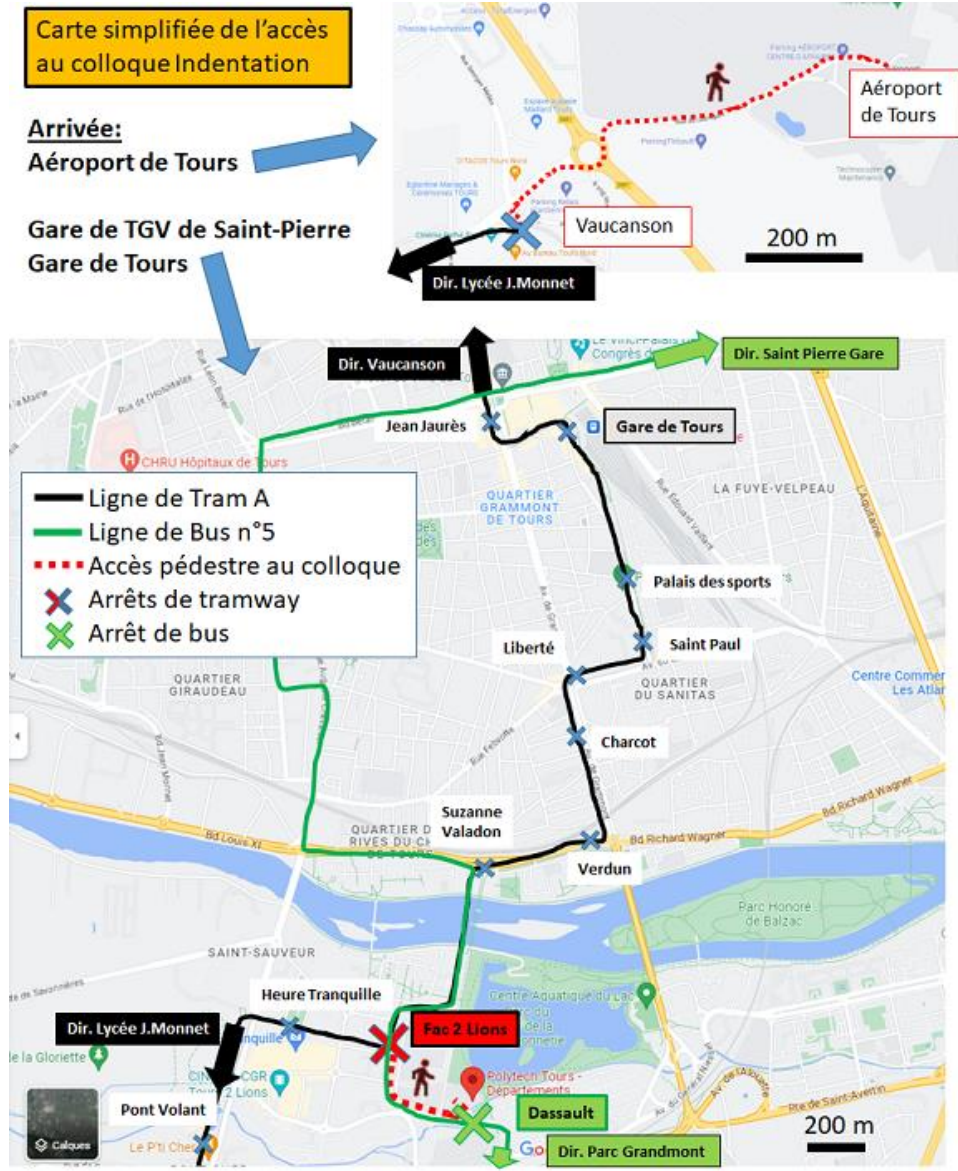


COLLOQUE INDENTATION



L'ensemble de l'événement se déroule à PolyTech Tours, 7 avenue Marcel Dassault, 37200 Tours, France.



Mardi 04/07


FORMATION		
13:30	Accueil	
14:00	Bases de l'Indentation	Benayoun Stéphane
16:00	Indentation et comportements de polymères : quelques aspects de la physique des polymères impactant la mécanique et la physique du contact	Gauthier Christian
18:00	Soirée libre	

Mercredi 05/07

08:00		TP expérimental sur Machines	
		Anton Paar	Berthout Guillaume
		Fischer Instruments	Roudier Jean- François
		Alemnis	Gennero Benoit
		Bruker	Hangen Ude, Paris Emmanuel
10:00		Outil de post-processing pour indentation	Charleux Ludovic
12:00		ACCUEIL / DEJEUNER FORMATION	
14:00	OUVERTURE		
14:20	Fondamentaux de la technique Chair: S. Benayoun	Strain-rate dependence of indentation induced dislocation distribution in copper	Kalacska Szilvia
14:40		Resolution limits of hardness mapping	Hangen Ude
15:00		Enriching nanoindentation with real-time electrical measurements and in-situ SEM monitoring	Volpi Fabien
15:20		New leads on the densification profile underneath nano indentation imprint in silica glass by means of a chemical dissolution technique	Guin Jean- Pierre
15:40	Pause Café		
16:00	SESSION POSTERS		
17:00	PRESENTATIONS INDUSTRIELS		
18:00	AG GIME		
19:00 – 22:00	Cocktail dinatoire + FoodTruck		

Jeudi 06/07

08:00	Accueil		
8:20	Polymères,élastomères et bio-matériaux Chair: C. Gauthier	Contact mechanics of pre-oriented amorphous polymers	Pecora Marina
8:40		Apport de la nanoindentation et de l'AFM pour la compréhension de la mécanique multi-échelle d'un polypropylène isotactique	Grondin Jérémy
9:00		Etude par nanoindentation et traction uniaxiale du vieillissement thermique d'un PDMS chargé	Feyne Florian
9:20		Vieillissement par thermo-oxydation des polymères exposés à des écoulements rapides. Caractérisation par nanoindentation.	Doriat Aurélien
9:40		Macro-, Micro- and Nanomechanical Characterization of Crosslinked Polymers with Very Broad Range of Mechanical Properties	Sedmak Pavel
10:00	Pause Café		
10:20	Matériaux Fragiles Chair: V. Keryvin	Microplasticité des amorphes silicatés - quelques développements expérimentaux récents	Étienne Barthel (invité)
11:00		Nano stamping of material surface during nano-indentation by the indenter: a fingerprint for contact area measurement	Charleux Ludovic
11:20	Simulation et modélisation Chair: G. Kermouche	Building the nanoMECommons Database: a workflow for indentation data digitalisation to numerical simulations	Mercier David
11:40		Mieux comprendre l'indentation avec deux modèles revisités	Hernot Xavier
12:00	DEJEUNER		
13:20	Photo		
13:40	Simulation et modélisation Chair: G. Kermouche	The effect of initial backstress on instrumented indentation test	Chen Hui
14:00		Multi scale in-situ micromechanical tests for BGA interconnections	Malkorra Sarasola Irati
14:20	Matériaux métalliques Chair: G. Mauvoisin	Acoustic Emissions as a Tool for the Exploration of Plastic Instabilities during Nanoindentation at the Nanoscale	Cech Jaroslav
14:40		Etude multi-échelle de l'effet de l'hydrogène et la nature des joints de grains sur les mécanismes de plasticité dans le nickel pur	Ben Jedidia Yasmine
15:00		Nanoindentation to reveal synergistic effects of hydrogen and precipitate on elastic/plastic properties of nickel-based superalloy	Murugan Siva Prasad
15:20		Understanding size effect and strain rate sensitivity in copper by In-situ pillar microcompression and dislocation dynamics simulation	Adogou Ame
15:40	Pause Café		

16:00	<p style="text-align: center;">SESSION POSTERS</p>
16:30	<p style="text-align: center;">GALA & REMISES DES PRIX Départ de bus à 16h30 de Polytech</p>
<p style="text-align: center;">visite/ cocktail/ diner au Château Villandry</p> <div data-bbox="520 524 1267 958" data-label="Image">A photograph of the gardens of Château Villandry in France. The image shows a large, white stone chateau with a prominent tower on the left. In the foreground, there are meticulously maintained formal gardens with geometric patterns of hedges, flower beds, and topiary trees. The sky is blue with scattered white clouds.</div>	
22:30	<p style="text-align: center;">Retour par bus avec dépose à Polytech</p>

Vendredi 07/07

08:00	Accueil		
8:20	Environnement : température, humidité, dynamique Chair: E. Barthel	High Strain Rate Nanoindentation: Recent Advances and Perspectives	Widmer Remo
8:40		High-temperature scanning indentation. Une nouvelle méthode pour étudier les changements microstructuraux en température	Tiphene Gabrielle
9:00		Effet de l'irradiation électronique sur la plasticité des verres d'oxyde lors d'essais micromécaniques in-situ.	Kermouche Guillaume
9:20		How to Measure Precisely Surface Shear Properties at the Micron Scale?	Guillonneau Gaylord
9:40		Investigation of carbon fibre mechanical behaviour at micro/nano scales	Keryvin Vincent
10:00	Pause Café		
10:20	Couches minces et matériaux hétérogènes Chair: A. Montagne	A method to determine anisotropic elastic constants in a metastable titanium alloy based on nanoindentation	Idrissi Youssef
10:40		Mechanical property changes accross the spin transition in a molecular film probed by nanoDMA	Nasimsobhan Maryam
11:00		Challenges in Small-Scale Testing of Energy Materials up to 1000°C	Hangen Ude
11:20		On the identification of elastic-plastic thin film properties using numerical nanoindentation tests and finite element modeling (FEM)	Jellad Ep Khelifi Asma
11:40	CLOTURE		
12:00	DEJEUNER		
14:00-17:00	VISITES Vieux Tours / Balade sur la Loire		



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NANO STAMPING OF MATERIAL SURFACE DURING NANO-INDENTATION BY THE INDENTER: A FINGERPRINT FOR CONTACT AREA MEASUREMENT

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ABSTRACT

The determination of the contact area is a key step to derive mechanical properties such as hardness or an elastic modulus by instrumented indentation testing. Two families of procedures are dedicated to extracting this area: on the one hand, *post-mortem* measurements that require residual imprint imaging, and on the other hand, direct methods that only rely on the load vs. the penetration depth curve. With the development of built-in scanning probe microscopy imaging capabilities such as atomic force microscopy and indentation tip scanning probe microscopy, last generation indentation devices have made systematic residual imprint imaging much faster and more reliable.

In this study, a post-mortem method is first introduced and further compared to three existing classical direct methods by means of a numerical and experimental benchmark covering a large range of materials. It is shown that the new method systematically leads to lower error levels regardless of the type of material. The pros and cons of the method vs. direct methods are also discussed, demonstrating its efficiency in easily extracting mechanical properties with enhanced confidence [Charleux et al, 2014].

In a second step, we take advantage of a geometrical feature left on the diamond indenter by mechanical machining. This nano-pattern leaves its imprint on the tested material (see Figure 1).

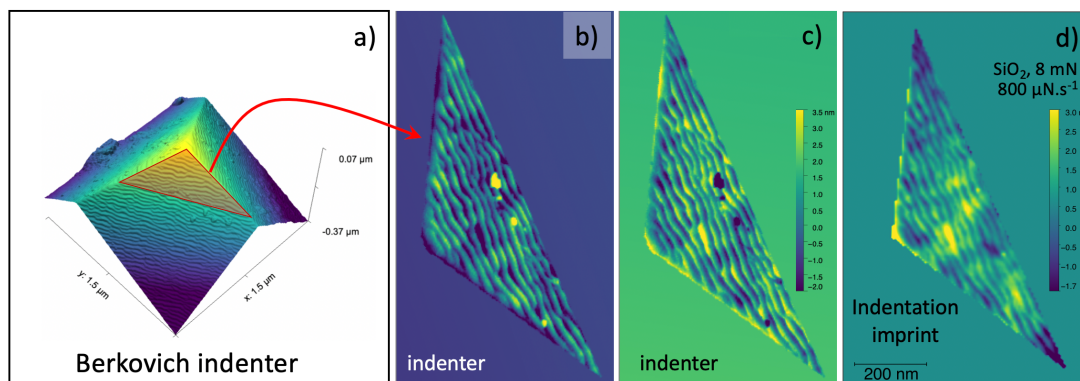


Figure 1. Nano-patterns on the indenter faces (AFM imaging; tapping mode) and on the faces of the imprint (silica glass)

The contact area is then measured by distinguishing the nano-stamping from the indenter and the rest of the material surface. This area measurement is finally compared to other classical methods (see Figure 2).

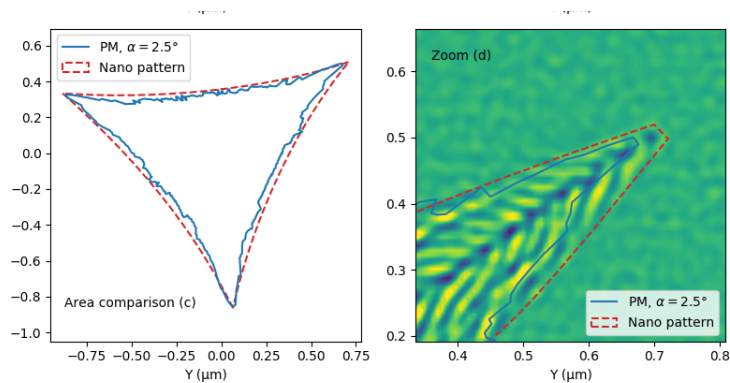


Figure 2. Determination of the contact area from AFM measurements on the imprint (silica glass) [Trenvouez et al, 2020], either from the nano-pattern or from another proposed in [Charleux et al, 2014] (PM)

KEYWORDS

Nano-indentation; contact area; imprint; elastic modulus; hardness

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Trenvouez Gwénoélé, Keryvin Vincent, Guin Jean-Pierre, Bernard Cédric. Comment on “elastic-plastic deformation in ion-exchanged aluminosilicate glass by loading rate dependent nanoindentation”. *J. Non-Crystalline Solids*, 2020, 528, p.119692, 2020.

Strain-rate dependence of indentation induced dislocation distribution in copper

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ABSTRACT

Indentation is routinely used for determining materials properties around the world. Even though many published works focus on the difficult interaction between the tip and the sample, the exact details on dislocation generation and stress fields evolving below the tip is still not fully understood.

In order to study the indentation induced dislocation distribution, a copper single crystal specimen was prepared (heat treatment, mechanical and broad beam Ar-ion polishing) for the experiments. Two different tips (conical with 1 μm radius and cube corner) were used to make indents close to the edge of the bulk sample at various strain rates (from quasi-static up to $10^4/\text{s}$). Focused ion beam cross-sections were prepared, and high (angular) resolution electron backscatter diffraction (HR-EBSD) was used to map the local geometrically necessary dislocation (GND) density [1] below the indents. Differences revealed by the HR-EBSD technique will be discussed in details.

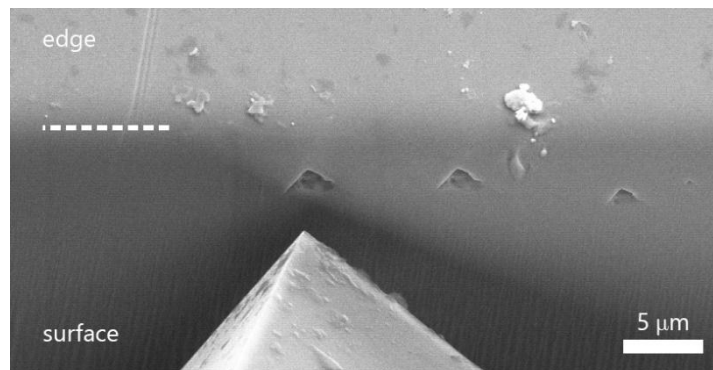


Figure 1. SEM image of the cube corner tip with some indents close to the edge of the sample (12° tilted view).

KEYWORDS

High strain rate indentation, HR-EBSD, dislocation distribution, FIB

REFERENCES

[1] Szilvia Kalácska, Johannes Ast, Péter Dusán Ispánovity, Johann Michler, Xavier Maeder. 3D HR-EBSD characterization of the plastic zone around crack tips in tungsten single crystals at the micron scale. *Acta Materialia*, 2020, 200, p. 211-222.

Alternative to the Oliver & Pharr's calibration in nanoindentation.

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KEYWORDS

Nanoindentation; Calibration; Contact area function.

ABSTRACT

The accurate determination of the mechanical properties by nanoindentation, especially for the lowest indentation depths, strongly depends on the size of the indenter tip defect (blunted tip). That is why, a proper calibration of both the compliance of the measuring system and the contact area function is required. This is usually achieved by following the Oliver and Pharr's proposal (1992). This calibration methodology is based on a relationship expressing the reduced Young's modulus of fused silica ($E_R = 70 \text{ GPa}$ calculated from the Young's modulus, $E = 72 \text{ GPa}$), as a known value, as a function of the inverse of the total compliance and the reciprocal square root of the contact area function where eight coefficients C_i are fitted till obtaining the constant value for the reduced elastic modulus. This standardized approach gives very satisfactory results even if the values of the C_i coefficients depend on the starting values of the fitting process. Despite this, these authors suppose that the corrective term of the frame compliance (C_m) has a fixed value for all the indentation experiments whatever the tested material, which is controversially admitted in the literature.

In this work, we propose to extend an alternative methodology which has been already successfully applied in the microindentation loads range on a large variety of ceramics and metals (Chicot *et al.* 2022). This methodology consists in applying, first, a Force Function (FF) to determine the corrective compliance term, C_m . In a second step, this last value is introduced into an Area Function (AF) to estimate the indenter tip defect, h_b , and the reduced modulus, E_R . Finally, the instrumented hardness, HIT , is then obtained by combining the two slopes of the AF and FF functions as shown hereafter:

$$\text{FF function: } C_T = C_m + \frac{\sqrt{\pi}}{2} \times \frac{\sqrt{HIT}}{E_R} \times \frac{1}{\sqrt{F_m}}; \quad \text{AF function: } C_T = C_m + \frac{\sqrt{\pi}}{2 \cdot E_R} \times \frac{1}{\sqrt{A_P(h_c)}}$$

$$\text{Oliver \& Pharr: } A_P(h_c) = 24.5 h_c^2 + C_1 h_c^1 + C_2 h_c^{\frac{1}{2}} + C_3 h_c^{\frac{1}{4}} + \dots + C_8 h_c^{\frac{1}{128}}$$

$$\text{Chicot et al.: } A_P(h_c) = 24.5 \left[h_c + h_b \left(1 - \exp \left[-\frac{h_c}{h_b} \right] \right) \right]^2$$

where C_T is the total compliance, F_m the applied force onto the indenter and $A_P(h_c)$ the projected contact area as a function of the contact depth, h_c . The analysis was performed by applying the two projected contact area models.

