

Plasticity mechanisms in ultrafine grained freestanding aluminum thin films revealed by in-situ transmission electron microscopy nanomechanical testing

Hosni Idrissi, Aaron Kobler, Behnam Amin-Ahmadi, Michael Coulombier, Montserrat Galceran, Jean-Pierre Raskin, Stéphane Godet, Christian Kübel, Thomas Pardoen, and Dominique Schryvers

Citation: [Applied Physics Letters](#) **104**, 101903 (2014); doi: 10.1063/1.4868124

View online: <http://dx.doi.org/10.1063/1.4868124>

View Table of Contents: <http://scitation.aip.org/content/aip/journal/apl/104/10?ver=pdfcov>

Published by the [AIP Publishing](#)

Articles you may be interested in

[The effect of solute segregation on strain localization in nanocrystalline thin films: Dislocation glide vs. grain-boundary mediated plasticity](#)

Appl. Phys. Lett. **102**, 241916 (2013); 10.1063/1.4811743

[In situ nanomechanical testing in focused ion beam and scanning electron microscopes](#)

Rev. Sci. Instrum. **82**, 063901 (2011); 10.1063/1.3595423

[Design and evaluation of a nanoscale differential tensile test device for nanofibers](#)

Appl. Phys. Lett. **94**, 103101 (2009); 10.1063/1.3095439

[Low-temperature plastic strain of ultrafine-grain aluminum](#)

Low Temp. Phys. **34**, 665 (2008); 10.1063/1.2967513

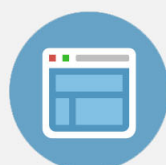
[Stress relaxation of free-standing aluminum beams for microelectromechanical systems applications](#)

Appl. Phys. Lett. **76**, 3415 (2000); 10.1063/1.126664



Re-register for Table of Content Alerts

Create a profile.



Sign up today!



Plasticity mechanisms in ultrafine grained freestanding aluminum thin films revealed by *in-situ* transmission electron microscopy nanomechanical testing

Hosni Idrissi,^{1,2,a)} Aaron Kobler,^{3,4} Behnam Amin-Ahmadi,¹ Michael Coulombier,² Montserrat Galceran,⁵ Jean-Pierre Raskin,⁶ Stéphane Godet,⁵ Christian Kübel,³ Thomas Pardoën,² and Dominique Schryvers¹

¹EMAT, University of Antwerp, Groenenborgerlaan 171, B-2020 Antwerp, Belgium

²Institute of Mechanics, Materials and Civil Engineering, Université catholique de Louvain, Place Sainte Barbe 2, B-1348 Louvain-La-Neuve, Belgium

³Institute of Nanotechnology (INT) and Karlsruhe Nano Micro Facility (KNMF), Karlsruhe Institute of Technology (KIT), Hermann-von-Helmholtz-Platz 1, 76344 Eggenstein-Leopoldshafen, Germany

⁴Joint Research Laboratory Nanomaterials (KIT and TUD) at Technische Universität Darmstadt (TUD), Petersenstr. 32, 64287 Darmstadt, Germany

⁵Matters and Materials Department, Université Libre de Bruxelles, 50 Av. FD Roosevelt CP194/03, 1050 Brussels, Belgium

⁶Information and Communications Technologies, Electronics and Applied Mathematics (ICTEAM), Microwave Laboratory, Université catholique de Louvain, B-1348 Louvain-la-Neuve, Belgium

(Received 29 November 2013; accepted 25 February 2014; published online 11 March 2014)

In-situ bright field transmission electron microscopy (TEM) nanomechanical tensile testing and *in-situ* automated crystallographic orientation mapping in TEM were combined to unravel the elementary mechanisms controlling the plasticity of ultrafine grained Aluminum freestanding thin films. The characterizations demonstrate that deformation proceeds with a transition from grain rotation to intragranular dislocation glide and starvation plasticity mechanism at about 1% deformation. The grain rotation is not affected by the character of the grain boundaries. No grain growth or twinning is detected. © 2014 AIP Publishing LLC. [<http://dx.doi.org/10.1063/1.4868124>]

