



# Size dependent fracture strength and cracking mechanisms in freestanding polycrystalline silicon films with nanoscale thickness

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## ABSTRACT

The size dependent fracture strength and fracture mechanisms of polycrystalline silicon films are investigated by analyzing a wide range of specimen lengths and widths with thicknesses equal to 240 and 40 nm. An on-chip method is used to deform the films and to determine the strength. The strength increases with decreasing external surface area. The thinnest films exhibit the lowest strength and a weaker size effect. The crack path changes from transgranular to intergranular with decreasing thickness. These results are related to differences in microstructure and surface roughness characteristics of the films as controlled by the fabrication process and thickness.

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## 1. Introduction

Polycrystalline silicon (poly-Si) thin films constitute a key structural component for various applications such as displays and microelectronics devices [1], nano- and microelectromechanical systems [2] as well as pressure [3,4], infrared [5] and biological sensors [6]. Poly-Si can be deposited and patterned over a wide variety of substrates and has the advantage of low production costs. A range of electrical properties can be targeted by varying the doping level and implanted species [1,2]. The good mechanical properties make poly-Si versatile in terms of the combination of functional and structural performances [1,2]. As for any structural element, mechanical failure of thin poly-Si components compromises device operation. The understanding of the fracture mechanisms occurring in poly-Si films as well as the characterization of the fracture properties are critical concerns for supporting the design, development and fabrication of efficient and reliable applications.

At room temperature, poly-Si thin films fail in a brittle manner by cleavage [7]. The fracture typically initiates from critical defects present on external surfaces [7–13]. The fracture is reported to be transgranular, with the crack propagating quasi-instantaneously along the compact crystal planes [7,9,12,14]. The intrinsic toughening mechanisms due to local grain orientation changes and grain boundary (GB) toughening are not very effective [14]. The fracture strength of poly-Si films is

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**Nomenclature**

|                     |                                                                                             |
|---------------------|---------------------------------------------------------------------------------------------|
| $a$                 | crack length                                                                                |
| $E$                 | Young's modulus of the specimen material                                                    |
| $E_a$               | Young's modulus of the actuator material                                                    |
| $K_{Ic}$            | fracture toughness                                                                          |
| $L$                 | specimen beam length                                                                        |
| $L_a$               | actuator beam length                                                                        |
| $Ra$                | arithmetic average of absolute values of the profile height deviations from the mean line   |
| $Rms$               | root mean square of the absolute values of the profile height deviations from the mean line |
| $S$                 | section of the specimen beam after the release step                                         |
| $S_a$               | section of the actuator beam after the release step                                         |
| $u$                 | displacement of the specimen beam                                                           |
| $u_a$               | displacement of the actuator beam                                                           |
| $t$                 | thickness of the specimen beam after the release step                                       |
| $Y$                 | non-dimensional geometrical factor                                                          |
| $\epsilon^{mech}$   | mechanical strain of the specimen beam                                                      |
| $\epsilon_a^{mech}$ | mechanical strain of the actuator beam                                                      |
| $\epsilon^{mis}$    | mismatch strain of the specimen beam                                                        |
| $\epsilon_a^{mis}$  | mismatch strain of the actuator beam                                                        |
| $\sigma$            | stress in the specimen beam                                                                 |
| $\sigma_a$          | stress in the actuator beam                                                                 |
| $\sigma_f$          | fracture strength                                                                           |
| ACOM-TEM            | automatic crystallographic orientation mapping in a transmission electron microscope        |
| AFM                 | atomic force microscopy                                                                     |
| c                   | crystallite                                                                                 |
| CSLB                | coincidence site lattice boundary                                                           |
| ED                  | electron diffraction                                                                        |
| FIB                 | focused ion beam                                                                            |
| g                   | grain                                                                                       |
| GB                  | grain boundary                                                                              |
| HAGB                | high angle grain boundary                                                                   |
| HF                  | hydrofluoric acid                                                                           |
| Mono-Si             | monocrystalline silicon                                                                     |
| LAGB                | low angle grain boundary                                                                    |
| LPCVD               | low pressure chemical vapor deposition                                                      |
| Poly-Si             | polycrystalline silicon                                                                     |
| RIE                 | reactive ion etching                                                                        |
| SEM                 | scanning electron microscope                                                                |
| SiN                 | silicon nitride                                                                             |
| TEM                 | transmission electron microscope                                                            |

governed by the size of the most critical defects which act as a collection of small precracks. The defect with the highest stress intensity factor initiates the fracture and dictates the overall fracture strength [8,11]. The nature, location and spatial distribution of the critical defects depend on the processing conditions and on the film thickness as the microstructure [1,2] and surface roughness [15] are governed by both of them.

Poly-Si films can be produced by various techniques [1], the most common being low pressure chemical vapor deposition (LPCVD). As-deposited LPCVD silicon films can be amorphous or polycrystalline [1]. Amorphous films are smoother due to the absence of large defects such as GBs, but still exhibit lower tensile strengths than polycrystalline counterparts [10]. Amorphous silicon contains a significant concentration of hydrogen and has a lower density which both participate to the creation of large surface defects when etching the sacrificial layer to release the films. For polycrystalline films, the grain size and surface roughness increase with increasing film thickness [1,15]. The effect of deposition temperature on the grain size and roughness is not easy to determine because these parameters are also influenced by deposition pressure [1,15–17]. From our knowledge, no information is available in the literature about the evolution of the tensile strength of as-deposited poly-Si films as a function of the deposition conditions and film thickness. Amorphous films crystallise during an additional annealing step. A rapid thermal annealing allows controlling the grain size over a wide range of dimensions [1,10]. The higher the annealing temperature, the larger the nucleation rate, the smaller the grain size. Tsuchiya [10] showed that

the fracture strength increases with the crystallisation temperature, hence with decreasing grain size. No correlation with surface roughness was provided. For poly-Si films, an additional annealing step induces an evolution of the grain size when the transferred energy is sufficient to promote grain growth and/or recrystallization [10]. A surface oxide present during the annealing step leads to enhanced grain growth and to a reduction of the surface roughness [1]. The presence and nature of doping species can also affect the grain growth mechanisms and then the surface roughness [1]. For poly-Si films with similar thicknesses, Kamiya et al. [18] found that films produced from an amorphous state exhibit larger fracture strengths compared to as-deposited polycrystalline films under the same annealing conditions. Although no information about the surface roughness and microstructure was provided, this result indicates that a poly-Si film produced from the amorphous state is smoother. This short literature survey illustrates the complexity of the relationships between microstructure, surface roughness, thickness and processing conditions which fix the magnitude of the most critical imperfections, ultimately dictating the tensile strength of poly-Si films.