This methodology has been applied to more than 40 calibration processes on fused silica for which each of them is the result of 25 indentation tests (5 by 5 square indentation mapping). **Figure 1** shows, as an example, the Young’s modulus (E) versus the indenter displacements lower than 100 nm allowing to better highlight the influence of the contact area estimation on the Young’s modulus variation. Note that this figure, which results from only one single indentation test on fused silica calibration process, clearly shows the similarity between the two approaches. It should be also noted that, in the alternative methodology, this work, the value of the Young’s modulus of the fused silica is not imposed in the regression analysis contrarily to the Oliver & Pharr’s proposal.

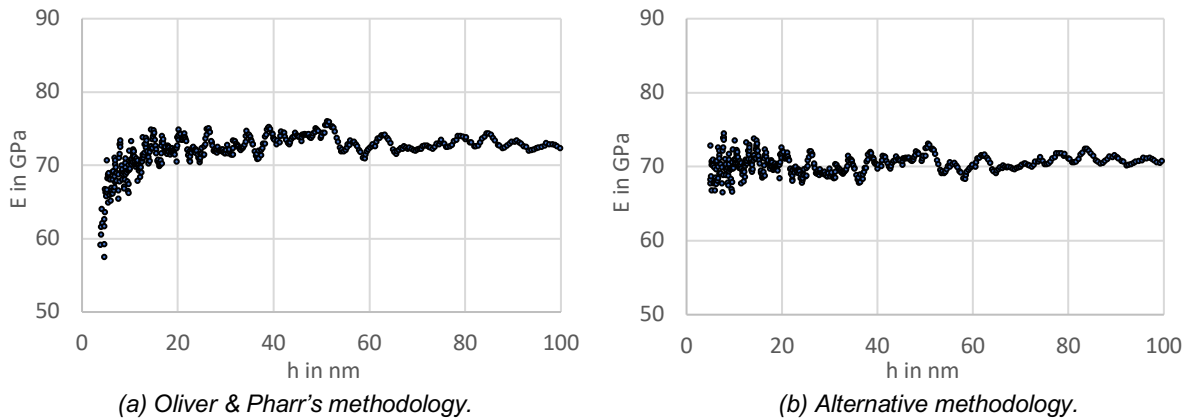


Figure 1. Young’s modulus (E in GPa) obtained by applying the methodologies proposed by Oliver and Pharr (a) and in this work (b).

Table 1 collects the mean values calculated with more than 1000 values of the Young’s modulus (E) and instrumented hardness (HIT) by calibrating different indenter tips used between 2012 and 2022 in the MSMP laboratory. Note that the alternative methodology allows the determination of the mechanical properties either from the two slopes of FF and AF functions or, similarly to the Oliver & Pharr’s method, from the computation of an average value considered between two arbitrary indentation depth limits; here for the two methodologies we have considered the same lower and upper limits, i.e. 100 nm and h_{max} (mostly 2000 nm) respectively.

Table 1. Means values of the Young’s modulus (E in GPa) and the instrumented hardness (HIT in GPa) calculated from the slopes of FF and AF functions or mean values between 100 nm and h_{max} .

	Oliver & Pharr		Alternative methodology		Alternative methodology	
	Mean values between 100 nm and h_{max}		Mean values between 100 nm and h_{max}		From FF and AF functions	
	E (GPa)	HIT (GPa)	E (GPa)	HIT (GPa)	E (GPa)	HIT (GPa)
Mean value	71,7	9,7	71,5	10,1	71,2	10,1
STDEVA	1,0	0,3	1,5	0,3	1,5	0,3

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Oliver Warren C., Pharr George M. An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments. *Journal of Materials Research*, 1992, 7, p. 1564-1583.

Resolution limits of hardness mapping

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ABSTRACT

Indentation experiments are simple, cost effective, and reproducible in comparison to tensile or compression testing. Indentation moduli measured on a homogenous material result in a standard deviation of around 1% at 10 nm penetration depth. These experiments can also be automated whereby hundreds of tests can be performed on a single sample. Moreover, the ability to test at very small penetration depths allows testing of thin films or regions of the sample microstructure independently.

Modern nanoindentation allows repeated control of maximum forces of a single μN or indenter displacement of single nanometers of depth. Even for very sharp diamond indenter tips and soft metals the transition from the elastic loading regime to plastic deformation can be studied and indentation tests can be performed just with the apex of the tip. The large ratio of Reduced Modulus (E_r)/Hardness(H) in metals will allow an early onset of plastic deformation which is supportive for measuring mechanical properties at the very early stage of deformation. When this indenter is combined with a piezo scanner these indents can also be spaced repeatedly at distances of single nanometers which gives access to measurement hardness maps with very high spatial resolution. It will be demonstrated that spacings of indentation tests of 50nm performed with the apex of a very sharp tip are practically manageable.

The focus here is twofold. First, an investigation of the theoretical and experimental limits of spatial resolution for the testing of multiple phases in a heterogeneous material, and secondly provide experimental strategies for operation.

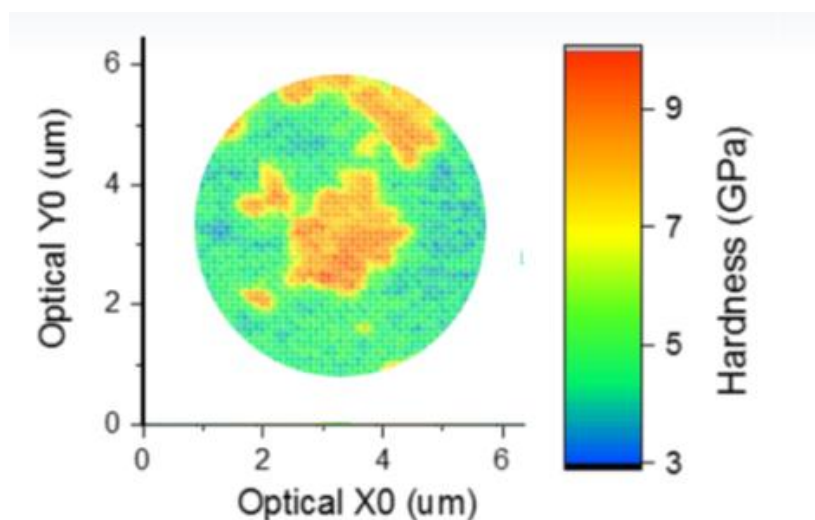


Figure 1. In-situ XPM mapping – crystalline phase in metallic glass; spacing of indentation tests 100nm

KEYWORDS: Hardness Mapping, Stress-Strain-Analysis, XPM, radius of indenter tip

Enriching nanoindentation with real-time electrical measurements and in-situ SEM monitoring

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ABSTRACT

Nanoindentation is a well-known technique dedicated to the local and quantitative mechanical testing of materials at nanoscales [1-3]. In the last decades, numerous efforts have been made to expand the capabilities of this technique [4]: real-time imaging (in SEM or TEM), high temperature nanoindentation, coupling with electrical measurements,... The latter development (usually referred to as ‘nano-ECR’, ‘resistive-nanoindentation’ or ‘electrical-nanoindentation’) was initially driven by a wide spectrum of motivations such as the local monitoring of phase transformation, the study of native oxide fractures, the characterization of piezoelectric materials,... However, the quantitative processing of experimental data remains delicate, and most of the reported works focus on metals [5] (the characterization of dielectric films other than piezoelectric materials is rarely addressed). In addition, even though the real-time imaging of nanoindentation tests has been widely reported, only few attempts have been made to combine both electrical measurements and real-time observations in-situ SEM [6,7,8].

The present talk will be structured in 2 complementary parts:

- First, the ability of this electrical/mechanical coupling to help the processing of raw nanoindentation data will be discussed. For instance, the monitoring of mechanical contact area will be shown on model and complex systems (Au and multiphased metallic alloys), with the support of SEM observations. The validity domains of this ‘electrical-nanoindentation’ method will be discussed, as well as the possible simultaneous extraction of both mechanical (modulus and hardness) and electrical magnitudes (local conductivity). Prospects on this topic will also be given.
- Then, ‘electrical-nanoindentation’ will be shown to be a precious tool to better understand the interplay between functional and structural properties of dielectric materials. For instance, it will be shown that the injection of electrical charges may induce the mechanical collapse of dielectric films, probably due to redox reactions that affect the atomic skeleton of the film. Finally, it will be shown that the plastic deformation of dielectric films can modify the conduction mechanism, probably through the build-up of a distributed space charge. On the last example, with the assistance of numerical modelling, a strain-dependent factor has been added to the standard Poole-Frenkel law to describe the dependence of leakage current with plastic deformation (Figure 1).

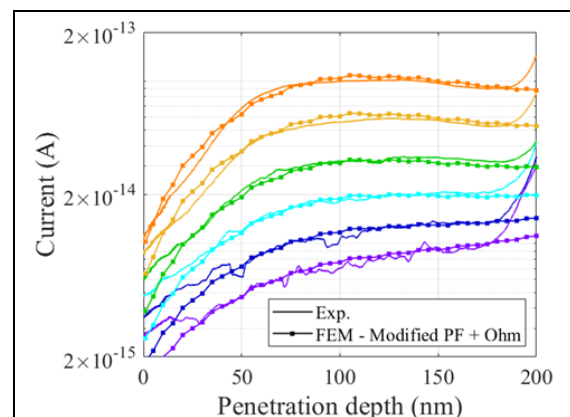


Figure 1. Evolution of leakage current during indentation under different voltages. Experiments and FEM modelling [9].

KEYWORDS

In-situ nanoindentation, electrical measurements

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Macro-, Micro- and Nanomechanical Characterization of Crosslinked Polymers with Very Broad Range of Mechanical Properties

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RÉSUMÉ

The present work focuses on the comparison of macro-, micro- and nanomechanical properties of a series of eleven highly homogeneous and chemically very similar polymer networks, consisting of diglycidyl ether of bisphenol A cured with diamine terminated polypropylene oxide. The main objective was to correlate the mechanical properties at multiple length scales, while using very well-defined polymeric materials.

By means of synthesis parameters, the glass transition temperature (T_g) of the polymer networks was deliberately varied in a broad range and, as a result, the samples changed their mechanical behavior from very hard and stiff (elastic moduli 4 GPa), through semi-hard and ductile, to very soft and elastic (elastic moduli 0.006 GPa). The mechanical properties were characterized in macroscale (dynamic mechanical analysis; DMA), microscale (quasi-static microindentation hardness testing; MHI) and nanoscale (quasi-static and dynamic nanoindentation hardness testing; NHI). The stiffness-related properties (i.e., storage moduli, indentation moduli and indentation hardness at all length scales) showed strong and statistically significant mutual correlations (all Pearson's correlation coefficients $r > 0.9$ and corresponding p -values < 0.001).

Moreover, the relations among the stiffness-related properties were approximately linear, in agreement with the theoretical prediction. The viscosity-related properties (i.e., loss moduli, damping factors, indentation creep and elastic work of indentation at all length scales) reflected the stiff-ductile-elastic transitions. The fact that the macro-, micro- and nanomechanical properties exhibited the same trends and similar values indicated that not only dynamic, but also quasi-static indentation can be employed as an alternative to well-established DMA characterization of polymer networks.

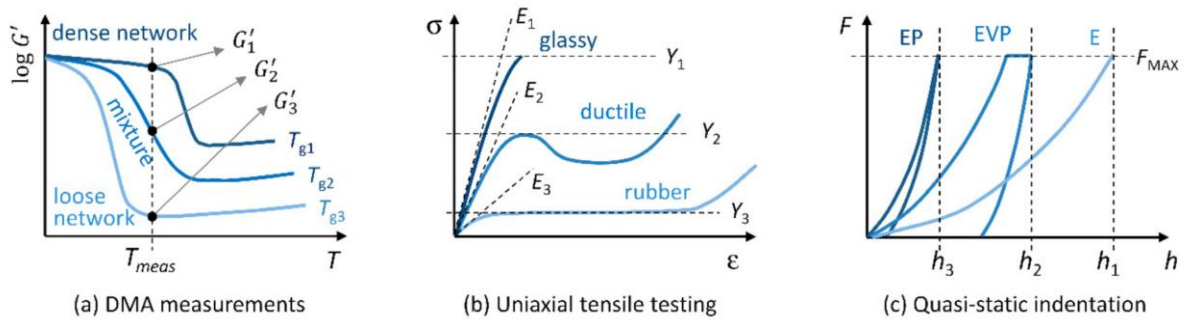


Figure 1. The scheme showing how the change of crosslinking density influences properties of crosslinked polymers measured by (a) DMA, (b) uniaxial tensile testing and (c) quasi-static indentation. The polymer with the highest crosslinking density (and the highest T_g) is marked with the darkest blue color, the polymer with the lowest crosslinking density (and the lowest T_g) is marked with the lightest blue color, and the mixture of the two polymers (with intermediate T_g) is marked with medium blue color.

MOTS CLÉS

Microindentation, nanoindentation, crosslinked polymers, stiffness

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Vieillissement par thermo-oxydation des polymères exposés à des écoulements rapides. Caractérisation par nanoindentation.

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RÉSUMÉ

Les matériaux composites à matrice polymère (PMC) sont de plus en plus utilisés et notamment dans des conditions environnementales difficiles. Il est alors nécessaire de maîtriser le processus de vieillissement du composite. Ce travail aborde un type de vieillissement environnemental : la thermo-oxydation. C'est le processus de diffusion-réaction de l'oxygène au sein du réseau moléculaire de la matrice polymère à haute température (autour de la température de transition vitreuse) [Shin et al., 2020]. Ce phénomène est strictement lié aux conditions environnementales (température, pression partielle d'oxygène, ...) et conduit à une dégradation du matériau, une fragilisation et une rupture prématurée [Audouin et al., 1994].

Lors de cette oxydation, une couche oxydée d'une épaisseur variable (de l'ordre de quelques centaines de micromètres) se forme dans le matériau [Zhang et al., 2016]. Ce gradient d'oxydation est le siège d'un gradient de propriété mécanique qu'il convient de caractériser expérimentalement. La nanoindentation se révèle être une excellente technique pour suivre le processus d'oxydation in-situ et notamment la rigidification du matériau dans les zones oxydées [Olivier et al., 2008, Gigliotti et al., 2016].

Dans ce travail expérimental, une pointe sphérique de 5 μm de diamètre indente le matériau avec une force maximale de 5 mN. Le module élastique d'indentation est extrait avec la méthode d'Oliver et Pharr [Oliver and Pharr, 2004]. La figure 1 présente en arrière plan une image d'une couche oxydée avec un réseau d'empreintes de nanoindentation. Un profil spatial du module élastique d'indentation (moyenne de 10 mesures) y est superposé afin d'illustrer la variation de cette propriété mécanique en fonction du niveau d'oxydation.

Dans ces nombreuses utilisations industrielles, les PMC sont soumis à des conditions aérothermiques de vitesses et de températures très importantes. Toutefois, jusqu'à présent, la thermo-oxydation des PMC n'a été étudiée qu'en effectuant des tests de vieillissement en laboratoire dans un four, c'est-à-dire en reproduisant artificiellement les conditions environnementales externes mais sans tenir compte de l'environnement aérothermique rencontré par certaines structures. Le défi est donc de caractériser le vieillissement thermo-oxydant d'un PMC soumis à un flux aérothermique important et d'étudier l'influence de ces conditions sur son vieillissement. Dans ce cadre, une température d'écoulement de 150°C et une vitesse d'écoulement transsonique (Mach 0,9) ont été appliquées comme conditions environnementales dans la soufflerie d'essais BATH de l'institut P'. Des mesures de référence en four (vitesse d'écoulement nulle) ont également été réalisées et des comparaisons pourront alors être faites.

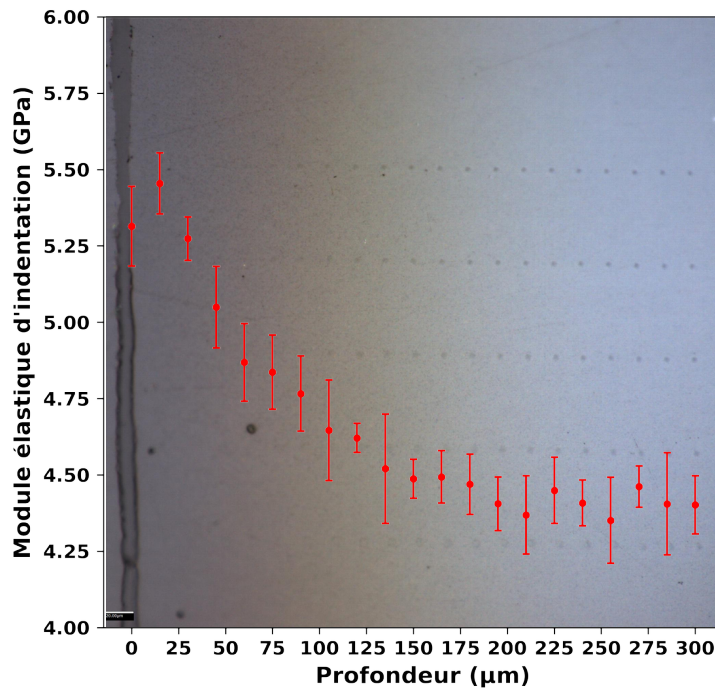


Figure 1. Mesure de module élastique d'indentation dans une couche oxydée vieillie à 150°C en four.

