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Study of recrystallization and activation processes in thin and highly doped Silicon-On-Insulator layers by nanosecond Laser Thermal Annealing

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Abstract

In this work, a thorough study of the phosphorus (P) heavy doping of thin Silicon-On-Insulator (SOI) layers by UV nanosecond Laser Thermal Annealing (LTA) is presented. The melting regimes and the regrowth processes as well as the redistribution and activation of P in the top-Si amorphized layer were studied as a function of the implant dose and laser annealing conditions. The results highlight the crucial role of the thin crystalline silicon layer preserved after amorphization of the top-Si layer, which provides nucleation seeds for the liquid phase recrystallization. The impact of the implant dose on the recrystallization process is investigated in detail, in terms of melt energy thresholds, crystallographic nature of the resolidified layer, defect formation, surface roughness and hillocks formation at the silicon surface. For all the implanted doses, optimized laser annealing conditions were identified, corresponding to the laser energies just preceding the onset of the full melt. Such optimized layers exhibit perfect crystallinity, negligible P out-diffusion, an almost perfectly flat P depth profile located below the segregation-induced surface pile-up peak and dopant active concentrations well above $1 \times 10^{21} \text{ cm}^{-3}$, close to the highest reported values reported for phosphorus in bulk Si substrates.

I. INTRODUCTION

The general evolution of technologies in microelectronics has been marked for several decades by the ceaseless miniaturization of its basic component, the MOS transistor, in order to obtain greater circuit complexity, better performances and reduced costs. Historically, the production of doped source/drain zones has required a continuous reduction in their size, accompanied by an increase in the concentration of active dopants. With the introduction of new MOS architectures such as FD-SOI or FinFET, which enabled increased speed, reduced parasitic capacitance and lower power consumption, the increase of dopant activation has become a particularly critical issue, especially because of its impact on the source/drain contact resistance [1]. Within this context, out-of-equilibrium, fast thermal annealing processes, such as laser thermal annealing (LTA), have therefore been explored as a replacement of conventional thermal annealing. In addition to allowing the production of ultra-thin and ultra-doped junctions, LTA annealing is able to achieve very strong local activation (on the surface and in depth) [2] and a uniform distribution of dopants [3]. This process, studied in the research community since the 1980s for the manufacture of solar cells [4], or for more fundamental studies such as the investigation of damage recovery in ion-implanted Si [5] (see also [6] and references therein), also offers new technological possibilities for the development of 3D architectures [7]. Indeed, in the case of nanosecond laser annealing, the short pulse duration as well as the short absorption length ensures that only the surface of the material is heated, which is particularly suitable for 3D sequential integration.

More generally, highly doped thin Si layers achieved by LTA are also attractive for possible applications in other fields, including (i) optoelectronics devices such as photodetectors [8] or photodiodes [9] and (ii) quantum devices based on the superconductive properties of highly doped silicon at low temperatures [10,11]. Infra-Red (IR) plasmonics is an additional application field to be considered. Indeed, Surface Plasmon Polaritons (SPP) have been demonstrated on p and n-doped Si layers [12,13] showing improved SPP confinement with respect to metal-dielectric interfaces. Overall, whether in microelectronics or plasmonics, the unique features of nanosecond LTA (uniform doping, high activation rate, local heating) make it an attractive technological solution.

Improved dopant activation by LTA in bulk Silicon substrates has been extensively studied and demonstrated for several dopants [14,15]. As for SOI substrates, differences in thermal processing with respect to bulk silicon (heat rate, dopant activation, solid phase epitaxial regrowth) have been mainly explored in the case of conventional furnace or RTA annealing schemes [16,17]. In contrast, fewer studies exist for laser annealing. For instance, Michalak *et*

al. [18] considered the case of millisecond laser anneals in sub-melt regime, while Chang *et al.* [19] investigated the activation of phosphorus in thick SOI layers (200 nm) by applying a complex sequence of furnace, RTA and nanosecond LTA.

The lack of studies becomes more critical for very thin SOI layers (25 nm or less), which are relevant for the most advanced technologies (FD-SOI and/or 3D sequential integration). In the works of Acosta Alba *et al.* [20] and Daubriac *et al.* [11], the process conditions for achieving high doping levels by nanosecond LTA in the melt regime were identified for phosphorus and boron, respectively. However, the complex relation between the structural properties and the corresponding doping levels were not fully investigated. Indeed, the deep understanding of the different melting regimes and regrowth processes when a thin and highly doped Si layer is submitted to nanosecond laser annealing is of outmost importance for predictive simulations and optimization of the future devices involving such processes.

In this paper, a thorough study of the heavy doping of thin SOI layers is presented, involving low energy and high dose P implantation coupled to UV nanosecond LTA for efficient dopant activation. For this purpose, a panel of multi-scale characterization techniques are carried out involving *in situ* time-resolved reflectometry, Time-of-Flight Secondary Ion Mass Spectroscopy, four-point probe, and Hall effect measurements coupled to atomic scale observations based on advanced Transmission Electron Microscopy and Atomic Force Microscopy (AFM). The different melting regimes and further recrystallization processes involving explosive crystallization and epitaxial liquid phase epitaxy are investigated as a function of the laser energy density and doping content. The concomitant P redistribution and dopant activation processes are discussed.

II. EXPERIMENTAL DETAILS

Investigated samples are 300 mm Silicon-On-Insulator (SOI) substrates with thin 23 $\pm$ 2 nm top silicon layer (001) and 20 nm-thick SiO₂ buried oxide (BOx) on top of an intrinsic Si substrate. In a first step, the top silicon is implanted with phosphorus at 4 keV (see Fig. 1a) with four increasing doses (see Table I). The projected range of the implanted profiles is 7.5 nm with a Full Width at Half Maximum (FWHM) at 10.6 nm as predicted by SRIM simulations. These high dose implantations give rise to the amorphization of part of the Si top layer (see Fig. 2a), with depths ranging from 14.5 nm for the lowest dose up to 18 nm for the highest (Table I).

TABLE I. Implanted doses, concentrations at the projected range (R_p) predicted by SRIM calculations and thickness of the Si amorphized region measured by TEM.

SOI	Q1	Q2	Q3	Q4
Dose (P/ cm ²)	10 ¹⁵	4x10 ¹⁵	7x10 ¹⁵	10 ¹⁶
Concentration at R_p (cm ⁻³)	8.5x10 ²⁰	3.5x10 ²¹	6x10 ²¹	9x10 ²¹
Concentration at R_p (at. %)	1.7	6.9	12	17
Amorphous thickness (nm)	14.5	15.5	17.5	18.0

In a second step (Fig. 1b), UV nanosecond laser thermal annealing (LTA) is performed using a SCREEN-LASSE LT3100 system involving a XeCl excimer pulsed laser ($\lambda = 308$ nm, 4 Hz) with a single pulse duration of 160 ns. The laser energy density has been adjusted in order to cover all the relevant phenomena investigated in this work (i.e. from the onset of melt at the surface to the full melt of the SOI layer). The corresponding energy density range (from 1.00 to 1.80 J/cm² with a step of 0.05 J/cm²) is relatively narrow, which is consistent with the extremely low thickness of the SOI layers used in this study.

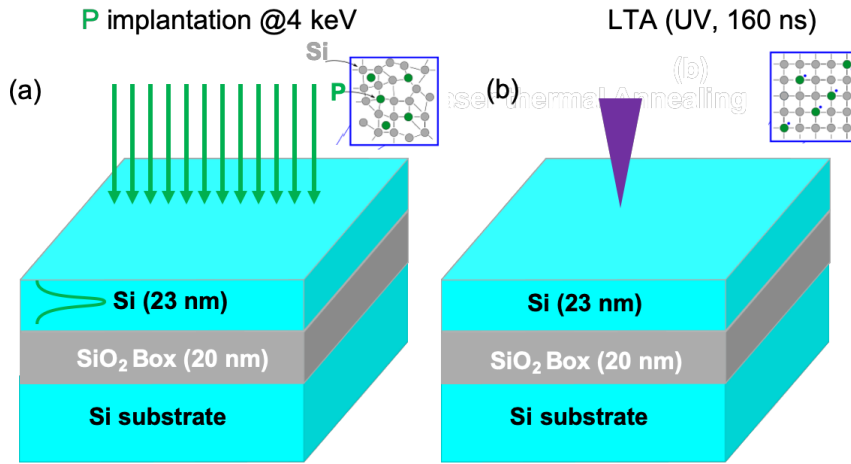


FIG. 1. Scheme describing the stack and steps for dopant introduction and activation: (a) P implantation and Si top layer partial amorphization and (b) Laser Thermal Annealing (LTA).

In situ time resolved reflectometry (TRR) measurements at 635 nm have been performed during the LTA process in order to determine the different annealing regimes. Modification in TRR signal is caused by changes in optical properties (refractive indexes). This occurs when the material temperature increases or because of a transition to the liquid phase, which exhibits a much higher reflectivity compared to the solid phase [21]. In addition, when the melt threshold is reached and then exceeded, the TRR intensity exhibits monotonic variations as the molten Si layer grows until reaching a plateau. Such plateau corresponds to the

reflectivity of a semi-infinite liquid Si layer. For Silicon, TRR saturation is obtained as soon as the melt front has penetrated ~ 20 nm, i.e. about two optical skin depths of the probe laser with a 635nm wavelength [22].

