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Gabriel Ferro, Didier Chaussende, Nikolaos Tsavdaris. Understanding Al incorporation into 4H-SiC during epitaxy. *Journal of Crystal Growth*, 2019, 507, pp.338-343. <10.1016/j.jcrysgr.2018.11.034>. <hal-02121592>

**HAL Id: hal-02121592**

**<https://hal.science/hal-02121592v1>**

Submitted on 22 Jan 2025

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# Understanding Al incorporation into 4H-SiC during epitaxy

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*KEYWORDS. Silicon Carbide, Aluminum doping, model, epitaxy, surface vacancy.*

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## ABSTRACT:

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In this paper are reviewed the main reported trends of Al incorporation as a function of the main 4H-SiC epitaxial growth parameters. Al incorporation was found to be limited by its high desorption rate for all the parameters investigated. Due to the lack of a general model describing reasonably the processes of Al incorporation, the concept of surface vacancy induced incorporation was proposed and applied to explain these experimental results. In this model, Al incorporation is mainly driven by the transient formation of Si vacancies at the surface or sub-surface of terraces. It was shown that for increasing Al incorporation, one needs to improve its stability upon adsorption on Si vacancy and/or to reduce its desorption time. In this model, the transient formation of C vacancies at the surface or sub-surface (favored at low C/Si ratio) is proposed to play also a role by destabilizing the incorporated Al atoms.

## INTRODUCTION

4H Silicon carbide (4H-SiC) based power devices are seen as the next revolution in power semiconductors to manage high power and high voltage in industrial and automotive applications, providing a significant gain in energy efficiency. The main devices on the market, for blocking voltage of few to several kV, are unipolar structures, like Schottky barrier diodes and metal-oxide-semiconductor field effect transistors (MOSFETs), in which the active part of the components are n type doped with nitrogen. For higher blocking voltages ( $>10$  kV), bipolar power devices, like pin diodes or insulated-gate bipolar transistors (IGBTs), are better suited [1]. On the material aspect, these bipolar devices require additionally to control the growth of p-type doped 4H-SiC epitaxial layers by injecting Al containing species in the chemical vapor deposition (CVD) chamber. Such doping is known to be more difficult than N one. Indeed, in addition to its memory effect from run to run, Al easily incorporates on Si face material so that controlling low p-type doping is not simple. On C face, high Al incorporation is difficult to achieve. Despite its technological interest and various studies on the subject, Al incorporation mechanism is still far to be well understood. As a matter of fact, it is difficult to extract from the literature a model satisfying each doping result. Ideally, this model should fit with both the site competition incorporation rule [2] and the "step-controlled epitaxy" process [3] (both will be detailed below). An example of a previously proposed model, in which step edges are the main sites for Al incorporation, can be found in ref [4]. But some researchers did not observe crystal off-orientation dependence [5] in addition to the fact that step edge incorporation is not always considered to explain the trends with growth parameters [5, 6, 7].

Recently, the present authors have proposed a new model for N incorporation into 4H-SiC during epitaxy. It involves N incorporation on terraces through the transient formation of C vacancies ( $V_c$ ) which are formed during statistical surface exchanges between the gas and the solid [8]. In this paper, we will transpose the same model to Al incorporation (via Si vacancy formation) and show how it can be adapted to explain the experimental trends observed so far.

### Parameters of the model

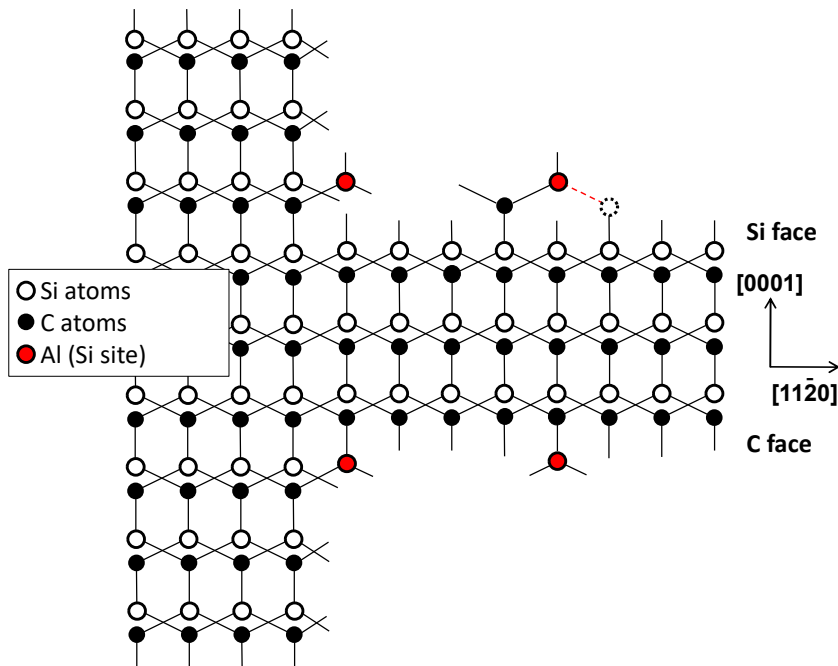
According to the "site competition" rule which was settled by Larkin et al [2], the covalent radius of impurities governs their incorporation site into SiC matrix. Small atoms (like N) incorporate on C atomic site while bigger ones (Al) do on Si atomic site. As a consequence, during epitaxial growth by CVD, tuning the C/Si ratio in the gas phase can favor incorporation of one or the other atomic site and thus limit or enhance the incorporation of specific dopants. For example, Al atoms are competing with Si ones for incorporation in SiC matrix so that Al incorporation can be significantly increased (reduced) by increasing (decreasing) the injected C/Si ratio.

