without detachment from the substrate

- This surface is almost completely stress relieved



OUR IDEA: MICRO-SCALE RING-CORE METHOD

- You can think to get a more relaxed structure by cutting out a **ring** (instead of slots and holes)
  - Higher relaxation strain for a fixed depth, which is also expected to be uniform over the stub's surface
  - Evaluation of all in-plane strain components
  - Measurement of stress depth-profile
  - High spatial resolution



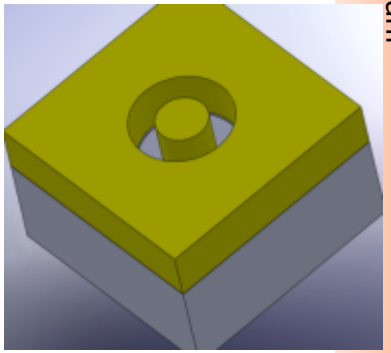
A. M. Korsunsky, M. Sebastiani, E. Bemporad

Materials Letters, 2009

EXPERIMENTAL IMPLEMENTATION (*IμRCM*)

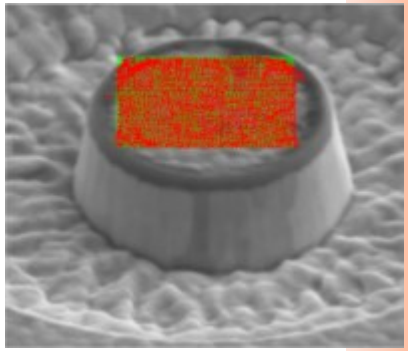
Focused Ion Beam controlled material removal, by the ring-core milling geometry

- Optimized stress relief
- Uniform relaxation strain
- Possibility of biaxial stress evaluation



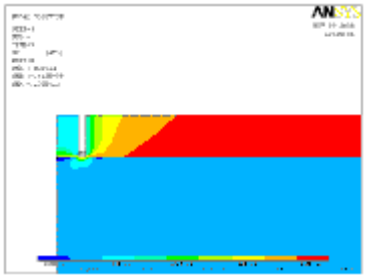
Digital Image Correlation for strain measurement

- Evaluation of the complete X and Y relaxation strain profile as a function of the milling depth



Residual stress calculation

- **Analytical** calculation for average stress analysis
- **FEM** for through-thickness stress gradient analysis;

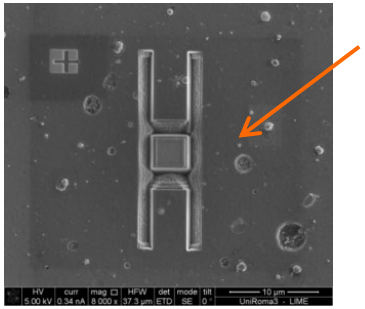


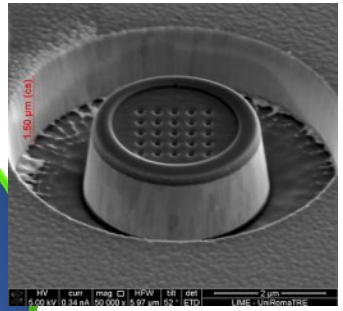
Statement of purpose

- The main objective of our work was to develop and optimize a FIB-DIC methodology for residual stress through thickness profiling in thin coatings and small scale structures, based on the micro-scale **ring-core** geometry.

METHOD DEVELOPMENT

IμRCM



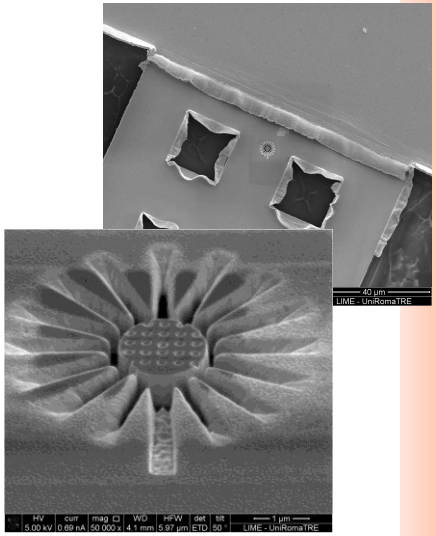


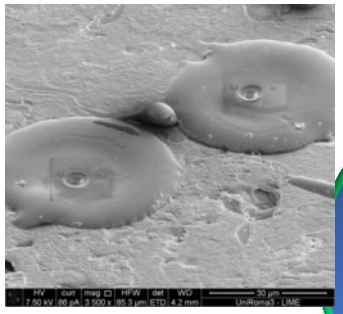
Average stress in **thin coatings**

Analysis of the Poisson's ratio

IμRCM

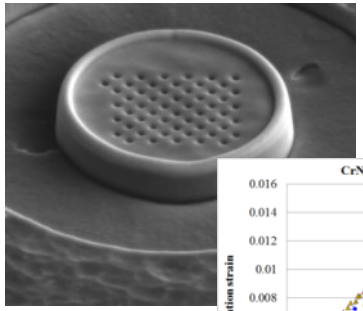
Stress in **MEMS**

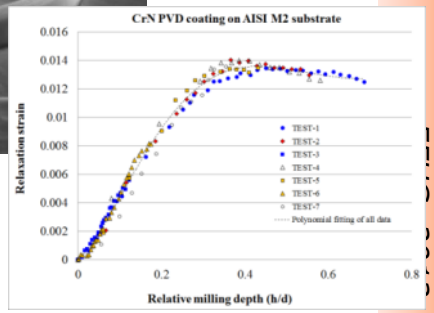




Stress in **amorphous** materials

Stress **gradients** (depth profiling)





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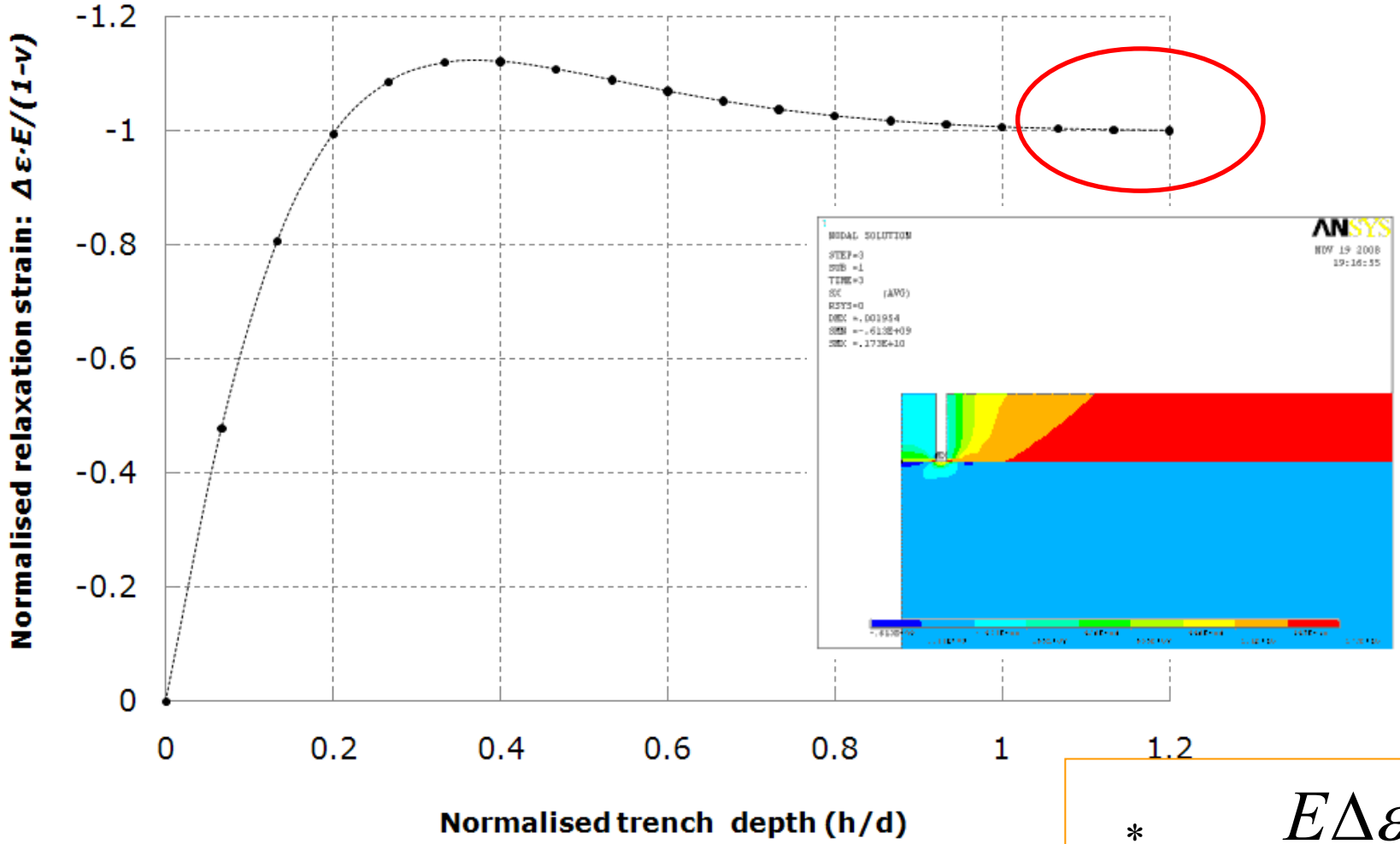
- FEM modelling of strain relief from an annular trench

Korsunsky, A.M., Sebastiani, M., Bemporad, (2010) **Surface and Coatings Technology**, 205 (7), pp. 2393-2403

FE Simulated strain relief curve ($d=t$)



$$\sigma^* = -\frac{E\Delta\varepsilon^*}{(1-\nu)}$$

Korsunsky, A.M., Sebastiani, M., Bemporad, (2010) **Surface and Coatings Technology**, 205 (7), pp. 2393-2403

FEM MODELING: AVERAGE STRESS MEASUREMENT

- FEM modeling predict a complete and uniform stress relief as the trench depth approaches the diameter of the remaining stub
  - **If the objective is to know the average stress in the coating, so strain should be acquired for h/d>1**
  - **NO need of FEM calculations to calculate the average stress in the coating**

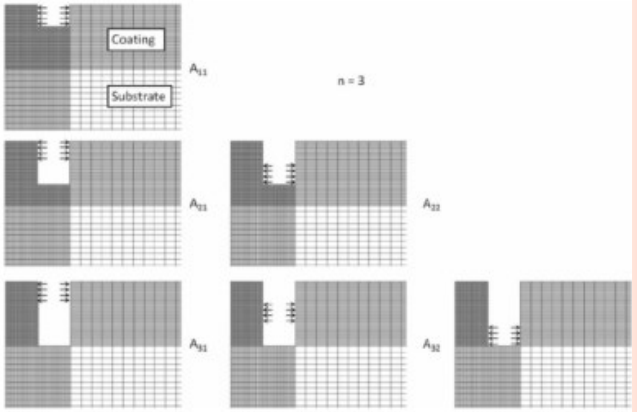
FEM - Calculation of stress depth profile