MOTS CLÉS

Thermo-oxydation ; Epoxy ; Couche oxydée ; Caractérisation expérimentale

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Étude par nanoindentation et traction uniaxiale du vieillissement thermique d'un PDMS chargé

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RÉSUMÉ

La technique d'indentation instrumentée, ou nanoindentation, s'est de plus en plus démocratisée durant ces 30 dernières années et s'impose actuellement comme une méthode de choix pour caractériser les propriétés mécaniques aux échelles micro et nanométriques. Cette technique a été développée initialement pour sonder les propriétés de matériaux au comportement élasto-plastique tel que la silice fondue ou l'aluminium. Récemment, le champ des matériaux à caractériser par nanoindentation s'est élargi aux polymères, et plus particulièrement aux élastomères [1-4].

Les matériaux élastomères sont connus pour leurs propriétés amortissantes, leurs capacités à s'allonger fortement de manière réversible et, dans le cas du polydiméthylsiloxane (PDMS), son excellente résistance thermique. Afin d'augmenter la rigidité de ces matériaux extrêmement souples, des particules renforçantes sont rajoutées à la matrice avant l'étape de réticulation. Dans le cas de la présente étude, il s'agit de deux populations de particules, SiO_2 et CaCO_3 .

L'utilisation de joints élastomères dans les milieux industriels au sein d'assemblages structuraux s'avère être un choix pertinent compte tenu de leur bonne coulabilité avant réticulation. Cependant, la caractérisation des propriétés mécaniques par méthodes conventionnelles (traction, compression) est difficile compte tenu des géométries complexes dans lesquels ils sont utilisés. La nanoindentation s'avère ainsi une excellente alternative à ces méthodes.

L'étude se porte sur l'évolution des propriétés mécaniques d'un joint élastomère confiné entre une structure en titane et un polymère, soumis à un vieillissement thermique. Les vieillissements appliqués sont échelonnés par incrément de 10°C entre 40°C et 70°C . Cette plage de température se trouve dans une gamme inférieure à la température de réversion de la réaction de réticulation. Les durées de vieillissement varient entre 10 et 200 jours selon la température de vieillissement considérée.

La caractérisation des propriétés mécaniques par nanoindentation est réalisée sur des échantillons de PDMS libres et confinés. Plusieurs protocoles sous chargement monotone et cyclique sont appliqués afin d'étudier les différents comportements exhibés par le matériau de l'étude en fonction de son vieillissement (Figure 1).

Des essais de traction sous déplacement monotone sont également réalisés afin de discriminer les phénomènes de viscosité, d'endommagement et de rigidification du matériau suivant les différentes températures et durées de vieillissement.

Ces essais à l'échelle macroscopique permettent de mieux comprendre les phénomènes observés via les essais de nanoindentation.

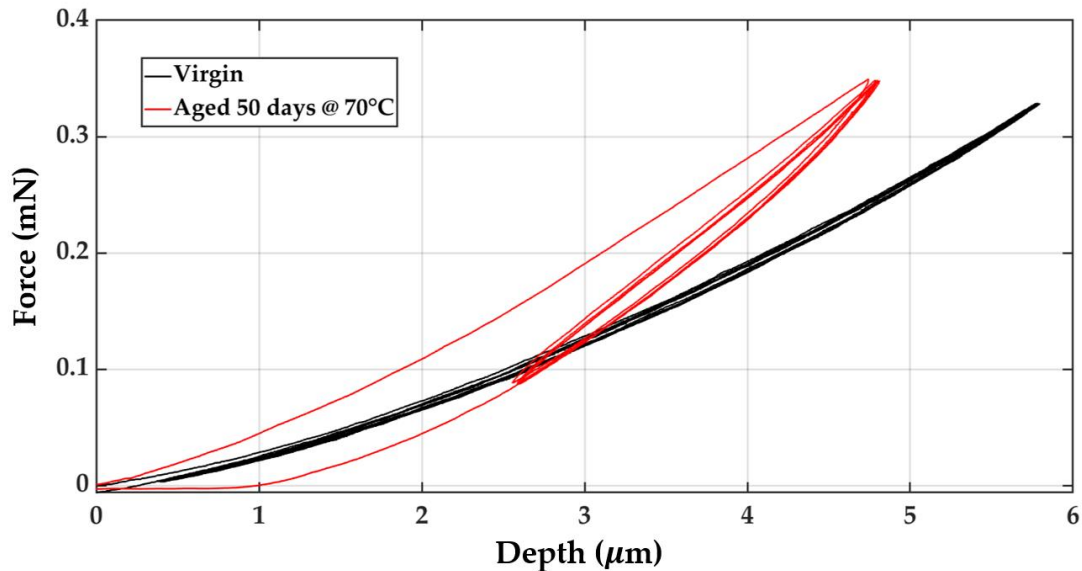


Figure 1: Courbes d'indentation du matériau non vieilli et vieilli à 70°C pendant 50 jours suivant un protocole cyclique

MOTS CLÉS

Indentation, élastomères, vieillissement thermique

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Apport de la nanoindentation et de l'AFM pour la compréhension de la mécanique multi-échelle d'un polypropylène isotactique

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RÉSUMÉ

Le polypropylène isotactique (iPP), est un polymère thermoplastique semi-cristallin qui est très largement utilisé depuis plusieurs décennies dans tous les secteurs industriels: de l'emballage à l'aéronautique en passant par le secteur médical.

A l'échelle nanométrique, l'iPP est caractérisé par la présence de deux phases - la phase cristalline et la phase amorphe, qui présentent toutes deux des propriétés mécaniques différentes. Ces phases sont présentes dans une organisation complexe de lamelles cristallines séparées par la phase amorphe. À l'échelle microscopique, les phases cristalline et amorphe forment une superstructure pseudo-sphérique appelée «sphérolite».

Bien que ces matériaux aient été largement étudiés à l'échelle macroscopique pour des trajets de chargement bien définis, leur comportement mécanique soulève encore de nombreuses questions. D'une part, le comportement de ce matériau à des échelles inférieures telles que l'échelle sphérolitique ou encore l'échelle des lamelles cristallines, n'est pas bien connu. D'autre part, peu de liens explicites entre microstructure et propriétés mécaniques ont été établis dans la littérature. Ceci est principalement dû à un accès expérimental délicat à ces échelles sub-micrométriques et à un manque de connaissance de la structure sphérolitique très complexe. Toutefois, quelques études mécaniques quantitatives existent [1,2].

Afin d'apporter un nouvel éclairage sur le comportement mécanique au sein d'un sphérolite, dans cette étude, un échantillon épais d'iPP avec une morphologie sphérolitique à la surface a été préparé à partir de granulés préalablement fondus dans un moule, puis poli et refondu. La microscopie à force atomique (AFM) et la microscopie optique ont été utilisées pour fournir des informations sur la morphologie et la topographie de la surface des sphérolites. La nanoindentation quant à elle a été utilisée afin de fournir des valeurs quantitatives de module et de dureté à l'échelle intra-sphérolitique. Ainsi, plusieurs cartographies de propriétés mécaniques ont été réalisées à partir de matrices d'indentation (pas $\delta_{x,y} = 3 - 5 \mu\text{m}$ et profondeur d'indentation $h_{\text{max}} = 180 - 200 \text{ nm}$) avec un indenteur Berkovich rigoureusement calibré. Les résultats démontrent que les propriétés mécaniques sont hétérogènes au sein d'un sphérolite. Il a de plus été montré que les valeurs de module peuvent directement être corrélées à la croissance sphérolitique - les régions correspondant à la fin de la cristallisation (extrémités des branches et zone de fermeture du sphérolite)

ont les valeurs les plus basses, tandis que celles correspondant aux premiers stades de croissance (zone centrale) ont les valeurs les plus élevées.

Afin d'avoir une caractérisation mécanique plus fine, des cartographies de module d'élasticité par AFM en utilisant la théorie du contact de Hertz (PeakForce QNM®) ont été réalisées. Contrairement à la nanoindentation, cette technique présente l'avantage d'avoir une grande résolution spatiale tout en sollicitant le matériau de manière élastique ($R_{\text{contact}} = 10 - 15 \text{ nm}$, $h_{\text{max}} = 2 - 5 \text{ nm}$). Les premiers résultats se montrent cohérents et complémentaires de ceux obtenus par nanoindentation et offrent un éclairage plus fin des hétérogénéités intra-sphérolitiques.

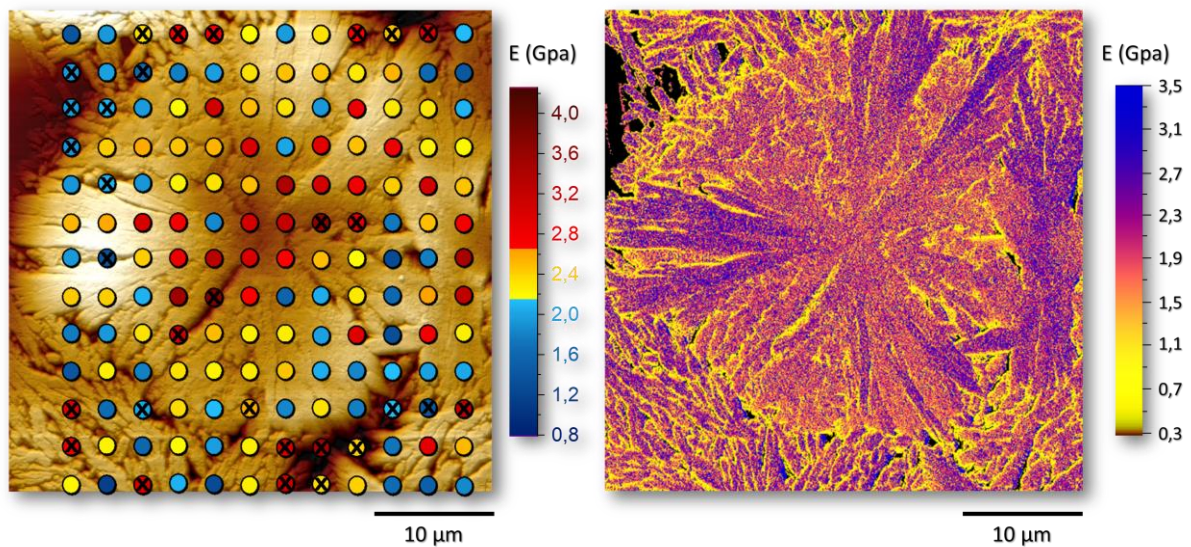


Figure 1. A gauche : Image AFM 3D en vue de dessus et cartographie de module obtenue par nanoindentation. Les données surplombées d'une croix correspondent à des indents étant impactés par la topographie locale. A droite : Cartographie de module de la même zone obtenue par AFM.

MOTS CLÉS

polymères semi-cristallins ; sphérolite ; nanoindentation ; AFM ; nano-mécanique

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Multi-scale characterization of the (visco)elastic properties of a semicrystalline polymer

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ABSTRACT

The high degree of heterogeneity of polymeric materials makes it difficult to describe their overall mechanical behavior. An access to information on a smaller scale is thus essential. Moreover, the mechanical response of these materials strongly depends on the microstructure developed during process especially for semicrystalline polymers. With the idea to better understand the relationships between structure and mechanical properties it is becoming necessary to develop multi-scale mechanical characterizations and instrumented indentation proves to be the most appropriate tool.

Initially developed for the study of metallic and ceramic materials, it appeared that its application to polymeric materials presented some difficulties because of the viscous nature of these materials. Thus, as regards the study of viscoelastic properties by instrumented indentation, two complementary paths can be adopted: static nanoindentation (quite commonly used) and dynamic nanoindentation. Nevertheless, rigorous protocols remain to be developed to study properly time dependent materials.

The aim of this work is to compare the local mechanical properties of a polypropylene obtained by compression molding and injection. The latter is known to exhibit a skin-core morphology [1] with a nonspherulitic skin and a typically spherulitic core. Afterwards, the viscoelastic properties are investigated at different scales using both dynamic nanoindentation [2] and dynamic mechanical analysis (DMA). Experiments were performed at temperatures between room temperature and 120°C and various frequencies over the range 0.1 – 100 Hz.

Regarding quasi-static indentation, the characterization of the local properties through the sample thickness highlights the heterogeneity of the morphology in the injected sample with a skin layer softer than the core whereas the compression-molded sample displays more homogeneous properties. If the elastic properties are different in the injected sample through the thickness, the frequency dependence is similar and no skin-core effect is highlighted.

From the nano-DMA results, it was possible to construct a master curve as it commonly done from DMA experiments for the injected polypropylene as illustrated in Figure 1. The frequency tests have been performed on the skin. The evolution of the storage

modulus is similar at the two scales with an increase with the frequency. The Arrhenius equation allows to determine the apparent activation energy for the relaxation process. This energy is in good agreement with values from literature regarding macro DMA investigations.

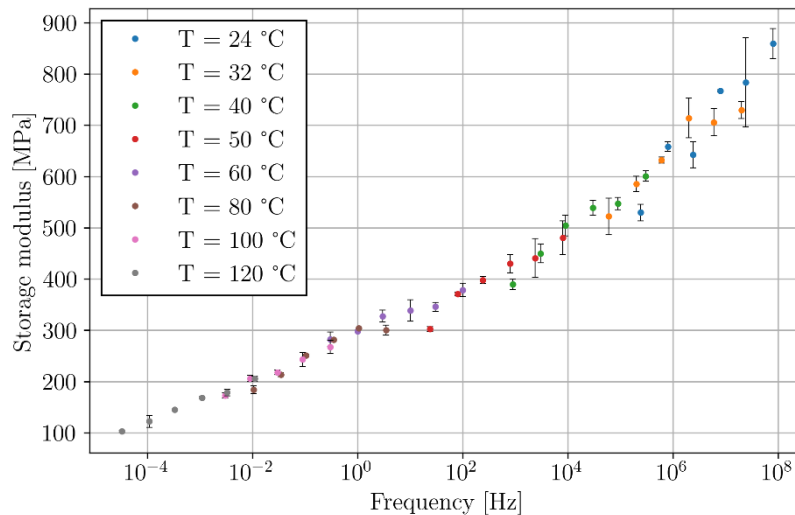


Figure 1. Master-curve constructed for a reference temperature of 60°C from nano-DMA results.

Future works will be devoted to test this method on softer polymers since it seems that nano-DMA overestimated the complex modulus by a factor two [2] when the modulus is of the order of MPa.

KEYWORDS

indentation ; semicrystalline polymer ; microstructure ; viscoelastic properties ; nanoDMA

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CONTACT MECHANICS OF PRE-ORIENTED AMORPHOUS POLYMERS

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ABSTRACT

Large strain involved during polymer processing can induce the orientation of macromolecular chains of the amorphous polymers along a preferred direction. The resulting mechanical properties will thus be dependent on the direction of load application, i.e. they are anisotropic. As a consequence, the material performances, can differ from the non-oriented isotropic materials. For that reason, since the late '60s, several studies were devoted to the characterization and modelling of the mechanical behaviour of oriented polymers [1-3]. These studies were mainly focused on the understanding of the link between structure and bulk and failure properties, while the link between structure and surface properties is still lacking. In this work, we have explored the effects of strain induced pre-orientation on contact mechanics problems through an approach which combines bulk compression tests, in-situ indentation experiments and finite element modelling.

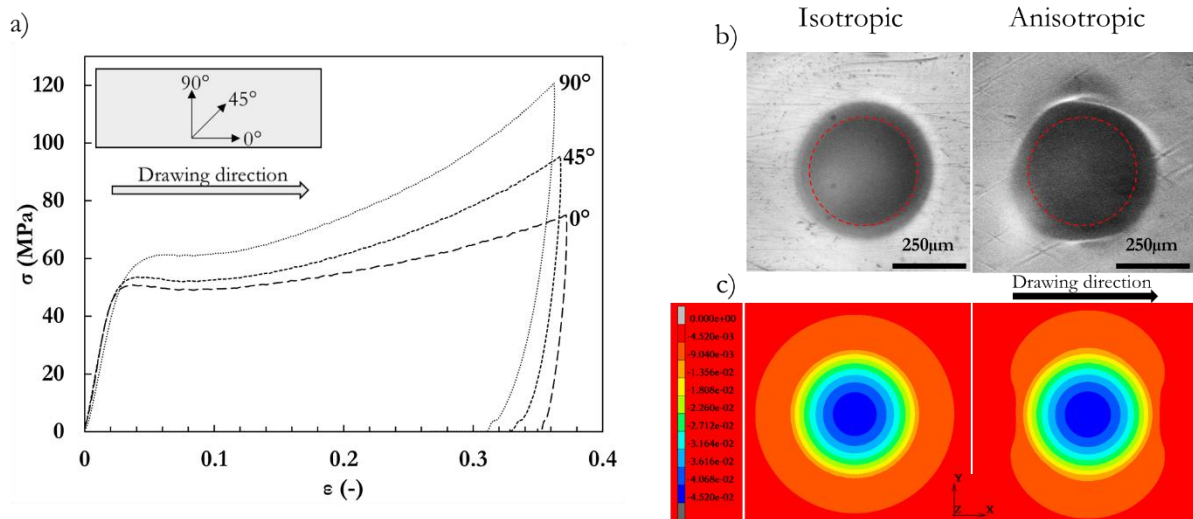


Figure 1. (a) Compression stress-strain curves anisotropic PC (stretch ratio $\lambda=1.75$) tested at 0°, 45° and 90° from stretching direction. (b) In-situ observations of the contact areas during indentation with a spherical tip ($R=500\mu\text{m}$) and an applied normal load of 15N on isotropic (left) and anisotropic (right) PC samples. (c) Vertical (z) displacement in mm resulting from finite element simulation of spherical indentation ($R=500\mu\text{m}$) for a 15N applied normal load in case of isotropic (left) and anisotropic (right) elastic-perfectly plastic rheology ($\frac{E}{\sigma_y} = 38$).

The material selected for this study is the Polycarbonate (PC) which, thanks to its ductility, allows for a strain induced pre-orientation in bulk form and thus enables the machining of samples for mechanical characterisation after orientation.