Thin metallic films with grain sizes ranging between 25 nm and 500 nm are commonly used in a variety of microelectronics, coatings, microelectromechanical systems (MEMS), or stretchable electronics applications. These films are subjected to mechanical loads originating from internal stress or external forces caused, for instance, by the thermal expansion mismatch with the substrate.^{1,2} The ductility, strength, creep, and fracture properties of these systems must be thus controlled or adapted to the in-service conditions to preserve functionality. The mechanical properties of thin metallic films are directly related to the fundamental atomistic plasticity mechanisms active at the nanoscale, the associated defect distribution and topology, and the resulting size effects. The most common microstructure-based size effect in polycrystalline materials is the strength dependence on the inverse of grain size, i.e., the famous Hall-Petch effect.^{3,4} When the grain boundary (GB) spacing is reduced down to 50 nm or even smaller, i.e., in so-called nanocrystalline (nc) materials, the Hall-Petch relationship has been shown to break down (i.e., inverse Hall-Petch)^{5–7} due to the activation of GB mediated processes. However, the specific defect properties controlling these processes at the nanoscale such as the GBs character, the local orientation of the grains and the nature of the mechanisms controlling the interactions of the GBs with the elementary atomistic deformation mechanisms, and the resulting size effects are not well documented. Several fundamental questions remain such as the nature of the elementary processes active just above the Hall-Petch breakdown, why these mechanisms shut down and how to

possibly control this transition by microstructure engineering? Recently, the development of a new generation of advanced instruments for *in-situ* transmission electron microscopy (TEM) nanomechanical testing has allowed establishing a one-to-one relationship between load-displacement characteristics and stress-induced microstructure evolution in TEM.^{8–10} Most of the studies reported in the literature have focused on the use of *in-situ* bright field TEM (BF-TEM) or dark field TEM (DF-TEM) and *in-situ* high resolution TEM (HRTEM) giving access to information about crack propagation,^{11,12} dislocation processes,^{13–16} or GB migration in a few well oriented grains.^{17–19} Nevertheless, quantitative analysis in terms of grain size distribution, character of GBs, twinning, and texture evolution using these techniques remains difficult. Very recently, Kobler *et al.*²⁰ have combined automated crystal orientation mapping in TEM (ACOM-TEM) to obtain full orientation maps with nanometer resolution and *in-situ* TEM nanotensile testing on sputtered nc Au thin films, closing the gap between electron back scattered diffraction (EBSD) and BF/DF-TEM. In the present work, it will be demonstrated that a step forward in the investigation of the elementary mechanisms controlling the plasticity of ultra-fine-grained (ufg) materials can be made by combining *in-situ* ACOM-TEM with *in-situ* BF-TEM nanomechanical testing allowing direct visualization of dislocations activity in the ultra-fine grains.

Electron beam evaporated Aluminum (Al) freestanding thin films have been produced using microfabrication techniques based on MEMS-type procedures.^{21–24} The experimental details of the synthesis can be found in the supplementary material.³¹ The investigation of the grain size distribution as

^{a)}Electronic mail: hosni.idrissi@ua.ac.be.

