Session Posters : liste des résumés

O. Castelnau [a], P. Goudeau [b], G. Geandier [b, c], N. Tamura [c], J.L. Béchade [d], M. Bornert [e], D. Caldemaison [e]

L. Clément [a], F. Cacho [b], R. Pantel [b], D. Delille [a], J.L. Rouvière [c], L. F. Tz. Kwakman [a], C. Wyon [d], C. Tavernier [b], H. Jaouen [b]

M. Eberlein [a,b], S. Escoubas [a], M. Gailhanou [a], O. Thomas [a], P. Rohr [b], R. Coppard [b]

E. Romain [a,c], M. Mermoux [a], A. Crisci [a], D. Delille [b], L. Kwakman [b]

*B. Malard [1, 2, *], T. Pirling [1], K. Inal [2, 3], S. Berveiller [2], E. Patoor [2]*

P. Martins [a], C. Malhaire [a], D. Barbier [a], J. Zimmermann [b]

R.M.S. Martins [a], N. Schell [a], R.J.C. Silva [b], K.K. Mahesh [b], F.M. Braz Fernandes [b]

R. Pesci [a], S. Berveiller [a], E. Patoor [a], K. Inal [b], J.L. Lecomte [c], E. Eberhardt [d]

L. Quémard [a], L. Desgranges [a], V. Bouineau [a], M. Pijolat [b], F. Valdivieso [b], N. Millot [c], J.C. Niepce [c], G. Baldinozzi [d] and A. Poulesquen [e]

D. Rouchon [1,3], M. Mermoux [3], J.M. Hartmann [1], J. Eymery [2], F. Andrieu [1], A. Crisci [3]

White beam microdiffraction experiments for the determination of the local plastic behaviour of polycrystals.

O. Castelnau [a], P. Goudeau [b], G. Geandier [b, c], N. Tamura [c], J.L. Béchade [d], M. Bornert [e], D. Caldemaison [e]

[a] *LPMTM (CNRS), Université Paris 13, av. J.B. Clément, F - 93430 Villetaneuse (France)*
[b] *LMP, Universite de Poitiers, Avenue Marie et Pierre Curie, 86962 Futuroscope (France)*
[c] *ALS, LBNL, 1 Cyclotron Road MS 2R0200, Berkeley, CA 94720 (USA)*
[d] *CEA-Saclay, DEN / DMN / SRMA / LA2M, 91191 Gif-sur-yvette Cedex (France)*
[e] *LMS - Ecole Polytechnique, 91128 Palaiseau Cedex (France)*

Contact : J-L Béchade, Tél. : (33) 1 69 08 41 42, Fax. : (33) 1 69 08 71 30
e-mail : jean-luc.bechade@cea.fr

The overall plastic behavior of polycrystalline materials strongly depends on the microstructure and on the local rheology of individual grains. The characterization of the strain and stress heterogeneities within the specimen, which result from the intergranular mechanical interactions, is of particular interest since they largely control the microstructure evolutions such as texture development, work-hardening, damage, recrystallization, etc. The influence of microstructure on the effective behavior can be addressed by physical-based predictive models (homogenization schemes) based either on full-field or on mean-field approaches. But these models require the knowledge of the grain behavior, which in turn must be determined on the real specimen under investigation. The microextensometry technique that has been developed at the LMS-X allows the determination of the surface total (i.e. plastic + elastic) strain field with a micrometric spatial resolution. On the other hand, the white beam X-ray microdiffraction technique developed recently at the Advanced Light Source enables the determination of the elastic strain with the same spatial resolution. For polycrystalline materials with grain size of about 10 micrometers, a complete intragranular mechanical characterization can thus be performed by coupling these two techniques. The very first results obtained on plastically deformed copper and zirconium specimens are presented.

Investigation of process induced strain in MOS transistor by CBED method.

L. Clément[a], F. Cacho[b], R. Pantel[b], D. Delille[a], J.L. Rouvière[c], L. F. Tz. Kwakman[a] , C. Wyon[d], C. Tavernier[b], H. Jaouen[b]

[a] *PHILIPS SEMICONDUCTORS,860 rue Jean Monnet, F-38920 CROLLES, FRANCE*

[b] *STMICROELECTRONICS, 850 rue Jean Monnet, F-38926 CROLLES, FRANCE*

[c] *DRFMC/SP2M/LEMMA, CEA-Grenoble ,17 rue des Martyrs, F-38054 GRENOBLE Cedex 9, FRANCE*

[d] *CEA-LETI, 850 rue Jean Monnet, F-38926 CROLLES, FRANCE*

Contact : L. Clément, tel : (+33) (0) 4 38 92 35 01
e-mail : laurent-renaud.clement@philipscrolles.st.com

Process induced stress occurs at several steps in the manufacturing of integrated circuits. As it is mandatory to characterize and control this stress experimentally, a method using Transmission Electron Microscopy (TEM) is well adapted to the scale of interest: the Convergent Beam Electron Diffraction (CBED) technique. It consists in focusing a convergent electron beam on the crystal and in examining HOLZ lines position in the diffraction pattern. Strain can then be determined with a resolution of 2.10^{-4} and with a spatial resolution of 1nm [1].

However, stress relaxation in TEM lamella can lead to CBED patterns distortions with HOLZ lines broadening [2]. So, accurate determination of strain from thin foil electron microscopy techniques requires quantitative stress relaxation effect analysis.

We propose a method to characterize stress in silicon substrates used for microelectronics devices by coupling mechanical modeling of the partially relaxed structure in the thin TEM lamella with dynamical electron diffraction theory, in order to reproduce experimental CBED patterns.