Processing imperfections involve ridges, crevices and pits generated during the reactive ion etching (RIE) of the sidewalls and/or the etching of the sacrificial layer by HF [9,10,19] as well as microstructural defects such as GB grooves emerging on external surfaces [8,11–13,20]. The stress intensity factor associated to a defect is mainly governed by its shape, size and orientation with respect to the main loading direction, spatial distribution and by possible residual stress [11]. The fracture toughness is a function of the crack plane with respect to the crystal orientation and of the chemical composition. The morphological and structural characteristics of the critical defects are statistically distributed. Therefore, the fracture strength of poly-Si films is scattered and size dependent. In case of non uniform loading conditions, the apparent strength depends on the size of the region undergoing the stress concentration as well: the larger the highly stressed region, the higher the probability to involve large critical defects for triggering fracture initiation, the lower the apparent strength. For instance, bending and uniaxial tensile tests on poly-Si films do not lead to the same fracture strength [21]. Similarly, the tensile strength of a straight beam is lower than the tensile strength of a beam with stress concentration such as induced by a notch or a perforation [19,22]. Tshuchiya et al. [9] observed that the tensile strength of 1.8  $\mu\text{m}$ -thick poly-Si beams varied between 2 and 2.8 GPa with the sample length and not with the external surface area. The critical flaws were identified as pits of 50 nm produced by the HF etching step and located at the vicinity of the upper-right sidewalls corners. Chasiotis et al. [22] reported the same dependency of the fracture strength with length for 2.5  $\mu\text{m}$ -thick poly-Si beams produced with the SUMMiT V<sup>TM</sup> process developed at Sandia National Labs. The mean strength of 1000  $\mu\text{m}$ -long beams was 2.9 GPa and increased to 3.4 GPa for a 250  $\mu\text{m}$  beam length. The critical flaws were located near the mid thickness of the sidewalls due to the bilayer nature of the film: the films were produced by successive deposition of two layers with a thickness of 1 and 1.5  $\mu\text{m}$ , respectively. Reedy et al. [12] observed the same trend for 2.33  $\mu\text{m}$ -thick, 2  $\mu\text{m}$ -wide specimens produced with the SUMMiT V<sup>TM</sup> process: the mean strength increased from 2.5 to 2.7 GPa for a beam length of 70 and 20  $\mu\text{m}$ , respectively. The GB grooves along the sidewalls were identified as the critical defects, preferentially located at the bottom edges. For all these studies involving micron-thick films, the tensile strength was found to depend more on the beam length than on the external surface area. But, the characteristics of the critical defects and of the resulting magnitude of the tensile strength are different, highlighting the decisive role of the fabrication process.

Recent applications such as piezoresistive pressure [4], thermopile infrared [5], and biological sensors [6] rely on poly-Si films with thickness well below 1  $\mu\text{m}$ . For instance, films thinner than 100 nm exhibit better piezoelectric and thermoelectric properties than thicker films due to significant tunneling piezoresistive effect [23,24] and phonon scattering [5] at GBs, respectively. The best piezoresistive properties are achieved for a film deposited at 620 °C with thickness in the range 80–100 nm and a boron doping concentration  $3 \times 10^{20} \text{ cm}^{-3}$  [23]. The best thermoelectric properties are achieved for a  $\sim 50$  nm-thick film deposited at 760 °C and heavily doped with phosphorus [5].

The objective of the present study is to investigate the cracking mechanisms in submicron poly-Si films and to determine to what extent the conclusions reached in the literature for micron-thick films can be transferred to films typically 10 times thinner. Recently, Vayrette et al. [20] found that the tensile strength of 240 nm-thick poly-Si films varies with the external surface area instead of the length. The fracture was transgranular and the critical defects were identified as GB grooves located on top and/or bottom surfaces. It was suggested that, for brittle thin films with the critical defects located on top and/or bottom surfaces, the decrease of thickness and of the critical defect size could be accompanied by an increase of the stress intensity factor associated with the critical defects, hence to a decrease of the tensile strength. From our knowledge, no effect of thickness reduction down to nanoscale on the tensile strength and on the fracture mechanisms occurring in poly-Si films has been reported in the literature yet. Nevertheless, as the thickness affects the microstructure [1,2] and the surface roughness [15], the morphological and structural features of critical defects hence the fracture mechanisms as well as the fracture properties could change when the thickness decreases down to tens of nanometers. In other words, the question addressed in this work is: do the size effect on strength and on the fracture mechanisms vary with thickness for nanoscale freestanding poly-Si films?

In order to answer this question, poly-Si films with thickness equal to 40 and 240 nm are produced by LPCVD. An on-chip uniaxial tensile testing technique [20,25–29] is used to deform freestanding films until fracture and to extract the fracture strength for a wide range of samples. The microstructure and the surface roughness are characterized by means of scanning and transmission electron microscopy (SEM and TEM), atomic force microscopy (AFM) and automated crystallographic orientation mapping in TEM (ACOM-TEM). The cracking initiation and propagation mechanisms are discussed based on microstructural and surface roughness data as well as post-mortem fractographic observations.

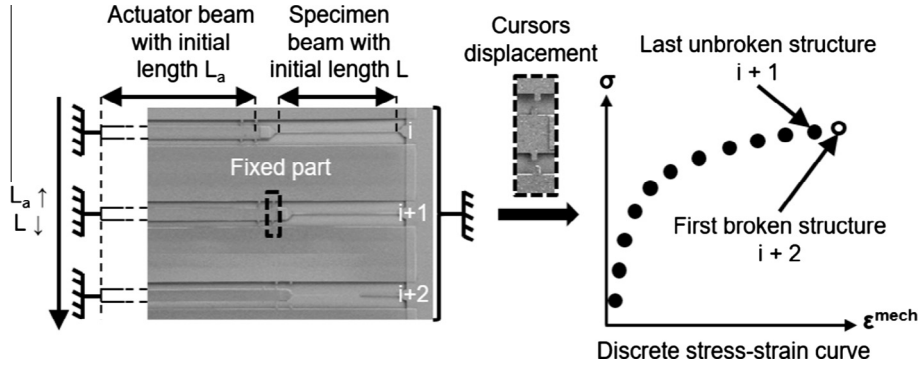


Fig. 1. Basic principle of the method for the extraction of the stress-strain curve up to fracture.

The outline of the paper is the following. Section 2 introduces the principles of the on-chip tensile testing technique, the fabrication process and the characteristics of the poly-Si films. Section 3 is devoted to the experimental results. First, the microstructure and surface topology are presented. The fracture strength and the SEM observations of the broken samples are then summarized. Finally, the nature, the location as well as the morphological and structural features of the critical defects are addressed in Section 4 in order to discuss their influences on the fracture process and properties. The discussion focuses on the decisive role of GB grooves on the fracture mechanisms when the thickness decreases.

## 2. Experimental procedure

### 2.1. Tensile method

An on-chip method illustrated in Fig. 1 is used to perform uniaxial tensile tests on poly-Si films. The working principle was described in Refs. [20,25–29]. The method relies on the relaxation of the tensile internal stress present in an actuator beam in order to deform another specimen beam attached to it owing to the etching of a sacrificial layer. Thanks to the use of micro- and nano-fabrication techniques, the specimen beam is directly deposited with an overlap on the actuator beam, hence avoiding any cutting and manipulation steps. Fixed boundary conditions are ensured. The damage induced by post processing steps and the misalignment errors are minimized. Besides, a very large number of simple elementary test structures with various in-plane dimensions are collectively produced on the same wafer so that numerous stress and strain levels up to fracture are simultaneously imposed.

The stress and strain in the specimen beam are estimated from the measurement by SEM of the displacement of cursors located on the actuator beam. The specimen beam stress  $\sigma$  is calculated from the stress in the actuator beam at free equilibrium  $\sigma_a$  multiplied by the ratio of the cross-section area of the actuator and of the specimen beams  $S_a/S$ , see Eq. (1).

$$\sigma = \frac{S_a}{S} \sigma_a \text{ with } \sigma_a = E_a \epsilon_a^{mech} = E_a \left( \frac{u_a}{L_a} - \epsilon_a^{mis} \right), \quad (1)$$

where  $E_a$ ,  $\epsilon_a^{mech}$ ,  $u_a$ <sup>1</sup> and  $\epsilon_a^{mis}$  are the Young's modulus, the mechanical strain, the displacement, and the mismatch strain of the actuator beam, respectively. The mechanical strain applied to the specimen beam  $\epsilon^{mech}$  is calculated as

$$\epsilon^{mech} = \ln \left( 1 + \frac{u}{L} \right) - \epsilon^{mis}, \quad (2)$$

where  $u$ <sup>1</sup> and  $\epsilon^{mis}$  are the displacement and the mismatch strain of the specimen beam, respectively. The data reduction scheme for the stress and strain extraction was recently improved, as detailed in Ref. [20]. The fracture strength is estimated from the last unbroken and the first broken test structures with the procedure described in Ref. [20].

