A novel methodology to determine the mechanical properties of amorphous materials through instrumented nanoindentation

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SUMMARY

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   - Objectives

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   - Main assumptions

3. NUMERICAL SIMULATIONS
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   - Validation of assumptions

4. INSTRUMENTED INDENTATION AS A CHARACTERIZATION TECHNIQUE

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Mechanical behavior of amorphous solids (ceramic glasses, metallic glasses and thermoset/thermoplastic polymers) presents a number of similar features:

- **Brittle in tension**
- **Ductile in compression/shear by the propagation of shear bands**
- **Yield strength is pressure-sensitive**
- **Limited strain hardening**

Determination of the mechanical properties by standard tests is sometimes difficult (brittle behavior in tension).

Instrumented nanoindentation emerges as an alternative with the additional advantage of in situ testing.
Mechanical behavior of amorphous solids is well represented by the Drucker-Prager model (rate effects are neglected):

\[
\Phi = \sqrt{3} J_2 - d + \frac{I_1}{3} \tan \beta = 0
\]

\[
I_1 = \sigma_{ii} \quad \sigma'_{ij} = \sigma_{ij} - I_1 / 3
\]

\[
J_2 = \frac{1}{2} \sigma'_{ij} \sigma'_{ji}
\]

\[
\frac{\sigma_{yc}}{\sigma_t} = 1 + \tan \beta
\]

Mechanical properties of amorphous solids are determined by:
(rate effects are neglected):

- Cohesion: \( d \)
- Friction angle: \( \beta \)
- Elastic constants: \( E, \nu \)

Can they be obtained from instrumented indentation tests?
INSTRUMENTED INDENTATION TEST

Four parameters: 
\( P_{max}, h_{max}, S, W_p/(W_e + W_p) \)
but only two are independent ...

This information is used to compute 
the hardness and the elastic modulus 

\[
H = \frac{P_{max}}{A} \\
E^* = \frac{S\sqrt{\pi}}{2\sqrt{A}} \\
\left( \frac{1}{E^*} = \frac{1 - \nu^2}{E} + \frac{1 - \nu_i^2}{E_i} \right)
\]
The inverse problem (determination of yield stress from hardness) is not trivial in general because different combinations of material properties (yield stress, pressure sensitivity, strain hardening) may lead to the same hardness.

Known results: Von Mises solid

\[
\frac{H}{\sigma_{yc}} = f\left(\frac{\sigma_{yc}}{E^*}\right) \quad f \approx 3 \text{ in the fully-plastic regime}
\]

Drucker-Prager solid:

\[
\frac{H}{\sigma_{yc}} = f\left(\frac{\sigma_{yc}}{E^*}, \beta\right)
\]

Main assumption: \(\sigma_{yc}\) and \(\beta\) are replaced by a characteristic indentation stress \(\sigma_r\)

\[
\sigma_r = d - \sigma_h \tan \beta = d - aH \tan \beta
\]

\[
\frac{H}{\sigma_r} = f\left(\frac{\sigma_r}{E^*}\right)
\]

\[
\sigma_{yc} = \frac{\sigma_r + aH \tan \beta}{1 - \tan \beta/2}
\]

The validity of this assumption and the value of the constant \(a\) can be obtained from the finite element simulation of the indentation test in a Drucker-Prager solid.
**FINITE ELEMENT MODEL**

- **Axisymmetric model, large deformations.**
- **Elasto-plastic Drucker-Prager material.**
- **Rigid conical indenter equivalent to a Berkovich tip (θ = 70.30°).**
- **Frictionless indenter/material contact.**
- **Discretization with first order elements with full integration (CAX4). Mesh refined at the contact area with the tip (40 nodes in contact).**
- **Simulations carried out with Abaqus/Standard.**
- **In cases of excessive distortion, simulations were carried out with Abaqus/explicit and arbitrary Lagrangian–Eulerian mesh adaptivity.**
**NUMERICAL RESULTS**

Elasto-plastic regime

\[ \frac{\sigma_{yc}}{E^*} = 0.02 \]

\[
\frac{H}{\sigma_{yc}} = f\left( \frac{\sigma_{yc}}{E^*}, \beta \right)
\]

\[
\frac{H}{\sigma_r} = f\left( \frac{\sigma_r}{E^*} \right)
\]
NUMERICAL RESULTS

\[
\frac{H}{\sigma_r} \approx 2.9 \\
H = f \left( \frac{\sigma_r}{E^*} \right) \\
\sigma_r = d - 0.29H \tan \beta
\]
$W_e/W_t$ is closely related to $\sigma_r/E^*$
Determination of $E^*$, $\sigma_{yc}$ and $\beta$ from instrumented indentation: $H_{ap}$, $S$, $W_e/W_t$

\[ H_{ap} = \frac{P_{max}}{A_{ap}} \]
Master curves are built as a function of parameters readily measurable and incorporate the effect of the pile-up.