Since Al incorporation is happening during 4H-SiC homoepitaxial growth, one needs to consider also the "step-controlled epitaxy" process. It implies the use of misoriented 4H-SiC(0001) or (000-1) seeds, of several degrees toward  $[11-20]$  or  $[1-100]$  direction, in order to favor step flow growth mechanism and thus replication of substrate polytype inside the growing layer. As a consequence, since the growth proceeds via step motion, it is logical to consider that Al incorporates at the same place as the impinging Si atoms, i.e. at the step edges. This was so obvious that the experimental studies on the effect of misorientation on Al incorporation are very few in the literature [4-5]. But the results are rather contradictory since one group [5] mentioned insensitivity to misorientation while the other group [4] showed Al moderate increase with increasing misorientation.

On figure 1 are summarized the different Al incorporation configurations proposed so far depending on polarity. It is assumed that Al atoms incorporate into the growing layer in an additive way, i.e. Al atoms

□

do not replace any atom of the seed but participate in the growth of the epitaxial layer. Since Al atoms occupy Si sites, their incorporation requires the formation of Al-C bonds on the surface. Thus, on Si face, an isolated Al atom can only attach to a step edge by forming only one Al-C bond with the matrix. In order to incorporate on a terrace, an Al atom needs first one or more C atoms beneath in order to sit on a Si site. But such incorporation on terrace is not consistent with the growth by step controlled epitaxy because it should favor occurrence of 3D nucleation and growth on terraces and thus lead to 3C-SiC inclusion formation. Considering now the C face, the situation is different since Al can directly attach on the terraces by forming only one Al-C bond. But at the step edge Al has a more stable incorporation site since it can create two bonds with the SiC surface.



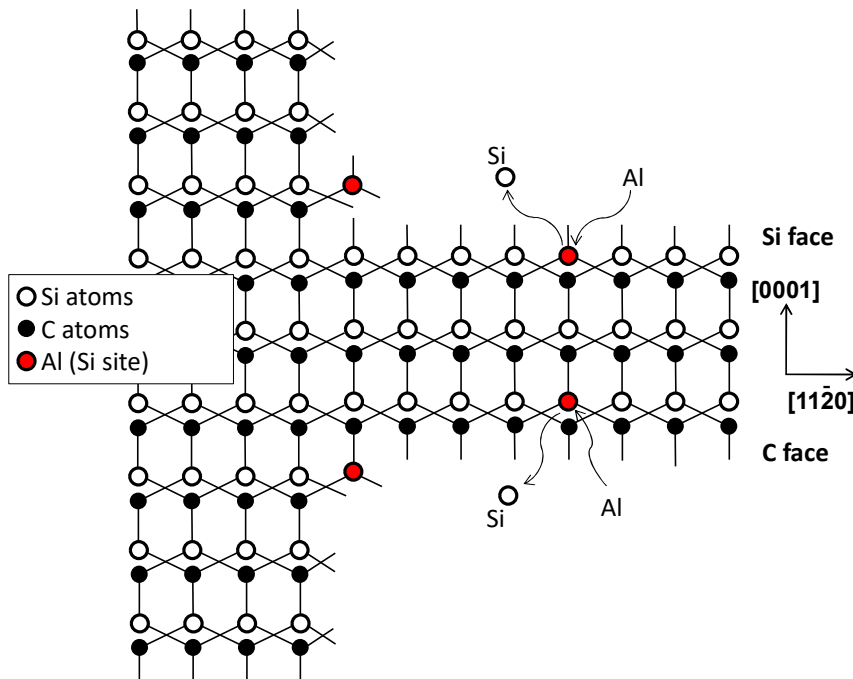
**Figure 1:** Schematic drawing showing the different possibilities of Al additive incorporation on both Si and C polar faces of a 4H-SiC crystal.

