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Polytype Transitions in Silicon Carbide: A Macroscopic View

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Abstract. Based on the analysis of the defect formation in silicon carbide polytypes in different semiconductor manufacturing processing steps, device operation and environmental-device interaction it is concluded that external material and energy fluxes are generally able to destabilize the polytype structure. The governing reason is the formation of stacking faults and instabilities of the partial dislocation associated with them. A new ansatz is proposed to describe the structural instabilities using none-equilibrium thermodynamics and entropy production. A criterial form for polyype transitions is proposed. The developed criterial form is applied to describe observed structural instabilities occurring under different external actions.

Introduction

Polytypism is a special one dimensional form of polymorphism and a universal property of a wide range of metals, elemental and compound semiconductors, insulators and superconductors [1-5]. Prominent polytypic crystals belong to the A^nB^{8-n} (SiC, ZnS), MX_2 (CdI_2) and ABX_3 (Perovskites) crystal structure families. One of the structural preconditions for the occurrence of this phenomenon is a relative low stacking fault energy of stacking faults formed in a glide plane (basal plane) [6-10]. Another, the location in the John-Bloch diagram at the border between two structural ground states, for A^nB^{8-n} the wurtzite (2H) and sphalerite (3C) modifications [11]. Additionally, the possibility to form different modes of stacking of two dimensional structural compatible units along a defined crystallographic direction is required [12].

Polytypic and polymorphic structural variants behave thermodynamically different. The polymorphic modifications represent well defined thermodynamic phases, with well specified stability ranges of temperature, composition and pressure. The transition from one polymorph to another is a first-order phase transformation. On the contrary, except for very few short period polytype modifications, no such physical factors have been found to govern the formation of polytypes. This is originated by small differences of formation and total energies between different polytypes structures [13-17]. The small energetic differences of the different polytype structures as well as the low stacking fault energies are less than the energetic disturbance in semiconductor technological processing steps and below the thermal energy at room temperature. As a consequence, different polytype appear under identical conditions of temperature and pressure and display syntactic coalescence and intergrowth of polytypes [18-21]. Additionally, this cause a specific type of defect formation, i.e. polytype lamella (syntactic inclusions) associated with stacking fault formation and local or global polytype transitions, during semiconductor device processing steps, i.e. thermal treatment [22, 23], thermo-mechanical stress [24], impurity diffusion [25], ion implantation and annealing [26-28], sputtering [29], etching [30], oxidation [31], metallization [32], crystal [33] and epitaxial [18, 34, 35] growth. Furthermore, polytype transitions occur during the operation of semiconductor devices [36-39].

According to published data polytype transformations are related to partial dislocation (PD) and stacking fault (SF) formation in the initial structure, i.e. structural “disorder”. It can be hypotesized that at the very early stage the transformation process is related to point defect formation and their

selective coagulation leading to dislocation and stacking fault formation. The transformation processes occurring at nonequilibrium conditions can be considered as nonequilibrium phase transitions. The local structural changes induced by the SFs are polytype inclusions, i.e. able to act as information centers for the nucleation and growth of extended regions with a different polytype structures. Collective and selective interactions between these defects result in a stability loss of the original structure. Reaching the critical state, nonequilibrium polytype phase transition occur which lead to the formation of a new polytype structure better adapted to the exterior conditions. Schematically, the nonequilibrium phase transitions can be described as: Order → point defect/dislocation generation → stacking fault generation → defined disorder → "catastrophe" → new order. In Fig. 1 a generalizing scheme of polytype phase transition in terms of thermodynamic quantities and defect interactions is given. Energy dissipation in the crystal originating from incoming fluxes of mass, energy, impulse and entropy lead to an information excess in the initial polytype structure as a precondition for structural changes.