An application to quantitative stress and strain maps determination in periodic MOS transistors integrating nickel silicide NiSi and tensile strained nitride Si_3N_4 Contact Etch Stop Layer (CESL) is then presented.

[1] *Strain determination in silicon microstructures by combined convergent beam electron diffraction, process simulation, and micro-Raman spectroscopy* V. Senez, A. Armigliato, I. De Wolf, C. Carnevale, R. Balboni, S. Frabboni, and A. Benedetti, Journal of Applied Physics, 94(9):5574-5583, 2003

[2] *Strain measurements by convergent-beam electron diffraction : The importance of stress relaxation in lamella preparation*' L. Clément, R. Pantel, L.F.Tz Kwakman, and J.L. Rouvière, Applied Physics Letters, 85(4):651-653, 2004

High Resolution X-Ray Diffraction measurement of the local strains induced in Si by periodic arrays of oxide filled trenches

M. Eberlein[(a,b)], S. Escoubas[(a)], M. Gailhanou[(a)], O. Thomas[(a)], P. Rohr[(b)], R. Coppard[(b)]

[(a)] *TECSEN CNRS, Université Paul Cézanne, 13397 MARSEILLE cedex 20, FRANCE*
[(b)] *ATMEL ROUSSET, Zone Industrielle, 13106 ROUSSET cedex, France*

Contact : M. Eberlein, tél : +33 (0)4 91 28 81 23, fax : +33 (0)4 91 28 27 93
e-mail :michel.eberlein@laposte.net

The continuous downscaling of microelectronic devices makes the issue of mechanical stresses increasingly critical. We propose investigating the periodic strain field [1] induced in a single crystal substrate by High Resolution X-ray Diffraction. Elastic calculations with a Finite Element Modeling (FEM) code are necessary to extract the displacement field, used for structure factor calculations [2]. The simulated diffraction maps are compared with experimental ones and allow for a complete validation of the strain field.

Investigated samples are periodic arrays of SiO_2-filled trenches in (001) Si single crystal (250-400nm deep) The Shallow Trench Isolation (STI) process induces a periodic strain field in silicon which gives rise to distinct satellites in reciprocal space. Their spacing is directly linked to the period, and their envelope is correlated to the strain field. In order to extract axial strains in the 3 directions, both 004 symmetric and 224 or 404 asymmetric Si reciprocal space maps were recorded in high resolution configuration. We have investigated the strain field evolution with decreasing period, from 2 to 0.2µm.

With a 2µm periodicity, on 004 reciprocal maps, the Si diffracted envelope intensity is shifted to a lower L value compared to the substrate one. This can be related to a tensile strain in the vertical direction ($\varepsilon zz>0$). On asymmetric maps, the envelope is also shifted to a higher H value, which indicates a compression across the trenches. FEM calculations show a strain gradient in Si. The higher strain values are located close to the sample surface and trench interfaces. Because the period is large satellites are closely spaced in reciprocal space and are thus difficult to separate.

For samples with a 0.58µm period, a secondary intensity maximum is evidenced. This can be understood from the FEM calculations. Indeed, the calculated strain depth profiles show plateaus or weak variations (ε_{xx} and ε_{zz} remains stable at +/- 10^{-4} on a depth of 180nm). Thus the secondary peak may be interpreted as coming from this homogeneously strained area. The measurement of the position of this secondary peak allows for a direct determination of the strain from the reciprocal space maps. For 0.24µm wide lines, we measured homogeneous strains $\varepsilon zz =1.61*10^{-3}$ (vertically) and $\varepsilon xx =1.6*10^{-3}$ (across Si lines). For increasing lines width (0.34µm), strains are ~20% lower.

When the period is scaled down to 200nm (close to dimensions of next generation devices), the secondary diffraction peak is still visible. The same interpretation is possible but requires synchrotron radiation for better intensity dynamics. Recent measurements performed at ESRF on BM32 beam line confirm the increasing strain value with decreasing Si lines width down to 50nm.

[1] T. Baumbach, D. Lübbert, and M. Gailhanou, *J. Appl. Phys.* 87 (8), 3744 (2000).
[2] A. Loubens, C. Rivero, Ph. Boivin, B. Charlet, R. Fortunier, O. Thomas, *Mater. Res. Soc. Symp. Proc.* Vol. 875 (2003).

Mesure de contraintes locales dans le silicium par spectroscopie Raman UV : expérience et simulation

E. Romain[a,c], M. Mermoux[a], A. Crisci[a], D. Delille[b], L. Kwakman[b]

[a] Laboratoire d'Electrochimie et de Physico-chime des Matériaux et des Interfaces, 1130 rue de la piscine, 38402 St Martin d'Hères, France, [b] Philips Semiconductors, 860 rue Jean Monnet, 38926 Crolles cedex, France, [c] Freescale Semiconducteurs, 870 rue Jean Monnet, 38926 Crolles cedex, France

Contact : E. Romain, tel : (+33) (0) 4 76 82 65 71, fax : (+33) (0) 4 76 82 66 77
e-mail : eddy.romain@lepmi.inpg.fr

La spectroscopie Raman est une technique de caractérisation généralement non destructive et très sensible aux états de déformation de la matière. L'utilisation de cette technique dans le cadre de mesures de contraintes locales autour de dispositifs microélectroniques est un enjeu important pour l'industrie microélectronique. L'effet des contraintes/déformations sur le spectre Raman du silicium est largement documenté. Toutefois, la résolution du tenseur de contrainte n'est pas directe et nécessite une modélisation. Nous montrons comment l'utilisation conjuguée de la spectroscopie Raman UV (363 nm) et de la modélisation mécanique permet de dépasser les limitations usuelles de ces techniques prises séparément : par simulation et fit sur les profils Raman UV expérimentaux (lignes larges L = 10 µm), un modèle mécanique est validé, puis redimensionné pour la prédiction des états de contrainte sur des structures plus petites (lignes étroites L = 0.3 µm).