We have then carried out bulk compression tests on both isotropic and anisotropic PC. Anisotropic samples have been machined from stretched bars ($\lambda=1.75$) at different angles from the stretch direction: 0° , 45° , and 90° . Nominal stress-strain curves (Figure 1a) show that pre-orientation induced anisotropy in the plastic response, visible in the yield stress and strain hardening. However, all the curves superpose in the elastic region, showing that pre-orientation does not affect measurably the Young's modulus. Indentation tests have been performed on our home-made experimental device which, thanks to a camera placed below the sample, allows in situ recording of contact surfaces during the tests through transparent materials. The contact surfaces during spherical indentation ($R=500\mu\text{m}$) for an applied normal load of 15N on isotropic and anisotropic PC are depicted in Figure 1b in which the contact area has been circled in red. The in-situ pictures highlight a direction dependency of the sinking-in around the tip in the case of anisotropic sample. This observation was confirmed by our finite element simulation of spherical indentation ($R=500\mu\text{m}$, $F_n=15\text{N}$), as illustrated in Figure 1c by the vertical displacement (along z), in which the parameters of the anisotropic Hill's flow rule [4], identified from the compressive yield stress values at different orientation angles were implemented. In such simulations, the post-yield behaviour has been assumed as perfectly plastic. We are currently working on a more complex model able to capture the direction dependency of the post-yield behaviour (strain hardening).

KEYWORDS

indentation; amorphous polymers; anisotropy; finite element modelling

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Stiffness of carbon black-filled polychloroprene after oxidative ageing: three experimental techniques

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ABSTRACT

Elastomer durability is a crucial issue to develop parts that withstand long-term environmental exposure. In marine renewables, the target lifetime of systems can reach more than 20 years, thus it is essential to understand the effect of ageing on the mechanical behaviour of materials.

Carbon black-filled polychloroprene (CR-CB) is a material widely used for this purpose because of its high extensibility and fatigue properties. When exposed to thermo-oxidative ageing, it has been shown that CR crosslink density increases because of radical reactions on the polymer alkene links [1]. This results in a consequent stiffening of the material. In this paper, we investigate and compare three experimental techniques to measure the stiffness of a filled CR-CB after ageing.

Practically, ageing is performed in air convection ovens, for five ageing conditions. Then, stiffness is measured using the following experimental techniques:

- **Tensile tests** are performed on aged samples. Mooney-Rivlin parameters are used to calculate the Young modulus.
- **Dynamic Mechanical Analysis** is used in strain sweep mode at 2 Hz frequency to determine the storage modulus of the material, E' .
- **Nanoindentation** is performed on a Bruker TI 980 machine, with a Berkovich tip. Experimental protocol is adapted from Fradet et al. [2], and Young modulus is calculated according to Oliver & Pharr [3]. Indentations are made both on the material surface and at the material core.

The corresponding values of the Young modulus are presented in Figure 1. Tensile tests and DMA clearly highlight the stiffening of the material with ageing, most of DMA values being higher than tensile tests ones because of the Payne effect. It is consistent with nanoindentation values and previous experiments [4]. Moduli measured by nanoindentation are uniform within the sample thickness up to 100°C, with satisfactory deviation for a filled elastomer. At 140°C, significant differences between the material surface and core are observed (factor 2). At this temperature, nanoindentation exhibits diffusion-limited oxidation profiles [5] that will be further examined in future investigations.

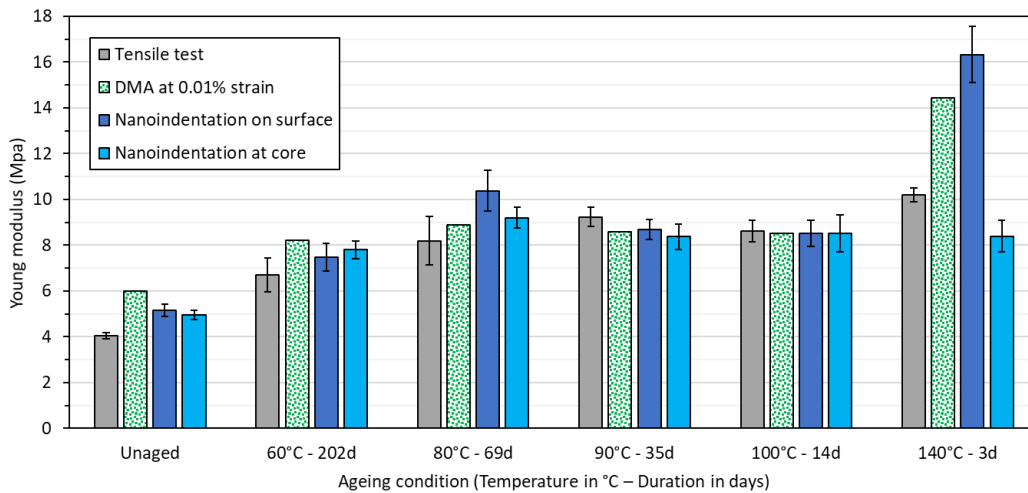


Figure 1. Young modulus of aged CR samples measured with different experimental techniques

KEYWORDS

Carbon black-filled polychloroprene, Ageing, Stiffness, Nanoindentation

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A METHOD TO DETERMINE ANISOTROPIC ELASTIC CONSTANTS IN A β -METASTABLE TITANIUM ALLOY BASED ON NANOINDENTATION

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ABSTRACT

Determining intrinsic properties such as the **anisotropic elastic constants** of a polycrystalline multiphase material is a real challenge. The latter properties are very important in order to model accurately internal stresses or to estimate local stresses from elastic strain measurements. This is especially hard in near β -Titanium alloys as it is impossible to produce a single crystal of β -phase at the same temperature and chemical composition as it exists in the α - β alloy. Instrumented spherical indentation solves this problem as it enables us to characterize every individual grain and obtain their orientation-dependent properties, such as the anisotropic indentation modulus E^* . Using our ultra-high resolution UNHT nanoindenter manufactured by Anton Paar, we are going to couple the local mechanical responses and the characterized material surface (EBSD) to compute E^* . This will allow us to use Bayesian inference to statistically extract the elastic constants by comparing our experimental results with the ones obtained by the theoretical framework developed by Vlassak and Nix^a. We will then be able to create detailed maps of the microstructure that characterize the elastic heterogeneity of our material. Before determining the elastic constants of a range of near β -Titanium alloys, we will first develop and test our approach on pure Nickel because it is a well-documented material with known elastic properties. The anisotropic indentation modulus of each grain can be calculated by fitting the initial elastic part of the indentation Force (P) – Depth (h) curve to Hertz's theory of contact^b:

$$h - h^* = k(P - P^*)^{\frac{2}{3}}, \quad k = \left[\frac{3}{4} \frac{1}{E_{eff}} \frac{1}{\sqrt{R_{eff}}} \right]^{\frac{2}{3}}, \quad \frac{1}{E_{eff}} = \frac{1}{E^*} + \frac{1 - \nu_i^2}{E_i} \quad (1)$$

where R_{eff} , E_i and ν_i are the effective radius, Young modulus and Poisson's ratio of the diamond indenter, respectively. h^* and k are obtained by the best linear fit between h and $(P - P^*)^{\frac{2}{3}}$ using a range of values for P^* . Hence, it corrects the force and depth of contact by providing h^* and P^* as the corrections. E^* is then deduced from the value of k through the effective modulus E_{eff} .

Vlassak and Nix's theory is based on anisotropic Green's surface function that was derived by Barnett and Lothe using Stroh's formalism^b:

$$E^* = 4\pi \int_0^{2\pi} \mathbf{n}_i \mathbf{B}_{ji}^{-1}(\omega) \mathbf{n}_j d\omega \quad (2)$$

where \mathbf{n} is the normal to the indentation surface and \mathbf{B} is a symmetric tensor that depends on the elastic moduli tensor of the material and that is computed by solving an eigenvalue problem.

The method of Bayesian inference considers the comparisons between experimental values of E^* and the theoretical ones E_{theo}^* computed through Eq.(2) for a range of different values of elastic constants in order predict the elastic constants with a quantified level of uncertainty. Applied to our current problem, the Bayes' theorem reads:

$$P(E_{the}^* | E^*) = \frac{P(E^* | E_{the}^*) * P(E_{theo}^*)}{P(E^*)}$$

where P and $P(/)$ denote probabilities and conditional probabilities, respectively. Our approach differs from previously developed approaches^c in that they used reduced models based on finite elements simulations, which is computationally expensive and extensive, to get E_{theo}^* .

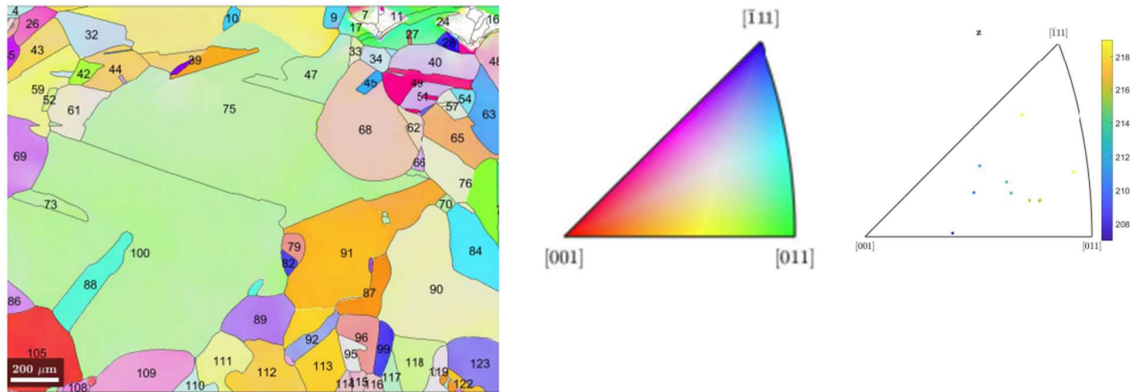


Figure 1. EBSD map of a pure Nickel sample, orientation IPF, and theoretical anisotropic modulus IPF

KEYWORDS

Near- β Titanium alloys, Pure Nickel, Nanoindentation, EBSD, Bayesian Inference

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On the identification of elastic-plastic thin film properties using numerical nanoindentation tests and finite element modeling (FEM)

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ABSTRACT

In this work, several numerical nanoindentation tests were conducted to study mechanical behaviour of thin films using Abaqus code. Numerical indentation methods were used to limit the substrate effect on determining intrinsic elastic-plastic properties of the films. The film's mechanical properties such as elastic modulus E , yield stress σ_y and strain hardening coefficient n were extracted using nanoindentation test and finite element modeling (FEM). The studied models were validated by comparing experimental and numerical results. Furthermore, the efficiency and accuracy of the numerical models was discussed using numerical verification. The mechanical properties of several elastic-plastic films deposited on elastic-plastic substrates were then identified using these models.

KEYWORDS

Thin films ; Nanoindentation ; Finite Element Modeling

Challenges in Small-Scale Testing of Energy Materials up to 1000°C

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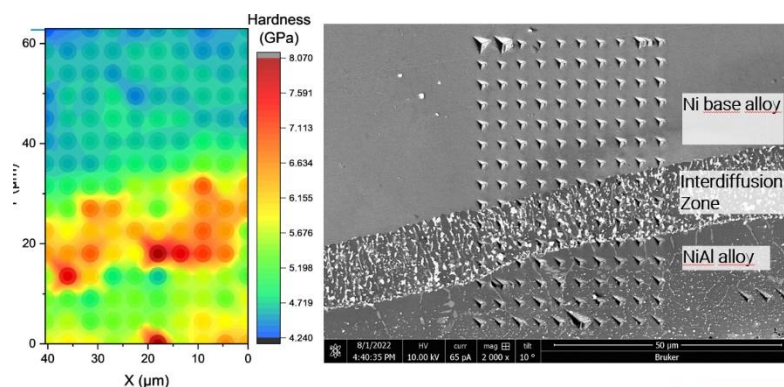
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ABSTRACT

The efficiency of energy conversion is improved by increasing the process temperatures. The increase of the process temperature also accelerates the creep, diffusion and corrosion processes which strongly depend on the temperature. Differences in the thermal expansion coefficient (CTE) induce internal stresses that can drive deformation processes – as well as the centrifugal forces found in turbines. It is therefore important to perform mechanical tests at process temperature level.

TRISO nuclear fuel product consisting of triple clad Pac pellets. In the center is a core of uranium (IV) oxide, or a uranium/thorium mixed oxide, clad with an inner layer of isotropic pyrographite, a layer of high-strength silicon carbide, and an outer layer of isotropic pyrographite. The silicon carbide layer acts as a diffusion barrier for some nuclides and has shown to be effective until 750°C. The temperature range of 950°C to 1000°C requires assessing the stability of the interfaces (graphite – silicon carbide – graphite) and the integrity of the silicon carbide layer at those temperatures. HT nanoindentation is the only way to determine the materials properties at these temperatures. It can be shown that the SiC hardness drops to a hardness of 10GPa at 1000°C at a controlled strain rate. The sample and tip stability and other limiting factors of these tests in vacuum in the SEM are discussed.

Thermal barrier coatings are used to protect nickel-based alloys in corrosive turbine environments. The nickel base structure is coated with an adhesive layer, which is then covered with the plasma sprayed thermal barrier coating that prevents corrosion and lowers the temperature of the turbine blade structure. The complex structure is tested at different high temperatures by XPM indentation mapping in the SEM to follow the changes in mechanical properties of the different microscale



elements of the thermal barrier coating system. Al is employed in the data analysis.

Figure 1. In-situ XPM mapping of the interface between Ni base alloy and NiAl bond coat at 600°C

KEYWORDS: 1000°C, indentation in SEM, XPM mapping, constant strain rate

Mechanical property changes across the spin transition in a molecular film probed by nanoDMA

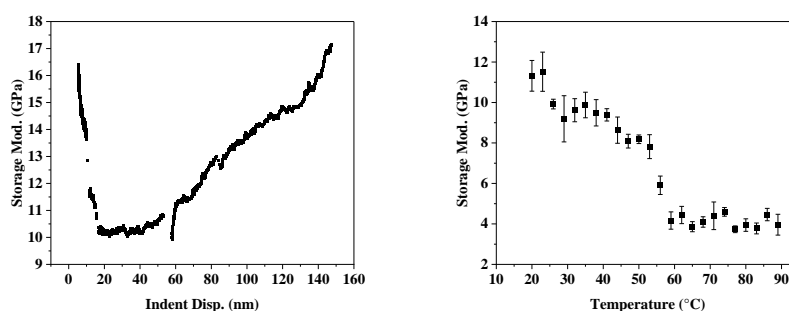
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ABSTRACT

The possibility of harnessing useful work from bistable molecular switches has inspired many scientists in the past two decades and led to the development of hundreds of compounds with a remarkable degree of sophistication [1]. In this context, we have shown recently that spin-crossover molecules of $[\text{Fe}(\text{HB}(1,2,4\text{-triazol-1-yl})_3)_2]$ (compound **1**), processed as thin films [2], can be used to actuate a silicon MEMS device, thus demonstrating good mechanical integration of the molecules to produce useful work under a controlled external stimulus [3]. The device also exhibits spin-state dependent resonance frequency and enhanced mechanical damping around the spin-transition temperature. To enhance the scope of these results, a detailed quantitative analysis of the mechanical properties and actuating performance of films of **1** becomes necessary. Viscoelastic property mapping by AFM revealed significant softening at the spin transition [4]. For a more quantitative assessment of this phenomenon, we conducted a variable temperature nanoindentation study on a ca. 1.3 μm thick film of **1** deposited on a silicon substrate. Continuous stiffness measurements (CSM) of the film give access to a continuous measurement of the mechanical properties through the depth of the sample (Fig. 1a), providing us with a tool that allows us to correlate mechanical properties to indentation depth and eliminate substrate and roughness effects. Using indentation parameters optimized by CSM, we conducted a nanoDMA study of the film between 20 and 95 $^\circ\text{C}$ with steps of 3 $^\circ\text{C}$ averaging 4 indentations at each step. The results reveal a pronounced and well-reproducible variation of the sample mechanical properties at the spin transition around 60 $^\circ\text{C}$. This is characterized by a drop of the storage modulus from 8.18 ± 0.2 GPa to 4.16 ± 0.4 and the hardness from 270 ± 11 MPa to 170 ± 22 MPa when going from the low temperature (low spin) to the high temperature (high spin) state (Fig. 1b-1c). In-situ scanning probe microscopy (SPM) images were also systematically acquired (Fig. 1d) in order to examine the surface features (e.g., pile-up, sink-in, roughness).



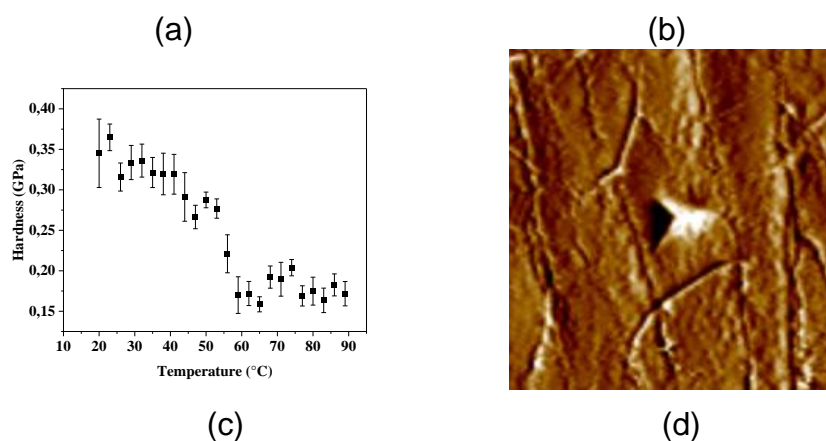


Figure 1. (a) Representative CSM curve of the film of **1** on silicon substrate. (b) Storage modulus (100 Hz) and (c) hardness as a function of the temperature for **1**. (d) Representative SPM image of an indent.

KEYWORDS

nanoindentation; nanoDMA; spin-crossover; elastic softening

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HOT HARDNESS AND YIELD STRESS MEASUREMENTS ON STAINLESS STEEL UP TO 700°C.

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ABSTRACT

In the aeronautical field, materials are used in severe environmental conditions (temperature, atmosphere), particularly for engine applications. In order to qualify new alloys compositions, ONERA is developing micromechanical characterization means to carry out nanoindentation tests from room temperature up to 700°C under conditions close to operating conditions.