The test structures are grouped by series of 30 with the same actuator and specimen widths and a constant total structure length, i.e. the actuator length plus the specimen length. Within one set of test structures, the length ratios between the specimen beam and the total structure vary from 0.65 down to 0.006, and the deformation imposed to the specimen increases in the same proportion. Each piece of wafer cut after all processing steps involves 44 different sets of structures corresponding to 4 different total structure lengths (500, 1000, 1500 and 2000  $\mu\text{m}$ ), 2 actuator beam widths (10 and 15  $\mu\text{m}$ ) and 6 specimen beam widths (1, 2, 4, 6, 8 and 10  $\mu\text{m}$ ).

<sup>1</sup> By convention,  $u_a$  and  $u$  are both set positive for an increase of the beam length and negative for a decrease of the beam length (which is not the convention used in some earlier papers [25–29]). Note also that  $|u|$  and  $|u_a|$  are not exactly equal due to the correction described in Ref. [20].

## 2.2. Processing and characteristics of the films

The fabrication process involves the successive deposition and patterning of three layers on top of a 3 in. silicon wafer: the sacrificial layer, the actuator and the specimen materials. First, a 1  $\mu\text{m}$ -thick silicon oxide produced by plasma enhanced chemical vapor deposition at 300 °C and under 675 mTorr pressure is deposited to play the role of sacrificial layer. Then a LPCVD SiN film is deposited at 790 °C and 250 mTorr. This layer is patterned by photolithography and by  $\text{SF}_6$  plasma etching. Then, a LPCVD poly-Si film is deposited at 625 °C and 200 mTorr with a  $\text{SiH}_4$  gas precursor at a flux of 95 sccm. The poly-Si films are doped with P by diffusion in a furnace at 900 °C using a  $\text{SiP}_2\text{O}_7$  solid source, the doping level being targeted at  $\sim 10^{19}$  at  $\text{cm}^{-3}$ . The doping time is 20 and 60 min for the 40 nm- and 240 nm-thick poly-Si films, respectively. The poly-Si film is then patterned by photolithography and by  $\text{SiCl}_4$  plasma etching. The test structures are released by wet etching in a 73% HF solution during 40 s. Hence, the poly-Si specimens do not involve surface oxide during the mechanical test. In order to keep the released structures freestanding and to avoid stiction onto the substrate, they are rinsed in isopropanol during 15 min and dried with a critical point dryer. This last step takes approximatively 1 h. The first measurement of the cursors displacement of a test structure with a SEM is performed usually 90 min after release. The test specimens are observed with an optical microscope after release and the last unbroken structures are identified. The same observation is performed with the SEM. We systematically check that no specimen fail during the transfer as one indication of the absence of environmental delayed fracture.

Two series of samples corresponding to two poly-Si film thicknesses have been produced. The first series is made of poly-Si specimens and SiN actuators with 240 and 290 nm thickness, respectively. The second series is made of poly-Si specimens and SiN actuators with 40 and 150 nm thickness, respectively. These thicknesses have been measured by ellipsometry after release. The uncertainty on the films thickness is equal to  $\pm 5$  nm for all films. The Young's modulus of the SiN films estimated by nanoindentation is equal to  $240 \pm 10$  GPa. The mismatch strain extracted from self-actuated tensile testing structures [30] is equal to  $-0.336\% \pm 0.01\%$  and  $-0.307\% \pm 0.01\%$  for the 290 nm-thick and 150 nm-thick SiN films, respectively. These experimental data on SiN films are in close agreement with values measured in earlier studies [30,31]. The mismatch strain of 240 nm- and 40 nm-thick poly-Si films extracted from the displacement of single-clamped cantilevers is equal to  $0.12\% \pm 0.02\%$  and  $0.2\% \pm 0.02\%$ , respectively, indicating an initial compressive state.

## 2.3. Microstructure characterization methods

The microstructure is investigated by means of the ACOM-TEM, bright field TEM and SEM observations. The ACOM-TEM method provides the local crystallographic orientation with nanometer resolution [32]. It gives access to the proportion of different components of the crystallographic texture and to the distribution of both the in-plane crystallite size and GB types. Bright field TEM imaging provides information on the presence of one or several grains over the thickness as well as on the possible presence of nanotwins lamella. SEM allows observing the surface morphology and the GB grooves emerging at the top surface of the films. It reveals also the in-plane shape and size of domains surrounded by GB grooves. The size distribution can be extracted by manually drawing the contour of these domains and using the free software ImageJ. For a sufficiently large scanned area, ACOM-TEM experiments and SEM observations provide data representative of the average microstructure. For the remainder, the grain size will refer to the size of a domain surrounded by GB grooves while the crystallite size will refer to the size of a domain with the same crystallographic orientation surrounded by any types of GB except by low angle grain boundaries (LAGBs). By convention a LAGB corresponds to GBs with a misorientation angle lower than  $15^\circ$  and larger than  $3^\circ$ . High angle grain boundaries (HAGBs) and coincidence site lattice boundaries (CSLBs) as  $\Sigma 3$  twin boundaries correspond to GBs with a misorientation angle higher than  $15^\circ$  without and with symmetrical lattice relationship, respectively.

The TEM and ACOM-TEM experiments are performed on a Philips CM20 microscope operating at 200 kV and equipped with a  $\text{LaB}_6$  gun. An external source device, DigiSTAR<sup>®</sup> developed by NanoMEGAS, is also used for the ACOM-TEM characterizations [32,33]. The freestanding films have been preliminary cut by Focused Ion Beam (FIB) and mounted on a TEM copper grid with a micromanipulator. Electron diffraction (ED) patterns are collected with an external charge coupled digital camera. For the ACOM-TEM experiments, the ED pattern is compared with the pre-calculated templates and the best match is selected [33]. ACOM-TEM orientation mapping recorded based on a 20 nm step size and an acquisition frequency of around 100 frames per second were first performed on an area of  $\sim 20 \mu\text{m}^2$  for the two film thicknesses so as to scan a large film area and to generate a representative information about the average crystallographic texture. In order to increase the quality of the orientation map and to highlight the microstructure of the sample as well as to accurately extract the crystallite size, the step size and the acquisition frequency were decreased down to 8 nm and around 60 frames per second, respectively.