In conclusion of the previously proposed models, among the four cases considered above, the most stable and thus probable incorporation site for Al should be on C face and at step edge. But, as will be shown below, all experimental data demonstrate a higher Al incorporation on Si face than on C face. As a matter of fact, one needs to consider a more realistic Al incorporation mechanism which could better fit with the main experimental trends. That is why we will apply our model developed for N incorporation into SiC [8] to Al incorporation into 4H-SiC.

Briefly, this model of impurity incorporation is based on the dynamic vaporization/condensation reactions at the surface of the substrate during epilayer growth. It is known that when heating up SiC material under inert ambient (either Ar or vacuum), the surface loses Si atoms up to complete graphitization of the surface. This process is currently used for the growth of epitaxial graphene [9]. If H<sub>2</sub> ambient is used instead, then the surface loses also C atoms by forming volatile CH<sub>x</sub> species. This loss is usually faster than Si one so that treating SiC under H<sub>2</sub> is generally accompanied by Si droplets formation [10]. Note that the accumulation of Si at the surface does not mean that Si loss from the surface is suppressed: it is still happening but at a slower rate than C loss.

□

When a Si atom vaporizes from the surface, it leaves behind a Si vacancy ( $V_{Si}$ ) at the solid/gas interface. But since SiC is in contact with Si-containing vapor phase during epitaxy, then the Si loss can be partially or completely compensated by the gaseous feeding, leaving an apparently stabilized surface. These atoms exchanges between the solid and the gas phases are constantly happening and the apparent equilibrium is thus dynamic. As a matter of fact, introducing in the gas phase an impurity which can replace Si atoms inside SiC matrix, Al for instance, can lead to some Al incorporation by this constant surface exchange of atoms between the solid surface and the gas phase. This vacancy driven Al incorporation is the main idea of the proposed model which is illustrated in figure 2.



**Figure 2.** Schematic drawing showing the proposed Al incorporation by replacement of a Si atom at the terraces of a 4H-SiC crystal, for both Si and C polar faces. Possible incorporation at step edges is also shown.

On Si face, Al replaces a surfacial Si atom on a terrace and forms thus three Al-C bonds with the matrix. This is a rather stable configuration which may not need any C additional atom unlike in Figure 1 (we will see later that even in this configuration additional C atoms may help). On C face, the simpler way of forming a  $V_{Si}$  at the surface is beneath the top most C layer. It is not a directly accessible incorporation site but it is surely the most stable situation for Al incorporation since it provides 4 bonding between Al atoms and the matrix. We will discuss later about the plausibility of this option according to the experimental trends observed in the literature.

Since the growth is supposed to occur at the step edges (for standard 4H-SiC homoepitaxial growth), Al can also incorporate at the step edges. But because of this growth, we can consider that the net balance exchange with the gas phase is locally largely shifted toward atoms incorporation rather than atom loss. As a confirmation, the sticking coefficient at step edges was reported close to unity [11]. As a matter of fact, we will neglect  $V_{Si}$ -mediated Al incorporation at the step edges and consider only the standard incorporation process (atom addition) at these places.

Finally, before confronting the model to the published experimental results, it is worth reminding that the