Samples have been prepared in cross-section by Focus Ion Beam (FIB) for Transmission Electron Microscopy observations with a decreasing voltage from 16 kV-50 pA to 5 kV-15 pA. A last cleaning step is performed at 2 kV-10 pA to avoid damage during the FIB preparation [23]. Structural properties were investigated by Scanning Transmission Electron Microscopy (STEM) observations using a JEOL ARM 200 F probe-corrected microscope with a cold FEG emission at 200 kV.

The surface roughness has been studied by Atomic Force Microscopy (AFM) in tapping mode, using a Dimension D3000 AFM with a Nanoscope III controller coupled with the Basic Extender module (Bruker). Experiments were performed in clean room, with Nanosensors PPP-NCH Si tips (typical force constant: 42 N/m, tip radius of curvature < 10 nm).

The P implanted profiles have been measured by Time-of-Flight Secondary Ion Mass Spectroscopy (TOF-SIMS) in a dual beam IONTOF IV system equipped with a Cs^+ ion gun for sputtering and a Ga^+ ion gun for analysis. Measurements have been accomplished by means of Cs^+ ions at 1 keV and 80 nA and Ga^+ ions at 25 keV and 1 pA. Accurate time-to-depth conversion has been performed by measuring the average sputter velocity in a 16 nm-thick SOI reference samples. Calibration of the $^{31}\text{P}^-$ signals in the Si matrices was performed on the basis of experimental protocols already reported in previous publications [24,25].

Sheet resistance measurements have been performed after LTA by Four-Point-Probe for each dose and laser energy density. These measurements were performed with a WS3000 tool from NAPSON. The tips are equally distributed with 1 mm spacing in between, and their curvature is about 150 μm . The active carrier dose and mobility have been measured by means of Hall effect technique, using a HL5500PC Nanometrics Hall bench equipped with a 0.3 T magnet. Considering the extremely high active dopant concentrations expected in the investigated samples (well above $1 \times 10^{20} \text{ cm}^{-3}$), the measured values of Hall carrier concentration and Hall mobility are corrected by using a Hall scattering factor, r_H , equal to 0.9 [26]. The experimental profiles measured by TOF-SIMS have been used as inputs for the Hall active dose extraction.

III. RESULTS AND DISCUSSION

A) Structural evolution upon laser annealing

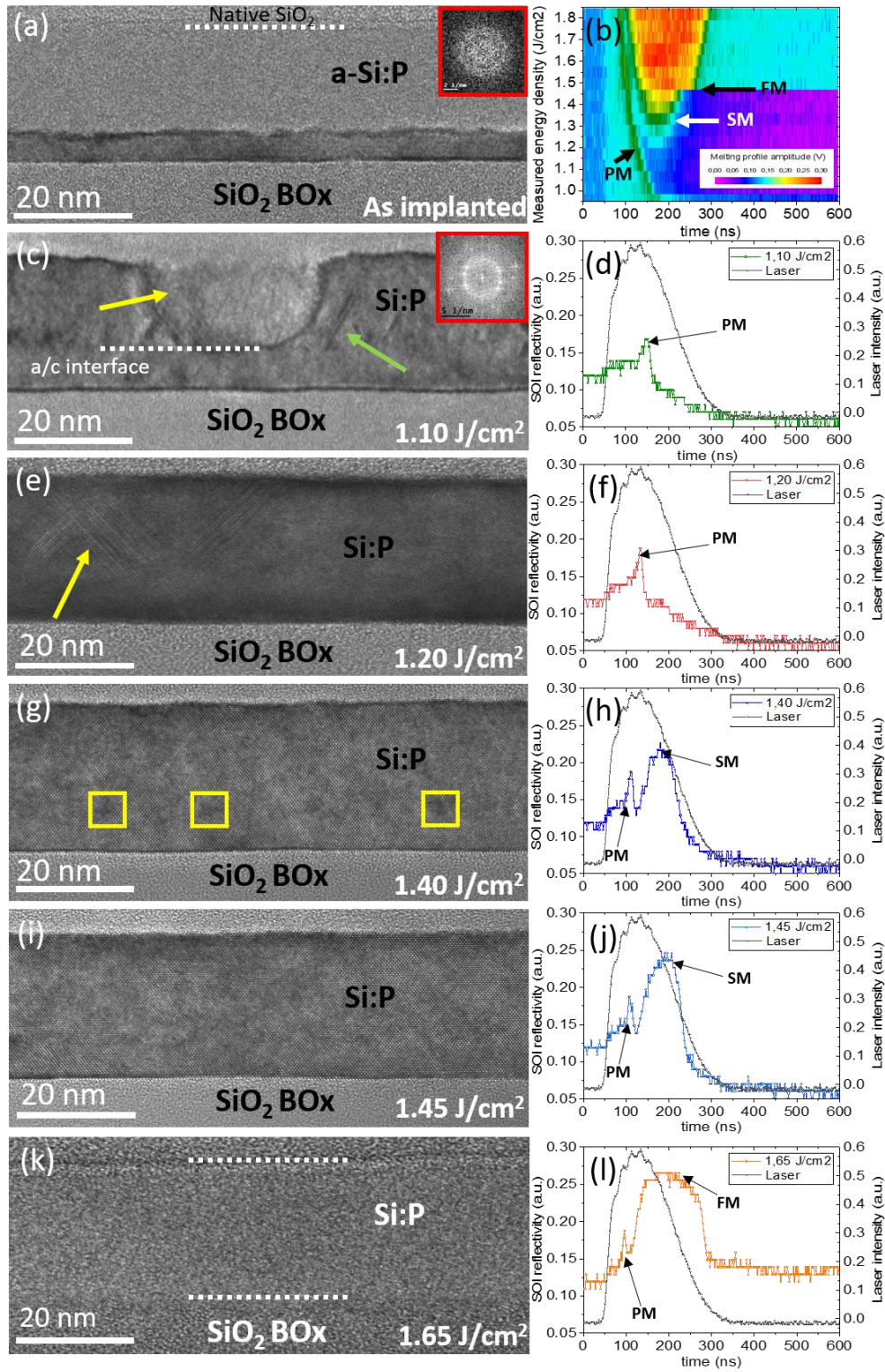
A.1 – Primary and secondary melt, explosive crystallization and liquid phase epitaxy

The different annealing regimes and final microstructures obtained after subsequent recrystallization of the Si layer have been investigated by coupling in situ TRR measurements and TEM observations, as shown in Fig. 2 for the sample implanted with the highest dose (Q4).

For the as-implanted sample (Fig. 2a), the interface between the amorphized and remaining crystalline region (a/c interface) presents a roughness of around 2 nm (the same value was found for all the implant doses). In Fig. 2b, the intensity of the reflectivity signal is plotted with a color map as a function of annealing time (x axis) and laser energy density (y axis). The TRR signals extracted at fixed energy densities corresponding to the different annealing regimes are plotted in Fig. 2d, f, h, j and l (colored curves, left axis). The laser beam intensity profile is also reported in these plots (black curves, right axis). The associated microstructures are shown in Fig. 2c, e, g, i and k.

Starting from an energy density of 1 J/cm^2 , the melt of the amorphous Si (a-Si) layer is observed in the form of a sharp peak in the TRR signal (labeled “PM”, for Primary Melt, in both the TRR color map and in the extracted profiles of Fig. 2). The primary melt is immediately followed by solidification at high velocities. This fast and transient process, known as Explosive Crystallization (EC), is due to the lower melt temperature of a-Si compared to c-Si [27], leading to the formation of an undercooled Si liquid layer. The latent heat released during its crystallization induces the melt of a thin layer of the underlying amorphous material. The fast solidification of this new undercooled liquid and the corresponding release of latent heat allows the crystallization process to propagate rapidly through the entire amorphized layer. Hence the regrowth of the a-Si takes place by a combination of upward and downward explosive crystallization, giving rise to a polycrystalline material in the entire volume of the amorphized film [28,29]. However, for a laser energy density of 1.1 J/cm^2 (Fig. 2c), only part of the film presents the expected polycrystalline structure, which occurs in the form of droplets (cf. yellow arrow in Fig. 2c) extending down to $16 \text{ nm} \pm 2 \text{ nm}$ (corresponding to the depth of the former a/c interface). Surprisingly, the surrounding areas are monocrystalline and epitaxially recrystallized with

180 respect to the c-Si substrate. Moreover, they contain several extended defects (cf. green arrow
 181 in Fig. 2c).



182

183 *FIG. 2. (a) HREM images of the as-implanted sample; (b) TRR signal colormap: intensity of*
 184 *the reflectivity signal as a function of annealing time (x axis) and laser energy density (y axis)*
 185 *for sample Q4; (d), (f), (h), (j) and (l) are the extracted TRR profiles at energy densities of*

respectively 1.1 J/cm^2 , 1.2 J/cm^2 , 1.4 J/cm^2 , 1.45 J/cm^2 and 1.65 J/cm^2 . The peak with black line corresponds to the laser pulse. The different annealing regimes, namely primary melt (PM), second melt (SM) and full melt (FM) are indicated by arrows. The associated HREM images are shown in (c), (e), (g), (i) and (k).