This method presents the interest of performing tests faster than classical mechanical tests like tensile or bending tests under severe conditions and with small amounts of materials.

In order to carry out screening tests, a 316L stainless steel is studied and the evolution of hot Vickers hardness properties and yield stress versus temperature are presented.

By increasing the applied load from 0.1 to 20 Newton, we show that we can neglect surface microstructural changes or possible contamination of the sample surface by oxidation. We show that from 0.5 Newtons, the hardness measurement is independent of load on 316L stainless steel. By using Tabor's law to express the mechanical resistance [1], we show that the hardness decreases by 40% when the test temperature goes from 20 to 600°C, which is close to the supplier's values.

A discussion about these measurements which could be performed on many different materials such as layers, coatings, composite materials, brazing cords, additive manufacturing materials is presented. The technical limits of this technology [2][3] and new ways to increase the test temperature in order to produce results up to 1000°C are explained.



Figure 1. Hot hardness apparatus – Inside the vacuum chamber – sample and indenter furnaces –

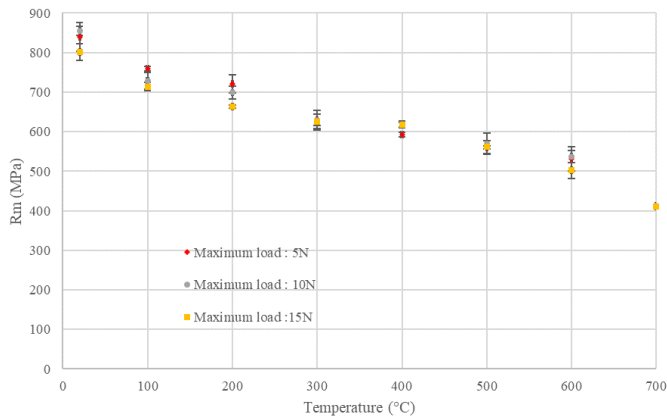


Figure 2. Hardness of stainless steel 316L versus temperature – Onera results

KEYWORDS

Hot hardness; indentation ; stainless steel ; mechanical resistance

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HIGH-TEMPERATURE SCANNING INDENTATION

Une nouvelle méthode pour étudier les changements microstructuraux en température

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RÉSUMÉ

Avec le développement de la méthode High-Temperature Scanning Indentation (HTSI), fondée sur un cycle d'indentation rapide spécifique, il est maintenant possible de déterminer l'évolution quasi-continue en température du module d'Young, de la dureté et des propriétés de fluage des matériaux et ce en quelques heures. Grâce à cette technique, on peut suivre les changements physiques ou microstructuraux des matériaux en température [1,2] à l'aide des évolutions des propriétés de dureté et de fluage au cours du cycle thermique appliqué.

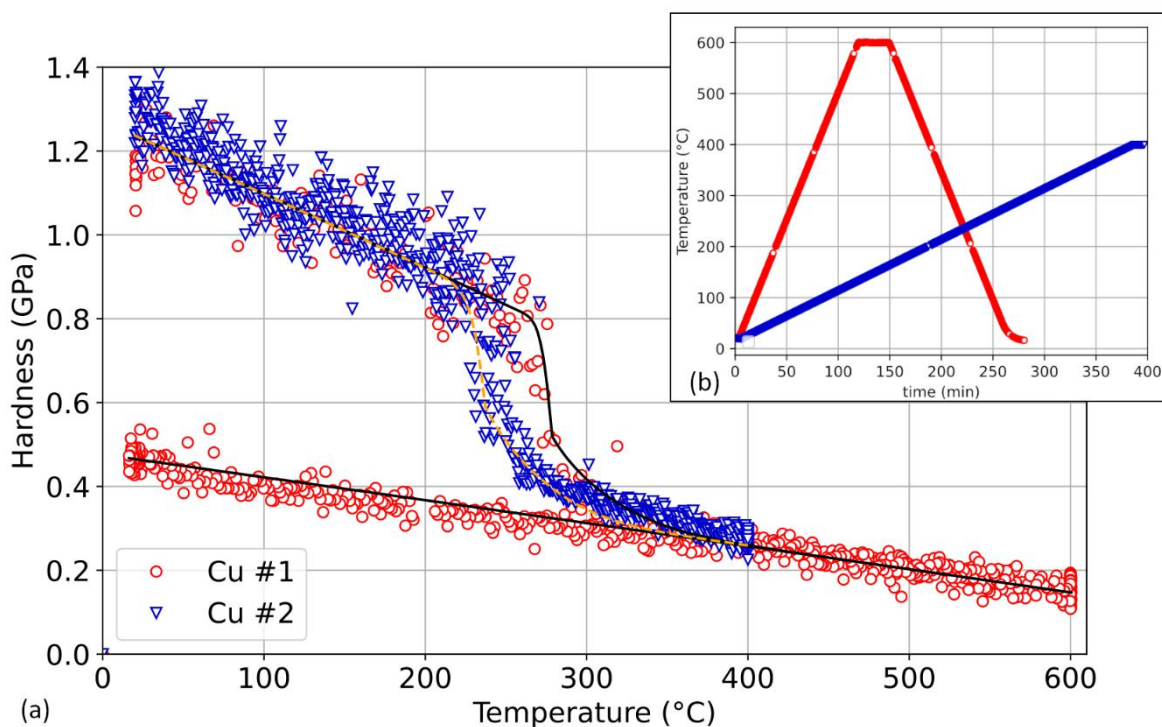


Figure 1. (a) Dureté fonction de la température d'échantillons de cuivre pur écroui, soumis aux cycles thermiques présentés en (b). Le modèle analytique permet de prédire l'évolution de la dureté en fonction du cycle thermique appliqué.

Dans cet exposé, on se concentrera sur l'étude d'échantillons de cuivre et d'aluminium purs, laminés à froid. L'évolution irréversible de la dureté de ces matériaux pendant le cycle thermique est liée aux processus de restauration statique et de recristallisation de leur microstructure. Afin de quantifier ces phénomènes, une modélisation est réalisée. Elle permet de déterminer leurs cinétiques. Les résultats obtenus (voir figure) sont cohérents avec les essais expérimentaux. On peut de plus observer l'impact de l'état de déformation initial des échantillons et du cycle thermique appliqué sur ces phénomènes. La comparaison avec les mesures EBSD post-mortem confirme les résultats obtenus par HTSI.

MOTS CLÉS

High-temperature nanoindentation, high-speed indentation cycle, anisothermal measurements, mechanical properties, microstructural changes

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Abstract

At the end of this work, it is important to note that the various aspects covered allowed to better understand the characteristics of marls and clays of post Miocene basin of Tizi Ouzou. The objective of our study is a mineralogical analysis of the marls and clays of five sites lying on an E-W direction line, which is located in the Tizi Ouzou basin which is part of the maghrebic chain.

We regroup under the Tizi Ouzou basin terminology all the Miocene subsoil formations which regroup the formations that outcrop in the region of Tizi Ouzou, but more broadly between the meridians of Thenia to the West, Azazga to the East and from Dellys to the North to the Kabyle Base to the South. Clays are sedimentary rocks composed of clay minerals with a low particle size between 2 and 4 μm , they can be studied by several methods, but the one we use is X-ray diffraction, because compared to other techniques, X-ray diffraction is a non-destructive technique that can allow us to recognize the symmetry of the crystal, determine the structure of its network parameters and identify a crystal. So it is to give us directly global characterizations and information on the crystalline structure of clay minerals. The results obtained following the processing of the samples by DRX allowed to characterize and identify the nature and mineralogical composition of each of them. And classified according to their content of calcite and clay minerals based on the Jung classification; most samples are classified as much as clay limestone has marl and clay-limestone. So it's concluded that the clays in the study area are essentially carbonates. It should start with a very brief background about why your study needs to be done, methods used, major results and implications of your research work.

Keywords: marls, clays, Miocene basin, DRX.

High Strain Rate Nanoindentation – Recent Advances and Perspectives

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ABSTRACT

The strain rate dependence of mechanical properties of materials, particularly at “high strain rates”, is of great interest for many applications including metal forming, machining, crashworthiness, projectile impact, etc. For strain rates above the quasi-static regime ($>1 \text{ s}^{-1}$), Split Hopkinson Pressure bar testing apparatus are most commonly used¹. While the uniaxial stress state in such experiments is desirable, the main drawbacks are the interference of elastic waves, low throughput, and extensive post processing of raw data. In view of these limitations, it is desirable to extend high strain rate capabilities of nanoindentation, thus taking advantage of relatively simple sample preparation, high-throughput testing capabilities and comparably small equipment.

This talk will focus on recent developments in instrumentation and testing protocols for high strain rate testing from 0.0001/s up to 20'000/s (8 orders of magnitude) with simultaneous high-speed actuation and sensing capabilities, with nanometer and micronewton resolution respectively. Moreover, we will elaborate on high strain rate testing performed at high and low temperatures as well as under variable humidity, and how the resulting multi-dimensional data is useful for understanding fundamental material behaviour and advancing engineering objectives. Specific case studies will include high strain rate nanoindentation at cryogenic temperatures and micro-compression experiments at elevated humidity. The challenges of such experiments and the associated technological and test protocol advances will be discussed along with select case studies.

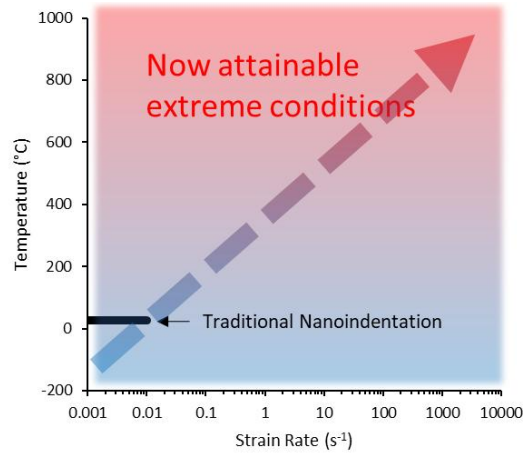


Figure 1. Schematic representation of extreme conditions at which nanoindentation can now be performed

KEYWORDS

Nanoindentation; High strain rate; Impact testing; Strain rate sensitivity

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Multi scale in-situ micromechanical tests for BGA interconnections

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ABSTRACT

In response to the demand for lead-free electronic equipment (due to the European directives), miniaturisation of the components and the need for a greater reliability, industry searches for new interconnections in microelectronics. New type of solder balls have been developed recently, Polymer Core Solder Balls (PCSB), which are multi material balls made of a polymer core covered with different metallic coating layers (Cu, Ni, Au...) (Figure1 a,b).

In this study, micromechanical tests have been conducted to analyse the behaviour of the solder ball PCSB at different scales. First, micro pillar compression tests have been performed to characterize the coatings mechanical properties (microscale) (Figure1 c). Then, the behaviour of the solder ball under compression was analysed (Figure1 d).

An ALEMNIS in-situ nanoindenter placed in a Zeiss Gemini supra 55VP Scanning Electron Microscope (SEM) was employed to carry out both compression tests. The size of the flat punch was of 5 μm for micro pillar, and 500 μm for the PCSB compression test.

In order to validate our experimental approach, finite element simulations were also performed.

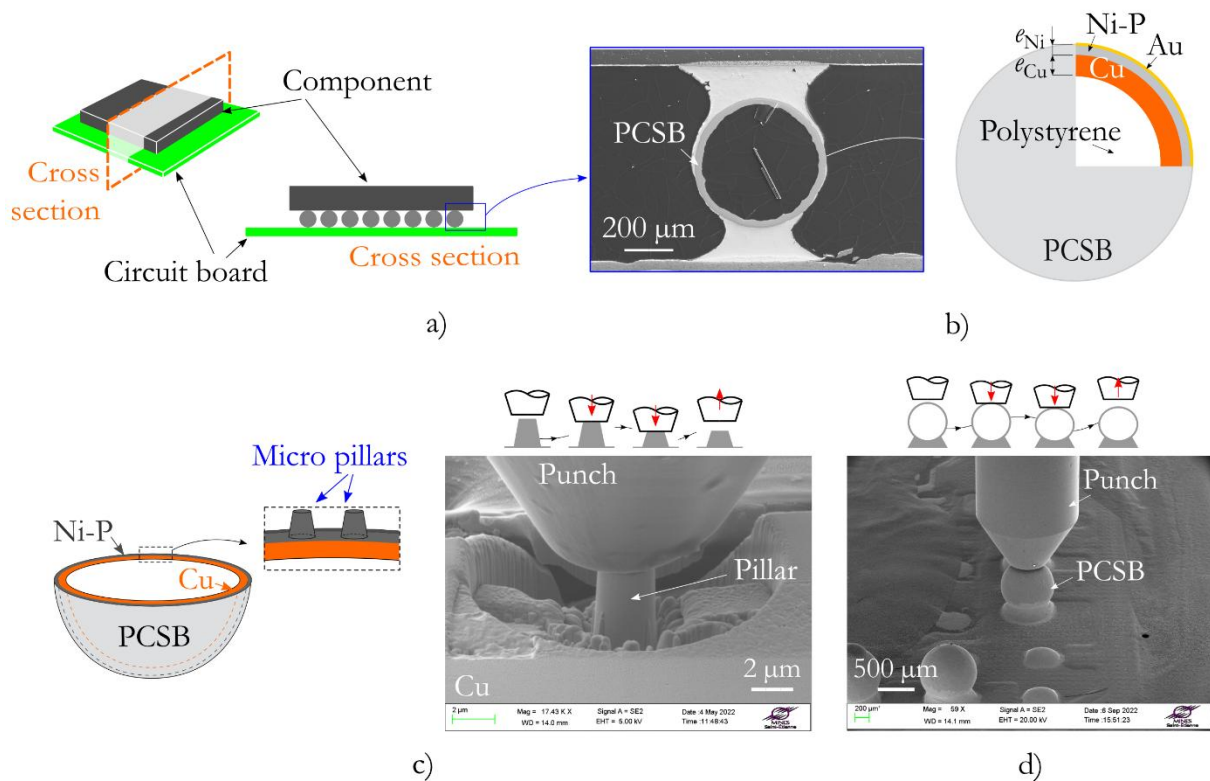


Figure 1. a) Description of a BGA interconnection with PCSB solder balls, b) detail of the coatings of the solder ball, c) micro pillar compression tests (microscopical scale) and d) PCSB compression tests (mesoscopical scale).

KEYWORDS

Solder ball; Coatings; Micro pillar compression; In-situ micromechanics; multi scale

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Building the nanoMECommons Database: a workflow for indentation data digitalisation to numerical simulations

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ABSTRACT

Nanoindentation is a widely used surface technique to measure mechanical properties of materials at small scale [1]. Recently, high-speed indentation is becoming more popular as real-time, in-line, and online testing are used to collect large datasets, on the load-depth response of materials, samples, and (also intermediate) products for quality control and quick manufacturing characterization of test specimens. Such approach can be for example extremely relevant to gain further insights into the process–structure–property correlations of highly heterogeneous materials [2]. However, large amount of data is usually generated during such experiments, requiring in parallel metadata storage for reproduction and reuse of such information. Indeed, in addition to the traditional nanoindentation approach of storing only the calibrated load–displacement curve, the metadata contain all the information on the sample, user, environmental conditions, calibration procedure and related data, raw data, analysis process and finally the analyzed data. In this way, a new data management strategy designed for nanoindentation results must be developed.

To tackle this challenge, the EU H2020 RIA funded project NanoMECommons (Grant Agreement 952869) has started in February 2021 [3]. With a consortium of 19 partners (11 from industry and 8 academia and research), coming from 10 countries and led by the National Technical University of Athens (NTUA), this project focuses on employing innovative nano-scale mechanical testing procedures in real industrial environments, by developing harmonised and widely accepted characterisation methods, with reduced measurement discrepancy, and improved interoperability and traceability of data. In this context, Ansys Inc. has been implementing a centralized materials information management platform to capture characterization data and protocols (experimental and virtual), ensuring FAIR principles for the project data and protocols [4-5]. It will also establish (a) software tools for complex data handling for characterization, model calibration and validation, and their availability through digitalized workflows and (b) a reference database for selection of materials and related protocols for reduced time to market, and improved resource efficiency. For the current presentation, the current database structure and workflows to capture nanoindentation data (see Figures 1 and 2) will be presented and discussed [6].