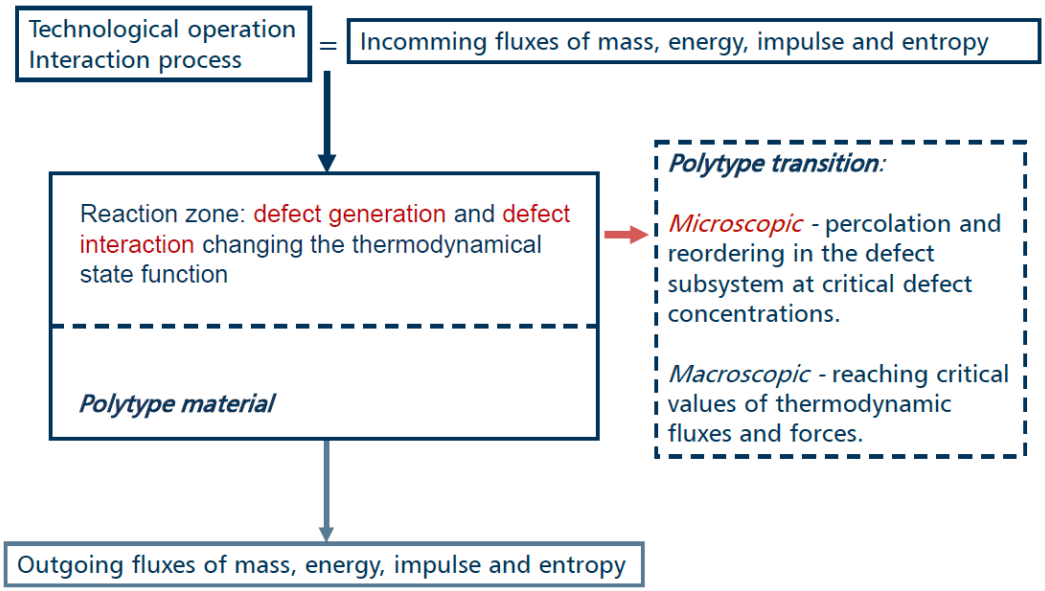

Fig. 1. Polytype transition as nonequilibrium phase transitions.

Theoretically, the stability of different polytype structures without point and extended defects was studied in the framework of total energy calculations using different simulation approaches [13-16, 40]. In [3, 40] the role of entropy for the stabilization of the polytype structure was discussed. In case of crystal growth the nucleation theory was used to assess the stability of the formation of different polytypes in dependence on supersaturation, Si to C ratio and doping [41, 42]. Alternatively, kinetic Monte Carlo [43-46] and molecular dynamic [47-48] approaches were applied. Another class of modelling polytype formation and transitions rely on dislocation and stacking fault reactions and their transformations [1, 49-51]. All this theoretical modelling approaches address singular initial material property conditions and specific material processing steps with a limited generalization ability to describe polytype transitions occurring at other semiconductor processing steps or during device operation.

In this contribution an ansatz is proposed which might pave the way for generalized macroscopic description of the defect related polytype transitions at arbitrary physical and chemical treatments.

Nonequilibrium Thermodynamic Model

As shown above polytype transformations are interconnected with defect formations and can be considered as a specific type of defect formation. The percolation of the defect subsystem at a critical point transforms the initial crystal structure *i* into a new polytype modification *j* independent on the

physical nature of the action in the semiconductor fabrication processing step or during device operation [50, 51]. The structural origin is the evolution of existing "disorder" (information excess) or the generation of "disorder" in the initial polytype structure *i*.

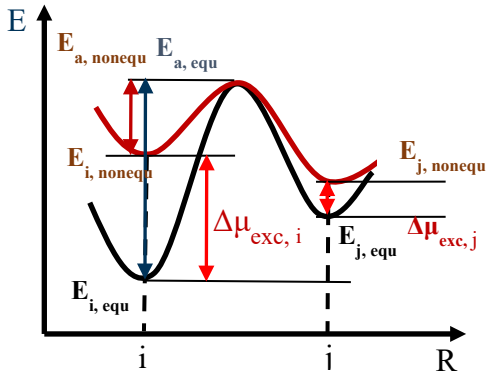

Fig. 2. Scheme of the energetic states between two different structures *i and j* under equilibrium and nonequilibrium conditions.