The EC phenomenon has been extensively studied for thick melt amorphous layers [28]. The initial liquid layer formed by the primary melt solidifies to form coarse-grained polycrystalline Si, meanwhile a thin, self-propagating liquid layer travels through the remaining amorphous Si, producing fine-grained polycrystalline Si. Finally, two polycrystalline layers with different grain sizes are expected to form as a result of the solidification process. However, in our case the thickness of the whole amorphized layer is so low that a distinction between the two poly-Si layers would be extremely difficult. In addition, the primary melt starts randomly at the silicon surface (nucleation) as in classical phase transitions [30,31]. For a laser pulse duration of 145 ns, it has been shown that melt droplets as thick as $\sim 15 \text{ nm}$ can be formed at the onset of surface melt while adjacent areas can be melted down to a lower depth or even remain in the solid phase [32]. In our case, due to the difference in melting temperature between a-Si and c-Si [27], the maximum thickness of the melted droplets in the surface melt regime might well correspond to the thickness of the entire a-Si layer. Based on such scenario, the different types of liquid droplets (L-Si) present in the thin amorphous Si layer within the primary melt regime are schematically represented in Fig. 3a. When the solidification of the undercooled liquid starts, the liquid droplets whose thickness is lower than the original a-Si thickness (cf. small liquid droplet on the left of Fig. 3a) will therefore recrystallize into a poly-Si phase according to the classical EC mechanism (see green arrows in Fig. 3a). In contrast, the undercooled liquid droplets whose thickness correspond to the whole a-Si layer, will solidify epitaxially into monocrystalline Si from the underlying crystalline seed (orange arrows noted “LPE” for Liquid Phase Epitaxy), and grow laterally by EC (green arrows). In this case, the lateral EC proceeds with thin liquid layers that are always in contact with monocrystalline Si so that the lateral solidification of such large droplets by EC also results in the formation of monocrystalline Si. This scenario is in accordance with the coexistence of polycrystalline droplets and monocrystalline Si regions observed by TEM in Fig. 2c.

By slightly increasing the laser energy density, it is expected that the entire amorphous layer is melted during the primary melt. In such case, LPE starting from the a/c interface would occur for the whole undercooled liquid layer during solidification, and no poly-Si droplets should be formed as a result of EC. Indeed, at 1.2 J/cm^2 (Fig. 2e), a monocrystalline structure

is observed with some twin defects (indicated by yellow arrows). These extended defects start at a depth of 16 nm from the surface and their formation is likely triggered by the roughness of the former a/c interface. Beyond 1.20 J/cm², the emergence of a high reflectivity region at $t \sim 200$ ns in the TRR color map (cf. Fig 2b) suggests that, after the LPE is completed, a second melt of the solidified material occurs (labelled SM in Fig. 2). It is important to note that unlike the primary melt, the second melt occurs at the “normal” melting temperature of silicon leading to a more stable liquid layer.

At 1.20 J/cm², such SM is not yet clearly detected (cf. Fig. 2f), probably because the molten thickness is still very limited. In contrast, for higher energy density (1.4 J/cm², Fig. 2g), the TRR signal (Fig. 2h), clearly exhibits a more intense SM peak, indicating a larger molten thickness. The final regrown structure (Fig. 2g) is a monocrystalline material with better quality compared to lower laser energies (absence of twins or stacking faults). In addition, some extrinsic dislocations loops are located at 21 nm from the surface, i.e. below the former a/c interface (yellow squares in Fig. 2g). Such defects are known to form during LTA at temperatures just below the melting point in self-interstitial rich regions [33,34]. In such conditions, some isolated and mobile liquid like droplets can be formed. They grow by a coalescence mechanism and transform into unconventional {001} loops. Their presence in the sample annealed at 1.4 J/cm² therefore reveals that the melt front did not reach the self-interstitial-rich c-Si region located below the a/c interface. After a slight increase of the laser energy from 1.4 to 1.45 J/cm², the melt front is deeper as indicated by the TRR intensity increase in Fig. 2j. For this last laser energy condition, the liquid front reaches the self-interstitials region and dissolves the dislocation loops. This gives rise to a further improvement of the Si top layer crystalline quality as observed by TEM in Fig. 2i.

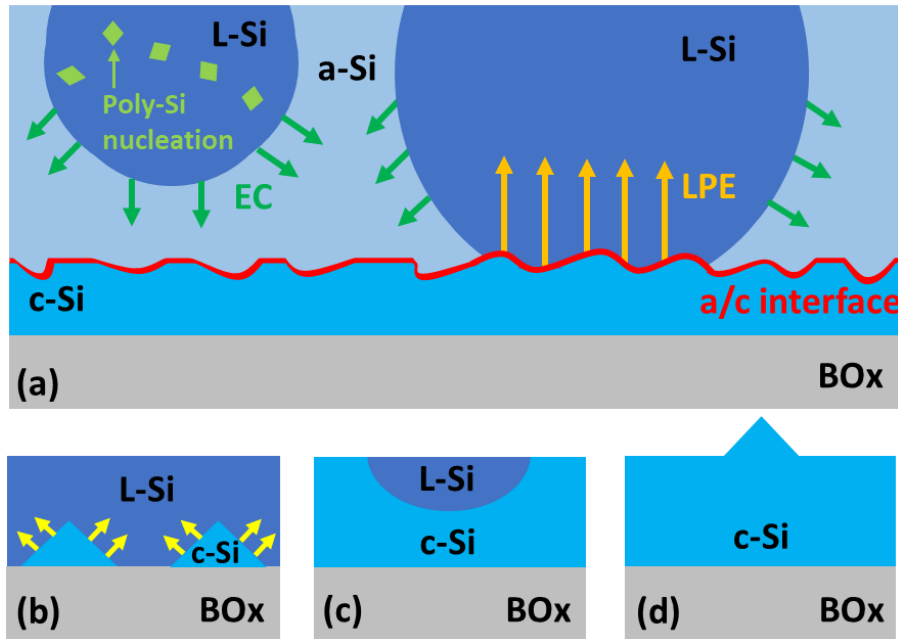


Fig. 3. (a) Schematic representation of the different types of liquid droplets (L-Si) present in a thin amorphous Si layer within the primary melt regime. In the case of small L-Si droplets (dark blue sphere on the left), the melt front does not touch the a/c interface. Explosive crystallization (EC, green arrows) is therefore expected to occur in all directions during solidification, leading to the formation of poly-Si grains. For larger L-Si droplets (dark blue sphere on the right), the lower part of the melt front reaches the a/c interface. The solidification occurs in this case by liquid phase epitaxy (LPE, orange arrows) starting from the underneath c-Si layer, leading to the formation of c-Si material; (b), (c) and (d) sketches illustrating a possible scenario for the formation of surface hillocks (as reported in Fig. 4). (b) resolidification (yellow arrows) starts from isolated crystalline seeds left at the c-Si/BOx interface at the end of the second melt; (c) when the upper part of the solidification front of two neighbour c-Si seeds reaches the surface, a liquid droplet (L-Si) is left at the surface; (d) surface hillocks are finally formed by the solidification of these residual L-Si droplets.

A.2 – Full melt and doping effect

For the melt/solidification regimes discussed so far (primary melt and second melt), the same recrystallization processes and final microstructures have been observed independently of the implanted doses. However, a decrease of the melt threshold when increasing the doping level is noticed (see Table SI, supplementary material). This systematic reduction of melt threshold is probably due to the lowering of the melting temperature for phosphorus-rich Si, from 1414°C (pure Si) down to 1140°C (SiP alloy). We cannot exclude an additional effect linked to an increase of the light absorption due to thicker amorphous layers for higher doses.

A different situation is found at higher energy densities ($> 1.5 \text{ J/cm}^2$, for Q4) when the secondary melt front reaches the BOx interface. This full melt (FM) regime corresponds to

the plateau observed on the TRR profiles in Fig. 2l for sample Q4. In the FM regime, the morphology of the regrown layers is strongly affected by the doping level. Hence, for samples implanted with the lowest doses (Q1 and Q2) the final structure of the Si top layer is found to be polycrystalline (see Fig. S1, supplementary material). On the contrary, the final structure is fully amorphous for the samples implanted with highest doses (Q3 and Q4) (see Fig. 2k for Q4). This is also confirmed by higher baselines in TRR profiles after recrystallization for Q3 and Q4 (Fig. 2l) compared to Q1 and Q2 (see Fig. S2, supplementary material) [35].

The polycrystalline morphology obtained after regrowth for the lower doping level (Q1 and Q2) is consistent with the estimated solidification velocity ($< \sim 2$ m/s), as extracted from the negative slope of the TRR signal, for which polysilicon nucleation and growth occurs during solidification [21]. In contrast, the formation of an amorphous layer is expected for much higher solidification velocities (typically higher than 15 m/s), which have not been measured in any of the observed samples. To this respect, the formation of an amorphous layer in samples implanted with high P doses (Q3 and Q4) is then an unexpected result. A possible explanation might be based on the reduction of the crystal nucleation rate in heavily P-doped silicon, as already found in the case of solid phase epitaxy experiments [36]. However further investigations are needed to confirm such hypothesis.