- Stress profiles can be obtained by the integral Method (Schajer)
  - The measured strain relief ***ε(h)*** is the integral of the infinitesimal strain relief components due to the removal of tractions at all depths in the range ***0 ≤ H ≤ h***

$$\epsilon(h) = \frac{1+\nu}{E}\int_0^h \hat{A}(H,h)\sigma(H)dH \quad 0 \le H \le h$$

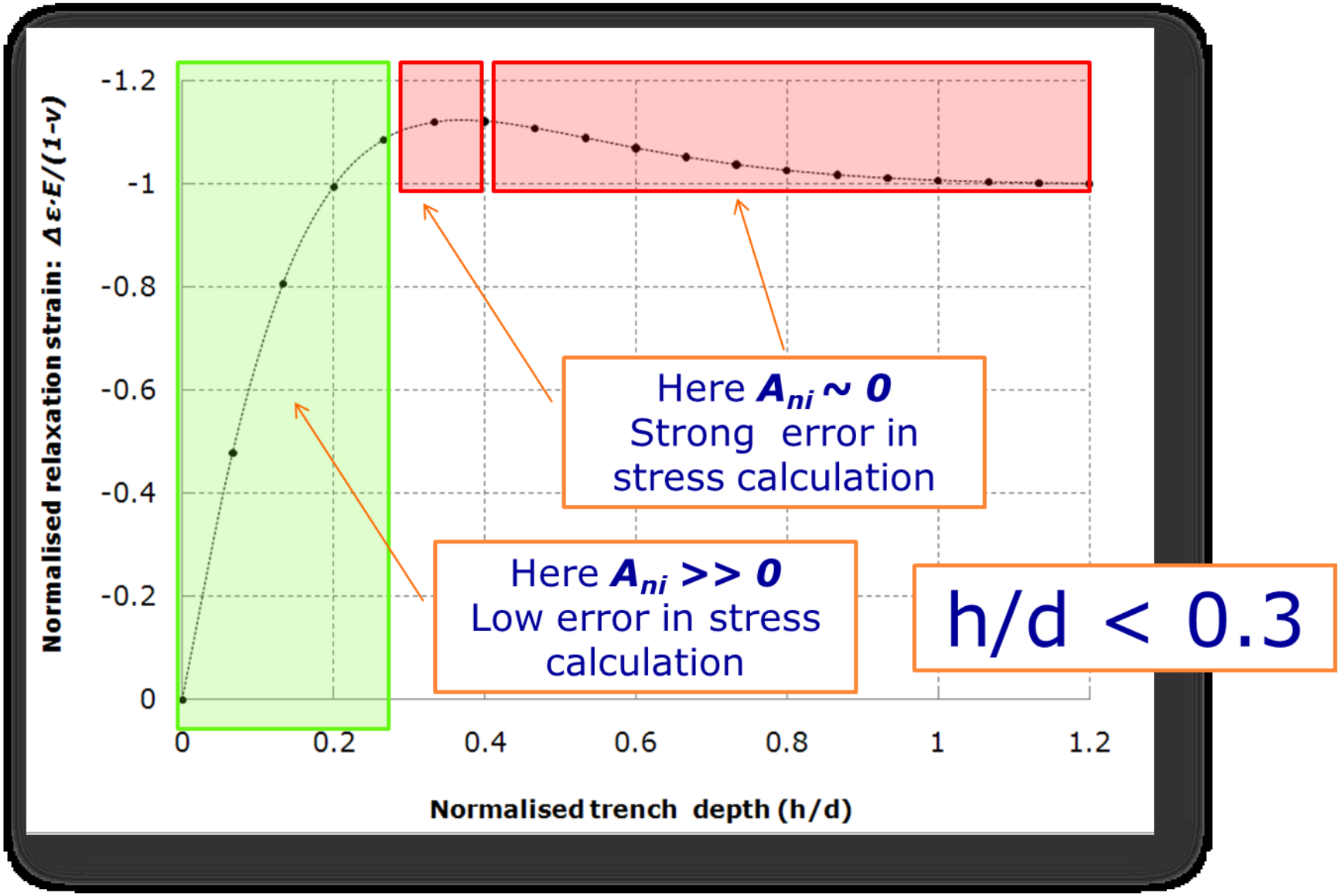
Schajer GS, J. Eng. Mater. Technol. 1988;110:33

$$\sum_{j=1}^{j=i} \bar{a}_{ij}\sigma_j = \frac{E}{1+\nu}\epsilon_i \quad 1 \le j \le i \le n$$



- Minimum error in stress calculation if ***a_{nn} = const***
- This is achieved by increasing the depth size of the calculation steps at deeper depths and limiting max calculation depth to ***h/d < 0.3***

Stress profiling: what the optimal milling depth?

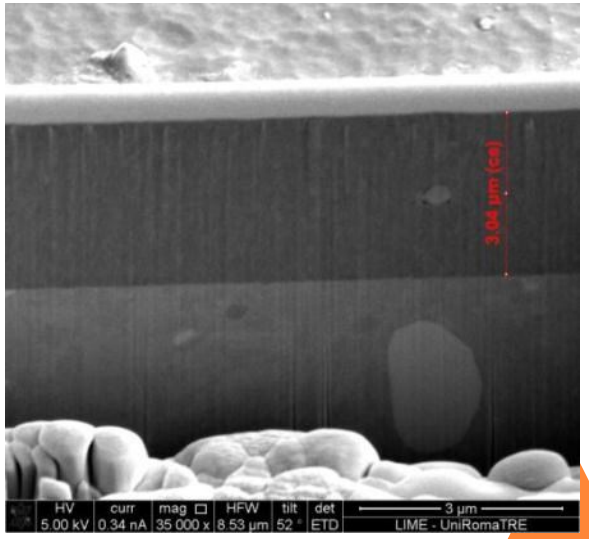


FEM MODELING: STRESS GRADIENT ANALYSIS

- IF the objective is stress profiling, then deformation data should be acquired at a sequence of trench depth increments and the integral method applied for ***h/d < 0.3***.

APPLICATION TO THIN FILMS FOR **AVERAGE STRESS** AND **STRESS GRADIENT** ANALYSIS IN THIN FILMS

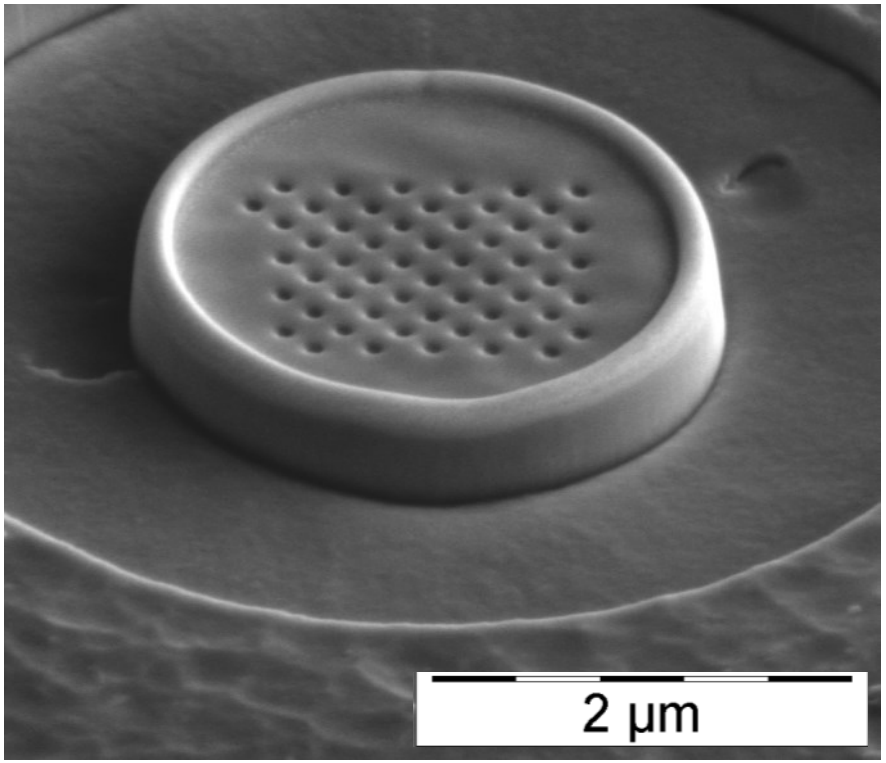
M. Sebastiani, C. Eberl, E. Bemporad, G. M. Pharr
Materials Science and Engineering A, 2011



3 µm CAE-PVD CrN on steel AISI M2

Micro-scale ring-core FIB milling

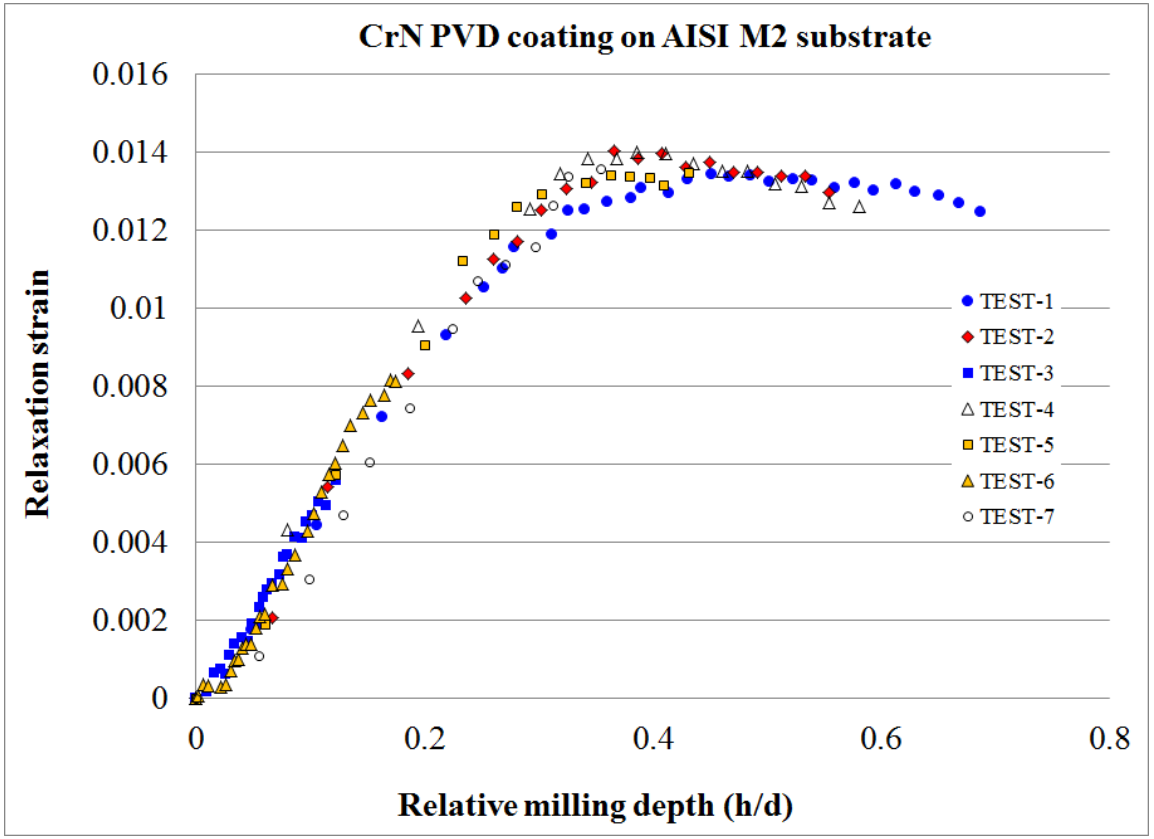
- Procedure for relaxation strain measurement:



- Strong efforts in terms of script development to cope with artifacts coming from FIBing:
- Correction of ion/electron drift
- Reduction of re-deposition due to an optimized milling strategy
- **Semi-automated procedure**

M. Sebastiani, C. Eberl, E. Bemporad, G. M. Pharr
Materials Science and Engineering A, 2011

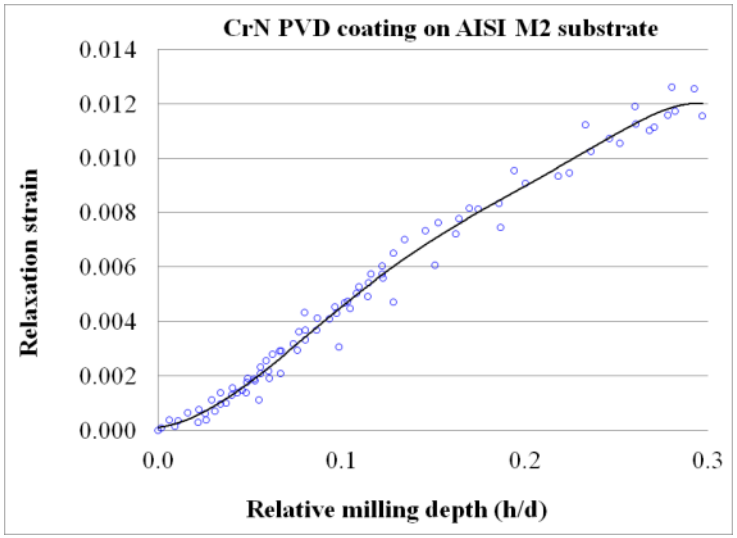
Relaxation strain-x profile: CAE-PVD CrN on tool steel

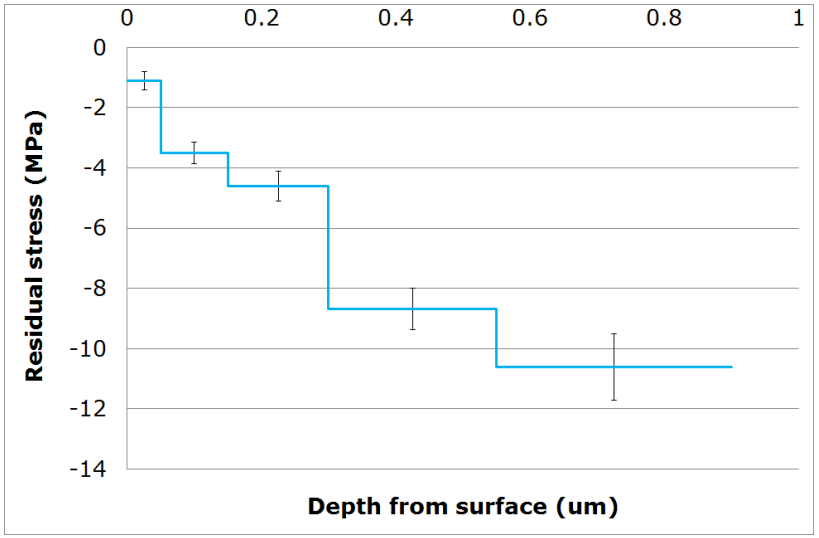


- **Good reprodicibility of relaxation strain data**

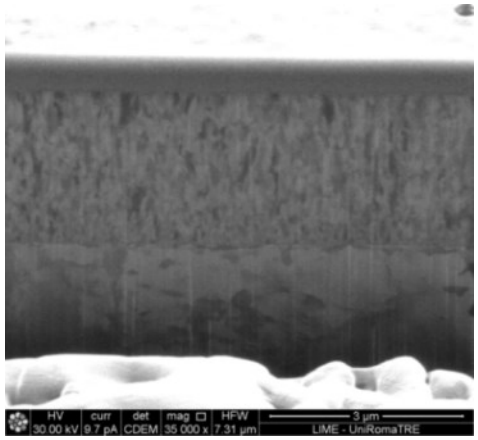
DEPTH PROFILING OF RESIDUAL STRESS: CrN

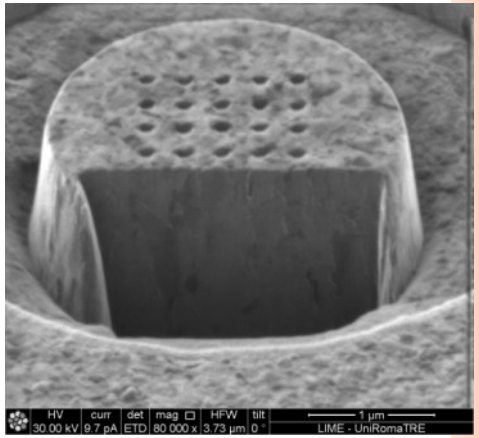
- Strategy for stress gradient calculation:
  - fitting with a polynomial expression of the strain data for ***h/d<0.3***
  - Calculation of the residual stress **DEPTH PROFILE** by FEM analysis (Integral Method)



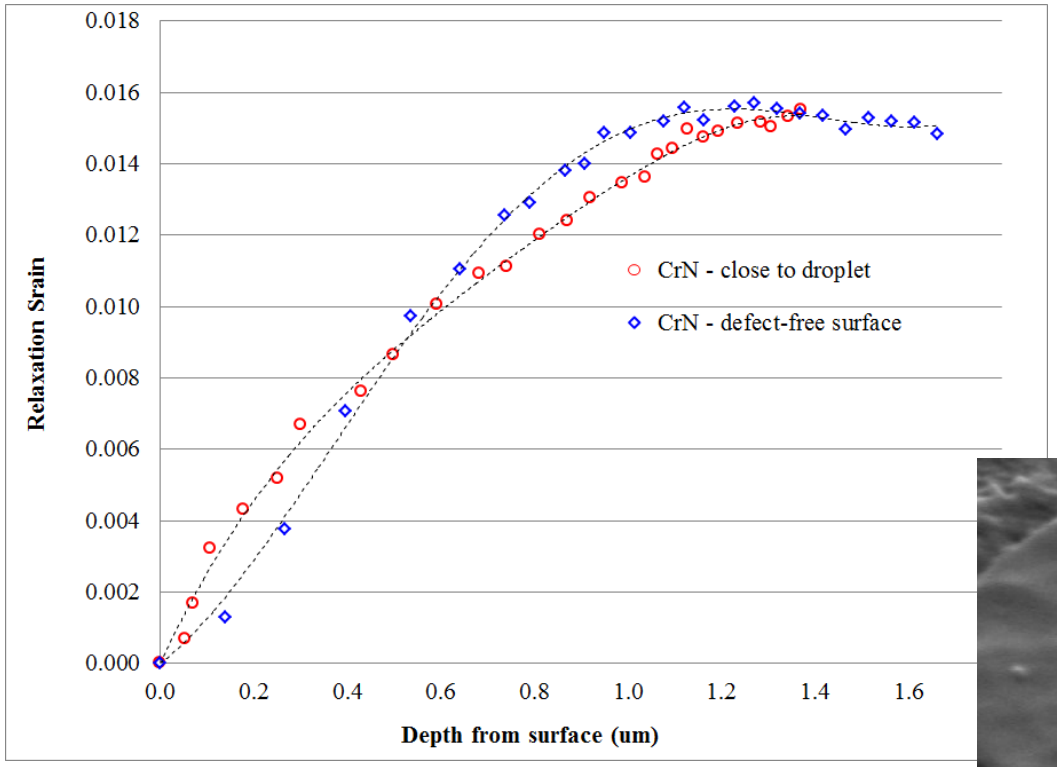


- **Significant gradient of stress is observed from surface to interface on this CrN coating**