Enfin, il sera également question de cartographie Raman, puisque celle-ci présente un intérêt considérable pour le repérage et la mesure de distributions de contrainte particulières. Là encore, l'excellent accord qui est obtenu entre l'expérience et la modélisation démontre la faisabilité et l'intérêt que présente une telle technique appliquée à la mesure de contrainte dans des dispositifs microélectroniques.

Stress determination during the mechanically-induced martensite phase transformation in the superelastic alloy CuAlBe by neutron diffraction.

B. Malard[1, 2,*], T. Pirling[1], K. Inal[2, 3], S. Berveiller[2], E. Patoor[2]

[1] *I.L.L / E.S.R.F - 6 rue Jules Horowitz – BP 156 - 38042 Grenoble Cedex 9, France*
[2] *LPMM / ENSAM - 4, rue Augustin Fresnel - Metz Technopôle - 57078 Metz Cedex 3, France*
[3] *Now at MECASURF / ENSAM - 2 cours des Arts et Métiers - 13617 Aix-en-Provence, France*

Contact : B. Malard: malard@ill.fr; Phone: +33-4.76.20.74.80

This paper focuses on the study of the superelastic behavior associated to stress induced martensite transformation in a Cu-11.5%Al-0.5%Be [wt. %] Shape Memory Alloy (SMA). Neutron diffraction was used to track the evolution of the transformation textures after martensite germination and the residual stress after loading. In-situ tensile tests were carried out on SALSA, the new ILL strain imager dedicated to the determination of residual stress, in order to determine the average distribution of strains and stresses in the austenite phase. Cylindrical specimens were used; having a grain size of about 100-150μm. Additional texture measurements were done on D20, the ILL high intensity 2-axis diffractometer equipped with a large microstrip detector.

Methodology: The first experiment involved the strain analysis of the bulk specimens, using the {422} plane family of the austenitic phase. We have used the $\sin^2\psi$ method with step of $\psi=5°$ ($0°<\psi<35°$ and $90<\psi<60°$) at 10 different applied stresses during a tensile loading cycle. The determined stresses from the bulk in the tensile direction obtained by this method were compared to: 1) The three principal stress method frequently used in neutron experiment and calculated using εxx, εyy, εzz. 2) The surface stress studies obtained by X-ray diffraction on the same type of alloy [1]. These comparisons were made in order to develop the methodology for further investigations. In the second experiment, texture in the austenite has been measured after unloading using an Eulerian cradle and tilting steps of $\Delta\varphi=\Delta\psi=5°$.

Results obtained: 1/ In the elastic regime the results of the $\sin^2\psi$ method correspond to the macroscopic applied stress. This result validates the methodology. 2/ The transformation starts when the applied stress reaches 240MPa. Then, the difference between the stress in the austenitic phase and the applied stress increases gradually until 80MPa at a total strain of 4.2% ($\sin^2\psi$ method). This is due to the load transfer between the two phases. 3/ After unloading some residual stress in the austenitic phase and a moderately marked fiber texture have been measured.
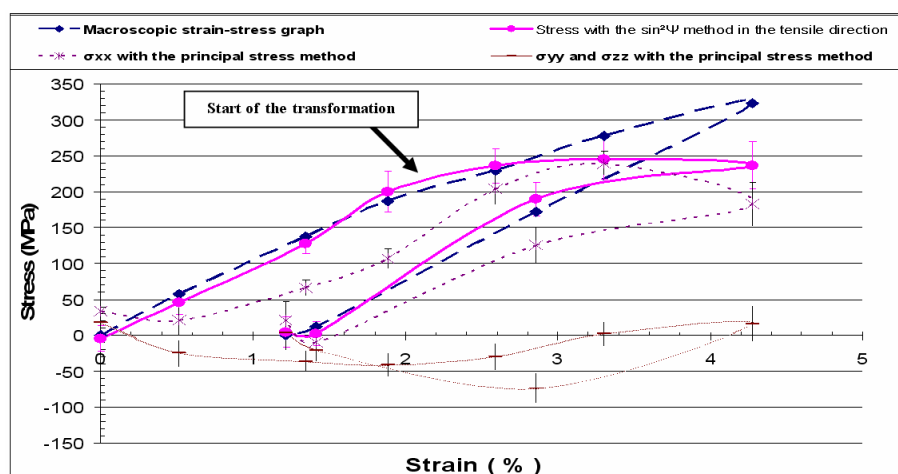
Evolution of the stress tensor components of austenite phase during a tensile cycle measured on SALSA.

[1] Stress analysis of martensitic transformation in Cu-Al-Be polycrystalline and single-crystalline shape memory alloy. B Kaouache, S. Berveiller, K. Inal, A. Eberhardt, E. Patoor. Materials science & Engineering A 378 (2004) 232-237.