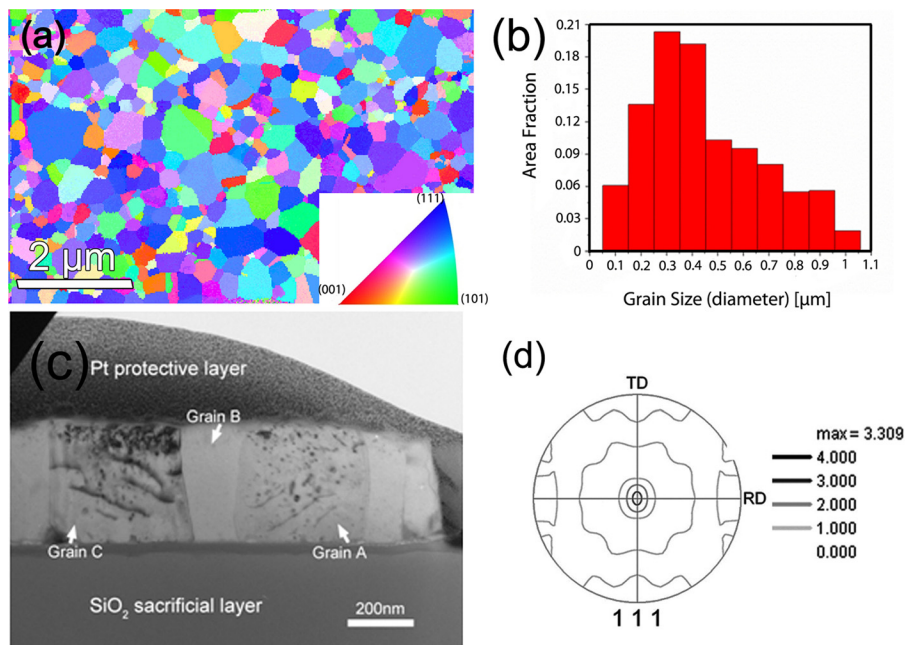


FIG. 1. (a) ACOM-TEM map of as-processed freestanding Al thin films. (b) Grain size distribution extracted from the ACOM-TEM data. (c) BF image obtained on cross-sectional FIB sample. (d) $\{111\}$ pole figure.

well as the crystallographic texture has been performed on the as-processed freestanding Al thin films using ACOM-TEM. The ACOM-TEM plane-view orientation map in Fig. 1(a) shows that the microstructure consists of equiaxed ufg grains and Fig. 1(b) displays the corresponding grain size distribution with a mean value of 342 ± 17 nm. The film thickness is about 300 nm and the film contains, thus, for most of the volume, only one grain over the thickness, as can be seen in the BF-TEM image in Fig. 1(c) obtained from a focused ion beam (FIB) prepared cross-section of an as-deposited Al film. The GBs are almost perpendicular to the plane of the film, indicating a continuous columnar growth. A few dislocations originating from the deposition process can be observed in Fig. 1(c) within well-oriented grains.²⁵

A strong $\{111\}$ texture parallel to the growth direction is revealed by the pole figure of Fig. 1(d). This ufg Al system exhibits grain sizes ranging between 100 nm and $1 \mu\text{m}$, and constitutes thus an ideal candidate to investigate the transition between competing or cooperating intragranular and intergranular deformation mechanisms. *In-situ* TEM uniaxial tensile experiments were performed on submicron Al beams using the PI 95 TEM PicoIndenter from Hysitron, Inc. (see supplementary material³¹). Figure 2 presents the true stress-

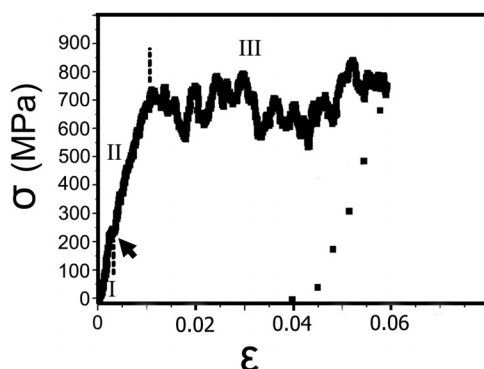


FIG. 2. True stress- true strain curve extracted from the uniaxial tension response of one Al beam (see supplementary material³¹).

true strain curve extracted from the *in-situ* TEM tensile testing of one Al beam (see supplementary material³¹) at a constant displacement rate of 0.1 nm s^{-1} . The deformation was stopped at a strain $\varepsilon \approx 0.06$, i.e., before fracture. Three deformation regimes can be distinguished in Fig. 2. Regime I corresponds to a purely elastic behavior. The Young's modulus measured in this regime is equal to 70 GPa in agreement with the value expected for bulk Al. A deviation from regime I can be observed at the position marked by a dark arrow in Fig. 2, i.e., regime II, at $\varepsilon \approx 0.002$ and $\sigma \approx 240$ MPa. The overall yielding of the sample (i.e., regime III) starts at $\varepsilon \approx 0.011$ and $\sigma \approx 750$ MPa. This regime clearly involves bursts of plasticity events leading to significant load drops. A Bauschinger strain of 0.005 was measured from the unloading of the specimen at $\varepsilon \approx 0.06$, see Fig. 2.