Within the framework of nonequilibrium thermodynamics system evolution and stability can be studied using change of entropy *S* [52-55]. The entropy can be linked to critical "disorder" (information excess) at the transition point from polytype *i* to *j*. The "disorder2 manifests itself in defects formation increasing the internal energy of the system (crystal interacting with the environment) and can be described by an order parameter $P_{i\rightarrow j}$ [50]. Therefore, the "disorder" generated during the interaction process changes the internal energy of the initial polytype *i* by a nonequilibrium contribution $\Delta\mu_{exc,\,i}$ as shown in Fig. 2. The change of the internal energy $\Delta\mu_{exc,\,i}$ can be linked to the entropy increase $\Delta S_{exc,\,i}$ (excess entropy of polytype *i*) using the expression $\Delta\mu_{exc,\,i} = T\,\Delta S_{exc,\,I}$ [56]. Then, a transition of a polytype *i* into a polytype *j* in an arbitrary process is possible if the total energy of polytype *i* is higher than the total energy of polytytpe *j* and the energetic barrier between the two states can be negotiated. Because the total energy differences between the different polytypes in the equilibrium state is small [13-17] the stability and transition between the states at nonequlibrium conditions is governed by the increase of the chemical potential at nonequilibrium state caused by the residual defects formed. This is schematically illustrated in Fig. 2. Therefore, for the critical point of the polytype transition $i \rightarrow j$ a critical excess entropy $\Delta S_{exc,\,crit}$ can be defined using the relationship between $\Delta\mu_{exc,\,i}$ and $\Delta S_{exc,\,i}$. From the of nonequlibrium thermodynamics point of view, the critical excess entropy must be determined in order to identify the critical conditions for the polytype transitions.

The excess entropy formed in polytype *i* $\Delta S_{exc,\,i}$ is associated with the energy dissipation in the polytypic single crystalline matrix material *i* due to the action of thermodynamic forces and linked fluxes of energy, mass and impulse of the physical interaction process. Introducing an ordering time of the initial polytype matrix *i* $\tau_{ord\,i}$ necessary to annihilate or incorporate the defects into the lattice and to rearrange the lattice stacking into the structure of the polytypic matrix under the conditions of the technological process under consideration. $\Delta S_{exc,\,i}$ can be determined using the entropy production *dS/dt* of the process and $\tau_{ord,\,i}$:

$$\Delta S_{exc,\ i} = (dS/dt)\tau_{ord,\ i}\,, \tag{1}$$

with $\tau_{ord,i} = \sum_{m=1}^{n}\tau_m$, and with τ_m as the ordering time of the ordering process *m*. In the linear approximation of nonequilibrium thermodynamics the general form of total entropy production with fluxes J_k and conjugated driving forces X_k is [52]:

$$dS/dt = \int \sigma_S\, dV = \int(\textstyle\sum_k J_k\, X_k)dV, \tag{2}$$

with σ_S as the entropy production per unit volume and *V* as the volume. Taking into account that forces X_l are affecting the fluxes J_k by $J_k = \sum_l B_{kl}X_l$ Eq. 2 takes the form:

$$dS/dt = \int(\textstyle\sum_k\sum_l B_{kl}X_l\, X_k)dV, \tag{3}$$

From Eq. 1 and 3 it follows that $\Delta S_{exc,\,i}$ is composed by the action of all fluxes and forces interacting with the polytypic crystalline matrix *i*. In table 1 a summary of thermodynamic forces and fluxes are given for which degradation of the polytype structure or polytype transitions were observed.

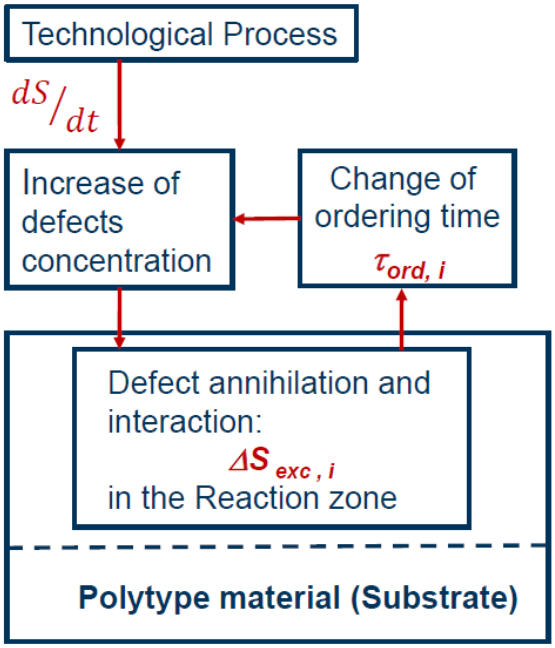

Fig. 3. Scheme of the excess entropy formation in the polytype matrix *i* due to disordering and ordering by a technological process.

