Plastic behaviour and deformation mechanisms in silicon nano-objects

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Abstract. Physical properties of nano-objects differ from what they are in bulk materials when the size decreases down to the nanometre scale. This behavioural change, named size effect, also applies to mechanical properties and has been evidenced in various materials. For instance, at low temperature, bulk silicon is known to be a brittle material while silicon nano-objects exhibit a ductile behavior. Although mechanical properties of silicon have been intensively studied over the last decades, the origin of this remarkable brittle-to-ductile transition at small scales remains, however, undetermined. In this article, a study of the plastic behaviour of nano-pillars is reported. The main results obtained from the combination of numerical calculations and experimental compression tests followed by atomically-resolved transmission electron microscopy imaging are described. We discuss the possibility for perfect dislocations to dissociate at low temperature and the underrated role of shuffle partial dislocations in plastic deformation of silicon. The formation of unexpected extended defects in the {115} planes with increasing plastic strain, also appears as a key-factor leading to the transition between ductile and brittle regimes at small scales.

1. Introduction

Silicon was probably one the most studied materials over the last half century and both its mechanic properties and structural defects have been widely investigated. The mechanical behavior of bulk silicon is notably characterized by an abrupt temperature-dependent brittle-to-ductile transition observed between 550°C and 800°C, depending on the solicitation conditions [1-5]. At low temperatures, the propagation of pre-existing cracks and the cleavage of the material are supposed to prevail on dislocations-mediated mechanisms leading to the observed brittle behavior of bulk silicon [6-8]. However, using specific deformation conditions that prevent the crack formation and propagation, such as deformation experiments under high confining pressure or using confining shielding media [9-12], some isolated plastic microscopic events have been evidenced in the brittle regime. Above the brittle-to-ductile transition temperature, mechanical stress allows inducing large macroscopic plastic deformation in silicon through the nucleation and propagation of dislocations [13]. Since the earliest observations of dislocations in silicon performed more than sixty years ago [14-15] and the first theoretical questionings about the possible types and slip systems in this material [16-17], numerous works have been devoted to the characterization of mobile extended defects in bulk silicon either in the ductile or in the brittle regimes. At high temperatures, experimental observations [13]
showed that plastic deformation of silicon is controlled by the propagation in the \{111\} planes of dissociated dislocations constituted by pairs of Shockley partial dislocations [18]. At intermediate temperatures, microtwins and extended stacking faults were also observed in deformed samples deformed under confining pressure [9]. At low temperatures, only perfect dislocations, i.e. non-dissociated dislocations, were revealed by post mortem analyses of silicon deformed under high stresses. The observed perfect dislocation segments have either a 30°, 41° or a screw character [10-12, 19]. However, although atomic structure of Shockley dislocations in silicon has been experimentally resolved using high resolution transmission electron microscopy (HRTEM) imaging [20], the core structure of the perfect dislocations observed in the brittle regime is still subject of debate [21-24]. Notably, the slip path of the dislocations, which can be localized either in between the widely spaced \{111\} atomic planes (i.e. in the shuffle set) or in between two adjacent close \{111\} planes (i.e. in the glide set) [17], has not been formally identified from experimental analyses. However, as numerical calculations predicted that the shuffle set perfect dislocation show a higher mobility than the glide set perfect dislocation when submitted to high stresses [24], mobile perfect dislocations observed in silicon deformed at low temperatures are usually assumed to propagate in the shuffle set. In addition, other numerical calculations also revealed that the core structure of mobile shuffle set perfect dislocations is not stable when the stress decreases, leading to a core transition toward a stable but not-mobile configuration [25], but this core transition has not been experimentally evidenced up to now. Deviating from this quite well-established scheme, plastic behavior of silicon objects exhibits unexpected features when size decreases. Indeed, contrary to their bulk counterpart, silicon nano-objects behave as ductile materials at low temperatures. This surprising behavior has been evidenced in silicon nano-spheres [26], nano-wires [27-32], micro- and nano-pillars [33-34], and nano-cubes [35]. Furthermore, the plastic deformation of silicon nano-objects at low and intermediate temperatures involves various types of dislocations, as Shockley dislocations [30, 32, 35-36], perfect dislocations [28-30, 32] or Lomer locks [30] and depends on the solicitation conditions: tensile vs compressive stress and solicitation axis [31, 37]. The origin of the size effect on mechanical behavior of silicon is then a complex issue as it refers both to the influence of the sample dimensions on the nature of mobile defects (ductile behavior) and to its impact on the fracture mechanism(s) (brittle behavior), that cannot be correlated by an obvious manner to the high surface-over-volume ratio which distinguishes the small-size objects from the bulk material.