Détermination des contraintes mécaniques dans les films minces submicroniques par l'utilisation de microsystèmes

P. Martins[a], C. Malhaire[a], D. Barbier[a], J. Zimmermann[b]

(a) Laboratoire de Physique de la Matière (LPM), UMR CNRS 5511, INSA de Lyon, 7 avenue Jean Capelle, bât. Blaise Pascal, 69621 VILLEURBANNE Cedex, FRANCE
(b) Enserg-IMEP-Minatec, Parvis Louis Néel, 38054 GRENOBLE Cedex 9, FRANCE

Contact: P. Martins, tel: (+33) (0) 4 72 43 87 95, fax: (+33) (0) 4 72 43 60 82
e-mail: paolo.martins@insa-lyon.fr

La réduction des échelles des systèmes microélectroniques, MEMS (Micro Electro Mechanical Systems) et NEMS (Nano Electro Mechanical Systems) ainsi que la réalisation de multicouches de plus en plus complexes requièrent des études mécaniques sur les matériaux utilisés. L'étude des microdéformations de micro-nano-structures mobiles permet de déterminer à la fois l'état de contrainte moyen et les paramètres élastiques (module d'Young, coefficient de Poisson,…) de films minces d'épaisseurs submicroniques.

Ce travail se déroule dans le cadre d'un projet financé par la région Rhône-Alpes ayant pour but d'étudier le comportement mécanique de structures submicroniques et de définir de nouvelles structures de tests.

Nous présentons deux méthodes de détermination des contraintes et paramètres élastiques. La méthode du gonflement de membrane (Bulge test) consiste à mesurer la déformation d'un film autoportant sous une pression appliquée. Un modèle ajusté au moyen de simulations 3D par éléments finis permet de remonter aux contraintes dans des films minces de nitrure (Si_3N_4) et un bicouche nitrure/oxyde (Si_3N_4/SiO_2) de respectivement 100 et 200 nm d'épaisseur. La réalisation de membranes de différentes dimensions permet de déterminer le coefficient de Poisson des films.

L'état de contrainte des films minces est aussi déterminé à partir de la méthode du recourbement de micropoutres bimorphes film/silicium (microbeam bending). La déformation statique des poutres, conséquence d'une relaxation partielle de la contrainte dans le film, est mesurée par microscopie interférométrique. Un modèle mécanique permettant de calculer la contrainte de films d'inconel à partir de la courbure de poutres de différentes longueurs est validé au moyen de simulations par éléments finis.

D'autres types d'études mécaniques peuvent s'appliquer à ces deux types de microstructures : mesure de fréquence de résonance par vibrométrie, déformation par nanoindentation, mesure en température…

In-situ study of Ni-Ti film growth by synchrotron radiation scattering.

R.M.S. Martins[a], N. Schell[a], R.J.C. Silva[b], K.K. Mahesh[b], F.M. Braz Fernandes[b]

(a) *Rossendorf Beamline (ROBL)-CRG, ESRF, B.P. 220, F-38043 GRENOBLE, FRANCE*
(b) *CENIMAT, Campus da FCT/UNL, 2829-516 Monte de Caparica, PORTUGAL*

Contact : R.M.S. Martins, tel : (+33) (0) 4 76 88 28 72, fax : (+33) (0) 4 76 88 25 05
e-mail : rui.martins@esrf.fr

Ni-Ti thin films are promising high performance materials in the field of micro-electro-mechanical system (MEMS) applications. Their preferential orientation is a crucial factor in determining the shape memory behavior since it has a strong influence on the extent of the strain recovery. The relationship between structure and deposition parameters is of extreme importance for future device applications. Our approach is *in-situ* x-ray diffraction *during* deposition carried out in a process chamber installed at a synchrotron radiation beamline. Near-equiatomic films ($\approx 800°\mu m$) were co-sputtered from Ni-Ti and Ti targets on heated substrates ($\approx 470°C$) without applying a substrate bias voltage. The texture evolution during deposition is clearly affected by the substrate type. On naturally oxidized Si(100) substrates the Ni-Ti B2 phase starts by stacking onto (h00) planes, and, as the thickness increases, evolves to a (110) fiber texture. An initial significant change of the lattice parameter, as calculated from $d_{(200)}$, is observed and its tendency for stabilization is coincident with the deposition time where the preferential stacking of B2 phase on (110) planes is starting.

For the deposition on thermally oxidized Si(100) there is a strong preferential stacking on (h00) planes of B2 leading to a (100) fiber texture. The measured lattice constants do not exhibit such a strong initial variation as for the sample deposited on naturally oxidized Si(100) substrate, but a continuous slight decrease of this value is perceptible [1].

Ni-Ti films were also deposited on top of a TiN buffer layer. There is a preferential growth of <110> oriented grains of the Ni-Ti B2 phase [<hkl> grains are defined as grains with a plane from the {hkl} family parallel to the film surface] from the beginning of the deposition, with a constant growth rate during the whole deposition for a Ni-Ti film deposited on TiN with a topmost layer formed mainly by <111> oriented grains. The Ni-Ti films deposited on top of a TiN layer where a dominating orientation could not be identified (primarily <001> and <111> oriented grains nucleate and grow) exhibit a different behavior. In this case <110> oriented grains of the Ni-Ti B2 phase dominate at small thicknesses while <211> oriented grains take over at larger thicknesses. The decrease of the lattice parameter suggests that the films experience compressive stress which is significantly relaxed with increasing film thickness.

[1] *'Growth of sputter-deposited Ni-Ti thin films: effect of a SiO$_2$ buffer layer'* R.M.S. Martins, N. Schell, M. Beckers, K.K. Mahesh, R.J.C. Silva & F.M.B. Fernandes, *Applied Physics A: Materials Science & Processing* **84** (2006) 285-289

Inter- and Intragranular Stress Determination with Kossel Microdiffraction in a Scanning Electron Microscope.