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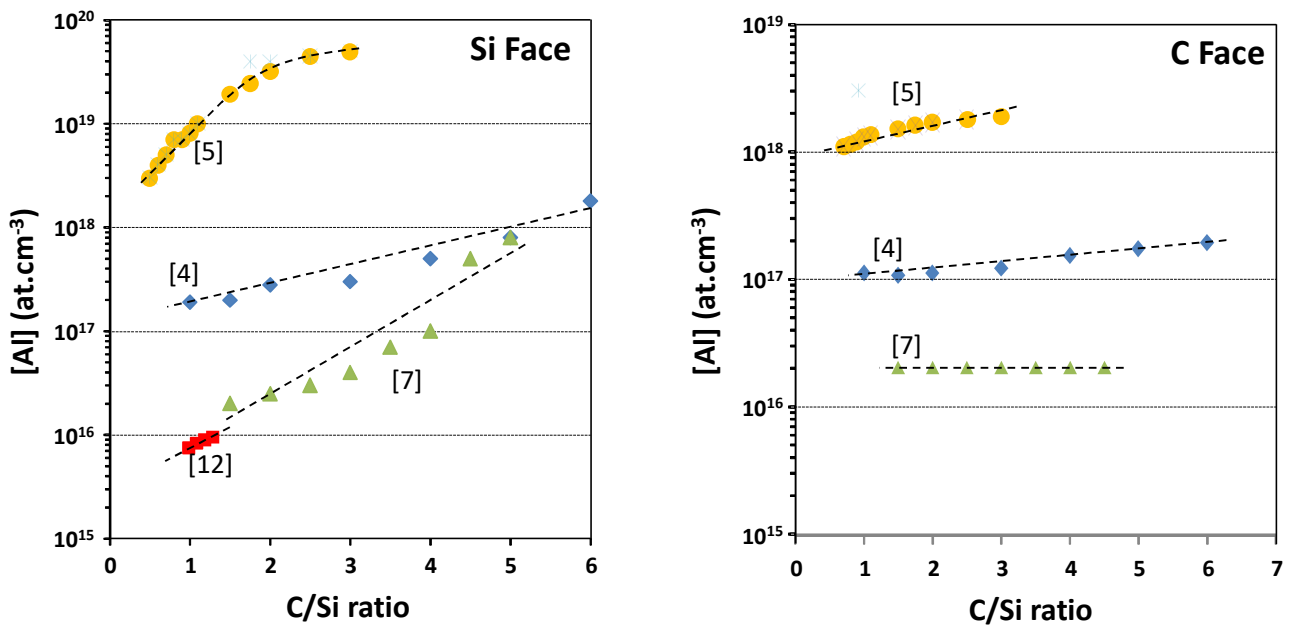
in situ p type doping with Al impurity is almost exclusively performed by adding trimethyl-Aluminum (TMA /  $\text{Al}(\text{CH}_3)_3$ ) precursor to the gas phase. We will consider here only the standard deposition chemistry  $\text{H}_2 / \text{C}_3\text{H}_8 / \text{SiH}_4$ , i.e. without chlorinated species.

## EXPERIMENTAL TRENDS

In order to test the validity of our model, one needs to confront it to the experimental Al incorporation trends found in the literature for each main separate growth parameter, i.e. C/Si ratio, temperature, pressure and growth rate. After describing all these trends, we will discuss on the mechanism which can favor them, in the framework of the surface exchange model proposed.

### Effect of C/Si ratio

The reported results about Al incorporation dependence on C/Si ratio show almost the same trend: [Al] increases when the C/Si ratio increases (figure 3). Also, the Al incorporation slope is always higher on Si face than on C face. Note the extreme case reported in [7] for which Al incorporation on C face was found independent on C/Si ratio. The observed trends are generally explained by the site competition rule in which Al incorporates on Si atomic site: lower C/Si ratio means Si rich conditions and thus stronger competition for Al to incorporate. However, this commonly used argument hides some unclear issues. Indeed, usually the C/Si ratio variation is performed by changing only the propane flux in order to maintain the same growth rate (in the usual growth conditions, SiC growth rate is Si-supply limited) and thus avoiding the interference with growth rate effect. It means that the Si partial pressure is roughly kept constant. As a matter of fact, assuming competition between Al and Si, how can Al incorporation change if the Si/Al ratio in the gas phase is constant?