In order to disentangle this puzzling question, it appears necessary to unambiguously characterize the nature of defects that govern the plastic deformation regime in silicon nano-objects deformed under controlled conditions, and to identify the conditions and corresponding microscopic events which favor the fracture of these objects. For this, we performed compression tests at very low strain rates on defect-free silicon nanopillars prepared by reactive etching lithography, and post mortem analyses of the deformation microstructures were done using HRTEM imaging. The experimental observations were compared to elementary deformation mechanisms revealed by molecular dynamics (MD) simulations, which allows deeply reinterpreting the results reported in the literature by emphasizing the role of the dislocations dissociation mechanism on the macroscopic mechanical behavior of silicon.

2. Methods

2.1. Sample preparation
The experimentally investigated samples consist in cylindrical nanopillars (see figure 1.a) designed by e-beam lithography and etched by reactive ion etching (RIE). The axis of the nanopillars is parallel to a <110> crystallographic direction so that only two \{111\} slip systems are activated during uniaxial compression tests, allowing to easily study individual plastic events. This sample orientation also permits to observe potential interactions between dislocations gliding in two intersecting slip planes.

2.2. Compression experiments
The compression tests were carried out at room temperature using an Anton Paar U-NHT nano-indenter
device, equipped with a diamond flat punch and operating in displacement-controlled mode ($\varepsilon_{pl} = 5.4 \times 10^{-4}$). This apparatus provides the very high mechanical and thermal stability necessary for the controlled deformation of nano-scaled objects. The force and the displacement were measured simultaneously using different sensors. The engineering stress and the engineering strain were calculated from the measured force and displacement and the dimensions of the pristine nanopillars.

2.3. **HRTEM imaging**

HRTEM imaging was performed using a geometrical aberrations-corrected FEI Titan 80-300 microscope, operating at 200 kV. Spherical aberration Cs was tuned to optimize the imaging conditions [38]. The HRTEM images simulations were calculated with the JEMS software using the multislice method [39]. The atomic structures used for the image simulations of defects were built using isotropic calculations obeying the classical elasticity theory [17], and then relaxed with numerical calculations using the Stillinger-Weber semi-empirical potential [40] for refining the atomic positions in the dislocation core regions. Deformation mappings were extracted from experimental and simulated images by the geometrical phase analysis (GPA) method [41] using the GPA for DigitalMicrograph plugin from HREM Research Inc.

2.4. **Molecular dynamics simulations**

The silicon atoms positions were calculated using a recently developed Stillinger-Weber potential [40]. The latter was specifically designed for an improved description of plasticity properties of silicon, and especially dislocation cores. The MD simulations were done using the LAMMPS code [42] in the NVT (canonic) ensemble. Atomic displacements were determined using the Verlet algorithm with an integration time step of 1 fs and the system temperature (300 K) was controlled with the Nosé-Hoover thermostat. The numerical compression experiments were performed at low strain rate for such kind of experiments (here $\varepsilon_{pl} = 10^6$ s$^{-1}$ as compared to usual MD strain rate of $10^8$ s$^{-1}$). Atomic configurations were extracted with the Ovito software [43].