The generalized interaction scheme explains the interrelation between dS/dt, $\tau_{ord,\,i}$ and $\Delta S_{exc,\,i}$ is shown in Fig. 3. It demonstrates that entropy production and the excess entropy formed in the polytypic matrix *i* $\Delta S_{exs,\,i}$ affects the ordering time $\tau_{ord\,i}$ by a positive feedback loop leading to an increase of $\tau_{ord\,i}$ through an increased defect density of the matrix crystal. Consequently, entropy production as a state function of nonequilibrium thermodynamics allows to formulate a stability criterial form of the polytype matrix *i* in an interaction process with an arbitrary environment. The polytype matrix is stable if the following inequality if fulfilled:

$$\Delta S_{exc\,i} = dS/dt\,\tau_{ord\,i} < \Delta S_{exs\,crit\,i}\ , \tag{4}$$

here $\Delta S_{exc\,crit,\,i}$ is the critical excess entropy causing the instability of the polytype matrix and the onset of polytype transition in form of $i \rightarrow j$.

Table 1 Collection of fluxes and forces in their thermodynamic description able to contribute to polytype defects formation or polytype transitions

Process	Thermodynamic force, X	Thermodynamic Flux, J
Heat flow	Temperature Gradient $\nabla(T^{-1})$	Heat Flux q
Current flow	Electric field ET^{-1}	Electrical Current I
Plastic Deformation	Stress σT^{-1}	Plastic Strain $\dot{\varepsilon}_p$
Diffusion	Chemical Potential $\nabla(\mu_C T^{-1})$	Diffusion Flux J_D
Deposition Vapour/Gas Phase	Chemical Potential $\nabla(\mu_P T^{-1})$	Diffusion Flux J_P
Chemical Reaction	Reaction Affinity $A_R T^{-1}$	Reaction Rate R_c
Particle irradiation	Particle Flux Density $A_R T^{-1}$	Target Atom Velocity $\dot{v}_p$
Polishing/Wear	Friction Force FT^{-1}	Relative Velocity V_r

μ_C and μ_P are the chemical potential due to a specific concentration and partial pressure, respectively. The forces and fluxes for deposition from vapour or gas phases are given for the diffusion limited case without convective or turbulent contributions.

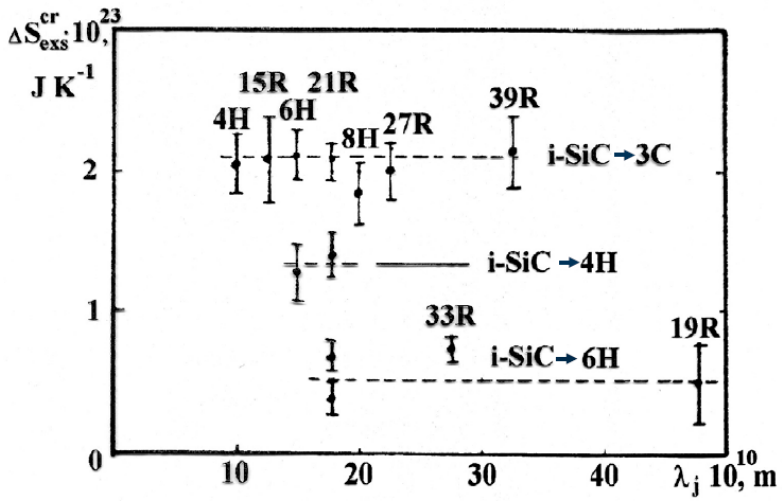

Fig. 4. Critical excess entropy of polytype transition $i \rightarrow j$ in the dependence on the shortest translation period λ_i of the initial polytype structure.

The critical excess entropy related with the polytype transition from polytype *i* to polytype *j* was determined using physical vapour transport experiments in a sublimation growth cell applying a special SiC vapour distributor allowing for supersaturation distributions over the crystal surface at constant substrate temperatures [18, 57]. For the investigations the following polytypes were used: 4H, 15R, 6H, 21R, 8H, 27R, 33R, 39R, 19R. The polytpes are ordered with respect to the shortest translation period which is the period containing fundamental stacking sequence characteristic for the given polytype, for example (22) or (32) for 4H and 15R, respectively. The expression for critical excess entropy $\Delta S^{cr}{}_{exc\ crit,\ i\rightarrow j,}$ for polytype transition $i \rightarrow j$ was derived using Eq. 1 and 3 for the conditions of physical vapour transport growth and has the form;

$$\Delta S^{cr}_{exc\ crit,\ i\rightarrow j} = (R/N_i) ln\left(1 + \left(v_{cr,\ i\rightarrow j}/v_{oi}\right)\right), \tag{5}$$

