Innovative method for residual stress analysis via FIB relaxation

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MECHANICAL PERFORMANCE OF THIN FILMS

The knowledge of **elastic properties** \((E, G, \nu)\) and **residual stress** \((\sigma_R)\) of thin film is a key issue for the assessment 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**
  
  \[
  K_C \propto \left(\frac{E}{H}\right)^n, \sigma_R
  \]

- **Load bearing capacity** \((H^3/E^2)\)

- **Adhesion** \((E, H, \sigma_R)\)

- **Wear resistance** \((H/E, \sigma_R)\)

- **Thermal shock resistance**
  
  \[
  R_{TS} = \frac{\lambda R_m}{\alpha E} (1 - \nu)
  \]
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**.
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.
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 using appropriate constitutive 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.

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**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)
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 ($I_\mu RCM$)**

- **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

Stress in MEMS

Stress in amorphous materials

Stress gradients (depth profiling)
FEM modelling of strain relief from an annular trench

FE SIMULATED STRAIN RELIEF CURVE \((d=t)\)

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

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
Stress profiles can be obtained by the integral Method (Schajer)

- The measured strain relief $\varepsilon(h)$ is the integral of the infinitesimal strain relief components due to the removal of tractions at all depths in the range $0 \leq H \leq h$

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

$$\sum_{j=1}^{n} \hat{a}_{ij} \sigma_j = \frac{E}{1 + \nu} \varepsilon_i \quad 1 \leq j \leq i \leq n$$

- Minimum error in stress calculation if $a_{nn} = \text{const}$
- 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?

Here \( A_{ni} \approx 0 \)
Strong error in stress calculation

Here \( A_{ni} \gg 0 \)
Low error in stress calculation

\[ h/d < 0.3 \]
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
Good reproducibility 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**
The results consistently show a significant modification of the LOCAL residual stress field induced by the droplet.
**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

Stress variation over the suspended membrane

Residual stress (MPa)

Distance from one side of the membrane (um)

Pos-1

Pos-7
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

$\text{Al}_2\text{O}_3$

- Single splats obtained by plasma spraying

Surface and Coatings Technology, 206 (23), pp. 4872-4880
**Al₂O₃ SINGLE SPLATS**

- Large micro-cracked splats
- Small splats without cracking

**Al₂O₃ and Al₂O₃-13%wtTiO₂**

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

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**Al₂O₃ 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: $-105.3 \pm 55.4$
  - Elastic modulus by nanoindentation
INNOVATIVE METHODOLOGY FOR THE MEASUREMENT OF POISSON’S RATIO IN THIN FILMS
IDEA FOR POISSON’S RATIO MEASUREMENT

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

\[ \Delta \varepsilon^{(I)}_x \bigg|_{h \to 1} = -\frac{1}{E} \left(1 - \nu^2\right) \cdot \sigma_R \]

\[ \Delta \varepsilon^{(II)}_x \bigg|_{h \to 1} = -\sigma_R \cdot \frac{1 - \nu}{E} \]

\[ \frac{\Delta \varepsilon^{(II)}_x}{\Delta \varepsilon^{(I)}_x} \bigg|_{h \to 1} = \frac{1}{1 + \nu} \]

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

\[
\frac{\Delta \varepsilon_x^{(II)}}{\Delta \varepsilon_x^{(I)}} \bigg|_{h/d > 1.2} = \frac{1}{1 + \nu}
\]

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**

- Average stress in thin coatings
  - *Surface and Coatings Technology*, 2010

- Stress in MEMS
  - *AIP proceedings*, 2010

- Stress in amorphous materials
  - *Surface and Coatings Technology*, 2012

- Stress gradients (depth profiling)

- Analysis of the Poisson’s ratio

Work in progress

- 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;
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](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](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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