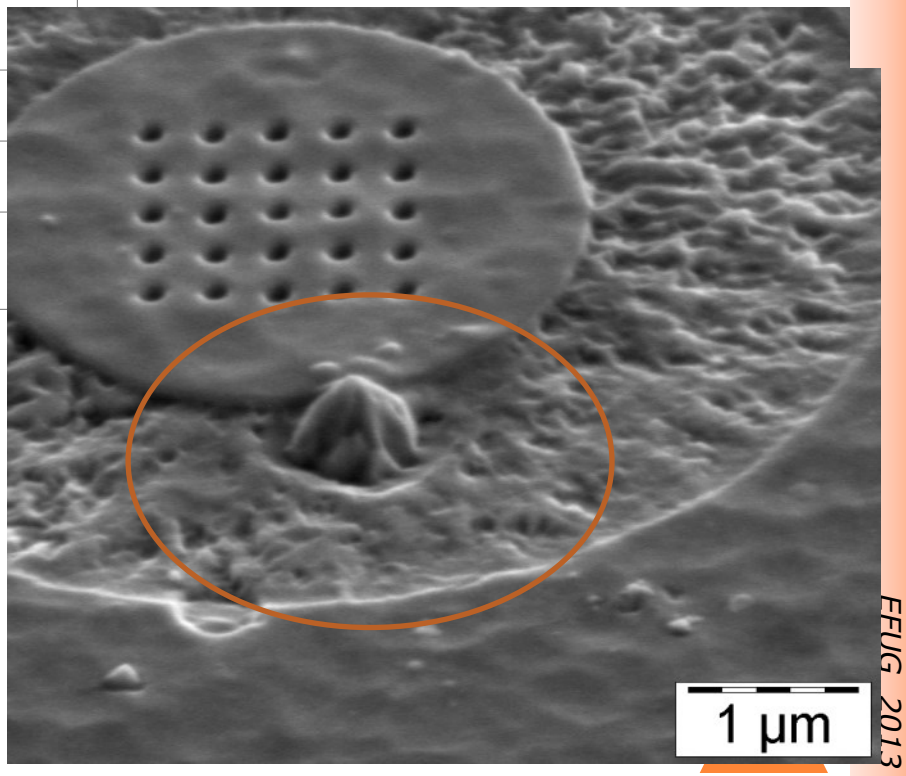




Local residual stress field in thin films

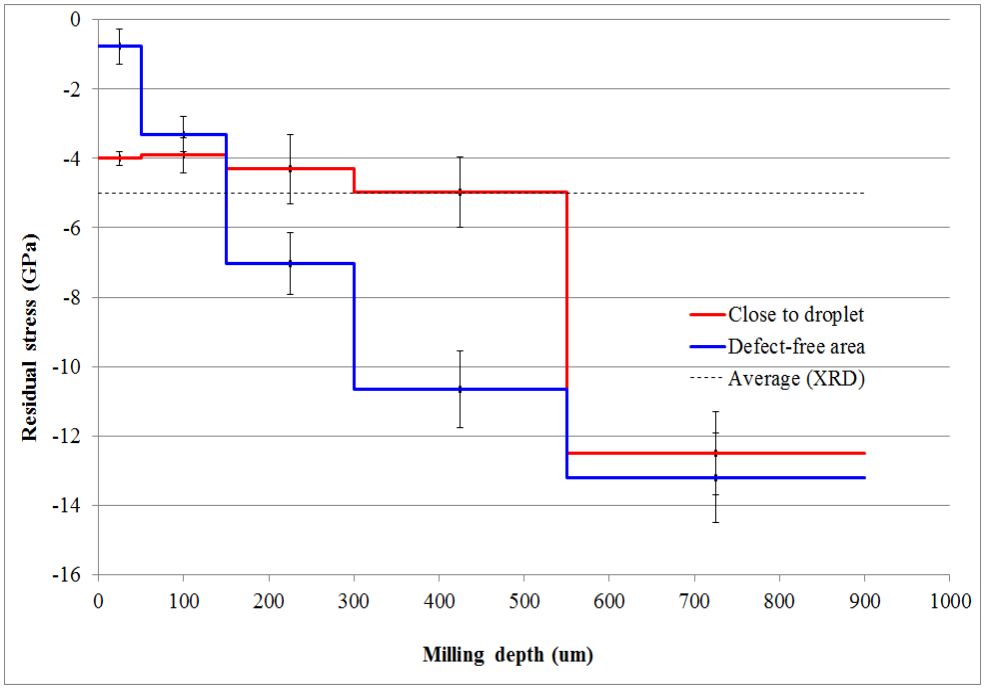


The results consistently show a significant modification of the LOCAL residual stress field induced by the droplet



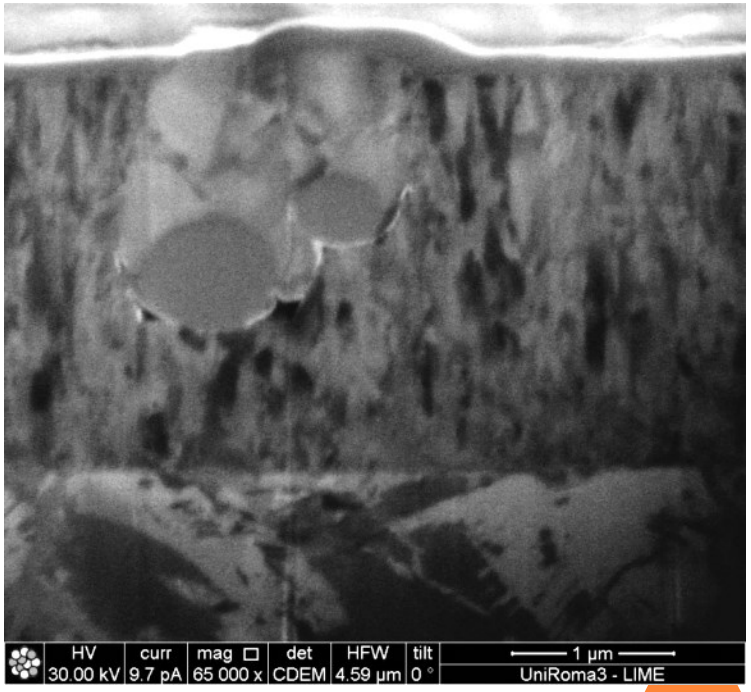
Surface and Coatings Technology, 2013

Local residual stress field in thin films



How to explain the observed differences:

(i) an additional thermal stress factor given by the presence of a metallic droplet



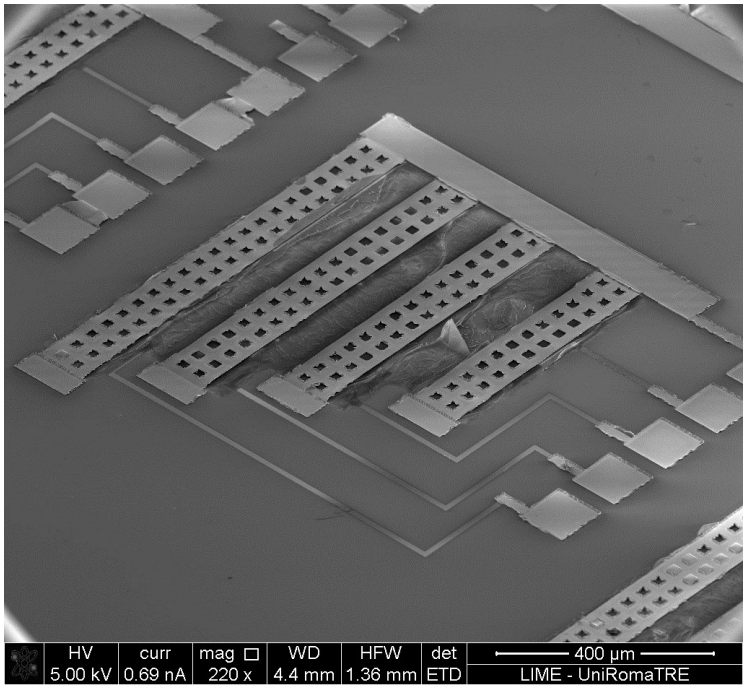
(ii) the induced coating re-nucleation process and modification of the growth mechanisms in correspondence of the defect

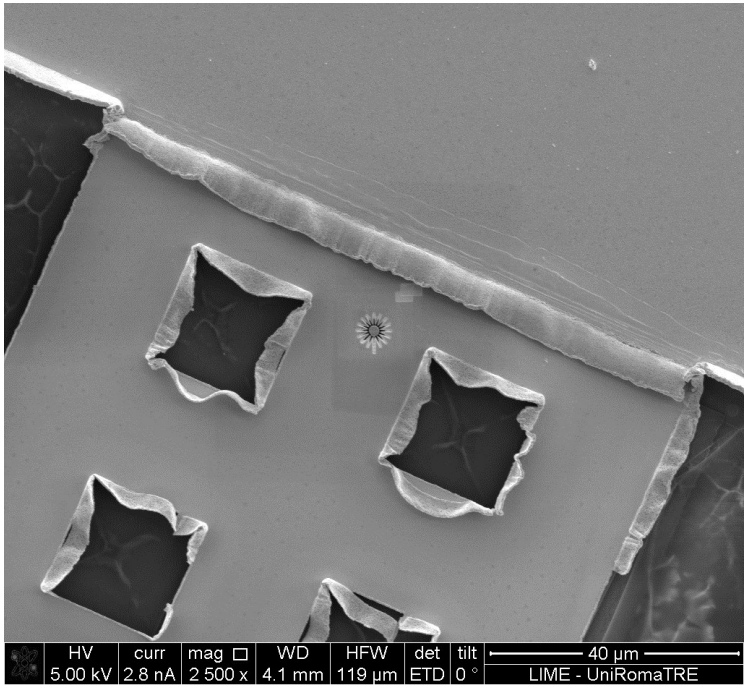
Surface and Coatings Technology, 2013

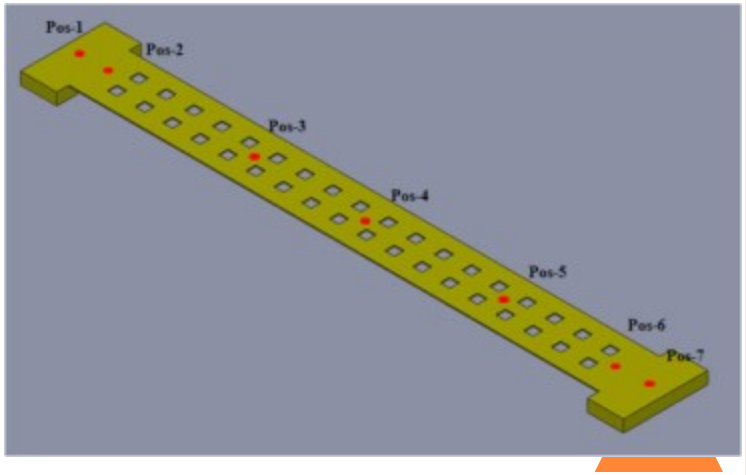
Application on suspended micro-bridges

Sebastiani, M., Bemporad, Korsunsky, A.M.
(2010) **AIP Conference Proceedings**, 1300,

