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4H-SiC(0001) surface faceting during interaction with liquid Si

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Abstract. The aim of this study was to find conditions allowing the "natural" formation of a regular and controllable step bunched morphology on a 4H-SiC seed without the need of any SiC deposition. This was performed by melting a bulk piece of Si on a 4°off 4H seed in the temperature range of 1500 - 1600°C, for 15 min. After etching the remaining Si, the 4H surface was found to be successfully highly step bunched with steps very parallel and regular. A mechanism of dissolution-precipitation was proposed, which could occur both on a short (step to step) and long (centre to periphery) range. This process is kinetically limited at low temperature (1500-1550°C) and considered to be close to the equilibrium at 1600°C.

Introduction

The formation of liquid Si droplets during 4H-SiC epitaxial growth is a common feature when using Si-rich chemical vapor deposition (CVD) conditions. Such growth conditions are usually to be avoided because the growth kinetics under the droplet is altered, resulting in a highly step bunched surface at the places where the Si droplets formed. On the other hand, it was shown recently that a step bunched surface can lead to threading dislocation conversion during epitaxial growth either by CVD [1] or liquid phase epitaxy (LPE) [2]. In ref [1] the step bunched morphology was artificially created by photolithography patterning while in ref [2] it is simply the consequence of growth from a liquid phase. The formation and control of a highly step bunched morphology is the study of recent works for the case of LPE [2-4] while this is almost a blank topic for CVD.

The aim of this preliminary study is to find conditions allowing the "natural" formation of a regular and controllable step bunched morphology on a 4H-SiC seed without the need of any SiC deposition.

Experimental

Considering the occurrence of a step bunched morphology after CVD growth below Si droplets, we investigated the intentional interaction between liquid Si and a SiC seed on a larger scale than what can be made using micrometer size droplets. Towards this end, a bulk piece of n type (phosphorus doped) Si substrate of 5x5 mm was molten on top of a n type ($5x10^{15}$ cm^{-3}) epilayer deposited on Si face 4H-SiC(0001), 4°off seed of ~1 cm^2. This created a circular liquid Si with an average diameter of 5-6 mm (see fig. 1). The experiments were performed in a homemade vertical cold wall CVD reactor working at atmospheric pressure. The interactions were done at 1500, 1550 and 1600°C for 15 min under high purity H_2. After cooling, the solidified Si was acid etched and the

surface morphology was then studied using optical and scanning electron microscopies. Mechanical profilometry was used to evaluate the macroscopic shape of the surface below the Si drop.

Results and discussion

The obtained morphologies after 15 min treatment are shown in Fig. 1. One can see that, for all these experiments, a step bunched morphology was successfully obtained, which evidences the effective interfacial reconstruction happening when SiC is put in contact with liquid Si. Furthermore, on all the samples, the steps are found parallel, very straight (with only slight long range undulation at 1600°C) and with very few disturbances. Obviously, the main difference between these samples is the step width: at 1500°C there is a high dispersion of step width while for higher temperatures the width is more regular. From a first glance, one can notice a general trend, i.e. a decrease of the mean step width with increasing temperature. In order to confirm this point, both SEM and optical images were quantitatively analysed for step width evaluation. The statistics obtained on a large number of steps (between 200 to 350) are shown in Fig. 2.