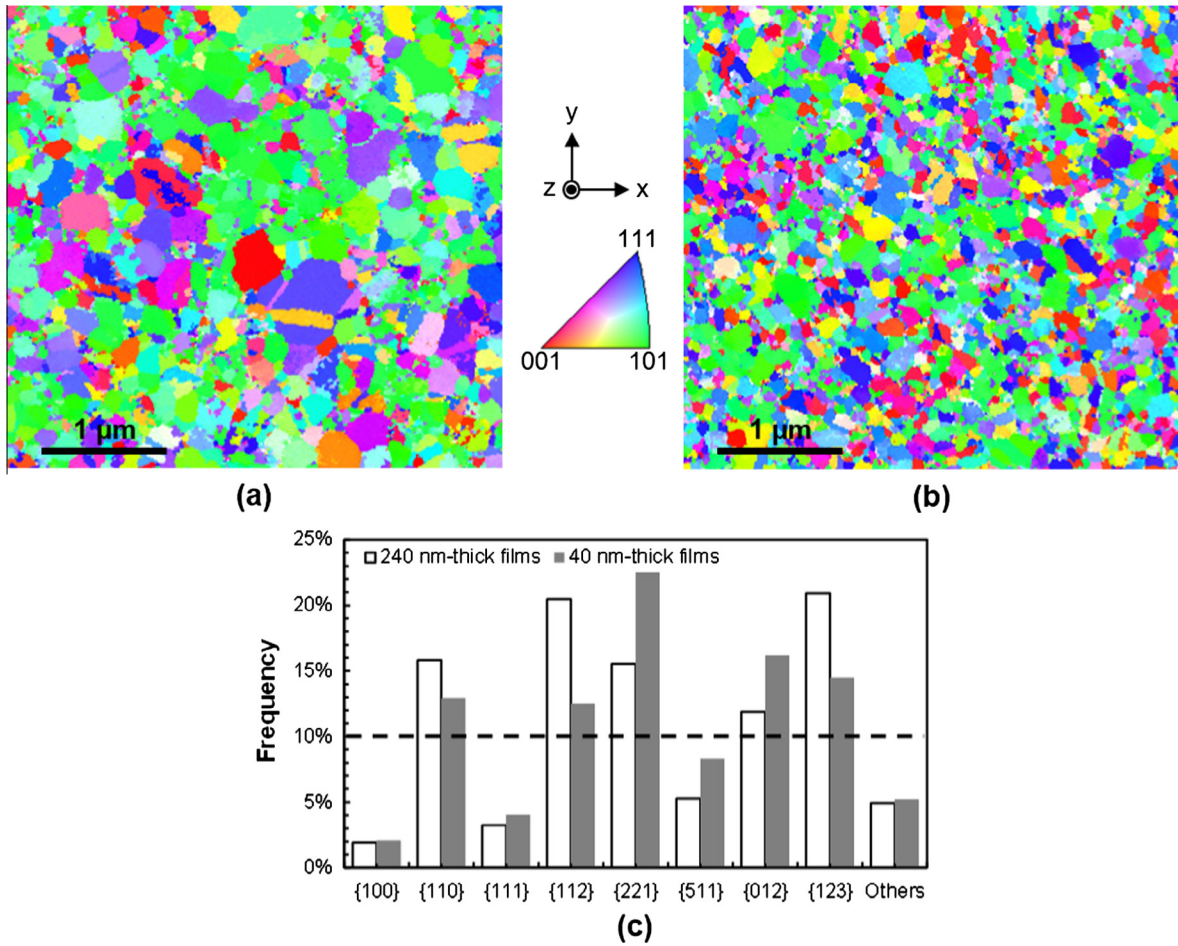
## 3. Results

### 3.1. Microstructure

#### 3.1.1. Crystallographic texture and distribution of GB character

Fig. 2(a) and (b) shows the out-of-plane orientation maps for the 240 nm- and 40 nm-thick poly-Si films, respectively. The two films exhibit a similar crystallographic texture. The in-plane texture is random with no preferential out-of-plane





**Fig. 2.** Out-of-plane orientation maps of (a) the 240 nm- and (b) 40 nm-thick poly-Si films performed in a TEM (step size of 8 nm, 60 frames per second); (c) proportion of the out-of-plane crystallographic texture components extracted from ACOM-TEM experiments performed on 240 nm- and 40 nm-thick films (step size of 20 nm, 100 frames per second). The total scanned area is  $\sim 80 \mu\text{m}^2$  and  $\sim 20 \mu\text{m}^2$  for 240 nm- and 40 nm-thick films, respectively. The angular tolerance compared to the Euler's angles of the ideal crystallographic orientation is fixed to  $10^\circ$ .

orientation. The out-of-plane texture is dominated by five crystallographic components with a contribution superior to 10%, representing  $\sim 80\%$  of the global texture: {110}, {112}, {221}, {012} and {123} (Fig. 2(c)). The two films have similar GB character distributions, see Table 1. The HAGB proportion is the largest, and slightly higher for the 40 nm-thick films. The significant percentage of CSLB, and more particularly of  $\Sigma 3$  twin boundaries in the proportion of  $\sim 10\%$  and  $\sim 15\%$  for the 40 nm- and 240 nm-thick films, respectively, indicates more pronounced twinning in the 240 nm-thick films.

### 3.1.2. Morphology of crystallites and grains

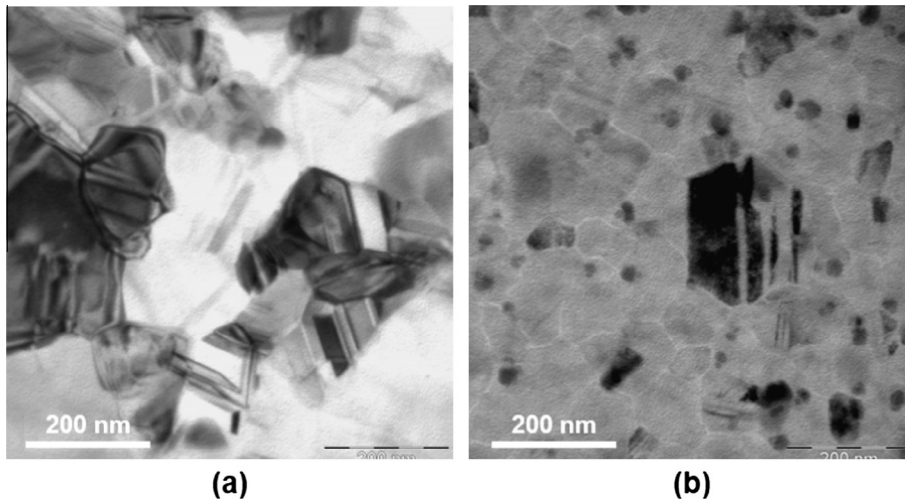
The ACOM-TEM analyses and TEM observations of the poly-Si films (Figs. 2 and 3) reveal a complex microstructure with more twinned grains in the 240 nm-thick films. This result agrees with the extracted GB character distributions summarized in Table 1. Besides, TEM observations (Fig. 3) and some ED patterns confirm the presence of one or two grains over the thickness for the 240 nm-thick films while there is only one grain over the thickness in the 40 nm-thick films.

The two films exhibit different top surface morphologies (Fig. 4). GB grooves are clearly visible on both films while hillocks are present in the thinnest ones only.

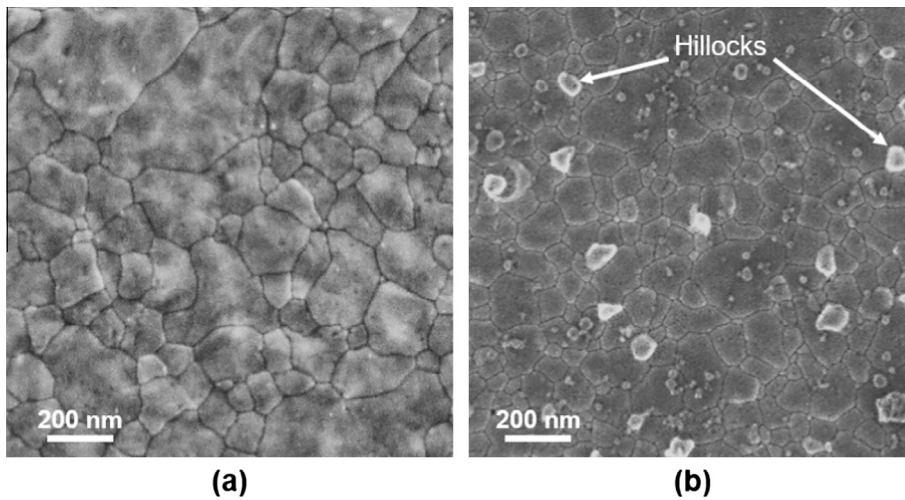
**Table 1**

Summary of the GB character distribution for the 240 nm- and 40 nm-thick poly-Si films extracted from ACOM-TEM analyses (step size of 8 nm, 60 frames per second).