Application on suspended double clamped micro-bridges

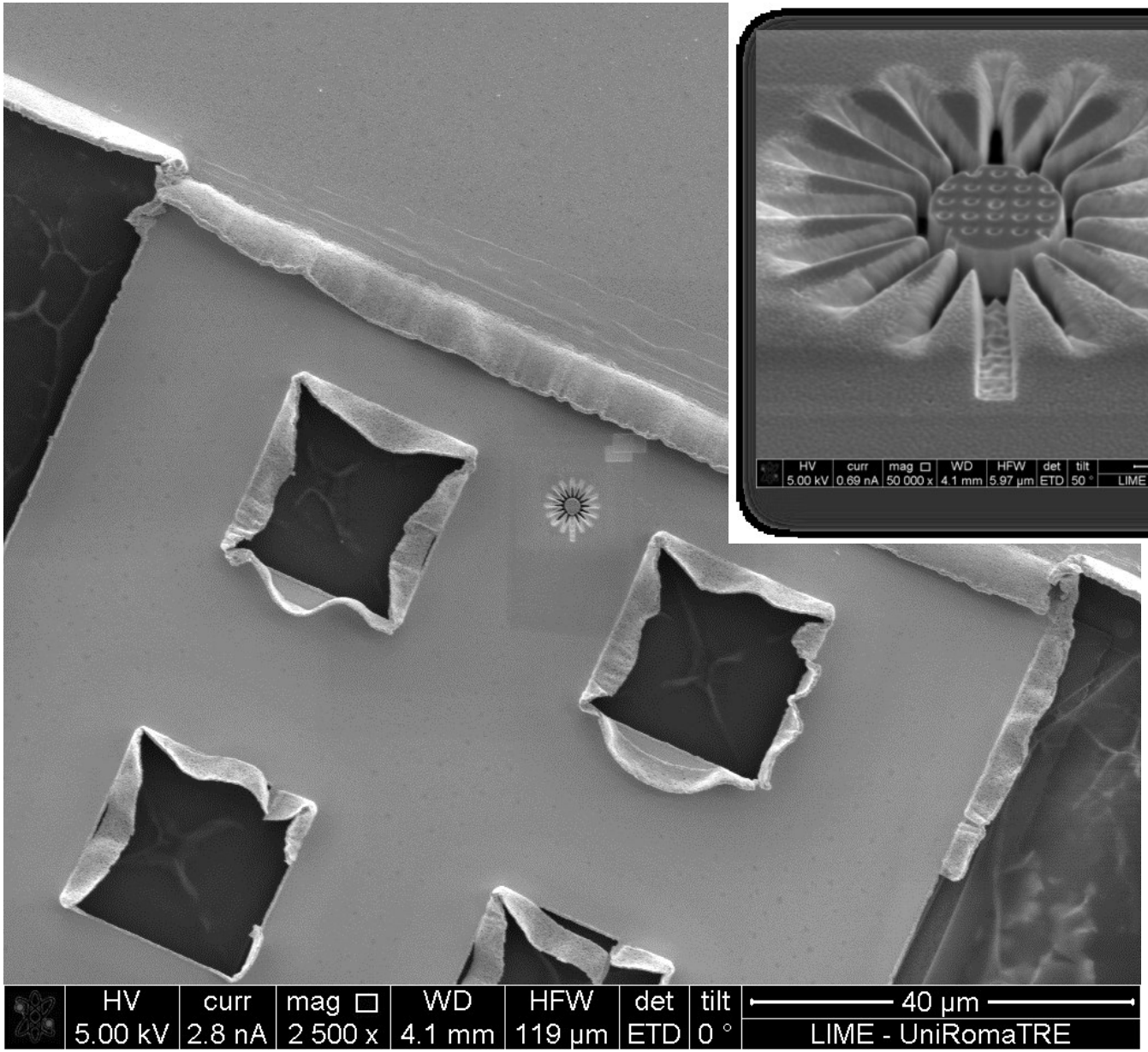


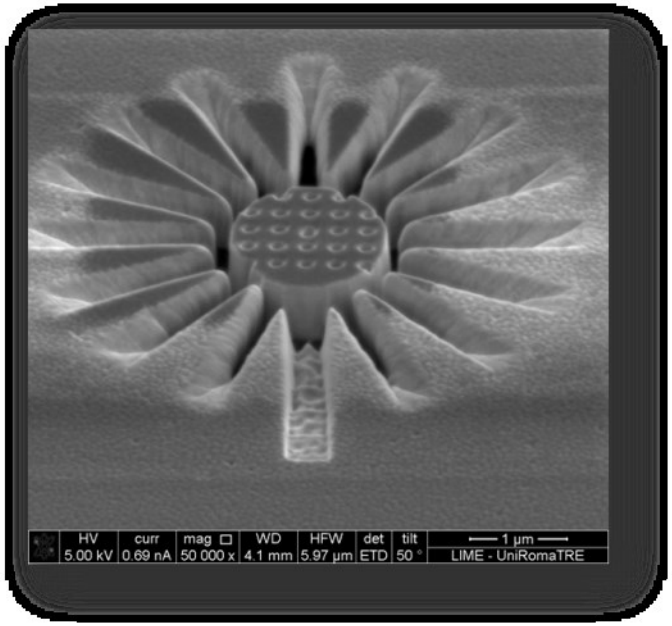




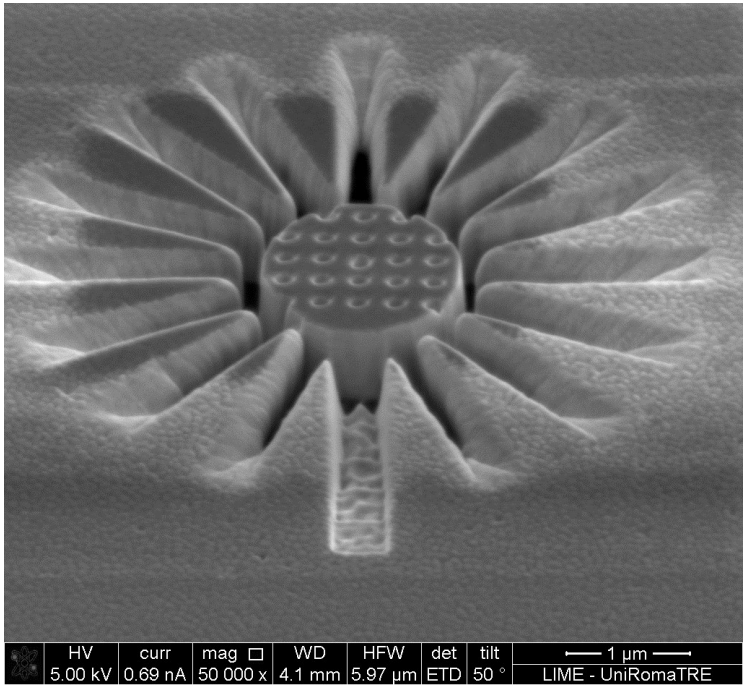
Sebastiani, M., Bemporad, Korsunsky, A.M. (2010) **AIP Conference Proceedings**, 1300,