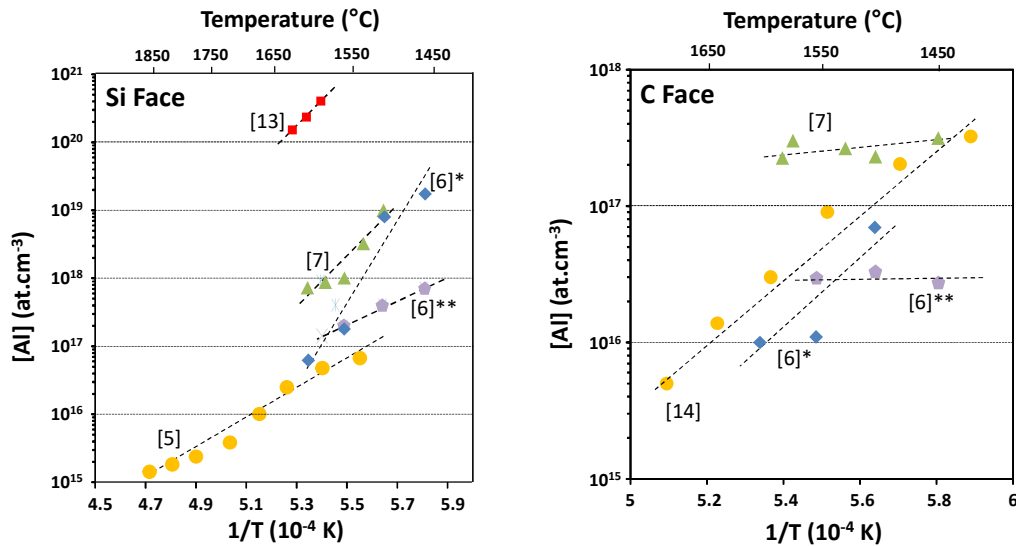


**Figure 3.** Selected results from the literature showing Al incorporation dependence on C/Si ratio during SiC epitaxial growth: for Si face (left) and C face (right).

In order to have a full picture of the process, it is necessary to consider also two other information which can be extracted from the graphs of Figure 3: i) the more pronounced dependence slope on Si face than on C face and ii) the lower Al incorporation levels on C face than on Si face (for identical conditions, see the related identical references). Differently said, Al incorporation on C face is not only less sensitive to C/Si ratio but it is also less important than on Si face. Surface bonding effect should be now considered to explain these trends, as will be discussed later.

### Effect of Temperature

Like for the C/Si ratio, the results reported on the effect of temperature on Al incorporation are rather homogeneous (figure 4). Indeed, in most cases, increasing temperature leads to a decrease of Al incorporation, except for one particular case in which independency is observed (C face and Si-rich conditions). This trend is commonly explained by Al-desorption limited incorporation: increasing temperature increases the desorption rate of Al atoms from the surface. Despite such apparent homogeneity in the literature results, the curves slopes, that is to say the apparent energies of activation ( $E_A$ ) of the limiting process, are varying significantly from 0 to -204 kcal/mol (see Table 1). However, each polar face seems to behave a bit differently since, in average, the absolute values of  $E_A$  are lower for C face than for Si face.



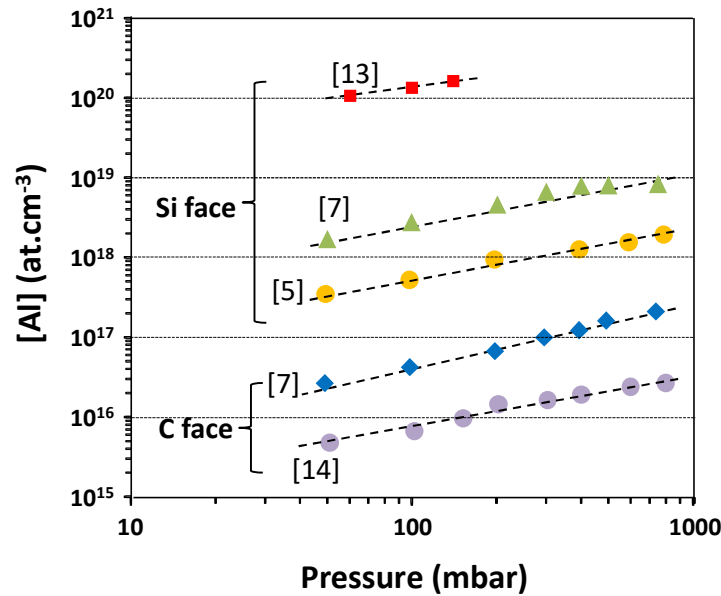
**Figure 4.** Selected results from the literature showing Al incorporation dependence on temperature during SiC epitaxial growth: for Si face (left) and C face (right). \*C-rich and \*\*Si-rich growth conditions [6].

**Table 1:** Apparent activation energies  $E_A$ , in eV, as a function of polarity, extracted from the curves of Figure 4.

$E_A$ Si face	$E_A$ C face	Reference
-3.7	-	[5]
-8.9	-5.9	[6]*
-2.4	0	[6]**
-6.3	-0.8	[7]
-7.1	-	[13]
-	-6.1	[14]

## Effect of Pressure

Concerning the effect of growth pressure on Al incorporation, the trends are even more homogeneous than for the previously discussed parameters (figure 5). One can see that Al incorporation decreases when decreasing growth pressure. This is usually ascribed to the decrease of the partial pressure of Al-containing species when reducing the total pressure. If one fits each curve with a power law ( $[Al] \propto P^{1/x}_{Alx}$ , where  $x$  is the number of Al atoms in the Al-containing species responsible for the incorporation), it can be found that the calculated value of  $x$  is varying from 1.2 to 2.1 (see Table 2). One would expect a value of  $x$  close to unity since TMA molecule contains only one Al atom (its dimer form  $Al_2(CH_3)_6$  decomposes into two monomers  $Al(CH_3)_3$  above 400 K [15]) while simulation suggests that TMA decomposition does not lead to any compound with more than one Al atom [16]. This shift from unity could be the indirect consequence of other parameters which are correlatively affected also by the reactor pressure, such as C/Si ratio or growth rate.