Finally, other structural differences can be evidenced depending on the doping level when the system approaches the full-melt regime. For the lowest implanted dose (Q1), hillocks on top of the resolidified surface appear at 1.6 J/cm^2 . At this laser energy, the secondary melt has reached the former a/c interface, as confirmed by the monocrystalline nature of the regrown layer and the presence of isolated loops indicated by yellow arrows in Fig. 4a. The observed hillocks are about 10 nm high and 80 nm wide. Atomic Force Microscopy (AFM) scans from this sample (see Fig. 4b) show the random hillocks distribution on the surface plane (better visible in the magnified view of the inset in Fig. 4b). When increasing the energy density up to 1.7 J/cm^2 , the melt front starts to overlap the interface between the Si top layer and the BOx (no loops observed in this case). At this stage, the hillocks are the most pronounced (25 nm high and 130 nm wide, Fig. 4c). The solidified layer mainly consists of defective c-Si material (a stacking fault is indicated by the yellow arrow), while some polycrystalline domains are also observed (inset of Fig. 4c). In the associated AFM image (Fig. 4d), the random network of hillocks (bright points) is clearly visible. Additional regions with a lower surface roughness (delimited by dashed white lines in the figure) correspond to the polycrystalline domains. A further increase of the energy density up to 1.8 J/cm^2 allows the melt front to fully cover the

c-Si/BOx interface. A polycrystalline layer is now formed and no hillocks are formed at the surface, as confirmed by both TEM (Fig. 4e) and AFM (Fig. 4f) analysis. For sample Q2, the same type of hillocks is also observed but their domain of existence is reduced and limited to the beginning of the full melt regime at 1.55 J/cm^2 (see Fig. S3, supplementary material). Finally, the samples implanted with the highest P doses (Q3 and Q4) do not show any surface roughness, neither at the end of the second melt (Fig. 2i for Q4) nor at the beginning of the full melt (Fig. 2k for Q4).

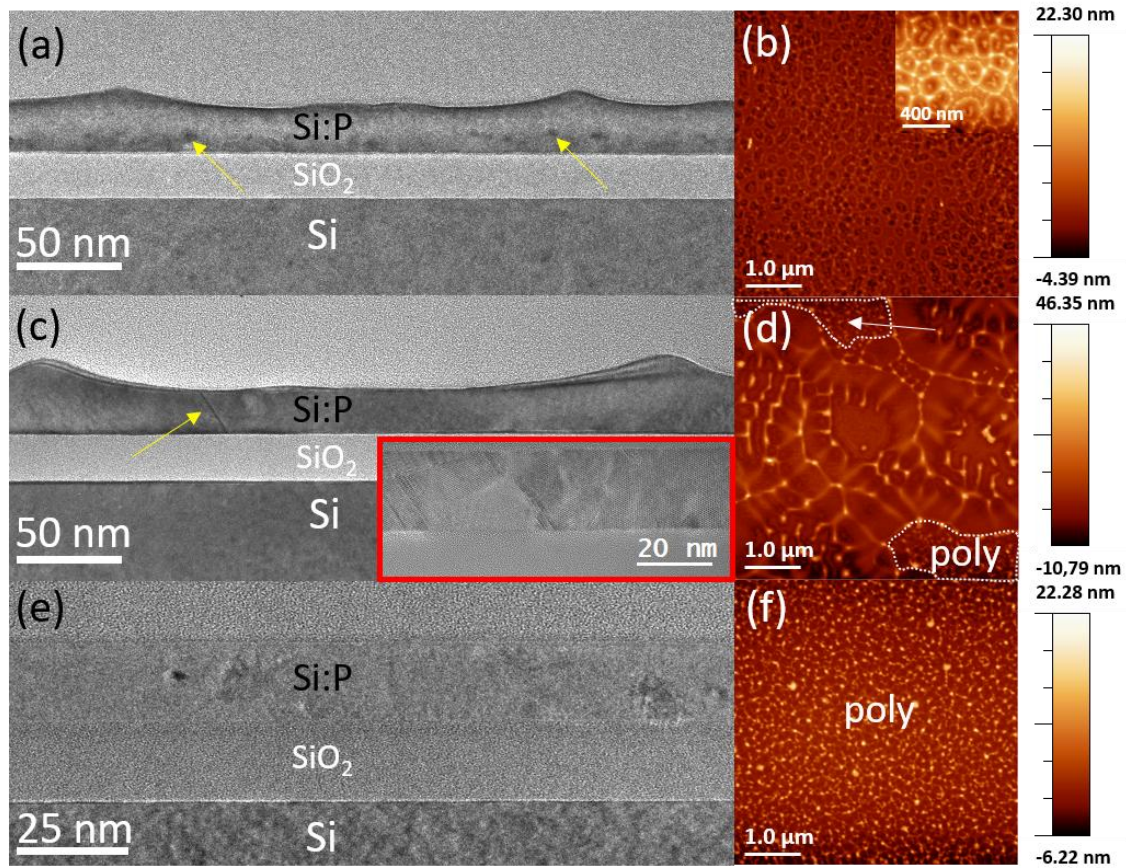


FIG. 4. Cross-section HREM images and corresponding AFM surface scans from sample Q1 showing the structural morphology of the SOI layer annealed at different energy densities. (a)-(b): 1.6 J/cm^2 (just below full melt). The yellow arrows in the TEM image indicate isolated loops formed in the solid region below the original a/c interface. The magnified view of the AFM scan (inset) shows the random hillocks distribution on the surface. (c)-(d): 1.7 J/cm^2 (full melt). Stacking faults (yellow arrow) and polycrystalline domains (inset) are observed by TEM in this sample. Poly-Si domains are also visible by AFM (cf. regions delimited by dashed white lines in the AFM scan). (e)-(f): 1.8 J/cm^2 (full melt). A polycrystalline layer is formed (with no surface hillocks).

A similar kind of surface roughness with submicronic hillocks has been observed at the onset of the full melt regime for amorphized SOI layers [37]. The formation of hillocks was attributed to the discontinuous nature of the buried crystalline seeds remaining after the melt,

which are organized in a dense array of isolated c-Si islands with the same crystalline orientation. When recrystallization occurs, these c-Si islands laterally grow leading to a network of Si hillocks at the surface.

The process of hillock generation at the surface, originating from isolated solid crystalline seeds when the melt front reaches the interface with the BOx, deserves to be deepened. A possible scenario is proposed in Fig. 3b, c and d. The isolated crystalline seeds trigger the recrystallization of the above liquid formed during the second melt (Fig. 3b). When the upper part of the solidification front of two neighbor c-Si seeds reaches the surface, a liquid droplet (L-Si) is left at the surface (Fig. 3c). Surface hillocks are finally formed by the solidification of these residual L-Si droplets (Fig. 3d). As demonstrated in [31], the characteristic surface contour of the re-solidified regions at the surface is driven by faceted solid-liquid interfaces and by the difference in molar volume between solid and liquid Si [38]. This surface contour presents a hillock shape, formed in the center of the transiently molten region surface, whose edge lies below the original surface level. The formation of the hillocks at the surface is therefore conditioned by the presence of remaining isolated c-seeds after the partial melting of the buried crystalline layer up to the BOx. The persistence of such seeds is highly probable at the beginning of the full melt, when the rough melt front only partially covers the BOx interface. This process should be quenched at full melt, when the melt front is flat and totally overlaps the Si/BOx interface, in good agreement with our observations.

To understand the effect of the doping of the surface hillock formation, it should be considered that the thickness of the buried crystalline region decreases from 8.5 nm for the lowest dose to 5 nm to the highest one. This is due to the increase of the amorphization depth with the implant dose (see Table I). As a consequence, the size of the remaining c-seeds after melt decreases when increasing the dose. This simple consideration could explain the absence of hillocks for the highest doses, as experimentally observed. Another possible explanation could be a reduction of the melt front roughness associated to the decrease of the melt threshold energy with P doping as previously discussed. However, the observation of hillocks for sample Q1 at relatively low energies with respect to the full melt regime (1.6 J/cm^2 , Fig. 4a) remains unclear at this stage. In particular, a higher roughness of the a/c interface can be ruled out, as it has the same value ($\sim 2 \text{ nm}$) for all the implant doses.

Finally, whatever the implanted dose, the morphologies obtained at the onset and after full melt (polycrystalline or amorphous layer, with or without surface hillock) do not fulfil the

requirements in terms of crystalline quality for future applications. In the following sections, we will therefore focus only on monocrystalline and defect free layers obtained before the end of the second melt regime for studying the phosphorus redistribution during the LTA process.

B) Phosphorus redistribution after LTA

The phosphorus concentration profiles were measured before and after LTA on selected samples by ToF-SIMS. Figure 5a shows the as-implanted profiles for all the investigated implanted doses. All phosphorus profiles exhibit a small peak (artefact) within the first 2 nm below the surface. Independently of this artefact, the phosphorus concentration is highest close to the surface for all the implant doses and progressively decreases as a function of depth until the top Si/BOX interface is reached at a depth of about 23 nm. A sudden increase of the ToF-SIMS signal occurs due to the substrate material change (cf. dashed line in the figure). It is important to note that the SIMS quantification procedure (and hence the corresponding concentration values indicated in the vertical axis) only applies to the top Si layer.