Figures 3(a)–3(c) exhibit three BF-TEM snapshots captured during the *in-situ* TEM loading within the regimes I, II, and III at $\varepsilon = 0.0015$, $\varepsilon = 0.008$, and $\varepsilon = 0.036$, respectively. Note that, in these figures, only few well oriented grains can be observed due to the variation of the diffraction conditions from one grain to another. In the elastic regime I, no changes of the diffraction contrast in the visible grains of Fig. 3(a) were observed during straining. In regime II, a few grains, such as the one labeled “G1” in Fig. 3(b), start to deform plastically. Indeed, a dark line indicated by a white arrowhead has formed in this grain. This line does not correspond to a dislocation line but to a bend contour which is an imaging effect observed in bent crystals. The bend contours indicate the position of crystallographic planes in Bragg conditions with the electron beam. The formation of these artifacts in grain “G1,” thus, confirms that the local orientation of this grain has changed due to the activation of plasticity mechanisms. After the macroscopic yielding of the sample (i.e., regime III), the number of grains exhibiting bend contours significantly increases as indicated by the multiple white arrowheads in Fig. 3(c), proving that the majority of the grains are deforming plastically. Based on these observations, the transition between regime I and II revealed

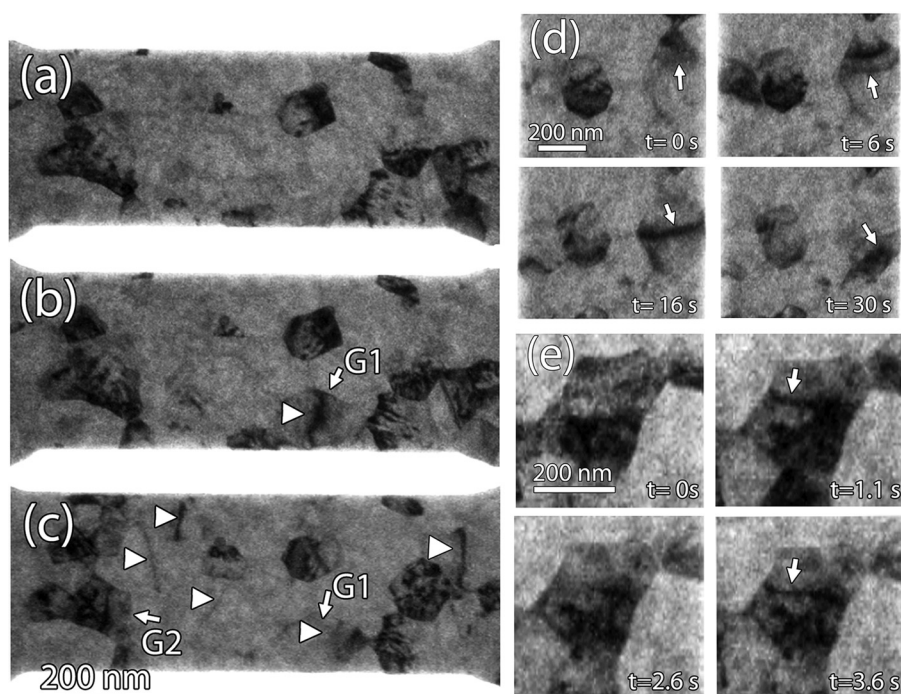


FIG. 3. BF images obtained at a strain (a) $\epsilon = 0.0015$, (b) $\epsilon = 0.008$, and (c) $\epsilon = 0.036$, respectively. (d) Dynamical BF-TEM sequence starting at $\epsilon = 0.036$ and showing the motion of bend contours. (e) Four BF snapshots starting at $\epsilon = 0.036$ and illustrating the dislocation starvation mechanism.

in Fig. 2 can be attributed to the initiation of non-elastic mechanisms in a few grains. Microstructure heterogeneities in terms of grain size and orientation lead to a long microplasticity stage.