R. Pesci[a], S. Berveiller[a], E. Patoor[a], K. Inal[b], J.L. Lecomte[c], E. Eberhardt[d]

[a] *LPMM UMR CNRS 7554 - ENSAM, 4 rue Augustin Fresnel Metz Technopôle, F-57078 Metz cedex 3, FRANCE,*
[b] *MECASURF - ENSAM, 2 cours des Arts et Métiers, F-13617 Aix-en-Provence cedex 1, FRANCE,*
[c] *LETAM UMR CNRS 7078,* [d] *ENIM, Ile du Saulcy, F-57054 Metz cedex 1, FRANCE*

Contact : R. Pesci, tel : (+33) (0) 3 87 37 54 30, fax : (+33) (0) 3 87 37 54 70
e-mail : raphael.pesci@metz.ensam.fr

Kossel microdiffraction in a Scanning Electron Microscope (SEM) is developed to determine the crystalline orientation as well as the inter- and intragranular strains and stresses on the micronic scale. When a focused electron beam excites a material, the latter emits its characteristic X-rays which are then diffracted on the crystallographic planes according to the Bragg equation, as so-called Kossel cones. Several reflections occur simultaneously, in all directions: one cone is emitted for each (hkl) diffracting plane. A CCD camera is placed so that its screen intercepts some of these cones, emitted from the area affected by the electronic spot (1 μm^3): one obtains thus Kossel lines that are transmitted to a computer for evaluation [1].

The aim is to benefit from the SEM environment to associate determined stress values with the microstructure evolution (slip line appearance, particle decohesion, crack). Indeed, this local analysis technique is very efficient because it also permits the observation of the microstructure, and in particular during in-situ tests, in order to realize for example stress profiles in the material (along grain boundaries or inside one grain, in different phases and even close to crack initiation). Besides, the analysis is quasi-instantaneous since it only lasts a few minutes.

In-situ tensile tests are therefore performed inside a microscope, using a small tension/compression machine equipped with a temperature regulating system [-160°C,300°C]. Several materials are successfully analyzed (copper, pure iron, pressure vessel bainitic steel): a shape memory alloy presenting a phase transformation (appearance of martensite beyond a certain stress rate) is investigated in particular, to show not only the different Kossel line patterns coming from each phase, but also the stress sensitivity of these Kossel curves (additional experiments realised with synchrotron radiation at the European Synchrotron Radiation Facility in Grenoble are also presented). Indeed, a lattice strain corresponds to Kossel line displacement which can be directly observed with the camera. All the Kossel curves are then indexed using a home-developed simulation program, which gives the Miller indices of all the diffracting planes and the orientation of the studied crystal in space. The determination of an unknown stress state finally remains to be introduced in the program using the single crystal Ortner's method and Hooke's law, and to be validated with the well-known strain/stress behavior of a single crystal.

[1] '*Inter and intra granular strain analysis by microdiffraction Kossel*' S. Berveiller, P. Dubos, K. Inal, A. Eberhardt & E. Patoor, Materials Science Forum **490-491** (2005) 159-165

Subdivision of the UO$_2$ Nuclear Fuel in Oxidizing Environment

L. Quémard[a], L. Desgranges[a], V. Bouineau[a], M. Pijolat[b], F. Valdivieso[b], N. Millot[c], J.C. Niepce[c], G. Baldinozzi[d] and A. Poulesquen[e]

[a] CEA Cadarache, DEN/DEC/SA3C/L2EC, Bat.315/316, 13108 St. Paul-lez-Durance.

[b] LPMG, Ecole Nationale Supérieure des Mines de St-Etienne, Centre SPIN, Equipe PROCESS, 158, Cours Fauriel, 42023 St-Etienne CEDEX 2.

[c] LRRS, Equipe MANAPI, UMR 5613 CNRS-Université de Bourgogne, 9 Av. Alain Savary BP 47870, 21078 Dijon.

[d] SPMS, UMR 8580 CNRS-Ecole Centrale Paris, Grande Voie des Vignes, 92295 Châtenay-Malabry.

[e] CEA Saclay, DEN/DPC/SECR/L3MR, Bat. 450, 91191 Gif-sur-Yvette.

Contact : L. Quémard, tel : (+33) (0) 4 42 25 70 00, fax : (+33) (0) 4 42 25 36 11
e-mail : ludovic.quemard@cea.fr

In a dry air storage facility, the oxidation of used/irradiated nuclear fuel is considered as the main risk induced by a leakage that would put the nuclear ceramics in contact with air. Indeed, the storage temperature can be up to 150°C for several years and the oxidation UO$_2$ ⇨ U$_3$O$_8$ (cubic ⇨ orthorhombic) can lead to a relative volume swelling of more than 35% which could cause the ruin of the fuel rod and the release of radionuclides in the environment [1].

Phenomenological and kinetic studies of the oxidation of fuel fragments and UO$_2$ single crystals are in progress to predict the long-term behavior of the used fuel [2]. Thus, a dramatic subdivision process of these samples is highlighted after only few hours at 285°C. It occurs by successive cleavages for the single crystals and by cracking of the grain boundaries followed by intra-granular failures for the fuel fragments.

In-situ measurements of the macro and micro-stresses in a UO$_2$ single crystal by X-Ray Diffraction are planned to complete the study of the damaging of the fuel in oxidizing environment. These results could improve the knowledge of the elementary mechanisms of the subdivision process and come in support of a modeling of the used fuel mechanical behavior.