| Thickness (nm) | HAGB (%) | CSLB (%) | $\Sigma 3$ (%) | LAGB (%) |
|----------------|----------|----------|----------------|----------|
| 240            | 64.2     | 30.2     | 14.5           | 5.6      |
| 40             | 70.4     | 23.7     | 9.8            | 5.9      |



**Fig. 3.** TEM top views of (a) the 240 nm- and (b) 40 nm-thick poly-Si films.



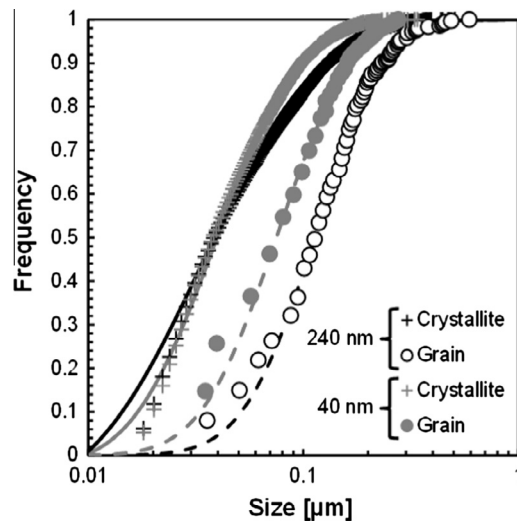
**Fig. 4.** SEM top views of (a) the 240 nm- and (b) 40 nm-thick poly-Si films.

**Fig. 5** shows the cumulative frequency of the in-plane grain and crystallite size distributions. The minimum in-plane crystallite size is set as the equivalent diameter of a circle with an area corresponding to 4 acquisition steps of the ACOM-TEM experiments. Then, an acquisition step size of 8 nm corresponds to a minimum equivalent diameter or in-plane crystallite size of 18 nm. Concerning the in-plane grain size, the minimum equivalent diameter is set to 36 nm. This value is determined by the magnification and the quality of the SEM images as well as the ability to draw small grains with the software ImageJ.

The crystallite size distributions are similar but the grain size distributions are clearly distinct: 50% of the crystallites have an in-plane size larger than 38 nm and the average size is 16% smaller for the 40 nm-thick films (it is  $61 \pm 55$  nm and  $51 \pm 36$  nm for the 240 nm- and 40 nm-thick films, respectively). The average in-plane grain size is  $\sim 35\%$  smaller for the 40 nm-thick films (it is  $127 \pm 82$  nm and  $82 \pm 47$  nm for the 240 nm- and 40 nm-thick films, respectively). For the 40 nm-thick films, 50% of grains have an in-plane size larger than 75 nm which is  $\sim 2$  times the film thickness (equi-axed grains). By contrast, the 240 nm-thick films are composed of 50% of grains with an in-plane size larger than 113 nm which is roughly half the film thickness (indicating a more columnar morphology). [Table 2](#) summarizes the data extracted from the crystallite and grain size distributions.

### 3.2. Surface topology

Contact mode AFM mappings have been performed in order to compare the top surface roughness and the depth of the GB grooves over a region of  $3 \mu\text{m} \times 3 \mu\text{m}$  with a resolution of  $512 \times 512$  pixels. Typical images of AFM and line scans are shown



**Fig. 5.** Cumulative frequencies of the in-plane crystallite and grain size distributions for the 240 nm- and 40 nm-thick films extracted from ACOM-TEM analyses (step size of 8 nm, 60 frames per second) and SEM top views, respectively. The straight and dotted lines are the log-normal fitting curves of the different sets of experimental data.

in Fig. 6. For the 40 nm-thick films, the height of hillocks ranges between 50 and 90 nm. The hillocks are not considered relevant for fracture initiation. They are not taken into account in the estimation of the surface roughness parameters  $R_a$  and  $R_{ms}$ . The top surface of the 40 nm-thick film exhibits a lower roughness. The  $R_{ms}$  roughness is  $\sim 4.3$  nm and  $\sim 5.6$  nm for the 40 nm- and 240 nm-thick films, respectively. The GB grooves depth is larger for the 240 nm-thick films. For the 40 nm-thick films, the deepest GB grooves (peak to valley) have an amplitude of 15 nm corresponding to  $\sim 37\%$  of the film thickness. By comparison, the GB grooves depth for the 240 nm-thick films is  $\sim 30$  nm which represents  $\sim 12\%$  of the film thickness. Concerning the bottom surface, it is not possible to estimate the GB grooves depth. Nevertheless, the top surface being free during the film growth and the doping process, the GB grooves should be significantly deeper on the top surface compared to the bottom one.

Fig. 7 shows SEM and TEM micrographs of the sidewalls for the two film thicknesses. The 240 nm-thick films involve significant sidewall roughness with the presence of crevices and GB grooves. Some of GB grooves are located in the middle of the crevices. The sidewall roughness is governed by the morphology of the crevices and seems to be larger than the top surface roughness, but we cannot quantify it more precisely. For the 40 nm-thick films, the sidewall topography is totally different. No crevice appears, probably due to the very small thickness. The sidewall roughness is dictated by the GB grooves morphology.

### 3.3. Fracture strength

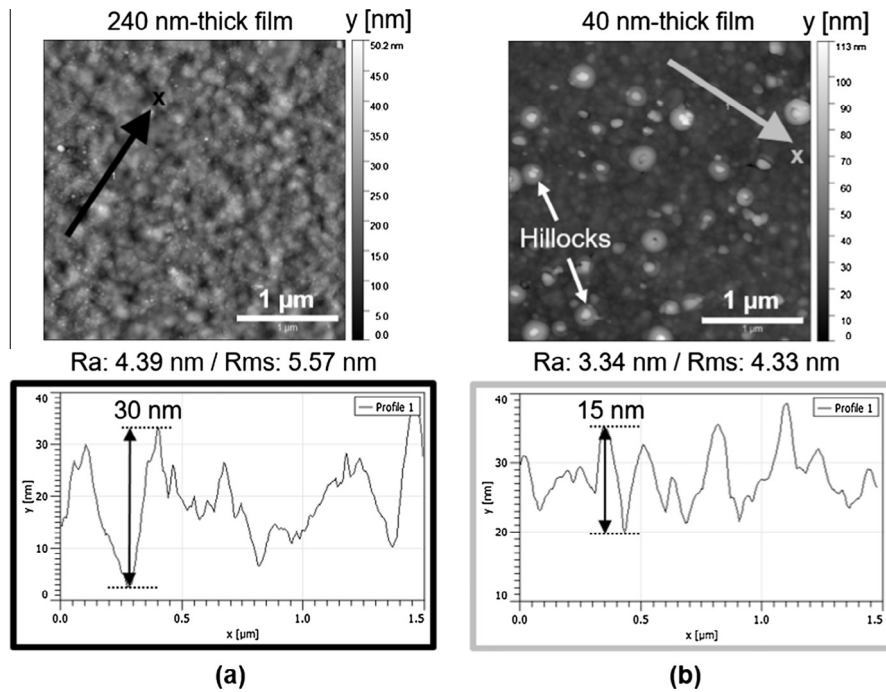
Fig. 8 shows the evolution of the fracture strength as a function of the external surface area and of the length for both film thicknesses. The fracture strength increases with decreasing external surface area for both film thicknesses, see Fig. 8(a). Fig. 8(b) shows that for the 240 nm-thick films, there is no dependence of the fracture strength on the beam length while the 40 nm-thick films exhibit an increase of the fracture strength with decreasing specimen length though with a larger scatter at small lengths. These observations suggest that for the 240 nm-thick films, the critical defects are located on the top and/or bottom surfaces. For the 40 nm-thick films, the critical defects are located on the external surfaces but no specific conclusion can be made. The cracking initiation process could originate from the GB along sidewalls, from the top and/or bottom surfaces, and/or from areas in the vicinity of hillocks. Besides, Fig. 8(a) shows that the fracture strength of