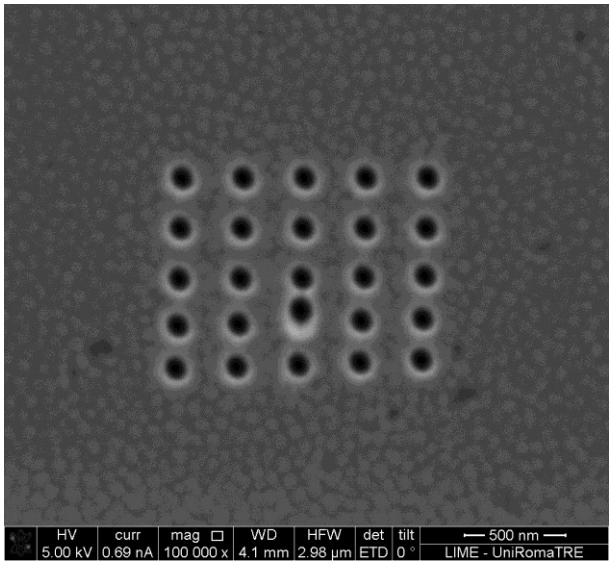
Application on suspended micro-bridges

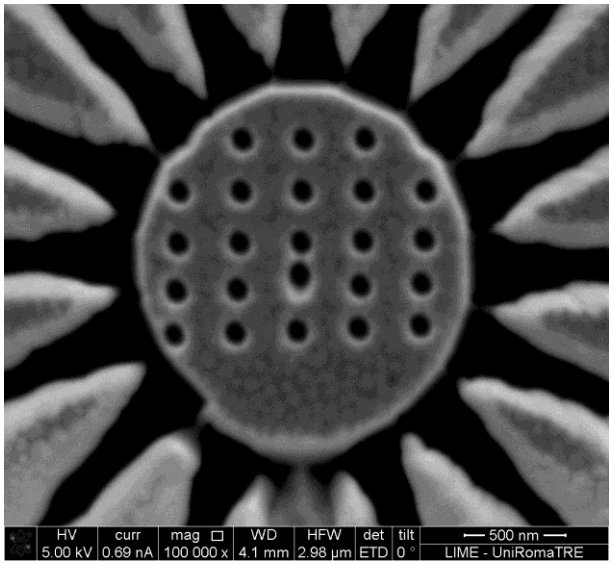




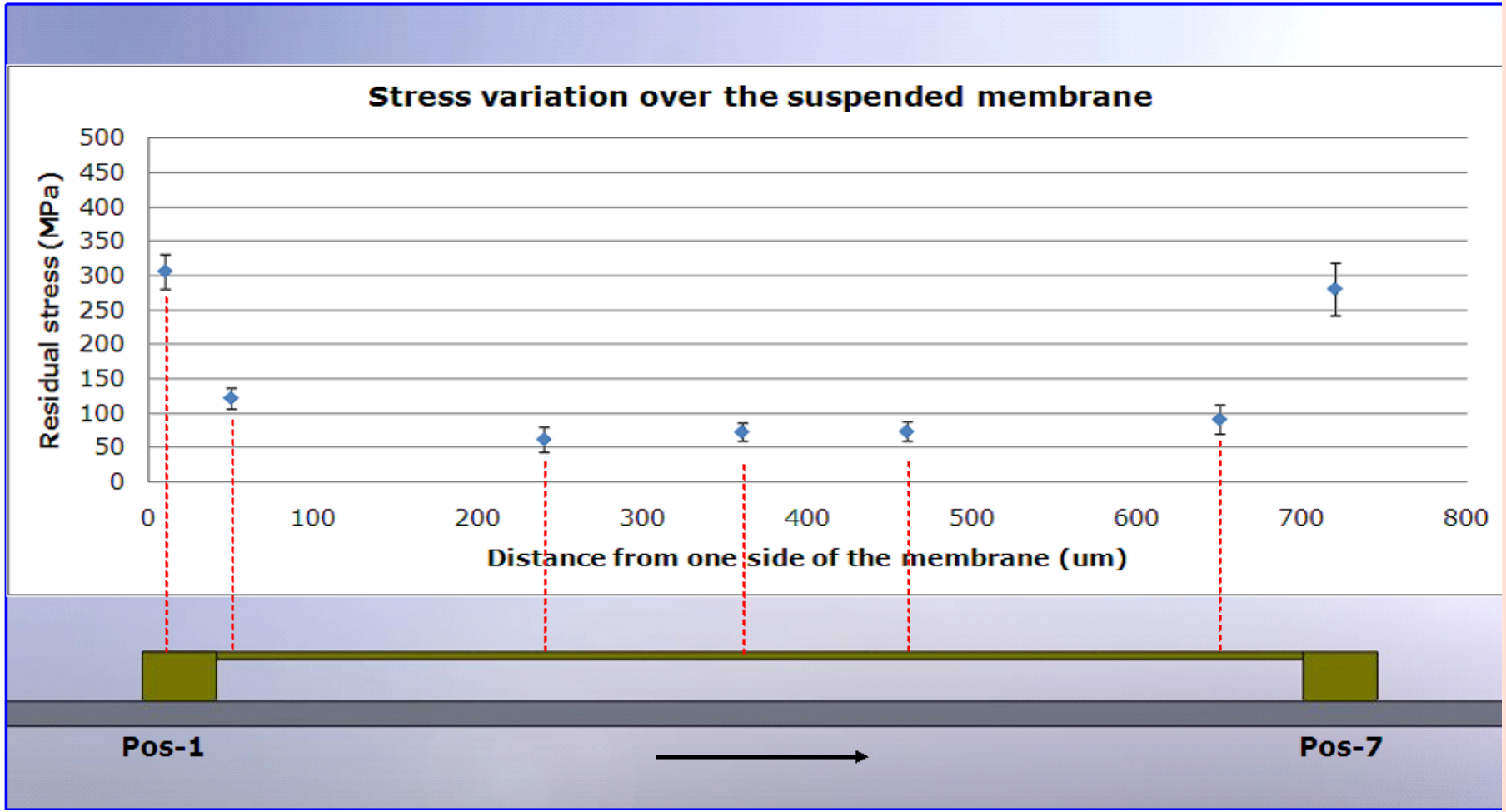
New milling strategy: "daisy" milling







Application on suspended micro-bridges

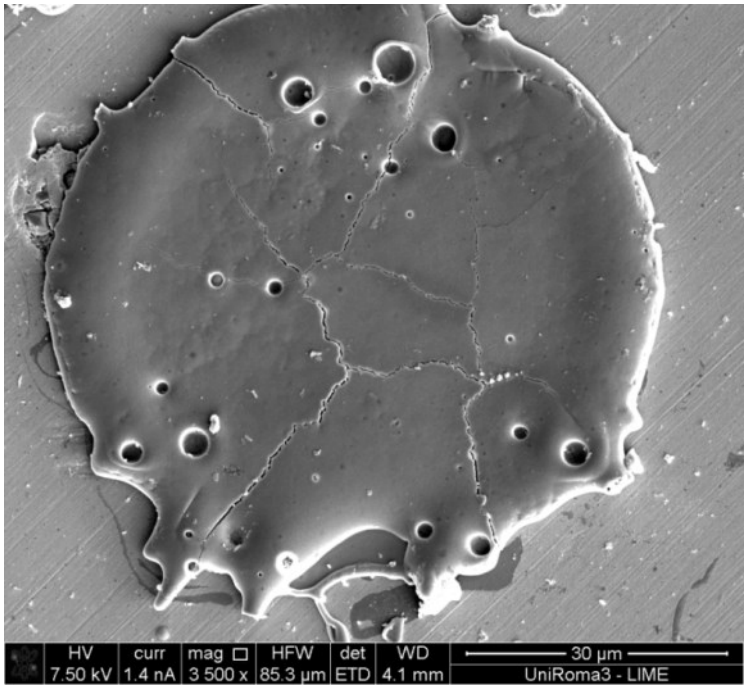


Application on **amorphous** plasma sprayed single-splats

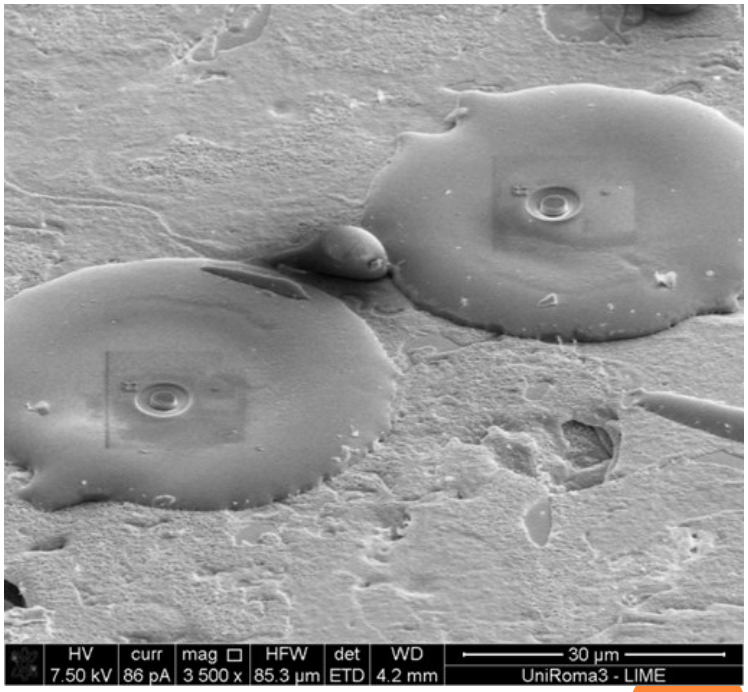
Sebastiani, M., Bolelli, G., Lusvarghi, L., Bandyopadhyay, P.P., Bemporad, E. **Surface and Coatings Technology**, 206 (23), pp. 4872-4880

Application to amorphous materials

Al_2O_3

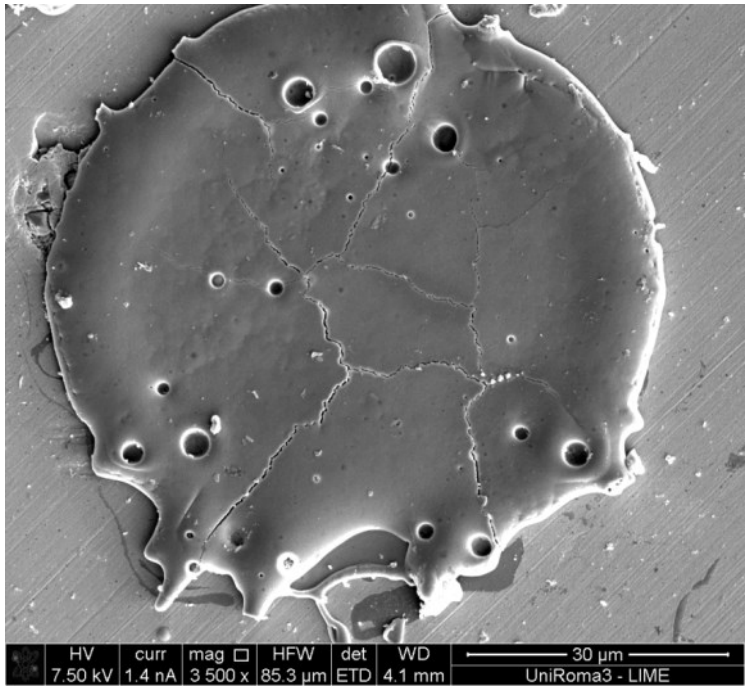


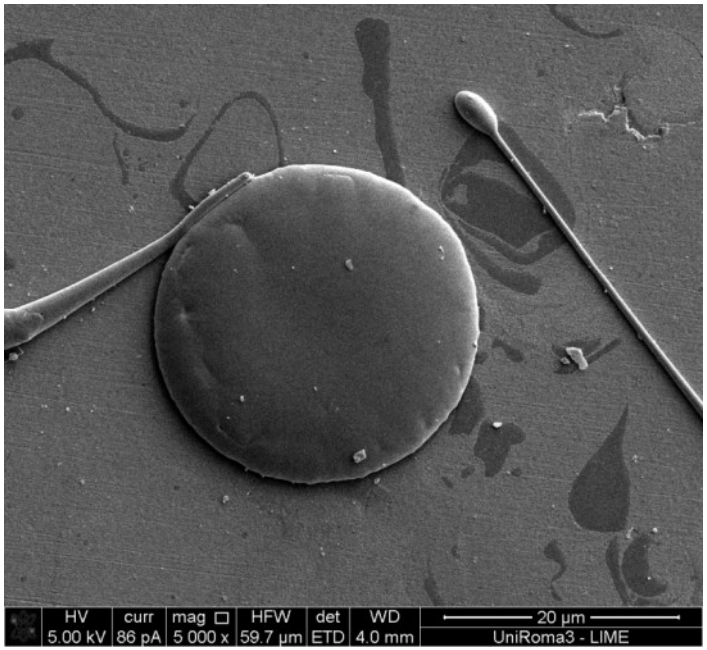
- **Single splats obtained by plasma spraying**