The master curves allow to determine $\sigma_{yc}$ from the indentation data when $\beta$ is known and vice versa.

In addition, it is possible to determine $\beta$ from the independent measurement of the real contact area $A_c$ if $W_e/W_t < 0.5$. 

 MASTER CURVES 

characterization by indentation
**MATERIALS**

- **Bulk metallic glasses**: Zr\(_{65}\)Cu\(_{15}\)Al\(_{10}\)Ni\(_{10}\), Mg\(_{58.5}\)Cu\(_{30.5}\)Y\(_{11}\) and Mg\(_{61}\)Cu\(_{28}\)Gd\(_{11}\)
- **Ceramic glasses**: Soda-lime glass (Starphire®) and borosilicate glass (Borofloat®)
- **Glassy polymers**: epoxy, PMMA and PVD

Wide range of \(\sigma_y/E\) and \(\beta\)

**EXPERIMENTAL TECHNIQUES**

- Experiments conducted using a Nanoindenter XP
- At least 10 indentations in each material with Berkovich tip at a strain rate 0.002 s\(^{-1}\)
- Residual imprints measured by AFM using a Park XE150
### Bulk Metallic Glasses

#### Analysis Strategy

- \( \frac{W_e}{W_t} \approx 0.31 \)
- No prior knowledge of \( \sigma_{yc} \) or \( \beta \) available

Measurement of Residual Imprint by AFM

\[
A_{ap} \rightarrow c_p \rightarrow \beta \rightarrow \sigma_{yc}
\]

### Ceramic Glasses

#### Analysis Strategy

- \( \frac{W_e}{W_t} \) in the range 0.52-0.58
- Oliver & Pharr provides \( A_c \) but \( \beta \) cannot be determined from \( A_c \)

\( \sigma_{yc} \) determined by mechanical testing

\[
W_e/W_t, H_{ap}/\sigma_{yc} \rightarrow \beta
\]
Glassy polymers

Analysis Strategy

- $W_e/W_t$ in the range 0.31-0.58
- Residual imprint cannot be measured because of viscoelastic effects

$W_e/W_t$, $H_{ap}/\sigma_{yc}$ → $\beta$

$\sigma_{yc}$ determined by mechanical testing
Experimental Range

- \( H_{ap} / \sigma_{yc} \) vs. \( W_e / W_t \)
- Materials: 
  - \( \text{Mg}_{58.5}\text{Cu}_{30.5}\text{Y}_{11} \)
  - \( \text{Mg}_{61}\text{Cu}_{28}\text{Gd}_{11} \)
  - \( \text{Zr}_{65}\text{Cu}_{15}\text{Al}_{10}\text{Ni}_{10} \)
  - PVC
  - PMMA
  - Starphire
  - Borofloat
  - Epoxy Resin

- Curves for different values of \( \beta \):
  - \( \beta = 30^\circ \)
  - \( \beta = 24^\circ \)
  - \( \beta = 12^\circ \)
  - \( \beta = 0^\circ \)
  - \( \beta = 36^\circ \)
  - \( \beta = 42^\circ \)
  - \( \beta = 48^\circ \)
Literature Comparison

Yield Compression Strength, $\sigma_{yc}$ (MPa)

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<tr>
<th>Zr$<em>{65}$Cu$</em>{15}$Al$<em>{10}$Ni$</em>{10}$</th>
<th>Mg$<em>{58.5}$Cu$</em>{30.5}$Y$_{11}$</th>
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Drucker-Prager Frictional Angle $\beta$ (°)

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Bulk Metallic Glasses

Ceramic Glasses

Glassy Polymers
A novel methodology based on instrumented indentation was developed to characterize the mechanical properties ($E$, $\sigma_{yc}$ or $\beta$) of amorphous materials.

The approach is based on the concept of a universal postulate that assumes the existence of a characteristic indentation pressure proportional to the hardness. This hypothesis was numerically validated.

This method overcomes the limitation of the conventional indentation models (pile-up effects and pressure sensitivity materials).


**Research Projects**

- Ministry of Science and Innovation, National Program on Materials (MAT09-14396)
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