**Table 2**

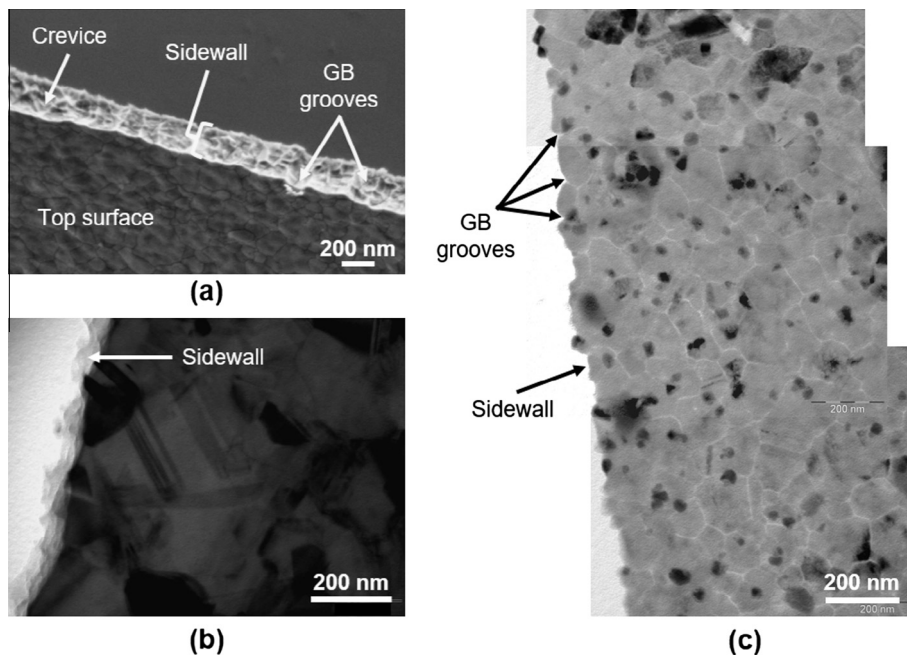
Summary of the data extracted from the in-plane crystallite and grain size distributions for the 240 nm- and 40 nm-thick films extracted from ACOM-TEM analyses and SEM observations, respectively;  $\pm x$  is the standard deviation. The mean size and the corresponding standard deviation are the input parameters for the log-normal fitting curves in Fig. 5; 'c' refers to crystallite and 'g' refers to grain.

|   | Thickness (nm) | Area ( $\mu\text{m}^2$ ) | Number of domains | Average size (nm) | Median size (nm) | Mean size (nm) |
|---|----------------|--------------------------|-------------------|-------------------|------------------|----------------|
| c | 240            | 42.5                     | 8104              | $61 \pm 55$       | 38               | $55 \pm 68$    |
| c | 40             | 16                       | 5185              | $51 \pm 36$       | 38               | $50 \pm 40$    |
| g | 240            | 7.1                      | 397               | $127 \pm 82$      | 113              | $138 \pm 90$   |
| g | 40             | 5.2                      | 739               | $82 \pm 47$       | 75               | $92 \pm 65$    |



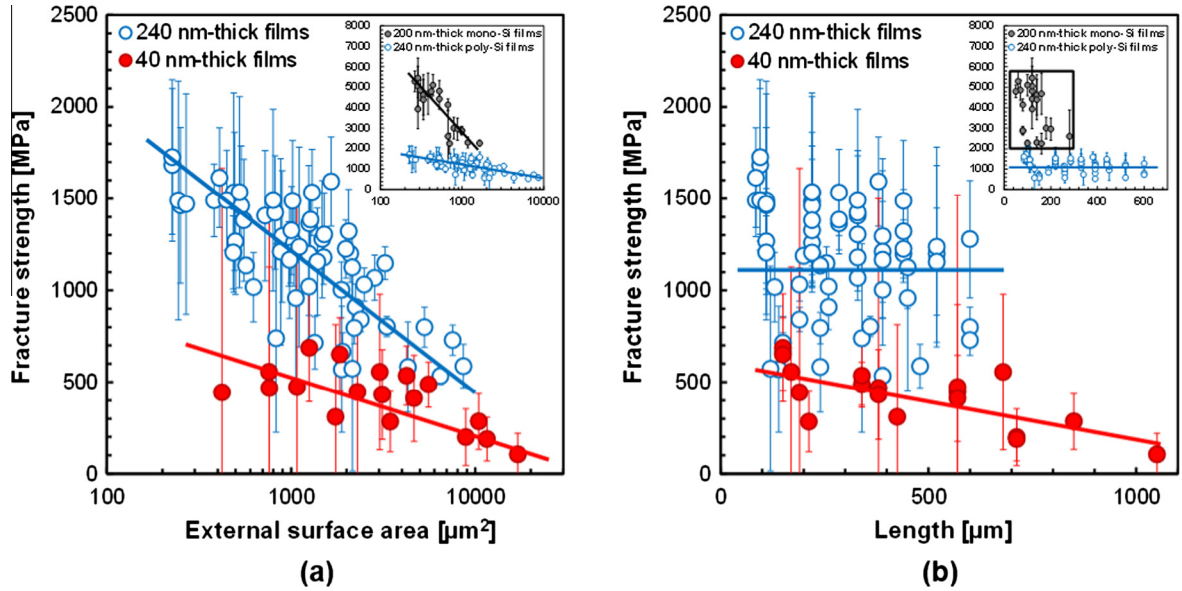


**Fig. 6.** Contact mode AFM mapping and line scan of the top surface of (a) the 240 nm- and (b) 40 nm-thick films. The straight line defines the x axis of the line scan.  $R_a$  is the arithmetic average of absolute values.  $R_{ms}$  is the root mean square of absolute values. For the 40 nm-thick films, the calculus of  $R_a$  and  $R_{ms}$  does not consider the hillocks. They are excluded of the data treatment by masking them with the AFM software.



**Fig. 7.** (a) SEM tilted view of the sidewall and top surface of a 240 nm-thick film; TEM top views of (b) the 240 nm- and (c) 40 nm- thick films showing the sidewall morphology.

40 nm-thick films is significantly lower than in the thicker films, and involves less variation with the external surface area. The 240 nm-thick films exhibit a maximum average fracture strength equal to  $1.73 \pm 0.48$  GPa for an external surface area of  $200 \mu\text{m}^2$ . This value drops down to  $0.59 \pm 0.19$  GPa for a beam of  $10,000 \mu\text{m}^2$ . The highest fracture strength in the

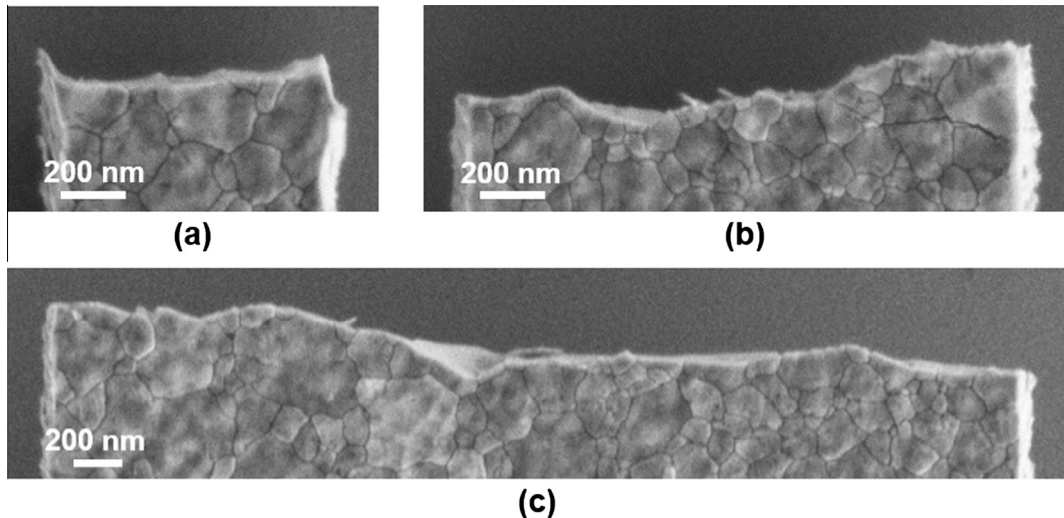


**Fig. 8.** Fracture strength of 240 nm- and 40 nm-thick poly-Si films as a function of (a) the external surface area and of (b) the length. The plot on the top right corner of each figure compares the fracture strength of 200 nm-thick monocrystalline silicon (mono-Si) [27] and the 240 nm-thick poly-Si films extracted with the same technique and patterned with the same RIE plasma etching process [20].