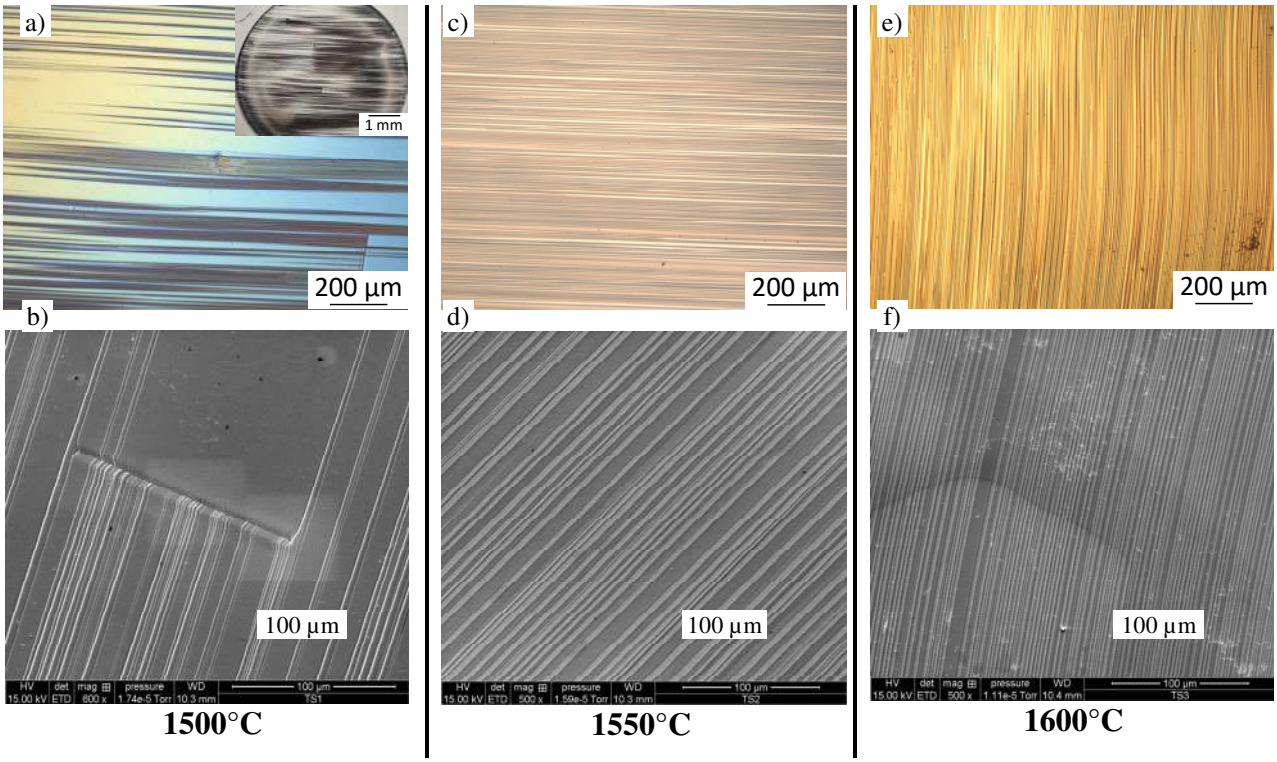

Figure 1. *Optical (up) and SEM (down) images of the 4H-SiC seeds surface morphology after liquid Si interaction at different temperatures: a), b) 1500°C, c), d) 1550°C and e), f) 1600°C. The insert a) is a low magnification observation showing the average shape and size of the original Si droplet.*

For 1500°C, despite the step width distribution being centred around 2-3 µm, steps width up to 37 µm could be found in the studied area (bigger ones could even be found in specific places, see Fig. 1a and 1b). It is important to mention that these very large steps observed for 1500°C are most probably not due to liquid Si induced step bunching. Indeed, when looking to Fig. 1b, if the large flat area on the upper part of the image was corresponding to a step bunching, then the associated step height at one extremity would be ≥ 7 µm. Such high step height would have been easily noticed both by SEM or even optical microscopy. We believe that these large flat areas are in fact unreacted zones, i.e. areas where mechanism leading to step bunching formation at the liquid Si/SiC has not occurred yet. Under this assumption and considering that the most kinetically advanced (or closer to equilibrium) sample is the one at 1600°C, one can propose that the evolution of step width distribution with increasing temperature reflects the progress of the interfacial reaction: at 1500°C some large areas are left unreacted; at 1550°C the large areas have reacted lately which gives steps

of intermediate width; while at 1600°C the reaction kinetics is so fast that all the intermediate width steps were converted into smaller ones.