Figure 3(d) shows a dynamical sequence of BF images starting at $\epsilon = 0.036$ (regime III) in grain “G1” of Fig. 3(b). These images demonstrate discontinuous and interrupted movement of bend contours which constitutes an indirect evidence of local orientation changes due to dislocation activity in the grain interior and/or plasticity events at GBs inducing grain rotation. The motion of bend contours cannot be attributed to the global tilting of the sample since simultaneous changes of diffraction contrast of all the visible grains (i.e., rigid body motion) was not observed. Occasionally, local distortion of the contrast of bend contours was observed during their movement, which may be related to their interaction with dislocations. However, such a feature is very difficult to confirm because, in contrast with the two-beam conditions commonly used to visualize dislocations, direct TEM observation of dislocation activity in randomly oriented grains exhibiting bend contours is not straightforward. Figure 3(e) exhibits dislocations activity starting at $\epsilon = 0.042$ (regime III) in the grain “G2” of Fig. 3(c), almost oriented in two-beam diffraction condition. In this grain, one dislocation line indicated by a white arrow appears and disappears with a waiting time close to 1 s. Such a feature is characteristic of the dislocation starvation mechanism^{8,14,26,27} with dislocations nucleating and immediately escaping at the surface of the sample. This mechanism is in agreement with the presence of only one grain over the thickness of the Al beam (Fig. 1(c)). The dislocation starvation mechanism also explains the flat macroplastic regime III (Fig. 1(d)) with almost no strain hardening because of the absence of a dense dislocation network in the interior of the grains. The waiting time of 1 s separating the escape of the dislocations through the surface (Fig. 3(e)) can be attributed to the presence of a 5 nm thick native oxide layer

at the surface of the Al thin films as revealed by electron energy loss spectroscopy (EELS) and energy filtered TEM (EFTEM) (see supplementary material³¹). Similar behavior has been reported in the literature during *in-situ* TEM testing of submicrometer Al single crystal and ufg Al thin films.^{14,28}

Although the TEM observations confirm the participation of the dislocation starvation mechanism in the overall plastic response, no conclusion can be made regarding the contribution of GB mediated processes such as grain rotation and GB sliding from the motion of bend contours revealed by *in-situ* BF-TEM (Fig. 3(d)) as well as the role of these mechanisms during the early stages of plastic deformation. Indeed, revealing grain rotation with quantitative parameters using *in-situ* BF-TEM is impossible because of the lack of local orientation information.

Figure 4(a) shows an ACOM-TEM orientation map of an Al beam before deformation. The beam contains 85.2% of high angle GBs (HAGBs) with misorientation angles higher than 15° , 4.2% of low angle GBs (LAGBs) with misorientation angles lower than 15° , 2.7% of $\Sigma 9$ coincidence site lattice (CSL) GBs, and 7.8% of $\Sigma 3$ growth twin boundaries. The positions of these twin boundaries are shown in Fig. 4(b). In order to avoid creep during an ACOM-TEM experiment with an Al beam under load,²⁹ the Al beam of Fig. 4(a) was strained to $\epsilon = 0.01$ at a displacement rate equal to 0.1 nm s^{-1} , unloaded, followed by the acquisition of the ACOM-TEM data. The same procedure was repeated with strain increments $\Delta\epsilon = 0.01$ up to $\epsilon = 0.04$. Thus, only the permanent plastic changes of the microstructure can be revealed using this method. Furthermore, because of the difficulty to observe changes in grain orientation visually, a numerical comparison of the orientation changes of individual grains based on the analysis of the evolution of the orientation density functions (ODFs) after each straining cycle was used.²⁰ Figures 4(c) and 4(d) show the changes in orientation of two selected grains (labeled “G1” and “G2” in Fig. 4(a))