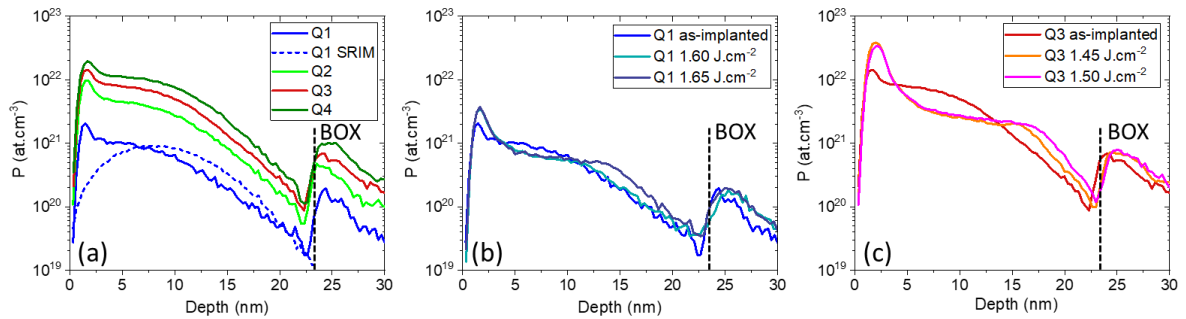


FIG. 5: (a) ToF-SIMS measurement of the P implanted profiles for the as-implanted samples; TOF-SIMS profiles for (b) Q1 (10^{15} cm^{-2}) and (c) Q3 ($7 \times 10^{15} \text{ cm}^{-2}$), at the two highest laser energy densities preceding the onset of the full melt (FM).

In all cases, the shape of the P concentration profiles slightly differs from the expected gaussian shape predicted by conventional SRIM simulations. This is probably due to the limits of such calculations that do not consider the matrix modification (P enrichment and sputtering effects) occurring during the implant. These matrix effects are particularly effective for such high dose/high current processes.

ToF-SIMS phosphorus profiles were measured after LTA for all samples annealed at the two highest laser energy densities preceding the onset of the full melt (FM). For sample Q1

implanted with $1 \times 10^{15} \text{ cm}^{-2}$ (Fig. 5b), these correspond to 1.60 and 1.65 J/cm², respectively. For sample Q3 implanted with $7 \times 10^{15} \text{ cm}^{-2}$ (Fig. 5c), the corresponding energies were 1.45 and 1.50 J/cm², respectively.

Independently of the implanted dose, all the annealed samples show a very similar behaviour. First, a strong P pile-up is observed in the first 5 nm below the surface. This surface peak is followed by a quasi-plateau region with concentrations above $5 \times 10^{20} \text{ cm}^{-3}$ for sample Q1 (Fig. 5b). The plateau value increases above $2 \times 10^{21} \text{ cm}^{-3}$ for sample Q3 (Fig. 5c) and is as high as $3 \times 10^{21} \text{ cm}^{-3}$ or more for sample Q4 (see Fig. S4, supplementary material). These values largely exceed the solid solubility limit of phosphorus in bulk Si at thermal equilibrium (lower than 10^{21} cm^{-3} at the Si melting temperature) [39]. The plateau region extends to a depth corresponding to the melt depth reached during the second melt (cf. section III.A.1). This depth increases, as expected, when increasing the laser energy density.

The P concentration profiles measured after LTA are in agreement with those typically observed for impurities with a low partition coefficient [40]. First, during melt, the implanted dopants rapidly redistribute within the liquid layer, leading to an almost perfectly flat depth profile. This redistribution is due to much higher dopant diffusivity in liquid Si compared to solid Si. Then, due to the low partition coefficient of phosphorus (~ 0.35 [41,42]), the phosphorus atoms are pushed into the remaining liquid layer during solidification, i.e. towards the surface. Hence, when the total solidification is accomplished, a substantial fraction of the total amount of impurities is accumulated at the surface (zone refining effect).

Finally, even if a strong diffusion of P toward the surface is observed after LTA, it is worth to notice that the integrated dose measured on the TOF-SIMS profiles after LTA is similar to the one of the as-implanted profiles. We can conclude that out-diffusion effects are negligible.

C) Phosphorus activation in thin and highly doped SOI layers after LTA

After LTA, sheet resistance measurements have been performed on all annealed samples, for laser energy densities ranging from 1.00 to 1.80 J.cm⁻². These resistivity measurements are shown in Fig. 6 for the four implanted doses. Similarly to the crystal structure, the sheet resistance also evolves through different stages related to the melt/recrystallization regimes determined by the laser energy density.

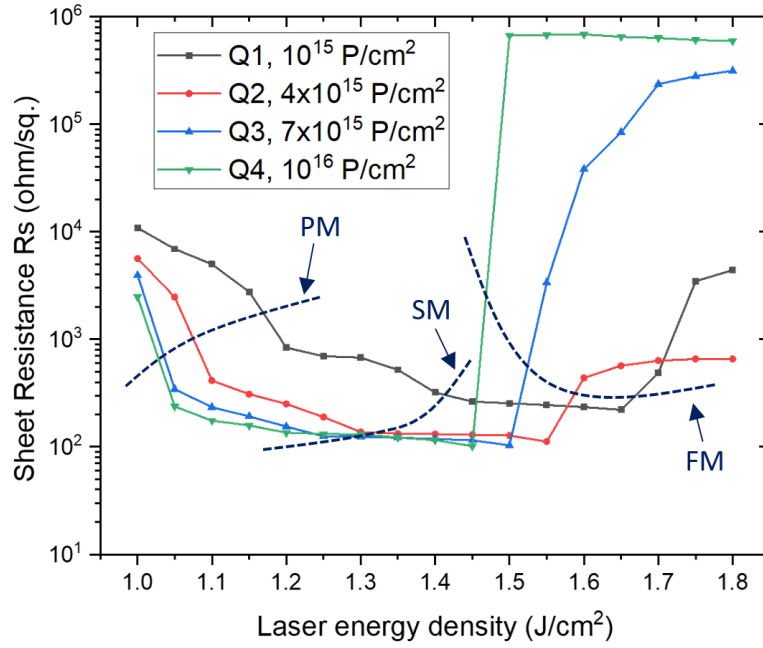


FIG. 6. Sheet resistance as a function of the laser annealing energy densities from 1.00 to 1.80 J.cm⁻² for the different implanted doses. The primary melt, second melt and full melt are indicated by respectively PM, SM and FM.

These different stages are qualitatively delimited by the dotted blue lines in Fig. 6. The first sudden reduction of the sheet resistance is observed in the low energy density region. This first region corresponds to the onset of the primary melt (cf. “PM” dotted line in Fig. 6) and the formation of the first polysilicon regions through explosive crystallization. This is followed by a slower decrease of the sheet resistance as a function of the laser energy density. This effect is presumably related to the progressive disappearance of polycrystalline regions and their replacement by defective c-Si through Liquid Phase Epitaxy. This is shown for example in Fig. 2a for sample Q4 at an energy of 1.2 J/cm². This process is completed when the secondary melt is triggered (cf. “SM” dotted line in Fig. 6). In the following stage, the sheet resistance is characterized by a quasi-plateau behaviour during which the monocrystalline nature of the material mainly determines the sheet resistance value. During this stage, there is almost no impact from the low density of residual defects (twins, stacking faults or isolated loops). Finally, when the full melt is reached (cf. “FM” dotted line in Fig. 6), a sharp increase in the sheet resistance occurs. This increase is much more pronounced for the heavily doped samples (Q3 and Q4, respectively blue and green curves in Fig. 6). This observation is in good agreement with their amorphous nature (Fig. 2k for sample Q4), compared to the samples implanted at lower doses, which instead exhibit a polycrystalline nature (cf Fig. S1, supplementary material).

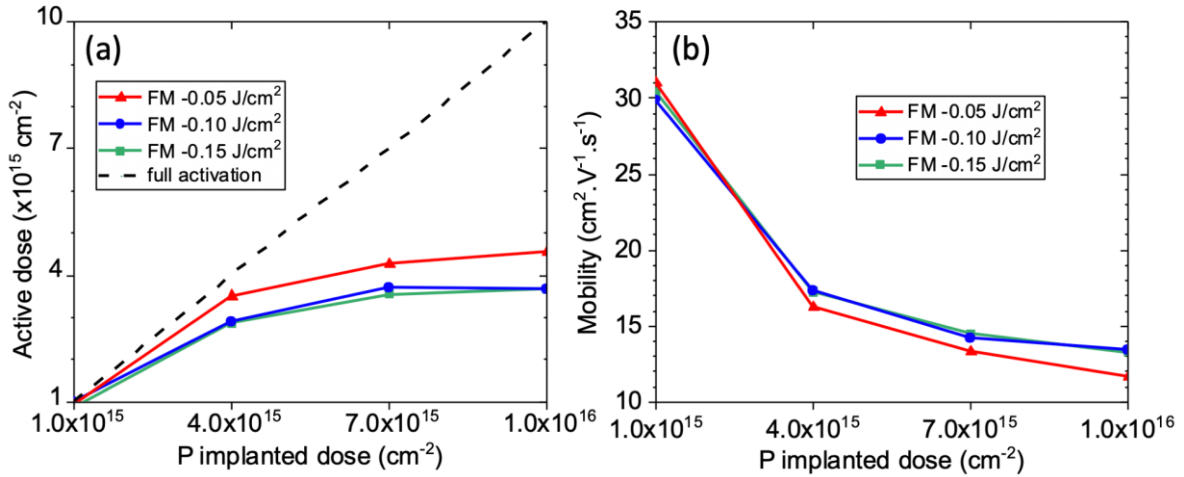
For all melt/recrystallization regimes prior to the full melt onset, the impact of the implanted dose is clearly visible from these results. Indeed, the sheet resistance decreases when increasing the implanted dose, suggesting an increase of the active dopant density. This effect is stronger when the dose is initially increased from $1 \times 10^{15} \text{ cm}^{-2}$ (Q1) to $4 \times 10^{15} \text{ cm}^{-2}$ (Q2). Then, it seems to saturate when the highest doses are reached (up to $7 \times 10^{15} \text{ cm}^{-2}$ in samples Q3 and $1 \times 10^{16} \text{ cm}^{-2}$ in sample Q4). This observation suggests that in the latter samples, a considerable fraction of the implanted dopants might no longer be accommodated in substitutional position.