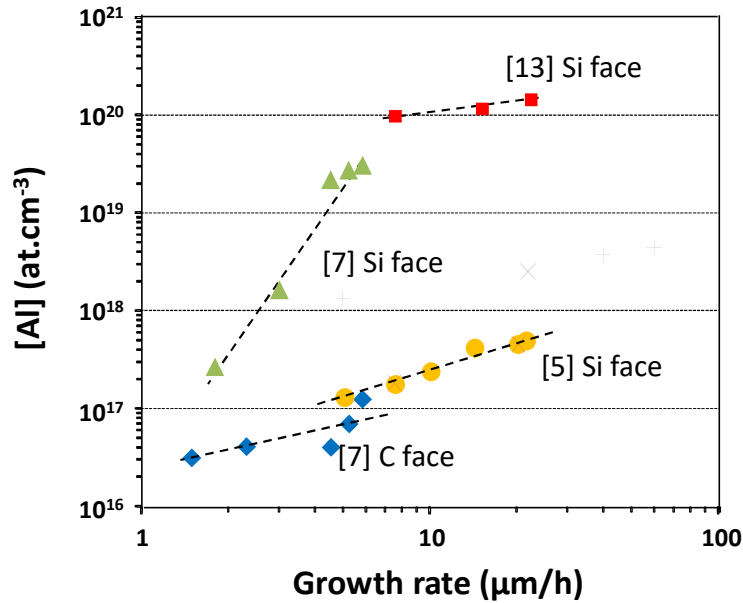
**Figure 5.** Selected results from the literature showing Al incorporation dependence on reactor pressure during SiC epitaxial growth.

**Table 2.** Evolution of the fitting parameter  $X$  of the curves in Figure 5, using a power law ( $[Al] \propto P^{1/x}_{Alx}$ ), as a function of other conditions.

<b>Polarity</b>	<b>Temperature (°C)</b>	<b><math>X</math></b>	<b>Ref-er-ence</b>
Si face	1600	2.1	[7]
Si face	1620	1.9	[13]
Si face	1650	1.7	[5]
C face	1600	1.2	[7]
C face	1650	1.7	[14]

## Effect of growth rate

Growth rate affects also the incorporation rate of Al inside 4H-SiC. As can be seen from figure 6, all the reported results demonstrate an increase of [Al] with increasing growth rate even though the increase slopes vary significantly from one data set to another. Note that this trend is true for both Si and C polarities.



**Figure 6.** Selected results from the literature showing Al incorporation dependence on SiC epitaxial growth rate.

## Discussion

As a preliminary remark of the discussion, it is interesting to note that the case of Al incorporation in 4H-SiC looks simpler than N incorporation case. Indeed, for each parameter, Al incorporation displays homogeneously the same trend (though with some slope variation), independently on surface polarity or on reactor type. This was clearly not the case for N incorporation [8].

According to the effect of temperature, Al desorption from the surface seems to be a prominent parameter limiting Al incorporation into SiC, for both polar faces. In fact, applying this Al desorption-limited process to other parameters like growth rate and pressure makes also sense. For instance, increasing growth rate leads to a decrease of the mean time before adsorbed Al atoms are incorporated to the crystal, which implies lower desorption probability and thus higher Al incorporation, as experimentally observed. Concerning pressure effect, decreasing growth pressure enhances desorption of every ad-species from the surface, especially those with high volatility like Al. This effect could also contribute to the shift from unity of the X exponential parameter discussed earlier.

The most difficult parameter to model for Al incorporation is the C/Si ratio. Indeed, as mentioned earlier, the Si/Al ratio in the gas phase does not change when adjusting the C/Si ratio, in order to keep constant growth rate. But this is the ratio C/Al which is changing and thus to be considered for proper understanding. Before going further in the discussion, it is worth reminding the two following aspects which could be relevant for the model: 1) contrarily to N impurity, Al and C atoms do not incorporate on the same atomic sites so that they are not in competition; 2) the aluminum precursor used (TMA) brings also three additional C atoms per Al atom so that the effective C/Si ratio has to be corrected if the flux of TMA is not negligible as compared to the C precursor one.