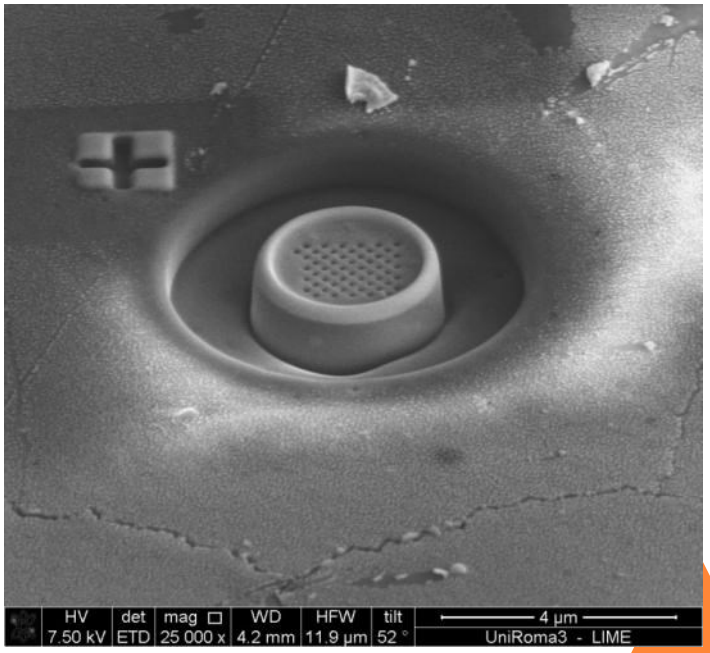


Surface and Coatings Technology, 206 (23), pp. 4872-4880

Al_2O_3 SINGLE SPLATS





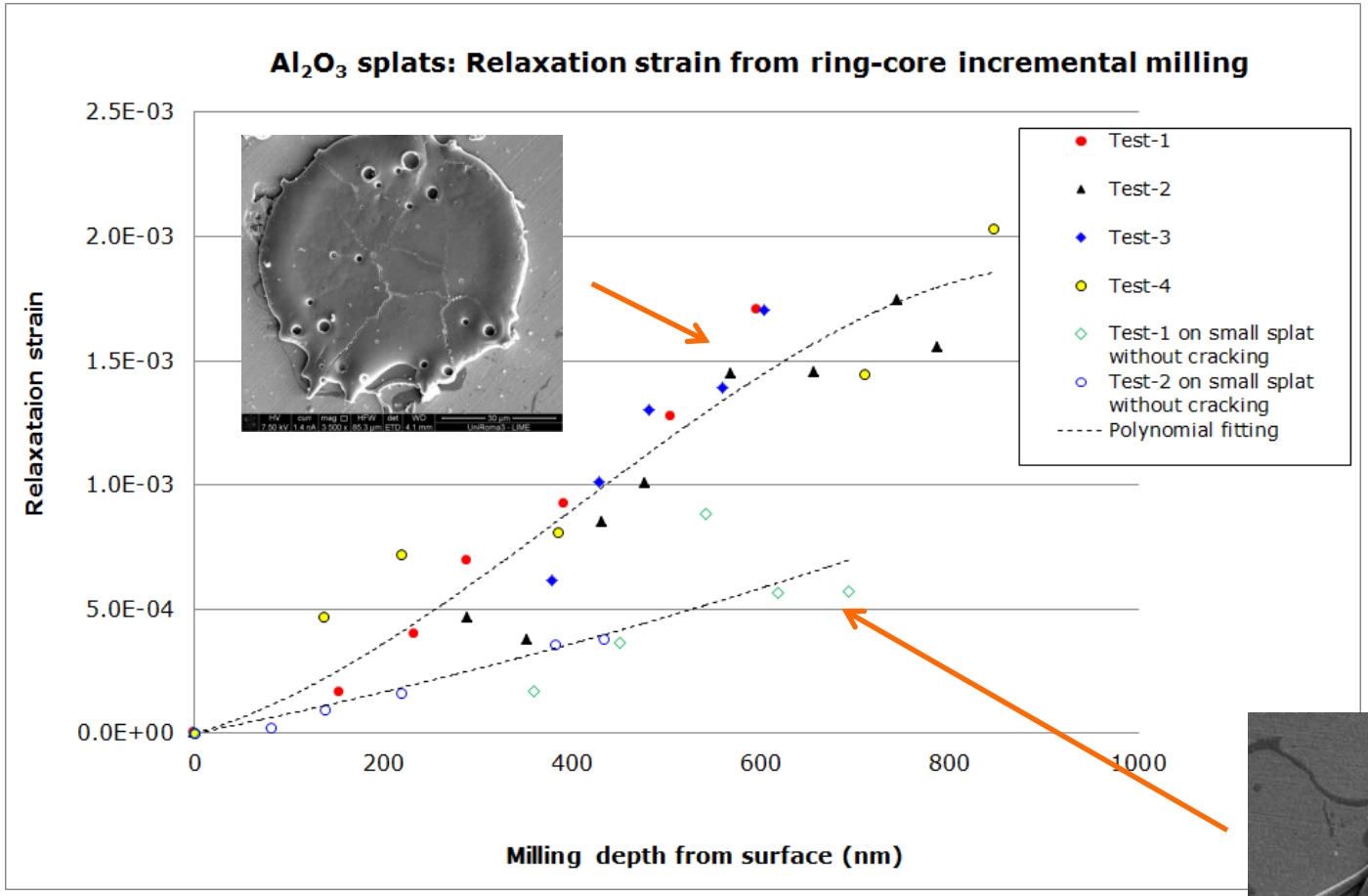


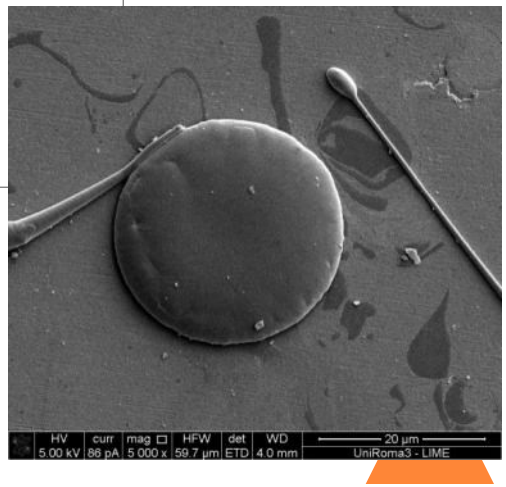
Al_2O_3 and Al_2O_3-13%wtTiO_2

- Large micro-cracked splats
- Small splats without cracking

Surface and Coatings Technology, 206 (23), pp. 4872-4880

Al_2O_3 SINGLE SPLATS



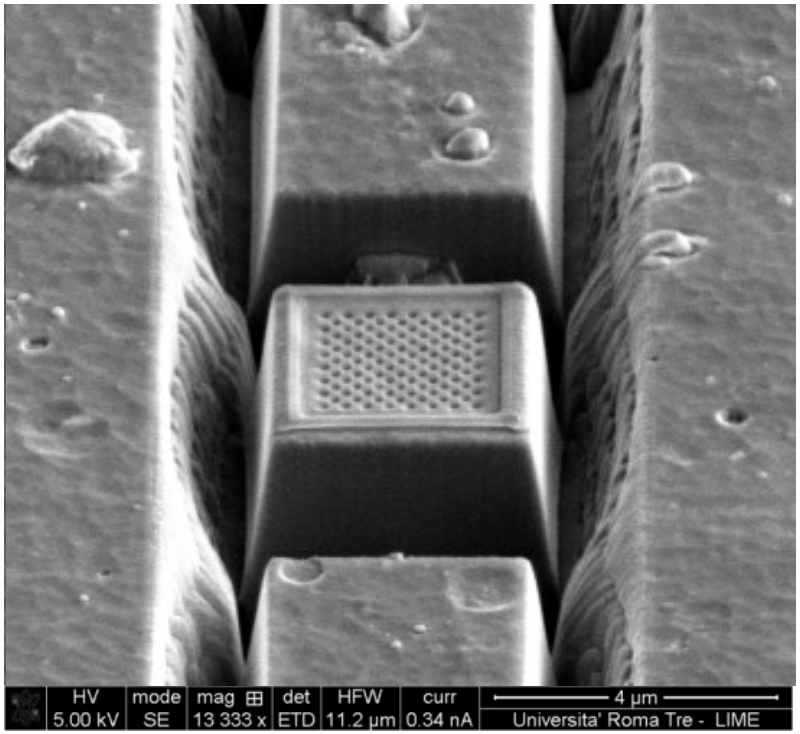


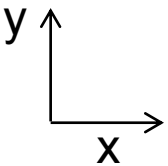
- Small splats (no cracks)
  - Interpolated strain at 400 nm: $+0.38 \cdot 10^{-3} \pm 0.20 \cdot 10^{-3}$
  - Average residual stress: -105.3 ± 55.4
  - Elastic modulus by nanoindentation

Innovative methodology for the Measurement of **Poisson's ratio** in thin films

Marco Sebastiani

IDEA FOR POISSON'S RATIO MEASUREMENT





$$\Delta\varepsilon_x^{(I)}\Big|_{\frac{h}{d}>1} = -\frac{1}{E}\left(1-\nu^2\right)\cdot\sigma_R$$

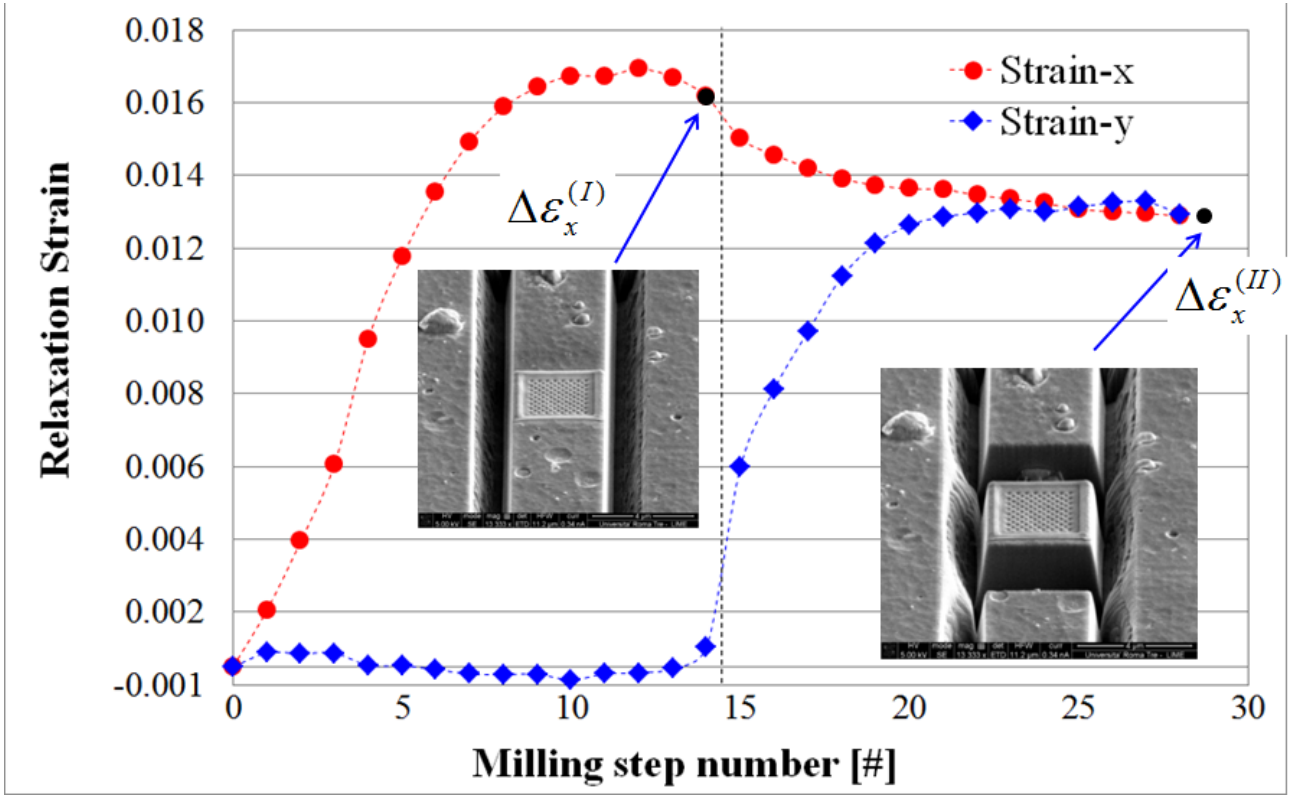
$$\Delta\varepsilon_x^{(II)}\Big|_{\frac{h}{d}>1} = -\sigma_R\cdot\frac{1-\nu}{E}$$

$$\left.\frac{\Delta\varepsilon_x^{(II)}}{\Delta\varepsilon_x^{(I)}}\right|_{\frac{h}{d}>1} = \frac{1}{1+\nu}$$

- New patented method for the simultaneous evaluation of the Poisson's ratio and the residual stress in thin films and small-scale structures

JMPS – submitted

DATA ON CRN – LONG SLOTS ($L=6\cdot D$)

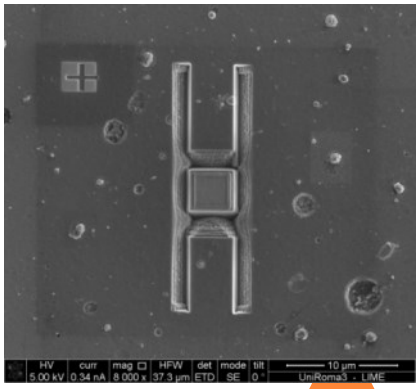


- In this case (5 tests):

$$\Delta\varepsilon_x^{(I)} = 0.0169 \pm 0.001$$
$$\Delta\varepsilon_x^{(II)} = 0.0136 \pm 0.0007$$
$$\nu = 0.248 \pm 0.007$$

- L = 6d
- Y-strain very close to zero
- No edge effects

$$\left.\frac{\Delta\varepsilon_x^{(II)}}{\Delta\varepsilon_x^{(I)}}\right|_{\frac{h}{d}>1.2} = \frac{1}{1+\nu}$$



JMPS – submitted

Why should we look at the Poisson's ratio?