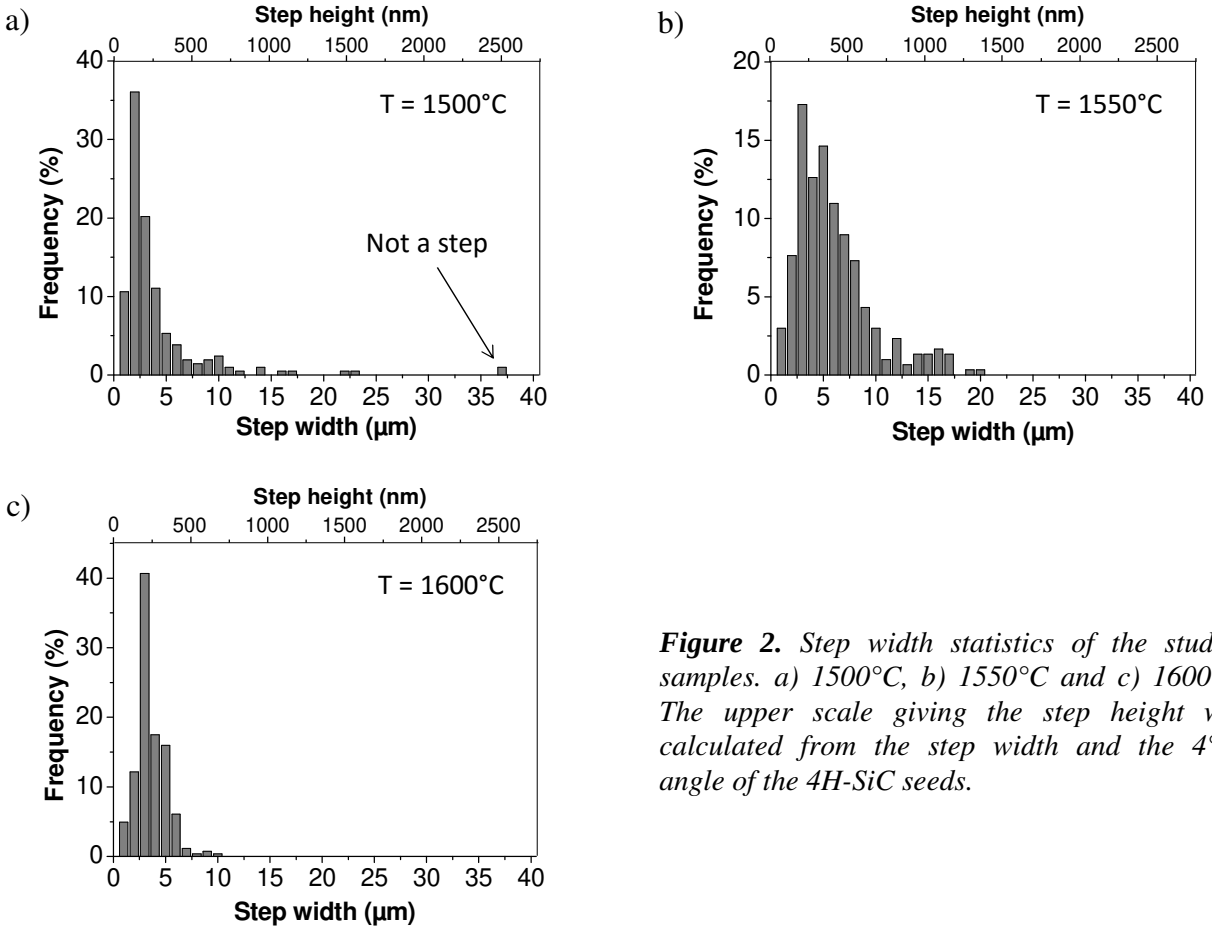

Figure 2. *Step width statistics of the studied samples. a) 1500°C, b) 1550°C and c) 1600°C. The upper scale giving the step height was calculated from the step width and the 4°off angle of the 4H-SiC seeds.*

Another interesting feature to be noticed on sample treated at 1500°C is the remarkable separation by a defect line between a step bunched area and an unreacted one (Fig. 1b). This defect line seems to act either as a blocking or a generating agent. According to the higher density of steps reaching from this defect line compared to the surrounding, we believe that the line is generating the step bunching. Also, from the same sample, many steps seem to be originating from the Si drop edges.