□

The C/Al ratio was never really considered (or calculated) when discussing on the mechanism of Al incorporation. But this parameter was mentioned indirectly using the term of "carbon coverage" on the surface [7, 16]. High carbon coverage was suggesting that the terraces on Si polar face are almost completely C-terminated so that it modifies the adsorption, bonding and thus incorporation of Al atoms on Si face. But as mentioned earlier, such incorporation was assumed to be additive and the possible Al incorporation on  $V_{Si}$  was not envisaged. Furthermore, adding atoms on the terraces (Al or Si on such C-terminated surface) suggests that growth can also occur by 3D nucleation which is not compatible with the step-controlled homoepitaxy model.

Let us now apply the  $V_{Si}$  driven Al incorporation model for explaining the experimental results. Firstly, the formation of a  $V_{Si}$  inside 4H-SiC is not easy to achieve, at least not as easy as  $V_c$ . Meanwhile the latter can be naturally found inside bulk crystals or epilayers, the former needs energetic treatments, like electron or proton irradiation, for appearing [18, 19]. But on the terraces of a Si-face SiC crystal, this could be easier since only three Si-C bonds have to be broken to form a surface  $V_{Si}$ . Experimental confirmations of such occurrence during high temperature (epitaxy compatible) thermal treatments are numerous, the more common being the surface graphitization of SiC under inert atmosphere by preferential Si evaporation from the surface, to create graphene for instance [20]. As a matter of fact, Si evaporation from the SiC surface should occur also during epitaxial growth, but in a transient manner since Si atoms are also coming from the gas phase (injection of Si-containing precursor). There should be thus a constant exchange of Si atoms between the surface and the gas phase: impinging  $SiH_x$  species compensating Si evaporation loss. But if one adds now Al atoms in the gas phase, element which can only sit on Si site, this constant exchange with the gas phase can lead to incorporation of Al atoms on the terraces surface of a Si-face seed, not in an additive manner but rather in a replacement manner (see figure 2). An Al atom sitting on such surface site would form three bonds with the surface and thus be more tightly attached than for the cases of additive incorporation (either at the step edges or on the terraces).

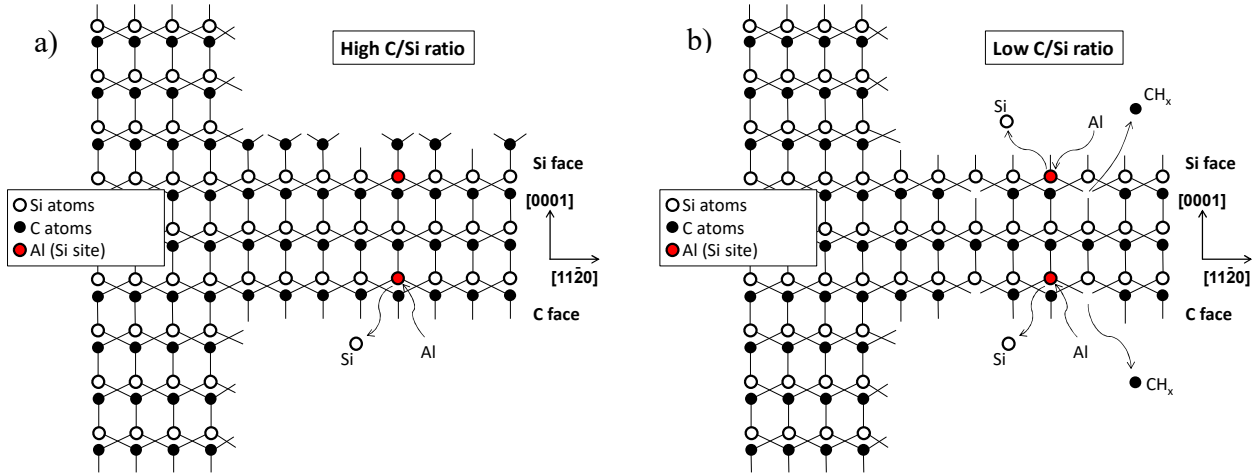
Since forming  $V_{Si}$  at the surface is obviously a thermally activated process, one would expect that increasing temperature would lead to Al incorporation increase due the enhanced availability of these vacancies. But the experimental data are showing the reverse trend. This does not contradict the proposed model but rather means that  $V_{Si}$  availability at the surface is not a limiting factor for Al incorporation. As mentioned earlier, Al desorption from the surface seems to be the prominent parameter limiting its incorporation so that increasing temperature would increase its desorption rate in a faster manner than forming additional  $V_{Si}$ . Considering growth rate effect, there is no trivial relationship between growth rate and  $V_{Si}$  at the surface. But as said previously, considering that the vacancy formation rate is apparently not the limiting process, the effect of growth rate seems more likely related to the decrease of desorption probability when the growth rate is increased.