- The knowledge of the Poisson's ratio is extremely important for thin films (and, in particular, for DLC amorphous coatings), and is mostly unknown

Indentation resistance

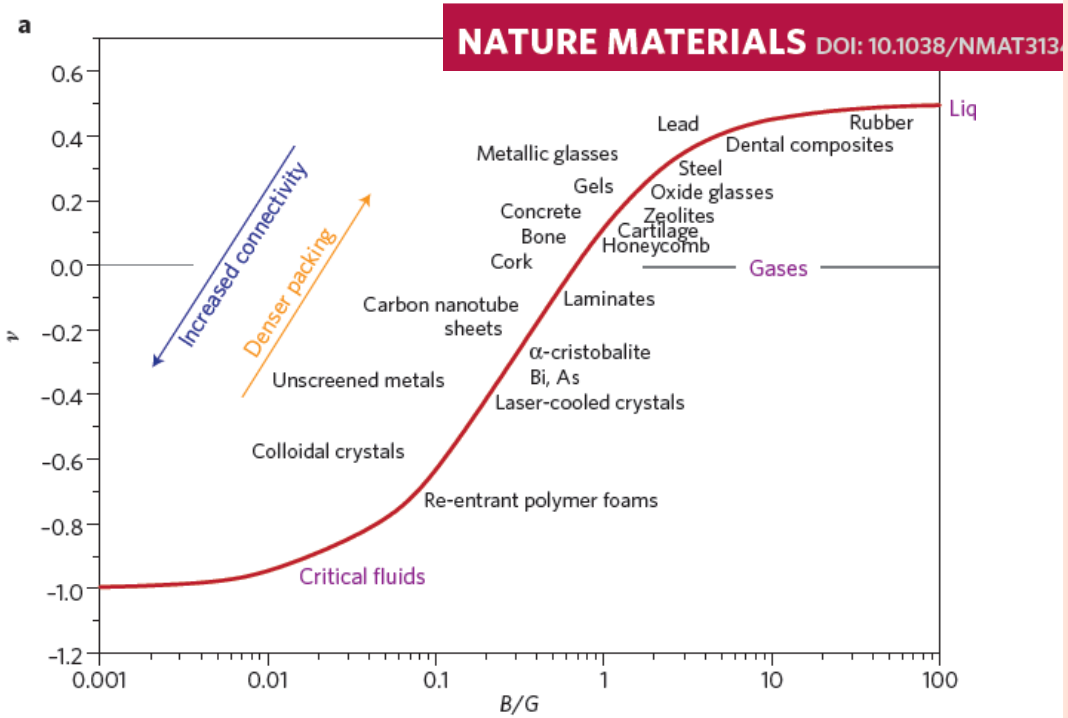
$$E^* \approx \frac{E}{1-\nu^2}$$

Shear modulus

$$G = \frac{E}{2(1+\nu)}$$

Thermal shock resistance

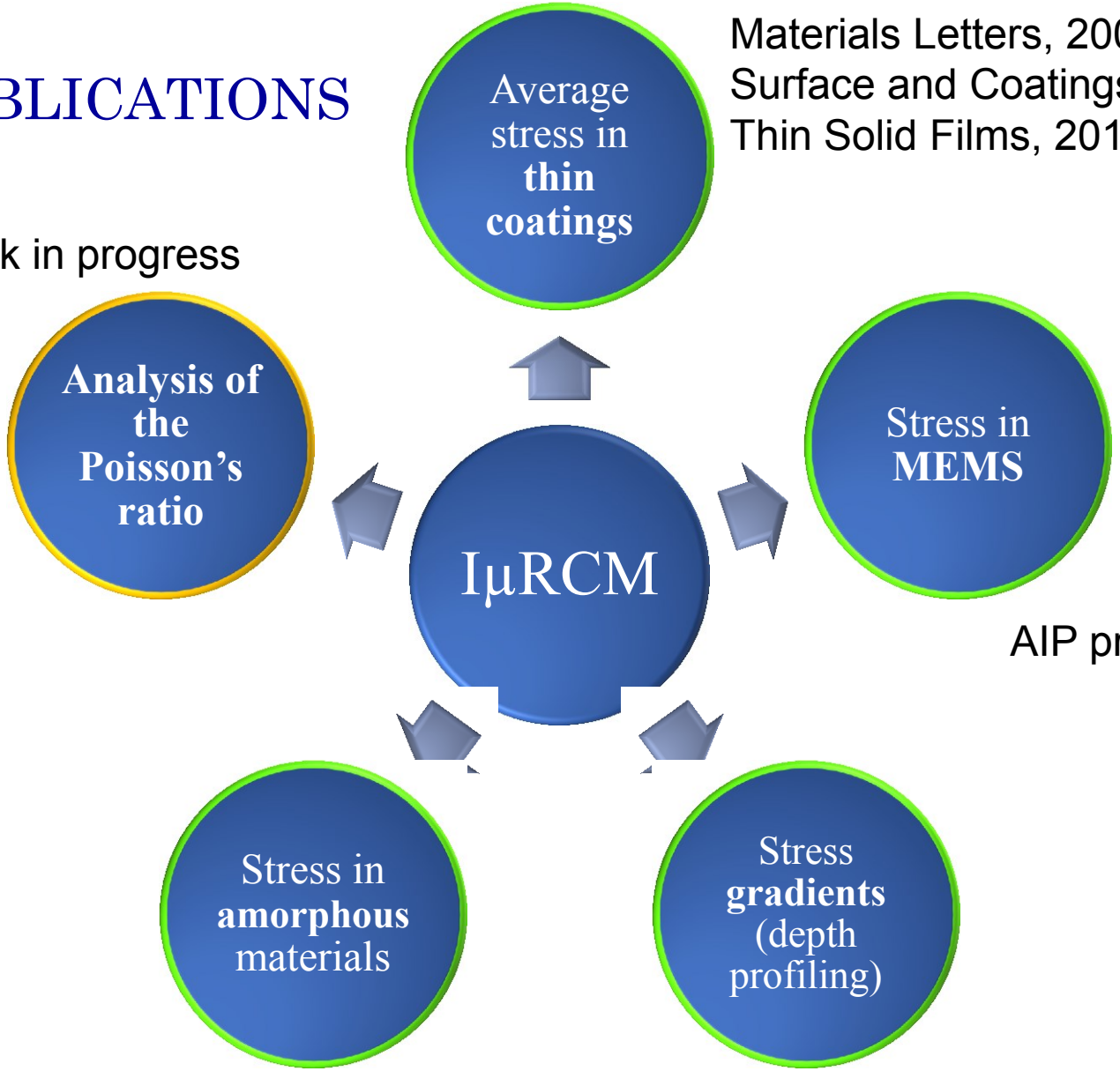
$$R_{TS} = \frac{\lambda R_m}{\alpha E}(1-\nu)$$



Shear vs bulk deformation attitudes

$$\nu \sim \frac{B}{G}$$

PUBLICATIONS



Materials Letters, 2009
Surface and Coatings Technology, 2010
Thin Solid Films, 2012

Work in progress

Analysis of the Poisson's ratio

AIP proceedings, 2010

Surface and Coatings Technology, 2012

Materials Science and Engineering A, 2011
Surface and Coatings Technology, 2013

CONCLUSIONS – FIB/DIC METHOD 1/2

- A ***TOOL*** for micron-scale 3D residual stress analysis on a micron scale has developed and optimized.

- Good results
  - The adopted geometry gives high depth resolution (≈ **100 nm**) and lateral spatial resolution (≈ **1 µm**)
  - The developed milling strategies have improved the repeatability and robustness of DIC procedures
  - LIVE strain depth profiling during FIBing and semi-automated procedure
  - Calculation of the through thickness stress profile;

Conclusions – FIB/DIC method 2/2

- Critical issues to be solved
  - Full automation of the procedure.
  - Does the FIB induces additional stresses??
  - Microstructure/anisotropy/plasticity effects??
  - Careful analysis of elastic properties for stress calculation.
  - Cross validation with other high-resolution methods (synchrotron-XRD, EBSD, μ-Raman).

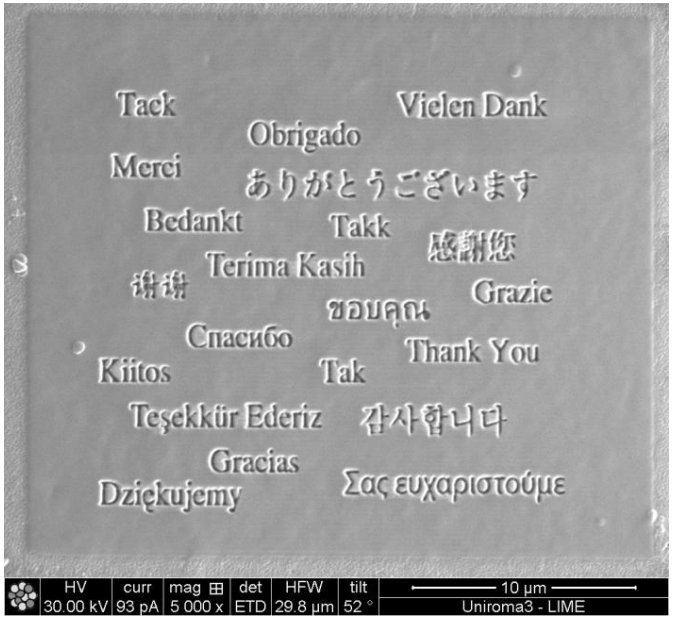
- Ongoing work: **establishment of a standardization activity through a European large project**

PRE-STANDARDISATION OF THE METHOD

- **EU project "ISTRESS" will start on Jan 1st 2014**
  - Pre-standardization of incremental FIB micro-milling for intrinsic stress evaluation at the sub-micron scale
- A new **VAMAS TWA** and project liaisons with **CEN TCs** are going to be established

ISTRESS PROJECT

- **Some info at this webpage:**
  - http://www.stm.uniroma3.it/iSTRESS
- MANY OPEN POSITIONS WITHIN THIS PROJECT: **WE ARE LOOKING FOR FIB EXPERTS TO WORK IN THE CONSORTIUM!**
  - http://www.stm.uniroma3.it/iSTRESS/Pages/Careers.aspx
- WE ARE LOOKING for **associated partners** to be involved in the ROUND-ROBIN activities.



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