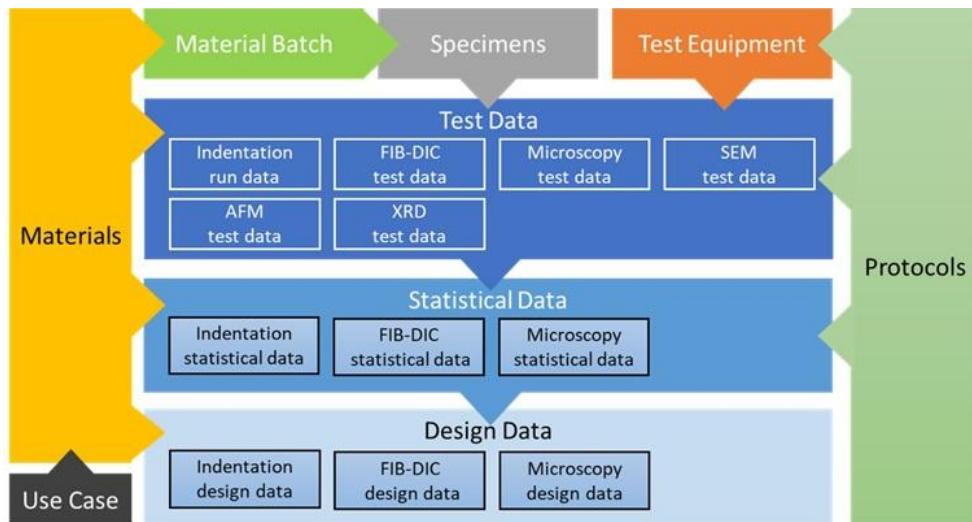


Figure 1. Database map designed for the NanoMECommons project

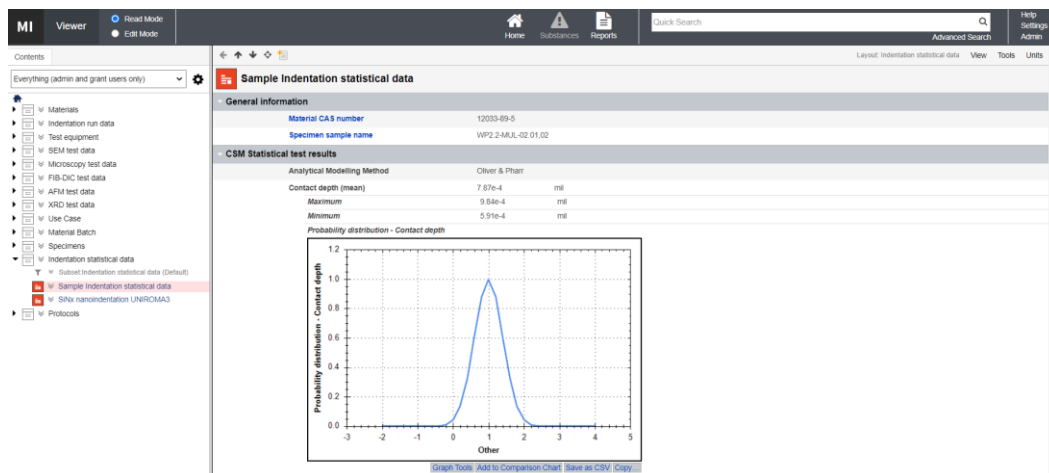


Figure 2. Screenshot of the database designed to help with data management, storage and analysis of nanoMECommons project, using Granta MI

KEYWORDS

Nanomechanical materials characterization; indentation data digitalisation; nanoindentation; materials information management; database

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The effect of initial backstress on instrumented indentation test

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ABSTRACT

Shot peening can introduce strong compressive residual stress and cumulated plastic strain simultaneously. Moreover, due to the repeated shot impacts, there is also strong backstress in the target material. To characterize the mechanical properties of the treated surface with instrumented indentation technique, the effect of backstress should be investigated. The distribution of backstress was obtained through shot peening simulations, and a series of indentation simulations using spherical tip were carried out with different backstress levels. The indentation direction is perpendicular to the shot-peened surface (i.e. the indentation test is performed on a cross-section). For Inconel 718, it was found that, the third backstress component has the greatest influence on the response. However, the difference of P_{max} and contact stiffness between the raw and treated material is 0.4% and -0.4% respectively. Compared to the effect of residual stress and cumulated plastic strain, the effect of backstress is quite small and can thus be neglected.

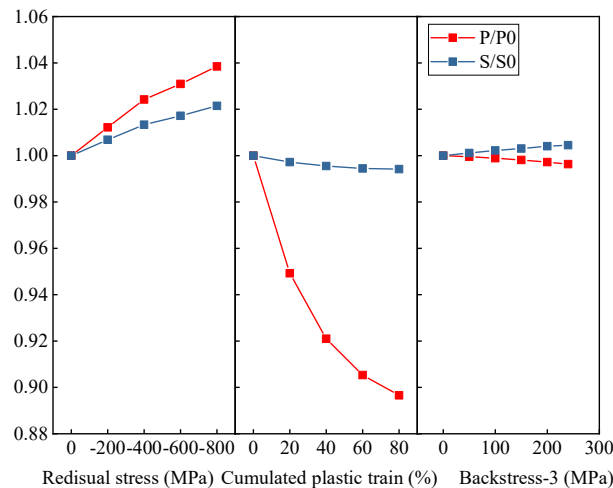


Figure 1. Nano indentation responses under different residual stress, cumulated plastic strain and equivalent backstress levels (Backstress-3 is the component of the backstress tensor along the normal to the treated surface)

KEYWORDS

Backstress, Finite element simulation, shot peening, instrumented indentation

Identification de paramètres de lois de comportement hyper-élastiques par indentation instrumentée d'élastomères.

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RESUME

De multiples recherches récentes dans le domaine de l'indentation ont montré un intérêt pour les matériaux polymères et plus particulièrement pour les élastomères [1]–[5]. En effet, la caractérisation de leur comportement hyperélastique, de leurs propriétés viscoélastiques, ou encore l'étude des différents phénomènes intrinsèques à ces derniers (dépendance à l'état de contrainte, effet Mullins, effet Payne), représentent un champ de recherche important. De plus, la composition particulière des élastomères, notamment la présence quasi-systématique de charges, induit une hétérogénéité à faible échelle qui fait de l'indentation instrumentée un procédé de caractérisation local intéressant.

C'est dans ce cadre que les travaux de O. Ezzaamari et al. se sont inscrits [1]. Ils mettent en évidence la possibilité d'identifier des paramètres de lois de comportement hyperélastique, à l'aide d'une approche par réduction de modèle. L'objectif est de réduire la dimensionalité de courbes $P-h$ d'indentation générées numériquement, puis, après interpolation, d'identifier des paramètres d'une loi de comportement à partir d'une courbe test.

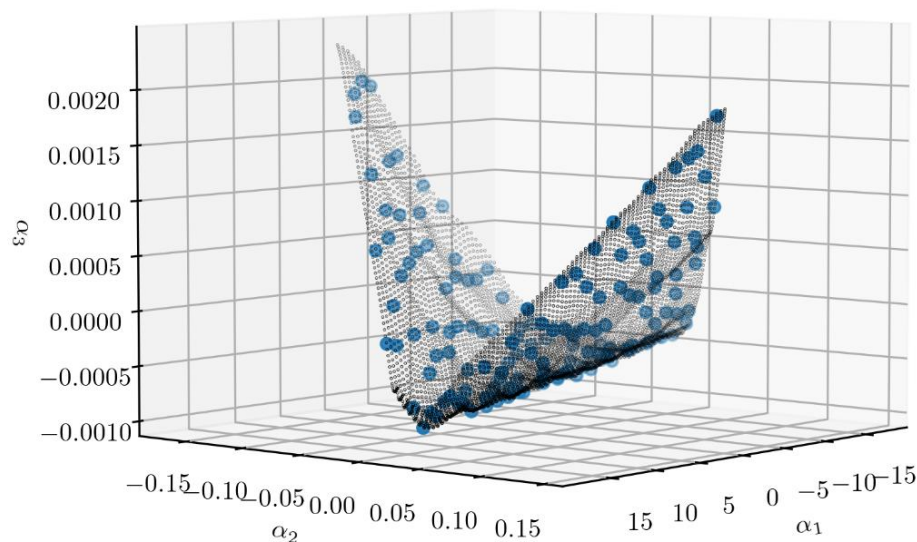


Figure 1: Représentation dans l'espace réduit 3D, de 150 courbes d'indentation générées à partir d'un plan d'expériences faisant varier les paramètres C_{10} , C_{01} d'un modèle de Mooney-Rivlin (points bleus). Visualisation de la variété de forme obtenue après interpolation dans l'espace réduit (pointillés noirs).

Les présents travaux font suite à ceux introduits précédemment. En effet, l'identifiabilité a été mise en évidence à partir d'études essentiellement numériques. Ainsi, un protocole expérimental permettant d'appliquer la méthode développée sera défini, pour identifier des paramètres correspondant à une courbe réelle et par conséquent au matériau caractérisé. De plus, un comparatif entre l'essai d'indentation et des essais macroscopiques « classiques » (traction uni-axiale, compression uni-axiale et cisaillement pur) sera proposée afin d'établir la validité des paramètres identifiés. Ces travaux seront dans un premier temps développés sur des élastomères faiblement visqueux à température ambiante. En effet, l'étude de matériaux visco-hyperélastiques ainsi que de l'impact de la température sur la méthodologie seront les sujets de travaux futurs.

MOTS CLES

Indentation instrumentée, Elastomères, Simulation numérique, Réduction de modèle

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MIEUX COMPRENDRE L'INDENTATION AVEC DEUX MODELES REVISITES

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ABSTRACT

On se propose de revisiter deux modèles de comportement relatif à l'indentation, le modèle d'expansion de cavité sphérique défini par Johnson et le modèle de ligne de glissement.

Le modèle d'expansion de cavité sphérique est défini par Johnson [1] et repris par beaucoup d'auteurs [2-5], en considérant deux états d'indentation infiniment proches et ne s'applique qu'à l'indentation conique. Nous proposons de lui substituer un modèle où, pour un état donné, le volume déplacé par l'indenteur se traduit par une compression des différentes zones du massif, le noyau hydrostatique, la zone plastique et la partie élastique. Cette formulation s'applique à toute géométrie d'indenteur et l'on montre que le paramètre pertinent de l'indentation X peut être définie par : $X = \frac{E}{2(1-\nu)\sigma_y} \frac{V_i}{\frac{2}{3}\pi a_c^3}$. Ce paramètre est identique à celui de Johnson dans le cas de l'indentation conique. Il est le produit du terme $\frac{E}{(1-\nu)\sigma_y}$, dépendant du matériau, et d'un terme représentant le rapport entre le volume déplacé par l'indenteur et le volume de la demi-sphère de rayon a_c .

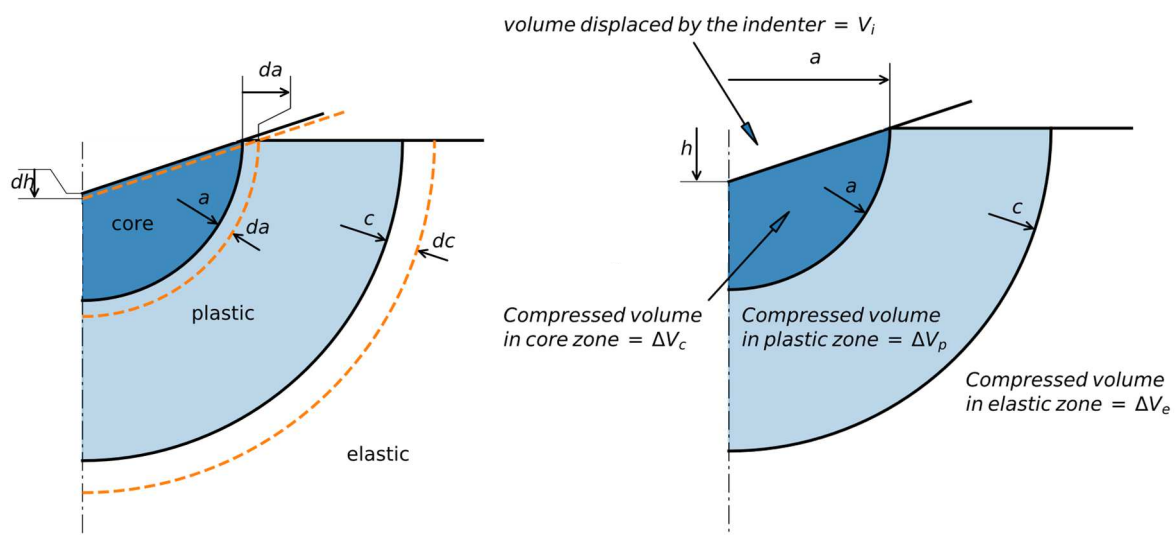


Figure 1. Comparaison entre les deux modèles de cavité sphérique : à gauche celui défini par Johnson et à droite le nouveau modèle

Le modèle de lignes de glissement [6-7] est quant à lui utilisé pour définir la chute de pression dans le régime plastique. Avec quelques hypothèses

simplificatrices, on montre que le cône équivalent est défini par un angle complémentaire au demi-angle au sommet donné par l'intégrale suivante :

$$\beta_{SL} = \frac{2}{a^2} \int_0^a \arctan\left(\frac{\partial z}{\partial r}\right) r dr$$

Expression dans laquelle z est la forme de l'indenteur, soit $z = f(r)$.

L'application de cette formule à l'indentation sphérique donne par exemple un angle β_{SL} égal à : $\beta_{SL} = \left(1 + \left(\frac{R}{a_c}\right)^2\right) \arctan\left(\frac{a_c}{R}\right) - \frac{R}{a_c}$ où R est le rayon de la sphère.

Ces deux relations, correspondant chacune à un régime particulier, le premier au régime élastique-plastique et le second au régime plastique, permettent d'exprimer des grandeurs indépendamment de la forme de l'indenteur.

KEYWORDS

Indentation, slip-line, ECM, Equivalent cone

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NEW LEADS ON THE DENSIFICATION PROFILE UNDERNEATH NANO INDENTATION IMPRINT IN SILICA GLASS BY MEANS OF A CHEMICAL DISSOLUTION TECHNIQUE

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ABSTRACT

The region of permanent densification beneath Berkovich nano-indentation imprint in silica glass is investigated using the chemical probe technique [1]. The later relies on the measurement of local chemical reactivity variations resulting from the structural modifications of the densified glass in the plastic zone by successive atomic force microscopy imaging of the same residual imprint through dissolution steps.

The use of the similitude regime in sharp indentation testing allows one to record reliable data with a good spatial resolution that makes it possible to deal with low loads (typically below 10 mN) and, more importantly, crack-free residual imprints. The densified zone dissolves much quicker than the non densified regions. The analysis of the results, along the vertical axis are conducted within the geometrical similar framework [2, 3] which makes it load independent but also re-scalable to any load for comparison if needed. Results indicate that the densification zone, along this axis is made of 2 zones: the first one just beneath the residual imprint is rather homogeneous in terms of dissolution rate thus in terms of densification ratio and the second one is a rather steep transition to the non densified zone.

A reactivity model linking densification ratio (through structural modifications) [4, 5] to dissolution rate has been developed [6]. It allows: on the one hand the validation of possible densification profiles such as two parameters sigmoidal functions for which the sole adjusting parameters to match the experimental points is the width of the transition zone as its position is provided by the experimental results; and on the other hand to gather all the densification profiles available in the literature wether they originate from Raman spectroscopy mapping (loads in the 1 N range) our from numerical modeling and compare them to the chemical probe results either in terms of imprint depth evolution through dissolution steps or in terms of densification profile all of this being treated within the similar geometry frame work of pyramidal indentation as, for example, described in references [2, 3] .

Agreements and disagreements with data from the literature are discussed in terms of possible densification profiles, geometrical similarity as well as Raman confocal

probe drawbacks. Limitations of both the chemical probe technique and Raman spectroscopy mapping are addressed and perspectives are drafted out.

KEYWORDS

Nano-indentation, AFM, geometrical similarity, glass, plastic zone

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RÉSUMÉ

Longueur maximale du résumé: 2 pages, 500 mots, 1 figure.

Figure 1. Titre de la figure

MOTS CLÉS

pas ; plus ; de ; cinq

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Bon Jean, Deuf John, Croche Sarah, Neymar Jean. Mechanical properties of amazing materials. *Journal of plants and trees*, 2014, 14, p. 239-243.

MICROPLASTICITÉ DES AMORPHES SILICATÉS - QUELQUES DÉVELOPPEMENTS RÉCENTS

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RÉSUMÉ

En tant que matériaux, les verres silicatés semblent irremplaçables pour leur rigidité mécanique, leur faible coût et souvent aussi pour leur transparence, et ce en dépit de leur faible résistance mécanique. En effet ils représentent le type même du matériau fragile, qui reste élastique jusqu'à la rupture. En fait, un certain nombre de résultats suggèrent que ce n'est pas tout à fait le cas et invoquer une plasticité localisée en pointe de fissure semble permettre d'en rendre compte. Cependant une approche plus quantitative visant à décrire cette plasticité à l'échelle locale se heurte à plusieurs difficultés : 1) la description de la déformation plastique dans les amorphes ; 2) la quantification de la plasticité, qui ne peut être faite que par des expériences très locales (à l'échelle du micron). De récents progrès expérimentaux ont permis certaines avancées, en particulier grâce aux approches couplant micromécanique et microspectroscopie, dont je présenterai quelques résultats saillants.

MOTS CLÉS

Silicates, microplasticité, fracture, spectroscopie Raman.

RÉFÉRENCES

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ACOUSTIC EMISSIONS AS A TOOL FOR THE EXPLORATION OF PLASTIC INSTABILITIES DURING NANOINDENTATION AT THE NANOSCALE

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ABSTRACT

Nanoindentation coupled with acoustic emission (AE) detection represents a very powerful combination of complementary methods providing a unique set of information about the deformation behavior of materials at small scales. The plastic instabilities of a silicon iron single crystal were explored via spherical nanoindentation together with detection of acoustic emission at the nanoscale [1]. Besides gradual development of plasticity without detectable AE activity and sudden onset of plasticity (pop-in) with a strong AE event, an elastic response with frequent AE activity was observed during both loading and unloading. This type of AE activity at the nanoscale was observed for the first time for purely elastic indentations and it suggests an action of reversible twinning. The reversible character of these events was confirmed by the analysis of the AE hit waveform.

Critical indentation stresses were identified for loading and unloading and together with AE hit parameters enabled unambiguous classification of observed events. Acoustic emissions proved to be a very powerful and complementary method for the examination and classification of different deformation mechanisms (slip and twinning) observed during indentation loading.

Acknowledgement: Financial support by the European Regional Development Fund in the frame of the project Centre of Advanced Applied Sciences (No. CZ.02.1.01/0.0/0.0/16_019/0000778) and Czech Science Foundation (project No. 21-11313S) is gratefully acknowledged.