In order to further investigate the dopant activation process, Hall effect measurements have been performed for all samples annealed at the three highest laser energy densities preceding the onset of the full melt (FM). The corresponding laser energy densities are respectively 0.15, 0.10 and 0.05 J.cm^{-2} lower than the FM energy threshold, whose actual value depends on the phosphorus implanted dose. In the following, these laser anneal conditions will be referred to as “FM – 0.15”, “FM – 0.10” and “FM – 0.05”, respectively. The corresponding laser energy density values are summarised in the coloured cells of Table II.

TABLE II. Description of the 12 samples used for Hall effect analysis. For each implanted dose (samples Q1-Q4), samples annealed at the three highest laser energy densities preceding the onset of the full melt (FM) have been analysed. The corresponding laser energy densities (in J/cm^2 units) are reported in the colored cells.

	FM - 0.15	FM - 0.10	FM - 0.05
Q ₁ (10^{15})	1.55	1.60	1.65
Q ₂ (4×10^{15})	1.45	1.50	1.55
Q ₃ (7×10^{15})	1.40	1.45	1.50
Q ₄ (10^{16})	1.35	1.40	1.45

It is important to note that, independently of the phosphorus implanted dose, these samples exhibit the best structural and electrical properties among all the investigated ones (i.e. defect-free c-Si with minimum sheet resistance values).



454

455 *FIG. 7. (a) Hall dose and (b) mobility as a function of the phosphorus implanted dose (Q1-*
 456 *Q4). For each sample, the three selected laser energy densities correspond to the highest*
 457 *energies preceding the onset of the full melt regime.*

458 Fig. 7 reports the measured Hall dose (Fig. 7a) and Hall mobilities (Fig. 7b), both corrected
 459 by the Hall scattering factor, r_H (cf. section II). For all annealing conditions, it is found that
 460 the Hall dose steadily increases when increasing the implanted dose. The Hall dose matches
 461 with the implanted dose (within the experimental errors) for sample Q1 implanted at 1×10^{15}
 462 cm^{-2} , suggesting a full dopant activation. However, for the samples implanted with the highest
 463 doses (Q3 and Q4) the measured active dose progressively saturates at values well below the
 464 total implanted dose (indicated by the dotted line in Fig. 7a). This observation confirms that a
 465 progressively larger fraction of the implanted dopants does not occupy substitutional positions
 466 after LTA in such samples. This behaviour is summarized in Fig. 8a, showing the dopant
 467 activation rate estimated in samples for which a ToF-SIMS measurement of the chemical
 468 profile was performed.

469 In parallel, and independently of the annealing conditions, the Hall mobility is found to
 470 decrease as a function of the implanted dose (cf. Fig. 7b). Being the carrier mobility inversely
 471 proportional to the carrier concentration, the observed mobility decrease might be simply due
 472 to the increase of the maximum active dopant concentration, C_{el} , when increasing the implant
 473 dose. However, a second mechanism leading to a decrease of carrier mobility might be related
 474 to the increased fraction of inactive dopants in samples implanted at high doses (cf. Fig. 8a).
 475 In such case, high concentrations of dopant clusters are expected to form, which may act as
 476 additional scattering centers and therefore strongly reduce the carrier mobility [43]. In any
 477 case, the mobility evolution clearly indicates that the observed decrease of the sheet resistance

as a function of the implanted dose (cf. Fig. 6) is solely due to the corresponding increase of the active dopant dose.

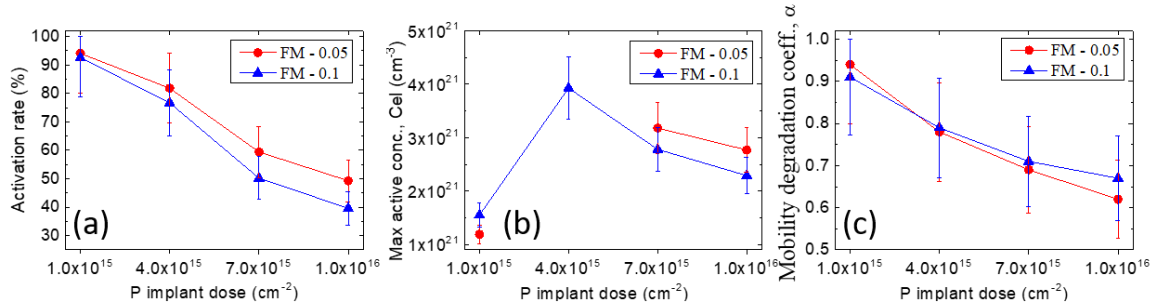


FIG. 8. (a) Activation rate, (b) maximum active concentration and (c) mobility degradation as a function of the implanted dose.

In order to clarify the origin of the observed mobility decrease and further analyse the evolution of dopant activation as a function of the annealing conditions, raw experimental Hall effect data were analysed using the method described in Ref. [43]. In practice, starting from the chemical phosphorus depth profiles measured by ToF-SIMS, the maximum active concentration, C_{el} , is first determined by fitting the measured Hall dose. The mobility degradation coefficient, α , is then adjusted in order to perfectly fit both the measured values of the Hall mobility and sheet resistance. The extracted C_{el} and α values are reported in Fig. 8b and 8c, respectively. For sample Q1, the results confirm that, within the experimental errors, the totality of the implanted P atoms is electrically active both at annealing conditions “FM – 0.10” and “FM – 0.05”. In the meantime, the measured carrier mobilities are consistent with the theoretical expected values (i.e. the mobility degradation coefficient, α , is close to unity). In contrast, for samples implanted at higher doses (Q2-Q4), the maximum active concentration C_{el} is found to decrease when increasing the implanted dose for both “FM – 0.10” and “FM – 0.05” annealing conditions (Fig. 8b). In addition, the mobility degradation coefficient also decreases (Fig. 8c) proportionally to the increase of the inactive fraction of implanted dopants in these samples. The observed decrease of the carrier mobility as a function of the implant dose (Fig. 7b) is therefore due to the formation of additional scattering centers related to inactive dopants, rather than to an increase of the maximum active concentration. In other words, when increasing the implanted dose, only the phosphorus atoms that penetrate deeper in the structure (cf SIMS profiles in Fig. 5b) are responsible for the observed increase of the active dose. In contrast, the progressive increase of the phosphorus concentration in the surface accumulation peak leads to an increased phosphorus

precipitation rate. This process has a double consequence: (i) the decrease of the maximum active dopant concentration, C_{el} , and (ii) the increased degradation of the carrier mobility.

Finally, we consider the effect of approaching the Full Melt threshold (by increasing the laser energy density) on dopant activation. For sample Q1, whose activation rate is always close to 100 %, the increase of the laser energy has little impact on both measured (Hall dose and Hall mobility, Fig 7) and extracted parameters (maximum active concentration and mobility degradation, Fig. 8). Again, a different behaviour is observed for samples implanted at higher doses (Q2-Q4). Considering for example sample Q3, implanted at a dose of $7 \times 10^{15} \text{ cm}^{-2}$, the Hall dose initially exhibits a relatively constant value ($\sim 3.6 \times 10^{15} \text{ cm}^{-2}$) for annealing conditions “FM – 0.15” and “FM – 0.10” (cf. green square and blue triangle in Fig. 7a). A similar behaviour is observed for samples Q3 and Q4, implanted with the highest phosphorus doses. Then, the Hall dose increases by about 20% to $\sim 4.3 \times 10^{15} \text{ cm}^{-2}$ for the highest laser energy density prior to full melt onset (“FM – 0.05”, red circle in Fig. 7a). This increase is accompanied by a comparatively smaller decrease (by only $\sim 9 \%$) of the Hall mobility from ~ 14 to $\sim 13 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$. This is in good agreement with the observed decrease of the sheet resistance (cf. blue triangles in Fig. 6). In addition, the C_{el} value also increases when the laser energy density increases (Fig. 8b). This observation suggests that the higher thermal budget associated to the increased laser energy density (both in terms of peak temperature and melt duration) favours the dissolution of phosphorus-related inactive clusters contained in the surface accumulation peak.

Finally, for such optimized LTA conditions, the obtained active dopant concentrations are 2 to 4 times higher than the solid solubility of P in bulk Si at thermal equilibrium [39] at the Si melting temperature. These values nearly reach the highest reported active P concentration in bulk Si obtained after pulsed laser annealing, with comparable mobilities [2].