[1] "*A review of the oxidation of uranium dioxide at temperatures below 400°C*", R.J. McEachern and P. Taylor, J. Nucl. Mater., **254** (1998) 87-121

[2] "*Fragmentation du combustible nucléaire en environnement oxydant: Etude cinétique et phénoménologique*", L. Quémard, L. Desgranges, V.Bouineau, F. Valdivieso, M. Pijolat, N. Millot, J.C. Niepce, G. Baldinozzi and A. Poulesquen, MATERIAUX 2006, Dijon, 13-17/11/2006 (2006)

Etude par spectroscopie Raman UV de systèmes multi-couches s-Si / Si$_{1-x}$Ge$_x$ et s-sol : effet de l'épaisseur et de la dimension des motifs sur la contrainte

D. Rouchon[1,3] , M. Mermoux[3], J.M. Hartmann[1], J. Eymery[2], F. Andrieu[1], A. Crisci[3]

[1]*CEA-DRT-LETI-CEA/GRE, 17 rue des martyrs 38054 Grenoble cedex 9*
[2]*CEA/DRFMC 17 rue des martyrs 38054 Grenoble cedex 9*
[3]*LEPMI/ENSEEG, 1130 rue de la piscine, Domaine Universitaire, BP 75, 38402 St Martin d'Hères*

La course à l'augmentation de la densité d'intégration et des performances des circuits intégrés impose la réduction constante des dimensions des composants en microélectronique. Cette miniaturisation conduit à proposer la mise en œuvre de nouveaux matériaux permettant d'optimiser les performances du transistor CMOS. En particulier, le canal du transistor placé entre source et drain, actuellement constitué de silicium massif, pourrait être remplacé par des couches minces contraintes de Si, Ge ou SiGe. La valeur de la contrainte dans le canal dépend alors de sa taille, de sa stabilité thermique, voire également de phénomènes d'interdiffusion entre le silicium et le germanium. Le but ultime recherché est l'augmentation de la mobilité des porteurs (trous et électrons) dans la couche contrainte entraînant un gain en courant des transistors sub-100nm supérieur à 10%.

Compte tenu de la réduction de la taille des composants, l'utilisation de techniques de caractérisations tels que la MET, le CBED et la spectroscopie Raman est devenue indispensable pour mettre en évidence et quantifier les contraintes locales.

Les atouts de la spectroscopie Raman sont connus : analyse non destructive, taille de sonde voisine du μm, possibilité de restreindre l'analyse à l'extrême surface en utilisant une longueur d'onde dans le proche UV, possibilité d'obtenir des images Raman que l'on peut traduire en images de déformations, …

Nous présentons quelques études réalisées sur les différents matériaux utilisables pour les futures technologies CMOS : silicium contraint épitaxié sur alliage silicium-germanium (s-Si/Si$_x$Ge$_{1-x}$) et silicium contraint sur isolant (s-SOI). L'effet de la dimension des motifs est discuté, les résultats issus des mesures Raman sont confrontés aux mesures de diffraction.

Mesures de macro et micro déformations de cuivre par rayonnement synchrotron

O. Sicardy[a], J.S. Micha[b], F. Rieutord[b], O. Robach[b]

[a]DRT/LITEN, [b]CEA/DRFMC - UMR 5819 SPRAM
CEA-Grenoble, 17 rue des Martyrs, 38054 GRENOBLE Cedex 9

Des mesures de macro et micro déformations d'interconnexions en cuivre électrolytique ont été réalisées par diffraction de rayons X sur le goniomètre multi-technique de la ligne IF-BM32 de l'ESRF. L'échantillon étudié est constitué d'un réseau de lignes d'épaisseur 350 nm et de largeur 300 nm, présentant une texture mixte composée d'une famille majoritaire orientée [111] et d'une famille minoritaire orientée [100].

Afin de pouvoir exploiter les profils de raies en termes de taille de grain et de micro-déformations, les conditions opératoires ont été optimisées pour réduire la largeur instrumentale des pics et augmenter la statistique de comptage. Le faisceau utilisé est très peu divergent, sa taille au niveau de l'échantillon a été réduite à 0,5 x 0,5 mm² et la fenêtre d'entrée du compteur, située à 830 mm de la zone d'analyse, a été limitée à 0,5 x 0,5 mm². Dans ces conditions, les volumes diffractant contiennent peu de grains et la statistique de comptage a été améliorée par des petites oscillations en θ (incidence de faisceau sur l'échantillon) ou en ω (rotation autour de la normale à la surface de l'échantillon) pour obtenir des profils de raies lisses.

Il a ainsi été possible d'analyser les déformations du cuivre sur les 2 familles d'orientation [111] et [100]. Cette analyse a concerné non seulement les plans cristallins parallèles à la surface mais aussi des plans inclinés orientés en azimut dans l'axe des lignes ou dans la direction transverse à l'axe.

Synchrotron Laue micro-diffraction: a new beam line project at SOLEIL for phase identification and mechanics of materials

P. Goudeau [(a)], O. Thomas [(b)]

(a) LMP UMR CNRS 6630 - Université de Poitiers, SP2MI, Bd Marie et Pierre Curie, BP 30179, 86962 Futuroscope Chasseneuil, France, (b) TECSEN UMR CNRS 6122 - Université Paul Cezanne (Aix-Marseille III), Fac. des Sciences et Techniques de St Jérôme, 13397 Marseille