3. **Results and discussions**

3.1. **Main features**

The experimental compression tests systematically showed that the elastic stage is followed by a purely plastic stage when the imposed stress reaches 8 to 10 GPa. This ductile behavior was even observed for total strain reaching values higher than 10% that clearly exceeds the strength limit of bulk silicon deformed at room temperature.

However, it must be noticed that in some rare cases, the deformation experiments leaded to the fracture of the nanopillars, but this brittle regime was only achieved after a few percent of plastic deformation. The imaging of deformed nanopillars by scanning electron microscopy showed that the plastic deformation is mainly localized at the head of the nanopillars, which exhibit large shearing parallel to the $\{111\}$ crystallographic planes (see figure 1).

The HRTEM analyses confirmed that the plastic events are mainly localized in the $\{111\}$ planes. In addition, in most cases, the observed shearing of the sample was likely produced by the propagation of several dislocations within a single slip plane, as shown in figure 2.

In this example, no extended stacking fault is visible in the shearing plane, indicating that the deformation involved the propagation of perfect or dissociated dislocations, leaving a perfect crystal in their wake.

3.2. **Nature of the mobile defects in the plastic regime**

A careful investigation of the HRTEM images in the sheared areas allowed us to evidence the presence of a few dissociated dislocations, extended in the $\{111\}$ shearing planes. The figure 3 shows a dissociated dislocation observed in the volume, in a shearing plane.
Figure 1. (a) Pristine nanopillar prepared by electron lithography and reactive ion etching and observed by SEM. (b) SEM image of a nanopillar deformed in compression. Framed image: Nanopillar seen along the compression axis. The trace of shearing at the top surface of the nanopillar is parallel to a <110> crystallographic direction. (c) TEM image of a deformed nanopillar. Framed zone: the head of the nanopillar is sheared parallel to the \{111\} planes.

The perfect agreement between the strain mappings extracted from the experimental image and from the calculated atomic structure unambiguously evidences the presence of a dissociated dislocation in the investigated area. This dissociated dislocation consists in a pair of 90° and 30° partial dislocations separated by a stacking fault but the nature of the dissociation plane (i.e. glide set or shuffle set) cannot be formally deduced from this observation.

Figure 2. Filtered HRTEM image (Low-Pass filter) of a nanopillar deformed in compression. The compression direction is parallel to the vertical axis. The shape of the step on the lateral surface of the nanopillar indicates that the plastic events are localised in a single slip plane. Here, the propagation of more than 40 perfect 60° dislocations was necessary to produce the observed shearing.

Surprisingly, perfect dislocations (i.e. non-dissociated dislocations) were also observed in the same samples (see figure 4). Their presence can be easily distinguished in the GPA mappings and their non-dissociated core is attested by the absence of the characteristic trail produced by a stacking fault (see for instance the trail observed between the two partial dislocations in figure 3). However, the peculiar localization of the observed perfect dislocations deserves to be underlined. Indeed, although the slip planes of these dislocations are \{111\} planes, their propagation did not occur in the main shearing planes but in secondary slip systems which intersect the main ones. In addition, these perfect dislocations are mostly found at the intersection with the main shearing planes and, in many cases, they form subgrain boundaries. Some isolated partial dislocations, extended stacking faults and nanotwins were also evidenced in deformed nanopillars, as shown in figure 5.
Figure 3. Top: HRTEM image of a dissociated dislocation observed in a shearing plane. Bottom: comparison between the strain mappings extracted from the experimental image (at the left) and from the simulated HRTEM image of a model atomic structure containing a dissociated dislocation (at the right), using the GPA method. The dissociated dislocation consists in two partial dislocations separated by a stacking fault responsible for the visible trail in the strain mappings. The localization of the observed area in the nanopillar and the crystallographic orientation of the image are indicated in the figure depicted at the left.