40 nm-thick films is equal to  $0.69 \pm 0.34$  GPa for a  $1250 \mu\text{m}^2$  beam and decreases down to  $0.11 \pm 0.06$  GPa for a  $17,000 \mu\text{m}^2$  beam. The uncertainty on the fracture strength is estimated based on an error propagation approach based on the partial derivative method. The uncertainties on the SEM measurements of cursors displacement and of the width of both poly-Si and SiN actuator beams are evaluated to be around 30 nm. The uncertainty on the beam length is set equal to 1 μm. Several 40 nm-thick poly-Si structures present a large uncertainty on the fracture strength due to the large difference of cross-sectional area between the specimen and actuator beams which amplifies the error, see Ref. [20]. Although for the 40 nm-thick films, the uncertainty on the strength of some structures can be larger than the absolute magnitude of the strength, a clear strength size effect emerges.

### 3.4. Crack path

In order to characterize the crack path, post mortem broken samples have been observed by SEM imaging. Figs. 9 and 10 show the typical SEM top views of the fracture surfaces of the 240 nm- and 40 nm-thick poly-Si beams, respectively, with



**Fig. 9.** SEM top views of the fracture surfaces of 240 nm-thick poly-Si beams of width (a) 0.9 μm, (b) 2.2 μm and (c) 4.2 μm.

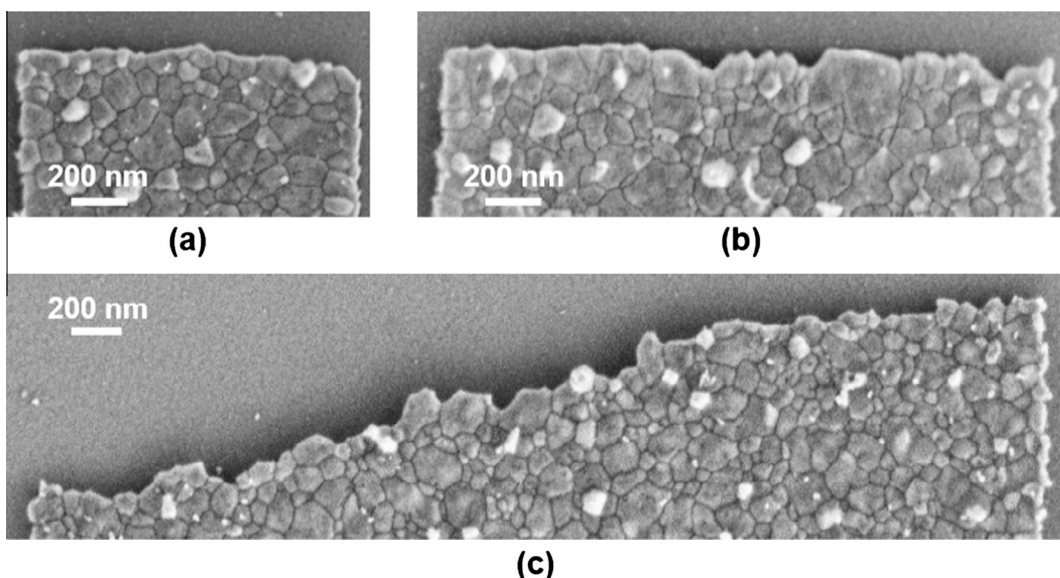
various widths. The fracture paths are clearly different for the two films. Cracking is transgranular for the 240 nm-thick films while it is mainly intergranular for the 40 nm-thick films independently of the beam width. For both films, the zig zag morphology of the crack path seems to be more pronounced for wider beams. For the 240 nm-thick films, the larger the width, the larger the number of crystallites encountered by the crack, the more the crack can deviate and lead to a zig zag morphology. For the 40 nm-thick films, the larger the width, the larger the number of GB grooves, the larger the number of potential paths for fracture propagation, the more pronounced the zig zag morphology.

#### 4. Discussion

The determination of the root causes of the fracture mechanisms requires to consider both of the driving force for fracture, i.e. the local values of the stress or stress intensity factor, and the resistive force to fracture, i.e. the local strength of the crystal and GB or the local fracture toughness of crystal interiors and GBs. The local stress level is dictated by the presence of stress concentration which can have both an extrinsic origin such as GB grooves created by the fabrication process or which can be more intrinsic as the one created by crystal orientation mismatch at GBs and triple points. The same applies to the resistive term. The local fracture stress is dictated by the intrinsic strength of Si and of the GB but can be modified by extrinsic factors like etchant diffusion along GBs decreasing the interface toughness. The following discussion tries to identify the different factors playing a role and, when possible, to quantify them. One of the difficulties is that it is not always possible to deconvolute the different contributions.

The nature of the critical defects in the 240 nm-thick films was previously identified in Ref. [20] as GB grooves emerging on top and/or bottom surfaces by comparing the fracture strength of mono-Si [27] and poly-Si films with similar thickness. This comparison is shown at the top right corner of Fig. 8(a) and (b). The transgranular mode of cracking indicates that the morphology of the GB grooves and, potentially, the local stress distribution in the vicinity of GB produce the conditions needed for cracking initiation and further propagation in the bulk of the crystal lattice. This result confirmed also that GBs are tougher than the crystal lattice, as found experimentally by Qiao and Chen [34]. The exact nature and location of the critical defects in the 40 nm-thick films is more difficult to determine with certainty. It could be GB grooves on external surfaces as well as GB grooves in the vicinity of hillocks. Fig. 10 exhibits the presence of hillocks along the crack path although it is not obvious that GB grooves in the vicinity of hillocks initiate cracking. In contrast, the intergranular crack path reveals clearly that GB grooves assist crack propagation. As the GBs are typically tougher than the crystal lattice [34] and the crystallographic texture is similar for both films, the stress intensity factor associated to GB grooves must thus exceed the GB toughness before exceeding the lattice value, leading to the intergranular fracture. But, the question of the GBs to be really intrinsically tougher than the bulk crystal and the question of the grooving process require further discussion.

For 240 nm-thick poly-Si films, the same fracture origin was put forward by Chasiotis et al. [22] on 2  $\mu\text{m}$ -thick films produced with the PolyMUMPS process. They showed that the depth of the GB grooves on the top surface increases and the fracture strength decreases with increasing HF etching time, the GBs being etched faster than the grains. They observed also a change of crack path from transgranular to intergranular which was attributed to the degradation of the internal structure of the GBs with increasing HF etching time. In our work, the HF etching time is lower than 1 min, hence very short compared to



**Fig. 10.** SEM top views of the fracture surfaces of 40 nm-thick poly-Si beams of width (a) 0.85  $\mu\text{m}$ , (b) 2  $\mu\text{m}$  and (c) 4.1  $\mu\text{m}$ .

the ones used in Ref. [22]. Then, the shape and structure of the GB grooves are not altered by HF etching and are only governed by the film growth process. Hence, the change of crack path has a different origin.