Micro-focused x-ray beams from 1 down to less than 0.1 micron in size have been one of the real success stories of 3rd generation synchrotron x-ray machines (SR), thanks to a large panel of focusing devices for hard and soft x-rays [1-5]. There is clearly a rapidly growing need for very small x-ray beams – 10 nm is theoretically possible - which allow for non-destructive local scale measurements of structure and chemistry. This need encompasses many different scientific fields: Microelectronics and microsystems, Metallurgy and mechanics, Environmental and earth sciences, Art and archaeology, Life sciences and soft condensed matter. In all these different research fields one would ideally like to get information on a local scale of the structure, the chemical composition and the local atomic environment. This implies performing at the submicron scale: XRD, XRF, EXAFS, XANES. However, the case of XRD technique is specific in the sense that the recorded signal does not only depend on the beam size but also on the ratio of the beam size to the grain size. If this ratio is large enough one is left with powder diffraction when using monochromatic x-ray beam (MB) and the diffraction information is an average over the size of the beam. On the other hand if the beam size over grain size ratio is small one gets single crystals diffraction. It is thus possible to obtain intra grain structural information. Transmission configuration with hard x-rays is done using either MB or white beam (WB) XRD with generally energy dispersive mode. For the proposed beam line, the working geometry is reflection. It is important to realize, however, that recording a significant number of diffraction spots from a single crystal requires either a movement of the sample under the beam (goniometry) or the use of a polychromatic incoming beam (Laue diffraction) combined with a two-dimensional area detector such as CCD type. Since there are no available goniometers with a sphere of confusion (SOC) radius smaller than the micrometer x-ray probe size it is thus necessary to use WB. Details concerning the different applications of the technique may be found for instance at the Advanced Light Source (ALS) web site address (http://xraysweb.lbl.gov/microdif/index.htm), this synchrotron source being close enough to the French facility SOLEIL. This beam line project is unique in Europe since there is almost nothing in European countries in terms of fully dedicated WB μXRD beam line except the BM32 project at ESRF (10 % of the full beam line time dedicated to microfocus WB during 3 years). The Scientific Advisory Committee (SAC) of SOLEIL approved the APS (preliminary beam line project*) in November 2005 and the SOLEIL Council decided in July 2006 to build this additional beam line and to provide ¼ of the total beam line budget which will be completed by external funds (not yet found).

* Available by e-mail at pgoudeau@univ-poitiers.fr (pdf file ~ 4 MO)

[1] F. Pfeiffer, C. David, M. Burghammer, C. Rieckel, T. Salditt (2002) *Science* **297** 230
[2] C. G. Schroer, B. Lengeler (2005) *Physical Review Letters* **94** 054802
[3] W. Chao, B.D. Hartenaeck, J.A. Liddle, E.H. Anderson, D.T. Attwood (2005) *Nature* **435** 1210
[4] B. C. Larson, W. Yang, G. E. Ice, J. D. Budai, J. Z. Tischler (2002) *Nature* **415** 887
[5] N. Tamura, R. S. Celestre, A. A. MacDowell, H. A. Padmore, R. Spolenak, B. C. Valek, N. Meier Chang, A. Manceau, J. R. Patel (2002) *Review of Scientific Instruments* **73** 1369

White (2-30keV) microbeam 0.5x0.7µm2 on the new BM32 beamline CRG-IF

X. Biquard[b], O. Geaymond[d], J.S. Micha[a], F. Rieutord[b], O. Robach[b], O. Ulrich[b], G. Rolland[c]

[a] CNRS / UMR SPrAM , [b] CEA-DRFMC, [c] CEA-LETI, [a][b][c] CEA-Grenoble ,17 rue des Martyrs, F-38054 GRENOBLE Cedex 9, FRANCE, [d] CNRS / Lab. de Cristallographie, 25 Avenue des Martyrs , B.P. 166 , 38042 GRENOBLE Cedex

Contact : X. Biquard, tel : (+33) (0) 4 76 88 29 23, e-mail : xavier.biquard@cea.fr

This poster presents the actual design and achieved performances on the new CRG-IF white beam microdiffraction beamline at ESRF.

The fundamental design point is our two-step demagnification of the source to prevent source vibration sensitivity, reduce µspot jitter and achieve stability of size and impact point going from white to monochromatic µbeam.

Concerning performance, we are now able to focus a white beam ranging from 2 to 30keV on a less than 1 µm diameter spot, with enough flux to saturate the Si(004) wafer diffraction peaks in less than 5s. We were also able to record a µ-Laue diagram on a 300nm wide Cu line and the smallest achieved µspot size is $0.5 \times 0.7 \mu m^2$

Thin film delamination study during in situ compressive testing by Scanning micro X-ray Diffraction

P. Goudeau[a], P.-O. Renault, J. Colin[a], C. Coupeau[a], F. Foucher[a], G. Geandier[b], N. Tamura[b]

(a) LMP-UMR 6630 CNRS, Université de Poitiers – SP2MI, F-86962 Chasseneuil
(b) ALS, LBNL, 1 Cyclotron Road, MS 2-400, Berkeley, CA, 94270, USA

The understanding of the mechanical properties of coated materials is a key factor in a number of technological applications. In particular, the delamination of compressed thin films is one of most limiting factor for the structural performance of the material and presents various interesting problems in physics and mechanics.

The delamination patterns result from relaxation phenomenon of high residual compressive stresses in the thin film mainly due to the deposition process and involve the propagation, from an initial buckle, of cracks at the film/substrate interface. They have been extensively investigated in various experimental and theoretical studies [1-4]. However, only a few experimental apparatus have been developed to measure the buckle growth dynamics. In fact, the additional compressive stress can be produced in the adherent thin film by using an experimental apparatus which allows the in-situ observation by AFM of the surface during deformation. Buckling patterns generated during the stress experiments are then described and discussed taking advantage of the high resolution offered by the atomic force microscopy (AFM). It has been observed that buckling structures may evolve from straight-sided wrinkles to either worm-like or varicose patterns when the applied stress is released. The formation of these two structures from the initial straight-sided wrinkles has been characterized. In particular, it has been found that one of the driving forces of delamination is the relaxation of the stresses along the longitudinal axis of the initial wrinkles. In the case of bubbles, the film does not *recombine* with the substrate; in situ picosecond acoustic experiments [2] would allow studying the adhesion between film and substrates in this region of interest. Finite element simulations have been done in order to investigate the delamination evolution [4]. Confrontations with in situ stress mapping measurements have to be achieved for validating and then improving these simulations.