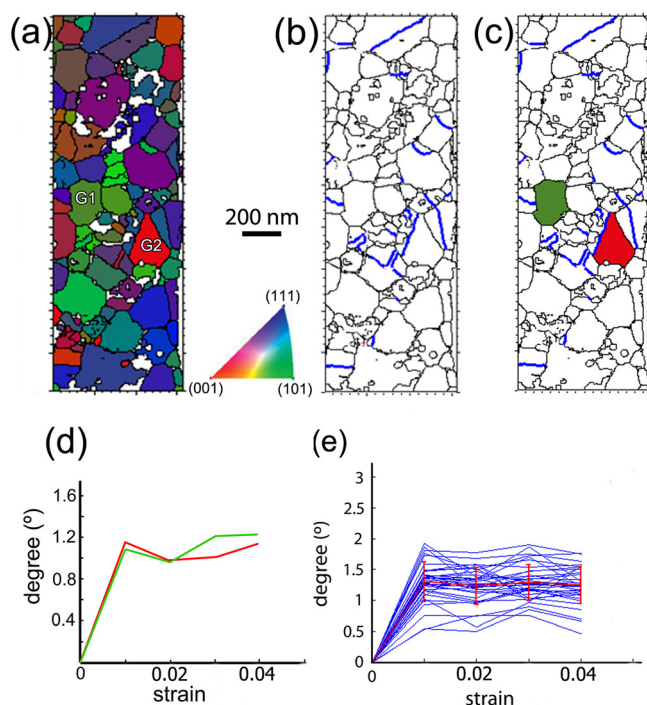


FIG. 4. (a, b) ACOM-TEM orientation and twins maps. (c) Selection of the two grains “G1” and “G2” shown in Fig. 4(a). (d) *In-situ* local orientation changes of the grains “G1” and “G2.” (e) *In-situ* local orientation changes of all the grains shown in Fig. 4(a).

with respect to their initial orientation as a function of deformation. It can be seen in Fig. 4(d) that these two grains rotate between $\varepsilon = 0$ and $\varepsilon = 0.01$ and keep nearly the same orientation until $\varepsilon = 0.04$. Figure 4(e) exhibits the same plot for all the grains of the beam. They all follow the same trend with an average orientation change of 1.3° between $\varepsilon = 0$ and $\varepsilon = 0.01$ followed by a nearly unchanged average orientation regime in which only slight variations of the local orientation of the grains can be observed. It is worth noting that, since the global change of the local orientation of the grains between $\varepsilon = 0$ and $\varepsilon = 0.01$ falls within the angular resolution limit of the ACOM-TEM technique ($\sim 1^\circ$), it was not possible to determine precisely the rotation axis of individual grains and thus to confirm (using the ACOM-TEM data) if these orientation changes correspond to collective grain rotation or to the global tilting of the sample. However, the global tilting of the sample can be excluded because, as explained above, simultaneous changes of diffraction contrast characteristic of rigid body motion of the sample, easily detected in TEM, was not observed. Moreover, similar global change of the local orientation of the grains can be expected when the sample is strained to higher deformation level, which is clearly not the case as can be seen in Fig. 4(e). Finally, it can be anticipated that any misalignment which can occur at the early stage of straining will affect the extracted Young's modulus which is clearly not the case in the present work (see Fig. 2). Grain rotation is thus the most plausible explanation of the global change of the local orientation of all the grains between $\varepsilon = 0$ and $\varepsilon = 0.01$ shown in Fig. 4(e). All the grains of the Al beam have rotated after 1% deformation confirming that this mechanism is not influenced by the character of the GBs.

The transition shown in Fig. 4(e) can be attributed to the transition of the plasticity mechanisms at 1% deformation from GB interfacial plasticity causing GB sliding to intragranular plasticity with dislocation glide and starvation revealed in Fig. 3(e) using *in-situ* BF-TEM. The sliding along the GBs leads to the bending or shearing of the grains involving thus a rotation. No grain growth or deformation twinning was observed during the present *in-situ* ACOM-TEM tensile testing.

Based on these results, the following scenario can be envisaged to explain the macroscopic deformation behavior of the ufg Al thin films tested in the present work.