These defects were observed in various samples, but they are assumed to minority contribute to the plastic deformation of the nanopillars as their density is widely insufficient to produce the observed macroscopic shearing. Nevertheless, the presence of Shockley partial dislocations (see figure 5c) was not expected at such low temperatures and confirms the observations reported in a previous study [35] suggesting the possibility for partial dislocations to be nucleated and to propagate in silicon nano-objects deformed at low temperature.

Despite the variousness of the defects observed in experimentally deformed nanopillars, the comparison between the experimental results and the MD simulations allowed us to clarify the origin of this microstructural diversity and to draw a possible chronic for the elementary mechanisms involved during the low-temperature and high-stress plastic deformation of silicon nanopillars. Indeed, the numerical samples deformed in compression at low strain rate exhibit structural features which are different than those revealed during tensile deformation experiments [44-45]. Notably, the load drop produced by the propagation and escape of the first nucleated shuffle perfect dislocations is responsible for the core transition of the trailing ones from an unstable but mobile configuration (S1) to a stable but not-mobile configuration (S3), as described in [25]. Upon reloading, the S3 dislocation core dissociates into one Shockley partial dislocation and one shuffle partial dislocation (see figure 6). Very surprisingly and contrary to the assumption established for years [17, 21], this shuffle partial dislocation is highly mobile for these deformation conditions (low temperature and high stress).
Figure 4. Top: Filtered HRTEM image (Low-Pass filter) of a deformed nanopillar. The compression direction is parallel to the vertical axis. Two main shearing planes are visible (dotted lines). The observed area contains three perfect dislocations gliding in various \{111\} planes (dashed lines) intersecting the main shearing planes. Bottom: the appropriate orientation of the x and y axes for the GPA allows to easily distinguish the strain lobes associated to the presence of the three perfect dislocations in the strain mapping. The localization of the observed area in the nanopillar and the crystallographic orientation of the image are indicated in the figure depicted at the left.

Thus, this dissociation mechanism and the unexpected high mobility of the shuffle partial dislocations can explain the experimental observations of both dissociated dislocations and extended stacking faults bounded at their extremity by a Shockley partial dislocation. The simultaneous observation of dissociated and perfect dislocations can also be explained by the previously mentioned alignment of the later ones, forming stable grain boundaries. Due to their stable position, contrary to the dislocations gliding in the main shearing planes, the observed perfect dislocations were consequently not re-activated under reloading and kept the S3 compact core configuration.

In addition, a few twinning events were also evidenced by the MD simulations (see figure 6c). The formation of these nanotwins involves the propagation of superpartial dislocations which were not experimentally identified. However, the authors consider the presence of this type of dislocations as a minority event in the deformation mechanism. In addition, the high mobility of these dislocations revealed by MD simulations lowers their chances to be identified by post mortem TEM imaging of deformed specimen. Nevertheless, this result confirms the possible occurrence of partial dislocations propagation and stacking faults formation in silicon nano-objects deformed at low temperature.

3.3. Ductile-to-brittle transition
Some nanopillars also showed some brittle behavior as shown in figure 7. In this case, the crack formation followed a first stage of plastic deformation. This suggests that a certain amount of strain is necessary to initiate the transition between the two regimes. As a rule, HRTEM post mortem analysis of
the fractured nanopillars revealed the presence of corrugated planar defects, extending parallel to the \{115\} crystallographic planes.

Figure 5. (a) Stacking fault and isolated 30° partial dislocation observed in a deformed nanopillar, and (b) corresponding schematic drawing of the observed defects. (c) HRTEM image of a nanotwin observed in another deformed nanopillar. The framed zone contains three 30° Shockley partial dislocations (see the induced rotation of the dumbbells orientation in the glide plane), as depicted in the atomic structure shown below.

Figure 6. MD calculations. (a) Numerical sample deformed in compression. The framed area contains various types of dislocations. (b) Top view of the defects contained in the framed area. The perfect dislocation segment exhibits a not-mobile S3 core configuration (at the left), but a part of this dislocation is dissociated under stress. This dissociated part is constituted by a 90° Shockley partial dislocation and a 30° shuffle partial dislocation which both are mobile. (c) Cross-section of the deformed nanopillar
showing the presence of the various types of defects. The defects observed in the framed area are visible at the bottom and the presence of a nanotwin is also visible at the top.