Reduction of Al desorption is thus a critical parameter for its incorporation during SiC epitaxy. This can be done for instance by reducing temperature or increasing pressure or growth rate. But one can also try stabilizing its position inside the Si vacancy for instance by increasing the C/Si ratio. Indeed, increasing carbon coverage can be beneficial for Al stability at the surface of a Si polar face, by forming a fourth (and out-of-plane) Al-C bond which leads to tetrahedrally bonded Al, alike inside the bulk SiC material (see figure 7a). Inversely, low C/Si ratio leads to destabilization of the Al atom sitting on Si surface vacancy not only by lack Al-C capping bond but also by C vacancy formation under the surface (Figure 7b), as was suggested in the model for N incorporation [8].

Let us discuss now on the case of C polar face. The  $V_{Si}$  that can form closest to the surface of the terraces are located below the first atomic row of C atoms. As mentioned earlier, comparison with graphene formation on SiC shows that C face can release Si atoms at similar temperatures even if located at the sub-surface. Such  $V_{Si}$  formation on C face is thus not unlikely and it can then allow incorporating Al atoms in a replacement manner as shown in Figure 7a. The effect of temperature on Al incorporation on C face

□

shows a similar trend as on Si face but with a smaller slope (lower absolute value of  $E_A$ ). As a matter of fact, the same balance between  $V_{Si}$  availability and Al desorption can be proposed. The lower absolute value of  $E_A$  on C face could be ascribed to the different bonding environment which should reduce  $V_{Si}$  availability but in the same time should reduce also Al desorption rate once an Al atom is sitting on such a site. The effect of C/Si ratio on C face is much less steeper than on Si face, probably because C coverage affects only the  $V_C$  availability at the surface. It suggests that low C/Si ratio could destabilize Al incorporated on  $V_{Si}$  site by lowering the numbers of bonds of already incorporated Al atoms (figure 7b).



**Figure 7:** Schematic drawing showing the C/Si dependence of Al incorporation by replacement of a Si atom at the terraces of a 4H-SiC crystal, for both Si and C polar faces, for a) high C/Si ratio and b) low C/Si ratio conditions.

One cannot exclude that Al atoms incorporate also at step edges. Most probably, both  $V_{Si}$  assisted Al incorporation on terraces and direct incorporation at step edges are occurring at the same time. But considering that for a  $4^\circ$  off seed the solid/gas interface is roughly composed of  $\sim 94\%$  of terraces and of only  $\sim 6\%$  of step edges [8], the processes involved on terraces should play a prominent part simply due to this proportion. As said earlier, experimental results on the effect of seed misorientation are rather contradictory since both insensitivity and Al incorporation increase were reported with increasing misorientation [4-5]. Note that the reported increase in [4] is rather moderate compared to the effect of the growth parameters discussed in this paper, effects which can be explained by processes occurring on terraces.

Finally, implementing this model into numerical simulation would obviously bring interesting insight for evaluating for instance the Si/Al and/or C/Al ratios in the reactors or for validating/ rejecting some assumptions made in this paper. However, we are using data from the literature, data which are intrinsically linked to each CVD reactor used (geometry, dimensions, real physical-chemical parameters at the layer surface during the process, and not just the input parameters). And since these essential information are generally not provided in the papers, simulation simply based on literature data cannot be reasonably performed. In addition, such simulation, besides being impossible without the reactor geometry, may not be appropriate in the present paper as we only discussed a surface mechanism. DFT based calculations and molecular dynamics would be more suited to assess the possibility of  $V_{Si}$  formation at the surface and the reaction pathway for Al incorporation in the Si vacancy. Performing such simulation is of course of the highest interest but it is an objective in its own. Present authors believe that it is out of the scope of the present paper, which only present a qualitative and phenomenological discussion and does not intent to quantify the incorporation.

## CONCLUSION

We have transposed to Al incorporation the model previously proposed for N incorporation during 4H-SiC epitaxial growth and involving transient formation of surface vacancy on the terraces. In the case of Al, one needs not only to take into account the formation of  $V_{Si}$  at the surface but also the high desorption rate of this element which dominates when considering the effects of the main epitaxial growth parameters. As a matter of fact, increasing Al incorporation means improving its stability upon adsorption on  $V_{Si}$  and/or reducing its desorption time. The transient formation of  $V_c$  at the surface or sub-surface plays also a role by destabilizing the incorporated Al atoms.

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