- (i) From $\varepsilon = 0.002$ to $\varepsilon = 0.01$: microplasticity mechanisms are activated as revealed by *in-situ* BF-TEM (Figs. 3(a)–3(c)) leading to a gradual elasto-plastic transition (i.e., regime II in Fig. 2). The ACOM-TEM results show that most of the plastic deformation in this regime is accommodated at the GBs. Indeed, clear collective grain rotation was captured using *in-situ* ACOM-TEM (Fig. 4(e)) whereas *in-situ* BF-TEM indicates that this collective orientation change is not due to a rigid body motion. The average local orientation change of the grains is equal to 1.3° . Such a feature is attributed to GBs sliding due to a dislocation activity confined at the GBs. Very recently, dislocation activity confined at the GBs at the early stage of plastic deformation has been reported by Mompou *et al.*²⁸ on ufg freestanding Al thin films. The grain rotation induced by GB sliding is facilitated by the limited geometrical constraints due to the presence of only one grain over the thickness (Fig. 1(c)). Also, it was demonstrated that the grain rotation in this regime is not affected by the character of the GBs indicating that the intergranular plasticity is probably triggered by the local stress concentrations associated to the GB grooves. Other mechanisms such as local strain accommodation at non-equilibrium triple junctions may also participate.
- (ii) After $\varepsilon = 0.01$, most of the dislocation activity is transferred from GBs into the interior of the grains with dislocations nucleating from intragranular sources and/or GBs and escaping the surface of the sample (Fig. 3(e)). Such a feature can be explained by the presence of one grain over the thickness and the resulting strong image forces acting on the dislocations (i.e., extrinsic size effect). The dislocation starvation mechanism also explains the lack of strain hardening in the macroscopic plastic regime (Fig. 1(d)). The coupling between the high ductility and the absence of strain hardening observed in Fig. 1(d) is a characteristic of strain rate sensitive materials (otherwise necking should occur) involving thermally activated plasticity mechanisms such as the dislocation starvation and the GB sliding mechanism revealed in the present work.

The present study demonstrates that both GB mediated processes and dislocation based mechanisms can be activated in ufg materials with a mean grain size close to 300 nm. Again this raises questions regarding the critical grain size reported in the literature (~ 40 nm, see recent review in Ref. 30) from which GB mechanisms are expected to start to

operate, yielding the inverse Hall–Petch phenomenon. The results, in addition to demonstrating the complexity of the small-scale plasticity processes controlling the deformation of materials with very small grains, also bring about the necessity to combine several advanced techniques to generate a complete physical picture. In the present work, we have demonstrated that the combination between *in-situ* BF-TEM nanomechanical testing and ACOM-TEM nanomechanical testing can play a pivotal role towards a better understanding not only of the fundamental mechanisms operating at the nanoscale in ufg materials but also of the competition and the synergy between these mechanisms. In the future, the focus will be on the use of the same method to investigate the elementary processes controlling the fracture of nc and ufg materials such as the interactions between GBs and the atomistic cracking mechanisms.

This work was carried out in the framework of the IAP program of the Belgian State Federal Office for Scientific, Technical and Cultural Affairs under Contract No. P7/21. The Karlsruhe Nano Micro Facility (KNMF) is gratefully acknowledged. The authors are grateful to Dr. U. Hangen from Hysitron., Inc., for the contribution on the *in-situ* TEM nanomechanical testing of the specimens.