Moreover, in a recent work [44], these optimized highly doped and thin layers have been successfully used as substrates for top-down processing of all-silicon-based plasmonic metasurfaces showing tuneable localised surface plasmon resonances in the IR range. Thirty years after the discovery of light emission from nano-silicon, this renewed the interest of silicon for optical applications as IR plasmonics

IV. CONCLUSION

Hyper-doping of SOI substrates thin over-layers has been realized by coupling low energy ion implantation of P and nanosecond UV-LTA. By varying the laser energy density, the

successive melting and recrystallization regimes involving explosive crystallization and classical liquid phase epitaxy have been identified. Our results highlight the complexity of such a thin stratified system for which a slight shift of the spatial position of the melt front or a small variation of the doping level are sufficient to strongly affect the regrowth processes and structural quality of the final microstructures. Optimized laser annealing conditions were identified, corresponding to the laser energies just preceding the onset of the full melt. Such optimized layers exhibit perfect crystallinity, negligible P out-diffusion, an almost perfectly flat P depth profile located below the segregation-induced surface pile-up peak and dopant active concentrations well above $1 \times 10^{21} \text{ cm}^{-3}$, close to the highest reported values reported for phosphorus in bulk Si substrates.

SUPPLEMENTARY MATERIAL

See the supplementary material for the table resuming the energy thresholds at the different melt regimes for increased implantation doses (Table SI). The microstructure of Q1 and Q4 at full melt and associated TRR are presented in Fig. S1 and S2 respectively. TEM image of hillocks in sample Q2 at the beginning of the full melt regime is shown in Fig. S3. ToF-SIMS phosphorus profiles after LTA for Q2 and Q4 doses preceding the onset of the full melt are reported in Fig. S4.

DATA AVAILABILITY

The data that support the findings of this study are available within the article and its supplementary material

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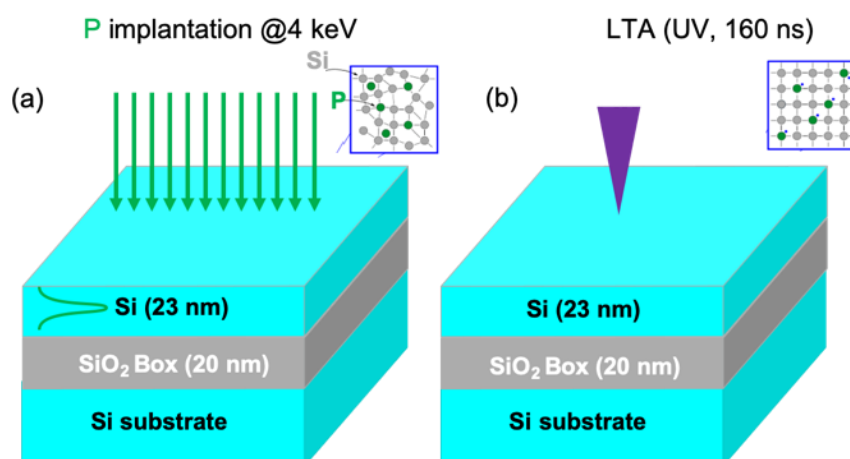
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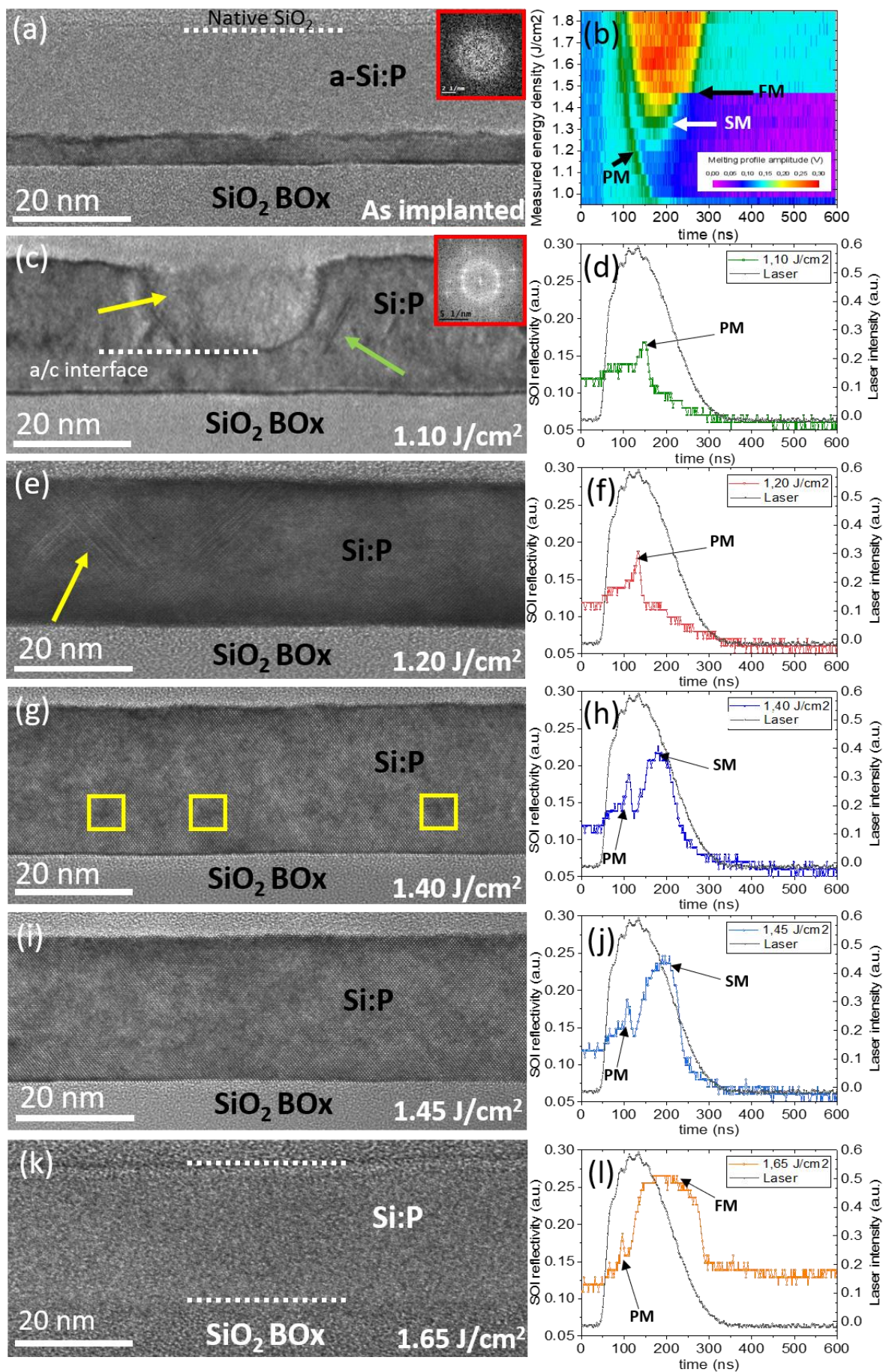
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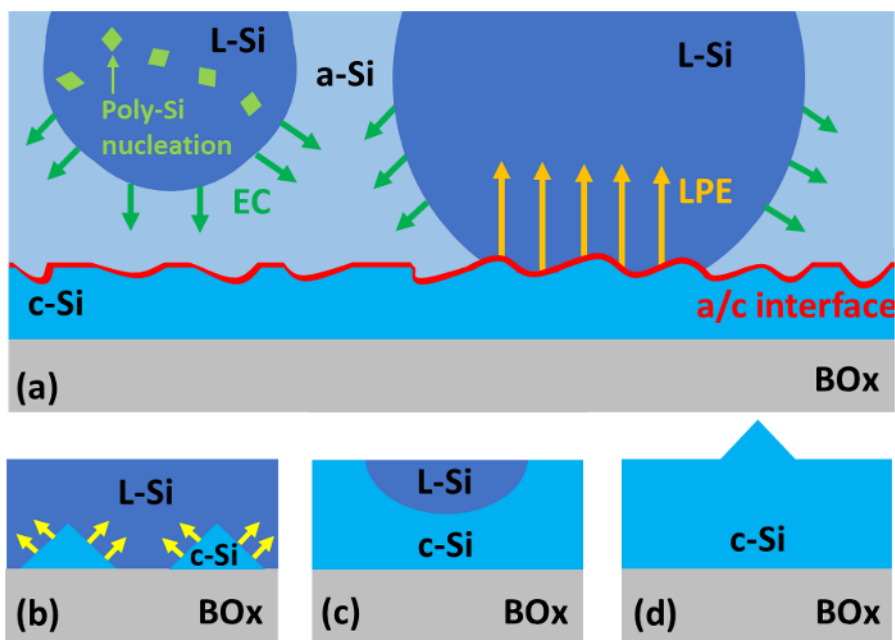
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691 Figure 2