In order to have complementary information on the overall mechanism occurring below the drop, mechanical surface profiling was performed on these samples. A typical result is given in Fig. 3. One can see that, compared to the mean altitude of the seed outside the drop, the surface at the periphery of the drop is several µm above it while it is ~1 µm below it at the centre of the drop. It suggests that some matter redistribution is occurring at long distance from the centre to the edges of the drop. Note also that at the centre the seed surface presents long range undulations.

Though it is most probable that the driving force leading to such reconstruction is the minimization of the interfacial energy, interpretation of our results in terms of interfacial energy is rather difficult. Considering all the observations made so far and the fact that the only reaction that may occur between liquid Si and SiC is SiC dissolution in the liquid, we can propose two distinct mechanisms based on the same process of dissolution-precipitation.

- The first mechanism would be a local one between neighbouring steps (transfer of atoms from one step to another via the liquid) and would result in the faceting of the steps. This mechanism could be activated by two factors: 1) kinetically activated by temperature and/or 2) catalysed by a surface imperfection. The latter factor is obviously predominant at low

temperature (1500°C). Note that the drop edge could be also seen as a local imperfection (solid/liquid/vapour triple line) since it was found to be also a source of step generation.

- The second mechanism would be a long range dissolution-precipitation process from the centre to the edge of the drop, with matter transport through the liquid.

Both mechanisms would happen simultaneously. The effect of treatment duration at 1500 and 1550°C is currently under investigation in order to have a clearer insight on the dissolution mechanism.

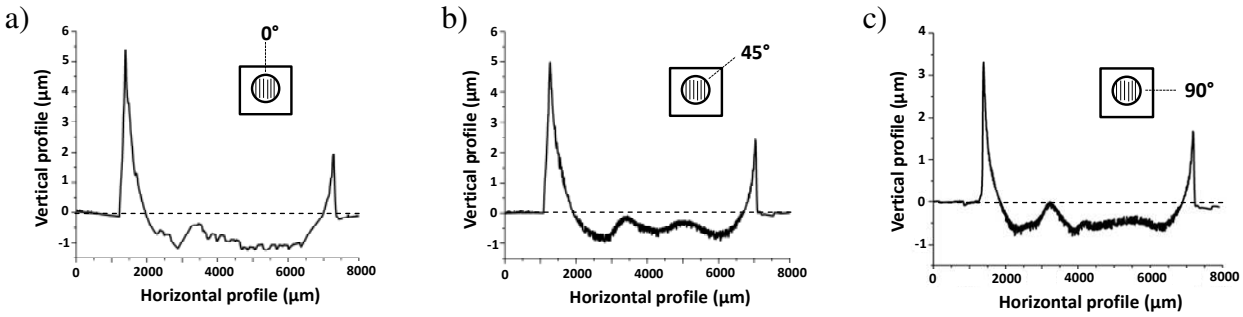

Figure 3*: Mechanical profiles of the surface of 1550°C treated sample a) parallel to the steps (0°), b) 45° rotated and c) perpendicular to the steps (90°). These profiles were found very similar to the ones obtained after treatment at different temperatures. The dashed line corresponds to the surface mean altitude outside the droplet.*

Finally, it is worth considering that Si evaporation from the drop was found be too small to have any significant influence on the mechanism discussed. Indeed, though some Si evaporation was effectively detected by the formation of a network of µm size droplets surrounding the big one, this did not drastically change the main Si droplet size, even at the highest temperature of 1600°C. Some slight receding (~50-100 µm) of the drop was detected from the step bunched print left by the droplet at the periphery, but this is clearly small compared to the drop diameter of 5-6 mm.

Summary

A parallel and highly step bunched surface was successfully obtained on a large scale by putting in contact a big Si drop to the 4H-SiC(0001) 4°off seed. The proposed mechanism of reaction involves a dissolution/precipitation process acting both in short and long range.

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