Figure 7. Images of a fractured nanopillar. (a) SEM image of the nanopillar. (b) SEM image of the TEM sample after FIB micromachining. The nanopillar is embedded in a platinum protective layer (on top). (c) TEM image of the nanopillar. Framed area: corrugated defects parallel to the \{115\} crystallographic planes are visible in the vicinity of the cracks. These defects are systematically nucleated from previously activated \{111\} planes.

The formation mechanism of this transition defects is still unclear but, as they are systematically associated with previously activated \{111\} slip systems, they could result from the interaction between several dislocation segments gliding in different slip systems, leading to locking or weekly mobile junctions [46]. This assumption was tested using MD simulations applied to atomic structures containing both perfect and dissociated dislocations in close interaction (see figure 8). This simulation shows that during thermal annealing at 300 K, the Shockley partial dislocation segment resulting from the dissociation of a perfect dislocation combines with the S3 core of the neighboring perfect dislocation to form a junction having a 1/6<411> Burgers vector whose slip plane is a \{115\} crystallographic plane. The formation of such kind of locking junctions could then initiate the observed plastic events propagating along the \{115\} planes and favor the formation of the cavities evidenced in few cases at their extremity by HRTEM imaging, which constitute favorable sites for crack nucleation, as revealed by previous work [44].

4. Conclusions

Both experimental study and calculations reported here demonstrate that silicon nano-objects exhibit a peculiar mechanical behavior when deformed in compression. Our results confirm the ductile behavior of the deformed nanopillars, followed in certain cases by a progressive transition toward a brittle regime. The controlled deformation process and the atomically resolved structural investigation of extended defects produced by the deformation experiments allowed to unambiguously identify the nature of the various defects involved during the plastic deformation of these objects. MD simulations revealed the possibility for shuffle perfect dislocations to dissociate under compressive stress, following a subtle mechanism resting on a core transition operating when the internal stress decreases due to the stress relaxation provoked by the propagation and escape of the first nucleated perfect shuffle dislocations. The dissociated dislocations are constituted by a pair of Shockley partial dislocation and shuffle partial dislocation, both of them being mobile under high stress. This mechanism permits to explain the presence of various types of dislocations, depending on the local stress field and the experimental solicitation conditions, as experimentally observed in this study and reported in the literature [28-30, 32, 35-36]. Finally, cracking seems to be correlated to the formation of unusual defects propagating in \{115\}
which are assumed to constitute the transition markers between the ductile and the brittle regimes at low temperature. These defects could result from the interaction between perfect and dissociated dislocations leading to the formation of locking junctions and then, constitute nucleation sites for crack nucleation.

Figure 8. Interactions between perfect and dissociated dislocations evidenced by MD simulations. Left: the numerical sample contains two perfect dislocations (in blue, on the two opposite sides) in two parallel \{111\} slip planes. The third dislocation is partially dissociated in two partial dislocations (green segments, in the middle) in a \{111\} slip plane intersecting the two others slip planes. Right: due to the elastic interactions between the various dislocations segments, one of the two partial dislocations combines with the neighbouring perfect dislocation to form a 1/6<411>{11-5} junction (in red).

The size effect on mechanical properties for small-scale objects is usually attributed to the increase of surface-over-volume ratio. Thus, the respective influences of either the surface or the reduced volume of materials may be invoked for explaining the behavioural changes exhibited by nano-objects. Here, the density of stored dislocations decreasing with the size reduction of the objects, the probability for locking interactions is reduced, and likely leads to the increase in ductility exhibited by silicon nano-objects for the smallest sizes.

Acknowledgements
The authors acknowledge the French national research agency (ANR) for financial support (grant reference ANR-12-BS04-0003-01).

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