KEYWORDS

indentation, acoustic emission, plastic deformation, reversible plasticity

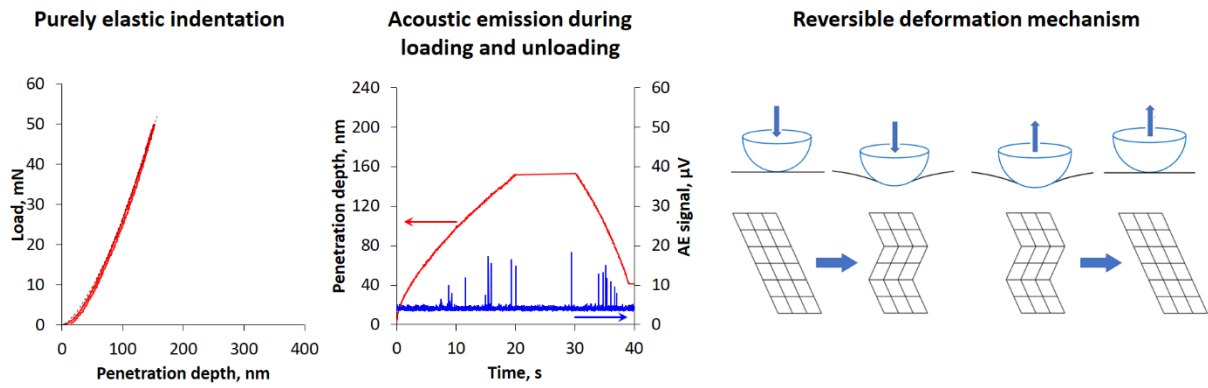


Figure 1. Acoustic emission detected for purely elastic indentation curve

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STRUCTURAL AND MECHANICAL CHARACTERIZATION OF CoCrFeNiX_y HIGH ENTROPY ALLOYS PREPARED BY MECHANICAL ALLOYING AND SPARK PLASMA SINTERING

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ABSTRACT

High entropy alloys (HEAs) represent a new class of materials with unique design possibilities and very promising physical and chemical properties. By definition, they are composed of at least 5 principal metal elements in equimolar or near-equimolar ratios. Depending on the actual composition and microstructure, HEAs can exhibit high strength and hardness, high creep and wear resistance, excellent performance at elevated and cryogenic temperatures together with superior corrosion and oxidation resistance, excellent irradiation tolerance as well as unique magnetic properties. This is the reason why they have attracted much interest in the last two decades.

This contribution focuses on the structural and mechanical characterization of CoCrFeNiX_y (X=Mn, Ti, Al, Nb; y=5÷35 at.%) high entropy alloys derived from the so-called Cantor alloy [1]. They were prepared by powder metallurgy technique of mechanical alloying (MA) and compacted by spark plasma sintering (SPS) (e.g. [2]). Scanning electron microscopy (SEM), transmission electron microscopy (TEM), and X-ray diffraction (XRD) were employed to characterize their microstructure and phase composition. Nanoindentation measurements were performed to determine mechanical properties (hardness, Young's modulus, etc.). Correlations between microstructure and mechanical properties (depending on the fifth alloying element and its amount) were found and analysed.

Acknowledgement: Financial support by the European Regional Development Fund in the frame of the project Centre of Advanced Applied Sciences (No. CZ.02.1.01/0.0/0.0/16_019/0000778) and Czech Science Foundation (project No. 21-11313S) is gratefully acknowledged.

KEYWORDS

high entropy alloy, microstructure, mechanical properties, nanoindentation

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Understanding size effect and strain rate sensitivity in copper by In-situ pillar microcompression and dislocation dynamics simulation

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ABSTRACT

High strain rates micromechanical testing of material raised a great interest over the last few years. In – situ pillar microcompression tests reveal for several materials especially copper, size effect and an orientation dependent strain – rate sensitivity [1, 2] that still need to be well understand.

As well known, dislocations activities are the source of plastic deformation of metals. In this regard, Discrete Dislocation Dynamics simulation can provide an important insight into the mechanical behavior of materials over a large panel of strain rates and can help unveil the dislocation behavior leading to the size effect and the strain rates sensitivity observed experimentally.

In this work, high strain rate tests are performed on copper micro-pillar at different strain rates (from quasi-static up to 10^3 /s). In addition, using a 3D Discrete Dislocation Dynamics (3D DDD) numerical simulation, we performed the micro-pillar compression simulation. The combination of these two methods brings new understandings to size effect and strain rate sensitivity in copper.

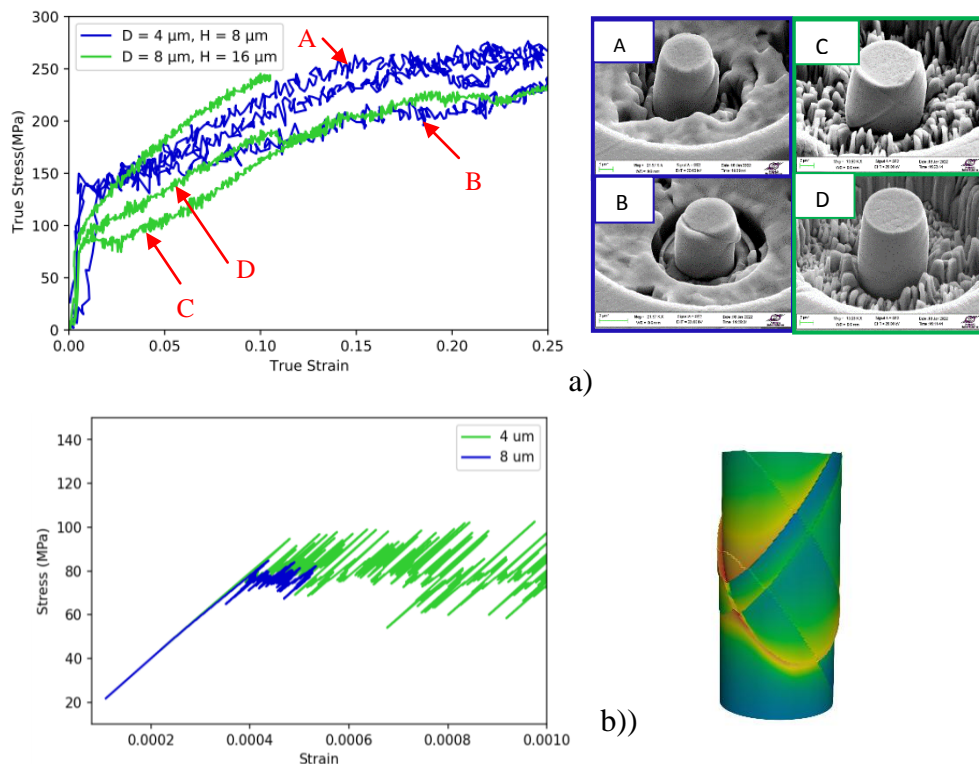


Figure 1. a) Pillar microcompression experimental results b) 3D DDD micro-compression simulation

KEYWORDS

High strain rates; size effect; strain rate sensitivity; pillar microcompression; Discrete Dislocation Dynamics.

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Nanoindentation to reveal synergistic effects of hydrogen and precipitate on elastic/plastic properties of nickel-based superalloy

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Nanoindentation is known to be capable of exploring the interaction of hydrogen with multi-scale defects and microstructural components such as vacancies, dislocations, precipitates, grain boundaries, etc., and the resulting implications on deformation mechanisms [1]. A handful of previous research have shown how to use the characteristic outputs of nanoindentation tests namely reduced modulus, pop-in attributes and indentation hardness to discover the deformation property dependency of hydrogen-defect-microstructure interaction. This work demonstrates the individual and synergistic effects of hydrogen on the elastic and plastic properties of Waspaloy with various γ' -Ni₃Al precipitation states (size and lattice misfit) in a hydrogen embrittlement perspective. A series of nanoindentation tests were performed on Waspaloy samples with precipitation size range of 0-100 nm and precipitate/matrix lattice misfit range of 0.15-45% in hydrogen pre-charged and uncharged conditions. The statistical analysis of indentation results showed distinctive effects of hydrogen and precipitates on deformation mechanisms: (a) different plasticity mechanism regimes according to the precipitate size (weakly coupled shearing, strongly coupled shearing and Orowan bypassing); (b) hydrogen induced hardening and/or softening in different above-mentioned regimes of plasticity. Microhardness test, tensile test and fractography were subsequently conducted in order to question these nanoscale behaviors obtained from nanoindentation in micro and macroscales.

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Etude multi-échelle de l'effet de l'hydrogène et la nature des joints de grains sur les mécanismes de plasticité dans le nickel pur

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RÉSUMÉ

L'effet de l'hydrogène sur l'interaction entre la plasticité et la nature de joint de grain a été étudié dans le nickel pur en réalisant des essais de traction et de nanoindentation. Les essais de traction ont été réalisés sur des échantillons polycristallins chargés ayant des tailles de grains différentes dans le but d'étudier l'effet de l'hydrogène et la distribution des joints de grains sur les mécanismes de plasticité. En présence de l'hydrogène, la limite élastique et l'allongement à rupture ont été affectés différemment en fonction de la taille des grains et la nature des joints de grains. On a observé deux effets antagonistes durcissement et adoucissement sous l'effet de l'hydrogène. Ces effets peuvent être reliés au rôle de différentes natures de joint de grain et leurs distributions sur la diffusion et le piégeage de l'hydrogène dans le nickel [1]. En outre, les analyses des fractographies montrent une rupture intergranulaire dans les échantillons chargés et la présence de pores à côté de certains joints de grains, ce qui peut indiquer la présence d'une accumulation de lacunes induites par l'hydrogène. Pour mieux comprendre l'interaction entre l'hydrogène et les joints de grains, des essais de nanoindentation ont été réalisés sur le nickel monocristallin, bi-cristallin et polycristallin. L'effet de l'hydrogène sur la charge de pop-in et la dureté, obtenues par nanoindentation sur différentes orientations de grain et à côté des joints de grains seront discutés.

MOTS CLÉS

Hydrogène ; Joint de grain ; plasticité ; rupture ; Nickel

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ENHANCING THE INFORMATION-RICHNESS OBTAINED FROM BERKOVICH NANOINDENTATION TO QUANTIFY SLIP SYSTEMS INTERACTIONS IN FCC CRYSTAL

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ABSTRACT

Determination of the slip systems interaction coefficients ($h_i, i \in \{1, \dots, 7\}$), plasticity laws core, is critical for reliable simulations applied to industrial fields. Those parameters are difficult to identify experimentally. Nanoindentation test seems to be a great choice for enhancing slip systems interaction identification (Renner et al., 2016, 2020). Nevertheless, the inverse method used to identify those parameters requires a Crystal Plasticity Finite Element Model of the test which is sustainable with the fewer computation time possible. The 10 hardening parameters (Q, b, τ_0 and h_i) must be identified simultaneously. To optimize and enhance the identification process, a prior identifiability study is performed to build an optimal experimental design. Identifiability study measure the information richness that could be given to the identification process using Finite Element Model Updating method.

Firstly, indentations simulations and experiments are performed within a single grain. We investigate the experimental crystal orientations and the relative orientation between crystals references frames and the Berkovich tip to simulate the nanoindentation test in the same configuration than the experimental one. To highlight the impact of the slip systems interactions coefficients on residual topographies, we first used three sets of interaction from literature (Madec & Kubin, 2017; Méric et al., 1994). Using those three sets of parameters, figure 1d) to f) illustrates the sensitivity of the residual topography to the interaction coefficients. We can observe on Figure 1 the difference between the three topographies in terms of height, shape and distribution of pile-ups. These simulations highlight the sensitivity of the topography and $P - h$ curve to the interaction coefficients.

To quantify the information richness, an identifiability analysis from some observables ($P - h$ curves, topographies) and tip/crystal experimental orientations is performed. The quantification of the information richness is based on an I-index (Renner et al., 2020). This I-index measures the parameters identifiability (i.e., the sensitivity of a set of parameters to the observables). It allows to compare the information-richness contained in each experiment and combinations of experiments.

This result will lead to an optimal experiment design, providing enough information to well-pose the inverse problem.

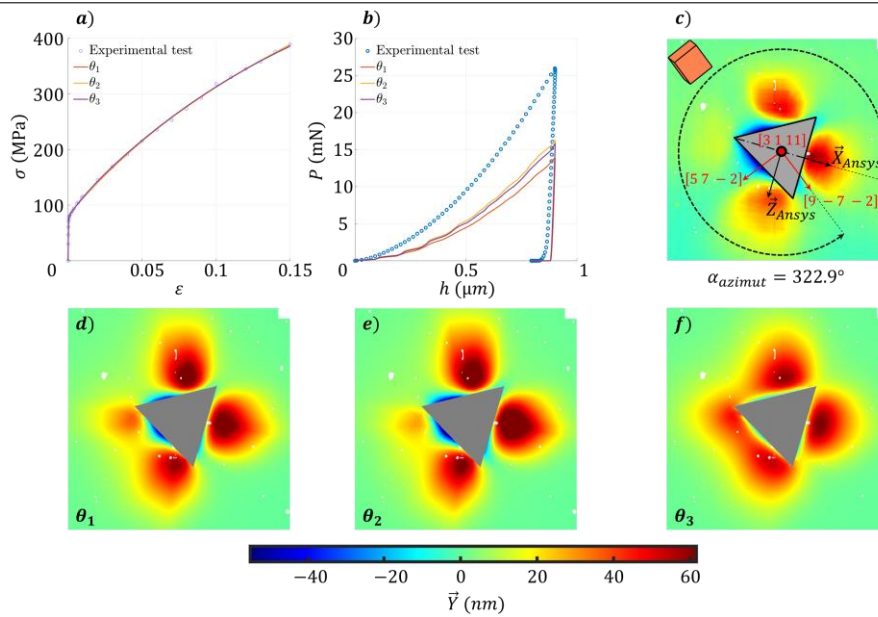


Figure 1 : a) Simulations and experimental tensile curves on polycrystal sample. b) Simulations and experimental $P - h$ curves. c) Crystal (red axis)/indenter tip configuration represented on the experimental topography. The \vec{X} and \vec{Z} component define the indentation plane and \vec{Y} the elevation. Crystal orientation is illustrated by a cube shape in the upper left corner. Figures d) to f) are the simulated topographies for the three θ_i sets of parameters.

KEYWORDS

Cristal Plasticity Finite Elements Model, Interaction matrix, Nanoindentation, Identifiability, Optimal experiment design

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Effet de l'irradiation électronique sur la plasticité des verres d'oxyde lors d'essais micromécaniques in-situ.

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RÉSUMÉ

Les verres d'oxyde sont l'archétypes des matériaux amorphes fragiles. A l'échelle du micron, ils peuvent s'écouler plastiquement et ainsi présenter une ductilité similaire à celle des métaux les plus mous. Il semble cependant exister un lien entre cette plasticité microscopique et la fragilité macroscopique, ce qui nécessite donc de mieux comprendre les spécificités de cet écoulement plastique local. Contrairement aux métaux, ils ne semblent pas présenter un seuil défini de plasticité, mais plutôt un comportement viscoplastique sans seuil avec toutefois une sensibilité à la vitesse de déformation relativement faible à température ambiante [BAR19]. Pour étudier la (visco)plasticité, il est donc nécessaire de recourir à des essais mécaniques permettant de solliciter ces matériaux à la bonne échelle, c'est-à-dire à l'échelle de quelques microns, voire de quelques dizaines de nanomètres [KER16]. Nous présentons dans un premier-temps les progrès réalisés dans ce domaine grâce aux essais micromécaniques in situ dans les microscopes électroniques à balayage. Nous montrons cependant que ces essais ne sont pas complètement neutres puisque à la fois les conditions environnementales (vide partiel) et l'irradiation électronique semblent affecter le comportement de ces matériaux en favorisant un écoulement viscoplastique. A partir d'essais micromécaniques mêlant indentation / compression / relaxation, nous montrons que l'irradiation joue un rôle équivalent à la température sur la viscosité du matériau, avec à la clé une potentielle équivalence temps-irradiation similaire à la « bien connue » équivalence temps-température. A terme, ces essais devraient nous permettre de mieux comprendre le comportement de ces matériaux à haute température ... mais tout en réalisant des essais micromécaniques à température ambiante.

MOTS CLÉS

Micromécanique, Viscoplasticité, Essais MEB in Situ, Matériaux amorphes

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How to Measure Precisely Surface Shear Properties at the Micron Scale?

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ABSTRACT

Industrial systems implies tribological contacts between surfaces and therefore friction and/or wear mechanisms at the sheared interface. The understanding of the tribological behaviour of these surfaces is closely related to the shear deformation between the two surfaces, which may be scale dependent.

To understand the shear behaviour of surfaces at the micron-scale, a new method to measure shear properties was developed, based on a specific geometry named shear compression specimen (figure 1.a). This method was validated on fused silica, enabling us the extraction of properties (especially the yield stress) at low and high strain rates [1]. However, numerical simulations of shear deformation does not match the whole experimental curve. Especially in the fully plastic regime, some differences were observed. To better understand this difference, a parametrical study was performed by FE simulations, on fused silica and nickel and will be presented. It revealed that the friction between the punch and the specimen seems to be the key parameter (figure 1.b).

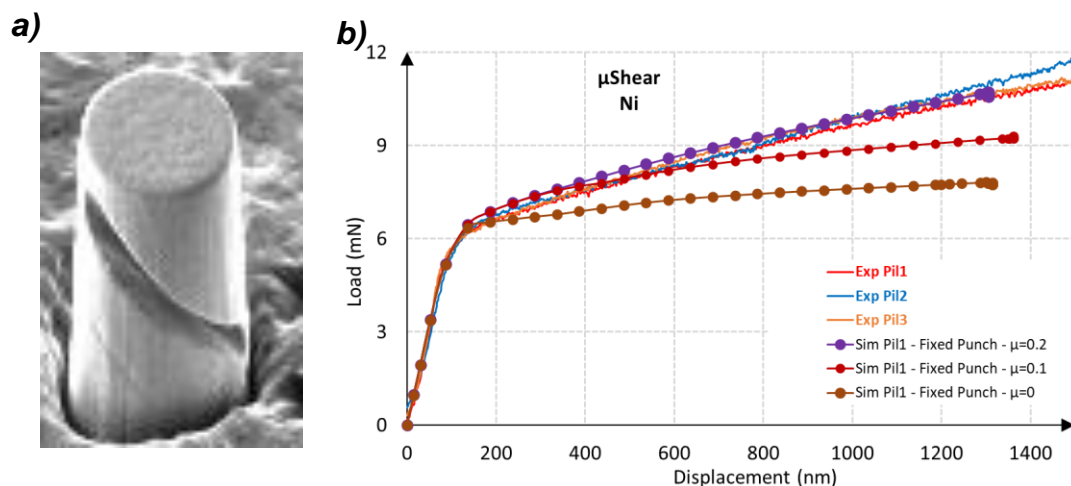


Figure 1. a) Microshear Compression Specimen geometry developed (nickel).
b) Comparison between experimental data and numerical simulations (nickel): friction between the punch and the specimen is present. In these FE simulations, the friction coefficient at the interface pillar head/punch bottom was varied from $\mu=0$ to $\mu=0.2$.