- ¹M. Legros, M. Cabie, and D. S. Gianola, *Microsc. Res. Tech.* **72**, 270 (2009).
- ²L. B. Freund and S. Suresh, *Thin Film Materials: Stress, Defect Formation and Surface Evolution* (Cambridge University Press, London, 2003).
- ³E. O. Hall, *Proc. Phys. Soc., Sect. B* **64**, 747 (1951).
- ⁴N. J. Petch, *J. Iron Steel Inst.* **174**, 25 (1953).
- ⁵M. A. Meyers, A. Mishra, and D. J. Benson, *Prog. Mater. Sci.* **51**, 427 (2006).
- ⁶R. Masumura, P. Hazzledine, and C. Pande, *Acta Mater.* **46**, 4527 (1998).
- ⁷K. S. Kumar, H. Van Swygenhoven, and S. Suresh, *Acta Mater.* **51**, 5743 (2003).
- ⁸M. D. Uchic, D. M. Dimiduk, J. N. Florando, and W. D. Nix, *Science* **305**, 986 (2004).
- ⁹D. Kiener and A. M. Minor, *Nano Lett.* **11**, 3816 (2011).
- ¹⁰Q. Yu, L. Qi, K. Chen, R. K. Mishra, J. Li, and A. M. Minor, *Nano Lett.* **12**, 887 (2012).
- ¹¹H. Rosner, N. Boucharat, J. Markmann, K. A. Padmanabhan, and G. Wilde, *Mater. Sci. Eng., A* **525**, 102 (2009).
- ¹²R. C. Hugo, H. Kung, J. R. Weertman, R. Mitra, J. A. Knapp, and D. M. Follstaedt, *Acta Mater.* **51**, 1937 (2003).
- ¹³S. H. Oh, M. Legros, D. Kiener, P. Gruber, and G. Dehm, *Acta Mater.* **55**, 5558 (2007).
- ¹⁴S. H. Oh, M. Legros, D. Kiener, and G. Dehm, *Nature Mater.* **8**, 95 (2009).
- ¹⁵M. A. Haque and M. T. A. Saif, *Proc. Natl. Acad. Sci. U. S. A.* **101**, 6335 (2004).
- ¹⁶J. Rajagopalan, C. Rentenberger, H. P. Karnthaler, G. Dehm, and M. T. A. Saif, *Acta Mater.* **58**, 4772 (2010).
- ¹⁷M. Legros, D. S. Gianola, and K. J. Hemker, *Acta Mater.* **56**, 3380 (2008).
- ¹⁸Y. B. Wang, B. Q. Li, M. L. Sui, and S. X. Mao, *Appl. Phys. Lett.* **92**, 011903 (2008).
- ¹⁹F. Momprou, M. Legros, T. Radetic, U. Dahmen, D. S. Gianola, and K. J. Hemker, *Acta Mater.* **60**, 2209 (2012).
- ²⁰A. Kobler, A. Kashiwar, H. Hahn, and C. Kubel, *Ultramicroscopy* **128**, 68 (2013).
- ²¹S. Gravier, M. Coulombier, A. Safi, N. Andre, A. Boe, J. P. Raskin, and T. Pardo, *J. Microelectromech. Syst.* **18**, 555 (2009).
- ²²M. S. Colla, B. Wang, H. Idrissi, D. Schryvers, J. P. Raskin, and T. Pardo, *Acta Mater.* **60**, 1795 (2012).
- ²³M. Coulombier, A. Boé, C. Brugger, J. P. Raskin, and T. Pardo, *Scr. Mater.* **62**, 742 (2010).
- ²⁴H. Idrissi, B. Wang, M. S. Colla, J. P. Raskin, D. Schryvers, and T. Pardo, *Adv. Mater.* **23**, 2119 (2011).
- ²⁵H. Idrissi, S. Turner, M. Mitsuhashi, B. Wang, S. Hata, M. Coulombier, J. P. Raskin, T. Pardo, G. Van Tendeloo, and D. Schryvers, *Microsc. Microanal.* **17**, 983 (2011).
- ²⁶M. D. Uchic, P. A. Shade, and D. M. Dimiduk, *JOM* **61**, 36 (2009).
- ²⁷Z. W. Shan, R. K. Mishra, S. A. Syed Asif, O. L. Warren, and A. M. Minor, *Nature Mater.* **7**, 115 (2008).
- ²⁸F. Momprou, M. Legros, A. Boé, M. Coulombier, J. P. Raskin, and T. Pardo, *Acta Mater.* **61**, 205 (2013).
- ²⁹M. Coulombier, G. Guisbiers, M. S. Colla, J. P. Raskin, and T. Pardo, *Rev. Sci. Instrum.* **83**, 105004 (2012).
- ³⁰J. R. Greer and J. T. M. De Hosson, *Prog. Mater. Sci.* **56**, 654 (2011).
- ³¹See supplementary material at <http://dx.doi.org/10.1063/1.4868124> for details on the synthesis and in situ TEM deformation of freestanding Al beams. The characterization of native oxide layer on the surface of the Al films is also presented.