In this work, we proposed to use the Scanning X-Ray microdiffraction technique developed at the ALS to perform in-situ spatially resolved stress measurements [1, 3] during a compression test on a film/substrate set. These measurements are expected to provide new insights on the first stage of the delamination process. Preliminary experiments have been done at ALS in February 2006 on gold thin films deposited on LiF substrates.

[1] Mesoscale x-ray diffraction measurement of stress relaxation associated with buckling in compressed thin films, P. Goudeau, P. Villain, N. Tamura, H. A. Padmore, Applied Physics Letters **83** (1) (2003) 51-53.
[2] Evidence of plastic damage in thin films around buckling structures, C. Coupeau, P. Goudeau, L. Belliard, M. George, N. Tamura, F. Cleymand, J. Colin, B. Perrin, J. Grilhé, Thin Solid Films **469-470** (2004) 221-226.
[3] Strain mapping on gold thin film buckling and silicon blistering, P. Goudeau, N. Tamura, G. Parry, J. Colin, C. Coupeau, F. Cleymand, H. Padmore, Mater. Res. Soc. Symp. Proc. Vol. **875** (010.4) (2005) 1-6.
[4] Effect of substrate compliance on the global unilateral post-buckling of coatings: AFM observations and finite elements calculations, G. Parry, J. Colin, C. Coupeau, F. Foucher, A. Cimetière, J. Grilhé, Acta Materiala **53** (2) (2005) 441-447.

Strain field and crystal coherence in SOI lines using high resolution x-ray diffraction

M. Gailhanou[1], A. Loubens[1,2], J-S. Micha[3], B. Charlet[4], R. Fortunier[2], O. Thomas[1]

(1) Laboratoire TECSEN UMR CNRS 6122, Université Paul Cézanne, 13397 Marseille cedex 20, France
(2) Ecole Nationale Supérieure des Mines de Saint-Etienne
CMP « Georges Charpak », Avenue des anémones, 13541 Gardanne, France
(3) CEA-Grenoble/DRFMC/SI3M/PCM, 17 rue des Martyrs, 38054 Grenoble Cedex 9, France
(4) CEA Leti 17 rue des Martyrs, 38054 Grenoble Cedex 9, France

Silicon on insulator (SOI) periodic arrays of lines are interesting systems for the study of two dimensional strain fields at the nanometer scale as well as objects of technological interest for semiconductor devices fabrication. The influence of local stress fields on the electrical properties of Si-based nanostructures is indeed of increasing concern. The experimental evaluation of stresses at the required scale (few nanometers) remains, however, a very challenging task.

High resolution x-ray diffraction measurements in the vicinity of the 004 reflexion from 100nm thick Silicon On Insulator (SOI) line arrays were recorded at the BM32 beamline of the European Synchrotron Radiation Facility (ESRF). A first result obtained from the comparison of different periods is that interferences between lines occur only if their distance is lower than a particular distance, identified as a an in plane crystal coherence length. This length is around 1 µm and is much lower than the X-ray coherence length. In the case of 1µm wide SOI lines (period 2 µm) there is no interference with the neighbouring lines, and the diffraction pattern recorded in the 004 reciprocal space map can be considered as very close to the diffraction pattern of a single line, because of the high uniformity of the sample.

This diffraction pattern has a very distinct shape (fig. 1) which is directly correlated with the displacement field in the lines. Calculations of the displacement field with Finite Element Modeling together with diffracted intensity calculations show a very good agreement with the measurements. These calculations indicate that the peculiar moustache-like shape is a direct consequence of the strain field inhomogeneity arising close to the edges.
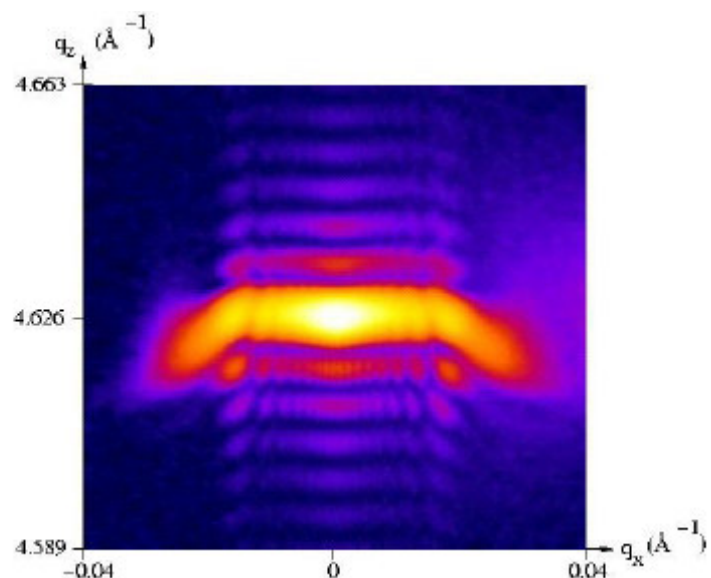

Fig. 1 : 004 reciprocal space map of 1 µm SOI lines