To avoid friction during the shear compression experiment, two developments were carried out:

- 1) A cross grooved specimen was developed
- 2) A dynamic loading mode was applied on classical shear compression specimen

The first technique must imply no sliding of the upper part of the specimen against the punch, and the second technique should minimise it. These two developments are very promising and will be presented and analysed during the conference.

KEYWORDS

microshear, shear compression specimen, parametric study, numerical simulation

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INVESTIGATION OF CARBON FIBRE MECHANICAL BEHAVIOUR AT MICRO/NANO SCALES

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ABSTRACT

Since carbon fibres are heterogeneous, anisotropic and have small dimensions, determining their mechanical properties is rather challenging. In this study, we investigate the elastic properties and the compressive strength of these materials.

The compressive strength of two different carbon fibres is determined from in situ Scanning Electron Microscopy (SEM) compression tests on micro-pillars (see Figure 1). The failure mode is found to be axial splitting (see Figure below). Large hysteretic loops are observed associated to the development of cracks but no or small permanent deformation is visible. A careful analysis of the test results is made, and the possibility of experimental artefacts is explored. The compressive strength of carbon fibres is found to be lower than their tensile strengths. The mechanisms involved during micro-compression tests are investigated and compared with those from various other experimental techniques. The scenario of nano-buckling of crystalline carbon stacks constrained by the shear stiffness of the fibre agrees with our results. In particular, the value of the shear modulus of the fibre seems to govern the compressive strength value.

Therefore, nano-indentation instrumented tests are carried out at shallow depths to identify the elastic parameters and especially the in-plane shear modulus. Indentation moduli are used to identify the elastic constants of the fibres, assuming a transversely isotropic behaviour, by minimising a cost function between measured and estimated values. Inconstancies between the identified in-plane shear and transverse moduli and reported literature values are pointed out, and some drawbacks of the nano-indentation method are highlighted. An improved method considering the buckling mechanisms of crystallites at stake during the indentation process, and visible in the hysteretic behaviour of force-penetration nano-indentation curves, is proposed. It allows to identify values of elastic constants that are in accordance with literature values. These elastic properties of carbon fibres are in turn used to estimate the elastic properties of epoxy matrix composites containing these fibres. Very good agreement is found with experimentally available values of unidirectional ply properties. An excellent correlation between experiments and Finite Element Analyses of the indentation response of carbon fibres is eventually found

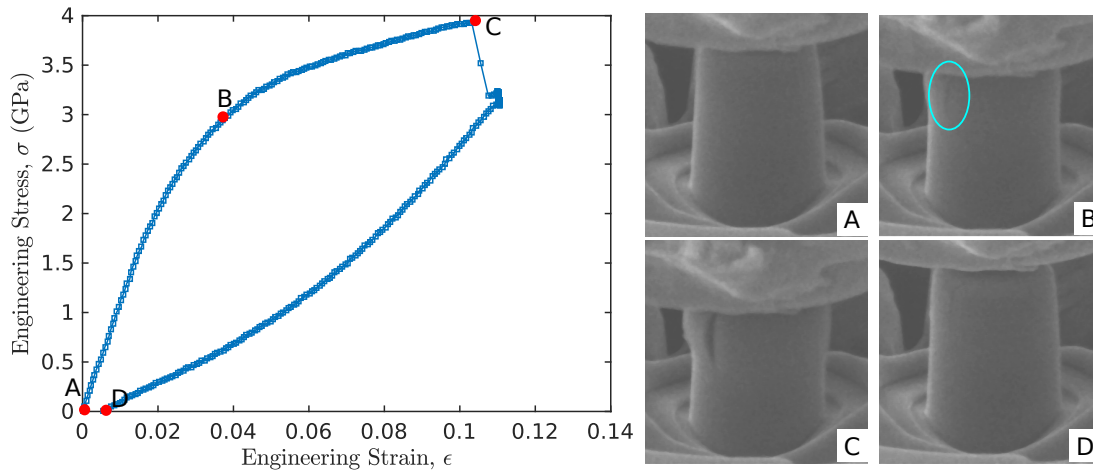


Figure 1. Micro-pillar compression on a carbon fibre

KEYWORDS

Carbon fibres; Micro-compression; Micro-pillars; Buckling; Compressive strength; Nano-indentation

Dureté et résistance à la fissuration de dépôt Cr-VI par macro-indentation : Vers un critère de fissuration.

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RÉSUMÉ

Cette étude a été réalisée dans le cadre du projet européen Interreg Alt-Ctrl-Trans dont l'objectif était de trouver une alternative au revêtement par chromage dur (Cr-VI), car la réglementation européenne REACH en interdit l'usage dès septembre 2024. Le projet a mis en évidence que les revêtements alternatifs, type NiW testé dans ce projet, ne satisfont pas aux exigences mécaniques dans tous les domaines d'application. Toutefois, ils peuvent être sélectionnés en fonction d'une utilisation visée contrairement au Cr-VI qui satisfait tous les critères, d'où sa grande popularité pour la fabrication de dépôts durs et résistants.

Dans une première partie de ce travail, les propriétés mécaniques (dureté et module d'Young) du revêtement Cr-VI, déposé sur un substrat en acier (ST37) sont déterminées par macro-indentation avec des charges allant de 0,2 à 20 N [1]. Les principaux résultats ont montré que la dureté est constante pour tous les niveaux de chargement si l'effet de taille en indentation et le défaut de pointe de l'indenteur, ici un Vickers, sont pris en compte de façon couplée. Quel que soit le modèle de dureté appliqué (Li & Bradt [2], Bull & Page [3] et Nix & Gao [4]) pour décrire l'effet de taille, les duretés équivalentes sont trouvées égales à 1,2 GPa pour le substrat en acier, et 8,5 et 8,0 GPa pour le dépôt de Cr-VI mesurées respectivement dans la section transversale ou prise perpendiculairement au dépôt.

Avec le niveau de chargement utilisé, c'est-à-dire pour des charges allant de 200 à 500 N, les essais d'indentation ont mis en évidence la présence de fissures circulaires autour de l'empreinte Vickers comme le montre la Figure 1. Il n'est donc pas possible d'appliquer les relations usuelles de ténacité par indentation que ce soit en considérant des fissures en mode radian ou en mode Palmqvist. C'est pourquoi pour caractériser le réseau de fissuration et montrer l'influence de l'interface sur la propagation des fissures, nous proposons un critère de fissuration défini par le rapport du diamètre du cercle circonscrit à l'empreinte résiduelle sur le diamètre de la fissure la plus éloignée de l'empreinte. L'évolution de ce critère en fonction de la

charge appliquée donne des informations intéressantes quant à la résistance à la fissuration du dépôt et au rôle de l'interface substrat/revêtement sur la propagation des fissures.

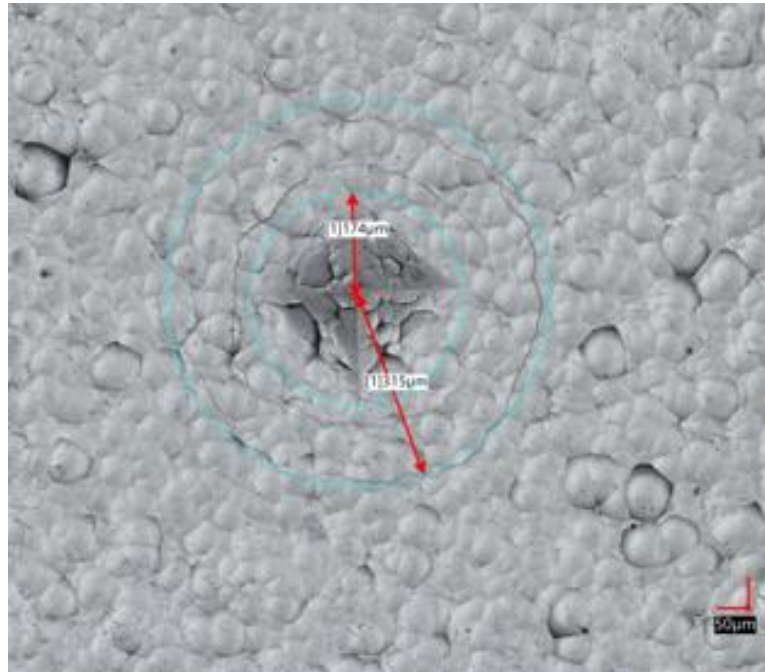


Figure 1. Détermination des diamètres extérieur et intérieur pour la charge d'indentation de 200 N appliquée au revêtement Cr-VI.

MOTS CLÉS

Revêtement dur ; ISE ; Défaut de pointe ; Critère de fissuration.

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Évaluation de la dureté en fonction de l'état de surface : détermination d'un critère de pertinence

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MOTS CLÉS

Dureté ; Morphologie de surface ; Dispersion

RÉSUMÉ

La plupart des modèles de dépouillement en indentation supposent un contact plan entre l'indenteur et la surface du matériau testé. Il est alors d'usage de préparer la surface de sorte à la rendre plane et lisse pour diminuer les effets de la rugosité. Cela passe la plupart du temps par une étape de polissage. Toutefois les différentes étapes du polissage, même si elles sont réalisées le plus soigneusement possible, peuvent modifier le comportement et les propriétés mécaniques en introduisant à la surface du matériau de l'écaillage et/ou des contraintes résiduelles, etc. De plus, dans certains cas comme celui des revêtements minces, il n'est pas possible ni même souhaitable de modifier la surface pour éliminer ou tout au moins tenter de réduire la rugosité de surface. Il devient donc nécessaire d'être prudent quant à l'interprétation des résultats de dureté et de module d'élasticité par indentation, encore plus dans le domaine de la nanoindentation sur des surfaces rugueuses.

Dans le travail présenté ici, nous cherchons à étudier l'influence de l'état de surface d'un verre initialement très plan ayant subi différentes attaques chimiques conduisant à des modifications notables de son état de surface. Les conditions d'attaques par acide sont présentées dans le Tableau 1. Elles ont été choisies de manière à ne pas modifier le matériau sous-jacent.

Les morphologies de surface sont étudiées par profilométrie tactile et les propriétés mécaniques (Module d'Young, E , et dureté instrumentée, HIT) par essais d'indentation à l'échelle nanométrique. Les courbes force – déplacement sont analysées en suivant deux méthodologies :

- 1) Le modèle d'Oliver et Pharr [1] qui est actuellement le plus utilisé par tous les utilisateurs de la nanoindentation. La détermination des huit coefficients de lissage de la fonction d'aire de contact sont obtenus par un étalonnage sur un échantillon de silice en supposant un module d'Young de 72 GPa.
- 2) Le modèle de Chicot *et al.* [2] qui présente l'avantage de ne faire intervenir qu'un seul paramètre, lié au défaut de la pointe, h_b , au contraire des huit coefficients de lissage du modèle d'Oliver et Pharr. De plus, ce modèle ne fait aucune hypothèse puisqu'il s'appuie sur aucun étalonnage préalable.

Les résultats sont d'abord comparés entre les deux modèles en terme de valeurs absolues puis en termes statistiques : valeur moyenne et dispersion mis en regard des critères de morphologie de surface. Le Tableau 1 rassemble l'ensemble des résultats des modules d'Young, E en GPa, et des duretés instrumentées, HIT en GPa. Il montre très clairement que les deux méthodes sont très comparables autant en termes de valeurs absolues que de dispersion. Ainsi, la dispersion des résultats constatée n'est pas due à l'application d'un modèle plutôt qu'à un autre mais elle est bien à relier à l'état de surface du matériau testé.

Tableau 1. Valeurs des modules d'Young, E en GPa, et duretés instrumentées, HIT en GPa, déduites des modèles d'Oliver en Pharr [1] et de Chicot et al. [2] appliquées aux verres ayant subi différentes attaques chimiques en vue de modifier leurs états de surface.

	Oliver & Pharr [1]		Chicot et al. [2]		
	E (GPa)	HIT (GPa)	E (GPa)	HIT (GPa)	hb (nm)
30 sec. HF40% dilué 50%	77,3 ± 1,0	6,74 ± 0,14	75,8 ± 1,0	7,37 ± 0,10	8,8 ± 1,9
1 min. HF40% dilué 50%	79,0 ± 13,9	6,69 ± 1,20	78,6 ± 14,0	7,38 ± 1,31	7,8 ± 2,9
2 min. HF40% dilué 50%	77,4 ± 0,8	6,71 ± 0,09	75,7 ± 1,2	7,34 ± 0,08	8,7 ± 1,3
4 min. HF40% dilué 50%	77,8 ± 1,6	6,77 ± 0,20	78,1 ± 3,3	7,45 ± 0,25	8,4 ± 1,7
3-4 min HF40% non dilué	74,9 ± 0,8	6,55 ± 0,09	74,5 ± 2,7	7,17 ± 0,09	9,1 ± 1,5

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Caractérisation par indentation de matériaux métalliques : comparaison des résultats à ceux de la traction

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Résumé

Dans ce travail nous présentons tout d'abord plusieurs techniques d'analyse des données de la macro-indentation, avec indenteur sphérique, afin d'identifier les paramètres d'une loi d'érouissage. Ces techniques sont basées sur la détermination de l'évolution de la déformation représentative.

Une première technique s'appuie sur la courbe d'indentation F-h [1]. Elle consiste à reconstruire la courbe d'érouissage point par point en déterminant une valeur de déformation représentative moyenne, et la contrainte représentative correspondante, pour plusieurs niveaux de chargement. Nos travaux exploitent une base de données issue de nombreuses simulations par la méthode des éléments finis de l'indentation de matériaux métalliques avec un indenteur sphérique. Un calcul d'écart, au sens des moindres carrés, entre la courbe d'indentation expérimentale, obtenue à partir d'un essai d'indentation réalisé sur le matériau à caractériser, et chacune des courbes de la base de données conduit à une cartographie de l'écart. Celle-ci présente une vallée dont le fond correspond aux couples de paramètres (σ_y et n) donnant une courbe d'indentation très proches de la courbe expérimentale. Plus précisément, les jeux de paramètres (σ_y et n) correspondant aux points situés sur le grand axe de la vallée correspondent à des lois d'érouissage qui se coupent toutes en un seul point dont l'abscisse constitue la déformation représentative moyenne [1]. La contrainte correspondante est ensuite obtenue à partir du modèle de Hollomon. Plusieurs "points représentatifs" peuvent ainsi être obtenus en considérant différentes portions de la courbe expérimentale.

Une deuxième technique s'appuie sur plusieurs couples F-h (effort-enfoncement) obtenus à différentes charges. Pour chaque couple de valeurs F-h issues d'une indentation, une déformation représentative instantanée est obtenue par un calcul de gradient de courbure (F/h^2), en utilisant un modèle F(h) interpolant la base de données évoquée ci-dessus.

Une troisième technique s'appuie sur plusieurs couples F-a (effort-rayon d'empreinte) obtenues à différentes charges [2]. Pour chaque couple de valeurs F-a issues d'une indentation, une déformation représentative instantanée est obtenue par un calcul de gradient de dureté ($F/\pi a^2$), en utilisant un modèle F(a/R) interpolant la base de données mentionnée plus haut, R étant le rayon de l'indenteur. Par cette méthode, on obtient pour des chargements comparables, des valeurs de déformation représentative plus élevées que par les deux techniques précédentes.

Enfin, une quatrième technique consiste à considérer l'ensemble des points représentatifs de la loi d'érouissage obtenus par la deuxième et la troisième technique. Cette procédure présente une meilleure robustesse car elle prend en

considération des "points représentatifs" plus nombreux que pour les techniques précédentes et répartis sur un domaine de déformation plus étendu.

Pour chacune de ces techniques, l'interpolation par une loi de Hollomon, des points représentatifs obtenus, conduit à une loi d'écroutissement comparable à celle obtenue par un essai de traction.

Ces différentes techniques sont ensuite appliquées à la caractérisation mécanique de matériaux à partir des données de l'indentation. La démarche est d'abord testée avec succès sur des données issues de simulations numériques de l'indentation sphérique. Ensuite la démarche est appliquée à une quinzaine de matériaux métalliques sur lesquels des essais de traction ont aussi été réalisés.

Pour la plupart des matériaux, nous observons une très bonne corrélation entre les résultats issus de l'indentation et ceux de traction. De moins bons résultats sont observés lorsque la loi d'écroutissement du matériau n'est pas bien représentée par la loi puissance de Hollomon.

Mots clés

Indentation sphérique, caractérisation mécanique, matériaux métalliques

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Identification of elasto-plastic properties of metals using spherical indentation force-contact radius data: Introducing a new definition of representative strain and a new indentation model

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ABSTRACT

Indentation tests are increasingly used to determine the mechanical properties of materials. Most methods in the literature are based on the concept of representative strain. This work proposes a new methodology based on the “gradient representative strain,” which was first introduced for the case of conical indenters only by Hernot et al.[1]. The indented material is assumed to be homogeneous and isotropic, with a hardening behavior following Hollomon’s power law.

The proposed definition of representative strain should be applied by first identifying the Hollomon law parameters (σ_y , n and E) using a predictive model of physical quantities from spherical indentation ($a - F$). Once these parameters are identified, the representative strain is obtained by calculating the gradient of the measured quantities to the identified mechanical parameters.

Applying the gradient representative strain to data obtained from numerical models using finite element commercial software leads to noisy signals and takes a long calculation time. To overcome these difficulties, we propose a new analytical model that covers most of the practical metals used in the industry. The values of the obtained representative strain strictly depend on the mechanical parameters of the assumed law. The results also show that it is possible to find points with coordinates (representative strains; representative stresses) very close to the hardening law obtained from a classical tensile test.

Notations: F : Force; a : contact radius; σ_y : limit of elasticity; n : hardening exponent; E : Young’s modulus

KEYWORDS

Spherical indentation, representative strain, gradient method, metals, mechanical properties

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scientifiques et de transducteurs piézoélectriques. Ces développements ont été réalisés conjointement avec l'EPFL (Ecole Polytechnique Fédérale de Lausanne) et l'EMPA (Laboratoire fédéral de science et technologie des matériaux, de Thoun). Les nouvelles fonctionnalités implémentées sont toujours validées avec le support de partenaires académiques reconnus.



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