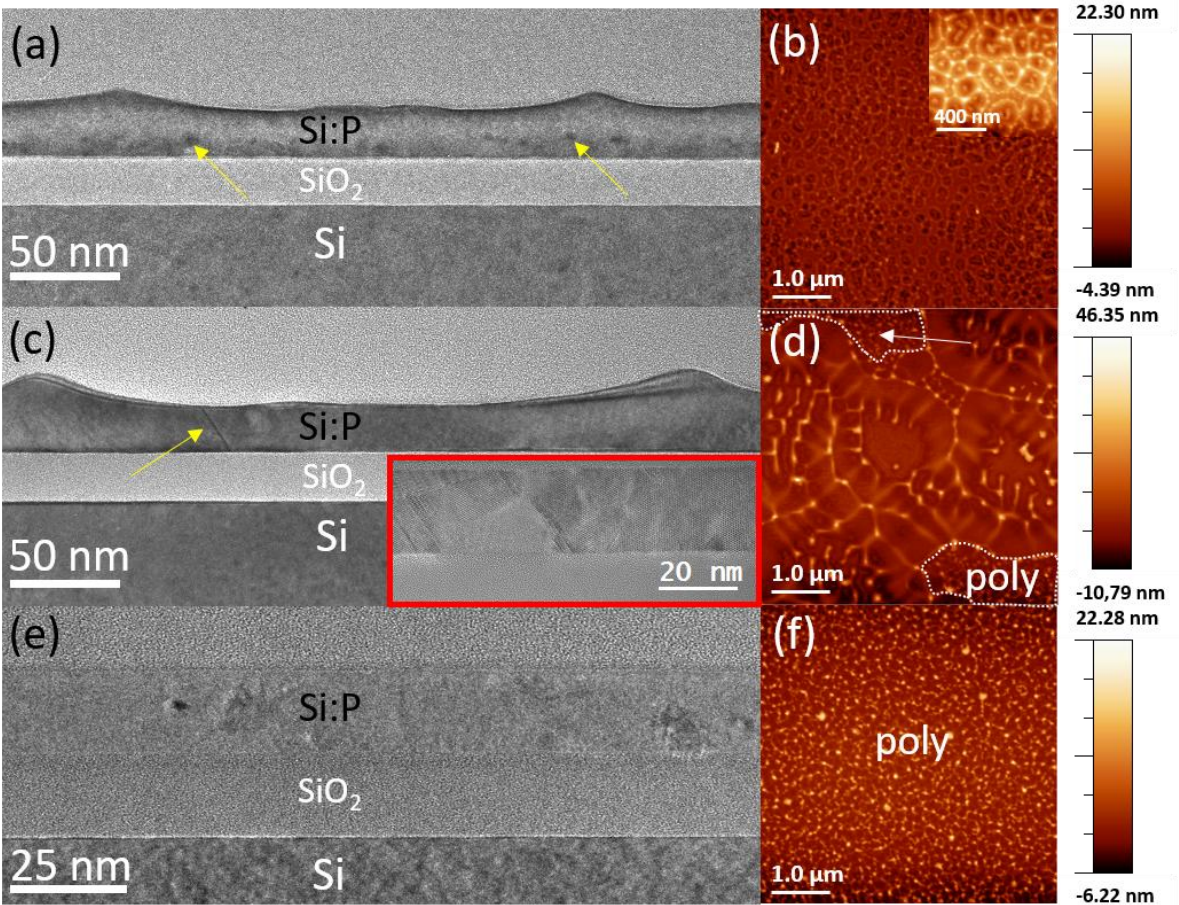
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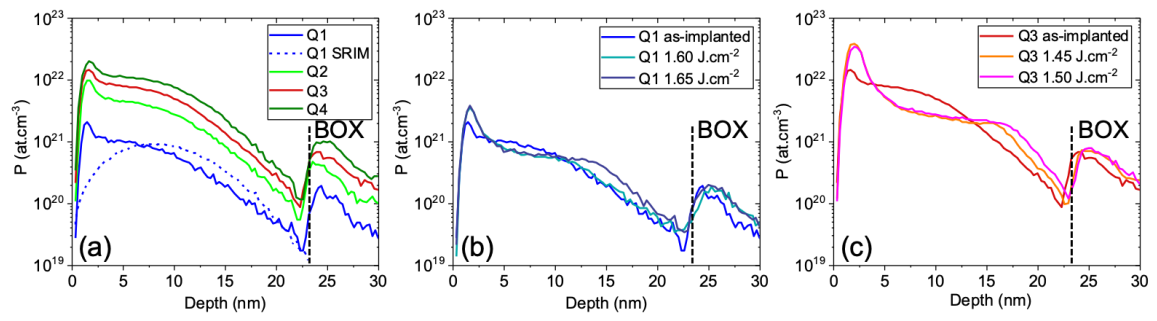
696 Figure 4



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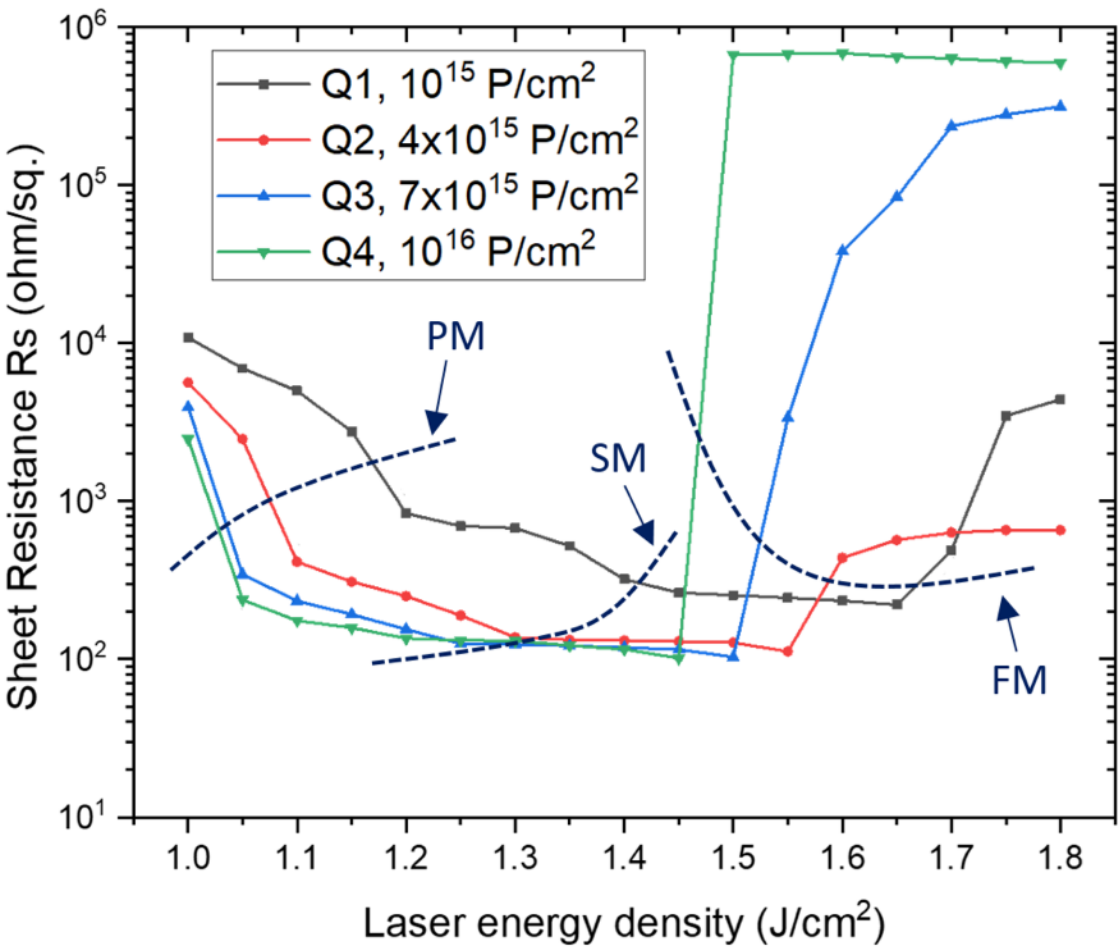
699 Figure 5



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702 Figure 6



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705 Figure 7

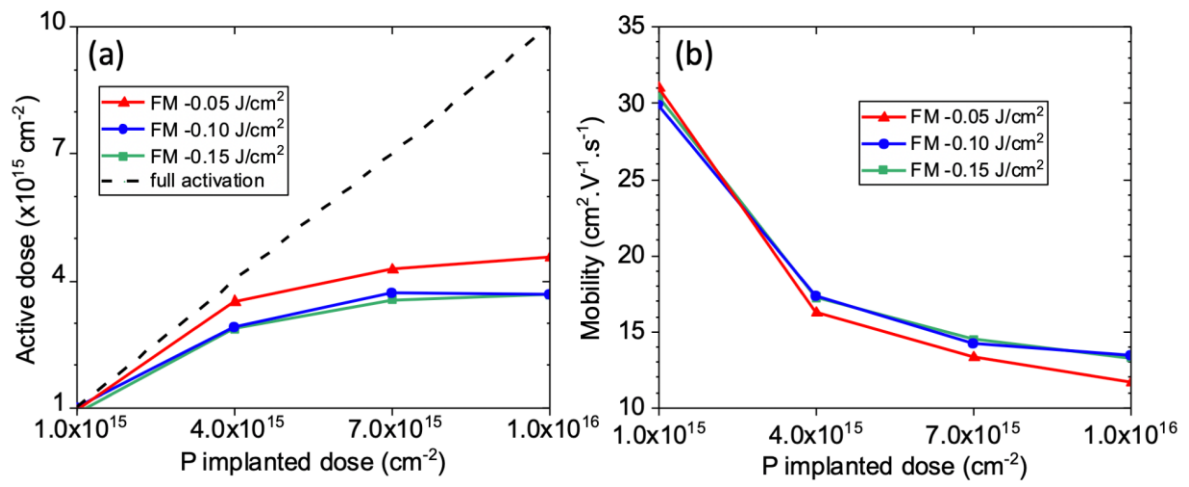


Figure 8