The HAGBs are characterized by a high defect density and a high degree of disorder. The structure of HAGBs is composed of regions with a good and poor atomic coherency governed mainly by the misorientation angle. The HAGBs with the lowest atomic coherency are more prone to grooving under heating because they constitute preferential paths for diffusion. In the same manner, the HAGBs are more susceptible to oxidation, hence to exhibit deeper grooves after HF etching. At the opposite, CSLB and LAGB involve a high atomic coherency because the crystallites on both sides of the GB have a symmetrical relationship. They are less prone to grooving and to oxidation than HAGBs at high temperature due to a more stable and dense structure. In other words, the most severe GB grooves can be associated as a first approximation to the HAGBs. In addition, with a lower average in-plane grain size, the 40 nm-thick films involve a relative proportion of HAGB higher than in the 240 nm-thick films. Assuming that GB grooves located on the top surface are the critical defects for the two films thicknesses, both characteristics support a weaker size dependence of strength in the 40 nm-thick films. The smaller the in-plane grain size and the larger the HAGB proportion, the larger the probability to exhibit deeper GB grooves, the lower the size effect on the fracture strength.

A simple fracture mechanics approach is used in order to more quantitatively analyse the contribution of GB grooves on the fracture initiation for both films. The fracture strength  $\sigma_f$  is related to the fracture toughness  $K_{Ic}$ , the critical defect size  $a$  and the non-dimensional geometrical factor  $Y$  associated to the critical imperfection as

$$\sigma_f = \frac{K_{Ic}}{Y\sqrt{a\pi}}. \quad (3)$$

Assuming that the critical defects are the GB grooves located on the top surface and that they can be assimilated to sharp cracks of length  $a$ , the expression of the non-dimensional geometrical factor  $Y$  is given by [35]:

$$Y = 1.122 - 0.231\frac{a}{t} + 10.550\left(\frac{a}{t}\right)^2 - 21.710\left(\frac{a}{t}\right)^3 + 30.382\left(\frac{a}{t}\right)^4, \quad (4)$$

where  $t$  is the film thickness (with 0.5% accuracy when  $a/t \leq 0.6$ ). For the 240 nm-thick films with a crack length of 30 nm equal to the deepest GB grooves detected on the top surface, the geometrical factor is equal to 1.22. With the average fracture toughness of  $1 \text{ MPa m}^{1/2}$  measured by Chasiotis et al. [14] for poly-Si films, the estimated fracture strength should be 2.66 GPa. For 40 nm-thick films with 15 nm crack length equal to the deepest GB grooves detected on top surface, the geometrical factor is equal to 1.97 and the corresponding fracture strength should be equal to 2.33 GPa. This simple approach suggests that for poly-Si films with GB grooves located on the top surface as critical defects, the theoretical fracture strength should be larger for the thickest films. This prediction qualitatively agrees with the experimental measurements. Nevertheless, the difference between the theoretical and the experimental tensile strength indicates that the stress intensity factor associated to the critical flaws must be affected by other contributions than the GB groove dimensions. This difference is particularly significant for the 40 nm-thick films. Several contributions can raise the stress intensity factor such as the interaction of critical defects and the local heterogeneous stress field in the vicinity of GBs. Although the crystallographic texture is similar for both films, the 40 nm-thick films involve the smallest average crystallite size. This aspect leads to an increase of the local stress heterogeneity in the GB vicinity and contributes to raising the stress intensity factor associated to the GB grooves. Besides, the equi-axed grain structure combined to the very low thickness suggest that the top and bottom grooves of GB could interact so as to facilitate the fracture initiation as well as its propagation by percolation through the GB grooves network. Finite element modelling can be very helpful and efficient to quantify and to analyse the role of these different contributions [36]. Finally, the fracture toughness of some HAGBs, where cracking takes place in the 40 nm-thick films, could potentially be lower than the transgranular toughness. All these aspects participate to explain the lowest fracture strength and the reduced size effect observed for the 40 nm-thick films as well as the intergranular crack path. The decrease of the fracture strength and the change of crack path from transgranular to intergranular with the film thickness decrease are not related to an intrinsic size effect but more to the polycrystalline nature of the material. They are induced by an indirect effect of both the fabrication process and of the film thickness which govern the film growth mode, hence the final microstructure. Very recently, Yagnarmurthy et al. [13] produced two types of  $1 \mu\text{m}$ -thick poly-Si films with different microstructures and surface roughness features. The first films had a coarse grain microstructure with an average grain size of 285 nm and one or two grains over the thickness. The second films involved a laminated microstructure with an average grain size of 125 nm. The laminated films had twice larger average tensile strength than the coarse grain films, respectively 1.31 and 2.44 GPa, due to smoother sidewalls. The critical defects were located on or in the vicinity of the sidewalls. For coarse grain films, the defects were GB grooves preferentially etched during the RIE sidewall patterning and located in the vicinity of the upper corners while for the laminated films they were not identified. Yagnarmurthy et al. [13] demonstrated that the tensile strength of the coarse grain films could be increased up to the tensile strength of the laminated films by smoothing of the sidewall roughness through FIB treatment. This result suggests that the minimization or even the suppression of the grooving process at the GBs would lead to larger tensile strengths for poly-Si films with nanoscale thickness. In conclusion, the production of poly-Si films with nanoscale thickness and large fracture strength requires an optimized fabrication process minimizing as much as possible the grooving at GBs. This will become more and more complex to achieve with thickness reduction down to the nanoscale.

## 5. Conclusions

The aim of this study was to investigate the effect of the specimen size on the fracture strength and fracture mechanisms occurring in submicron freestanding poly-Si films. Two film thicknesses were investigated: 240 and 40 nm. The fracture strength was extracted using an on-chip tensile testing technique. The fracture initiation and propagation were analyzed based on microstructural and surface roughness data as well as post-mortem observations of the broken specimens. The main conclusions of the study are the following:

- The critical defects controlling fracture initiation are the GB grooves for both thicknesses. For the 240 nm-thick films, the critical defects are located on top and/or bottom surfaces. This result is deduced from the fact that there is no dependence on specimen length but only on specimen external surface area, and from the comparison of the fracture strength of mono-Si and poly-Si films with similar thickness. For 40 nm-thick films, the critical defects location has not been elucidated, being either GB grooves on external surfaces or GB grooves in the vicinity of hillocks.
- The decrease of the film thickness is accompanied by a change of fracture mechanisms from transgranular to intergranular. Furthermore, the fracture strength for the same external surface area and beam length, and the specimen size dependence decrease with film thickness. These results are not related to an intrinsic size effect but more to the polycrystalline nature of the material and to microstructure difference controlled by the fabrication process and film thickness.
- For both films, the theoretical fracture strength estimated from fracture mechanics using as crack length the maximum experimental GB grooves depth detected is larger than the experimental fracture strength. This discrepancy is much larger for the 40 nm-thick films and suggests that the stress intensity factor associated to the GB grooves is raised by other contributions that the GB grooves depth and the film thickness involving the interactions between top and bottom GB grooves, the local stress heterogeneity in the vicinity of GBs induced by the very fine crystallite and grain size, and very local residual stress.
- For the present range of film thickness and the present fabrication process, the smaller the film thickness, the smaller the in-plane grain size, the higher proportion of HAGB, the larger the probability to exhibit deeper GB grooves and the lower the size effect on the fracture strength.
- The transition into an intergranular mode of cracking fracture in the thinnest films is most presumably a result of a percolation of the GB grooves network.

This study demonstrates that in the perspective of the development of reliable and efficient applications using polycrystalline brittle thin films with nanoscale thickness, the minimization or even the suppression of the grooving process at GBs is the critical issue to address.

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