with *R* as universal gas constant, N_i as the number of unit cells per mole of polytype *i*, v_{cr} as critical growth rate for transition $i \rightarrow j$ and v_{oi} as the vaporization rate of polytype *i*. The obtained results are summarized in Fig. 4. The main findings are as follows. The critical excess entropy $\Delta S^{cr}{}_{exc\ crit,\ i\rightarrow j}$ for polytype transition $i \rightarrow j$ is independent on the polytype structure of the initial polytype *i* and depends only on the final polytype of the polytype transition *j*. The value of $\Delta S^{cr}{}_{exc\ crit,\ i\rightarrow j}$ is decreasing if the shortest translation period λ_j of the transformation product is increasing. Consequently, the entropy barrier for transitions into a polytype structure with higher λ_j is lower. This means that the transformation into polytypes with larger λ_j requires a lower "disorder" (information excess), i.e. the formation and rearrangement of a lower defect concentration compared to the transition into a polytype with higher crystal symmetry, for example 3C-SiC. This is supported by the defect permutations needed to transform different hexagonal or rhombohedral polytypes into other hexagonal or rhombohedral polytypes compared to a transformation into the 3C or 2H structures [1, 3, 58]. As a consequence polytype transitions into polytypes with larger λ_j occur at lower critical growth rates as reported in [56, 59-61].

Assuming now a defective polytype matrix. Defects can be dopants, dislocations, stacking faults or inclusions. Then, these defects increase the excess entropy $\Delta S^{cr}{}_{exc}$ and chemical potential of the matrix to $\Delta\mu^{def}$. It follows that the vaporization rate v_{oi} changes into the vaporization rate of the defective matrix $v^{def}{}_{oi}$;

$$v_{oi}^{def} = v_{oi}\, exp(\Delta\mu^{def}/RT). \tag{6}$$

Combining Eq. 6 with 4 it follows:

$$\Delta S_{exc}^{def\ cr} = (R/N_i) ln\left(1 + (v_{cr}/v_{oi}) exp(-\Delta\mu^{def}/RT)\right). \tag{7}$$

Therefore, defective or strained substrates exhibiting an enhanced evaporation rate have a reduced entropy barrier for polytype transitions due to decreased disorder needed for the transition. As a consequence the critical growth rate for structural changes at a given substrate temperature decreases. This was observed in [57], where a reduction of the critical growth rate for the polytype transitions 6H → 4H and 6H → 3C on 6H modified by boron diffusion in dependence on the near surface boron concentration was observed. The increase of the boron concentration decreased the critical growth rate. The same effect was detected for nitrogen implanted 6H-SiC substrates in dependence on the implantation dose and for surfaces treated by polishing [57].

Thermo-plastic deformation of silicon carbide at different temperature and deformation conditions was studied in [24, 62-67]. In this studies it was revealed that beside dislocation related mechanisms, structural transitions in form of polytypic transformations are possible in the brittle [65, 67] and ductile deformation region [49, 62, 66]. The observed structural transformation series are formally similar to polytype transformation series observed in growth experiments. Generally, the shortest translation period of the transformation product is smaller than the initial one. In [67] the transition sequence 15R → 4H → 3C was observed in milling experiments with increasing milling time. In [24, 64] the possibility of polytype transitions i → 2H and polytype i → 3C was found. The polytype transformation of an arbitrary polytype into 3C is the most common one observed in mechanical treatments of SiC polytypes [24, 49, 62, 63, 65, 66]. In Fig. 5 the experimental critical initial stress as a function of the shortest translation period λ_i of the initial polytype is given. The critical stress values for the polytype transitions are decreasing with increasing λ_i. This behavior resembles the dependence of the critical growth rate of the onset of the polytype growth transitions in dependence on λ_i published in [56].

For thermo-plastic deformation the entropy production can be written as [68]:

$$dS/dt = T^{-1}(\tau\dot{\gamma} - E(d\rho/dt)) , \tag{8}$$

with τ as the sheer flow stress, $\dot{\gamma} = \rho_m v_m$ as the shear strain rate (ρ_m density of mobile dislocation segments, v_m as average velocity of the mobile dislocations), E as average dislocation energy per unit length, ρ as the dislocation density. Using Eq. 8 and the defect interaction model developed in [50, 51] the critical initial shear stress was estimated. The results of the analysis are given in Fig. 5. They show a reasonable agreement with experimental data

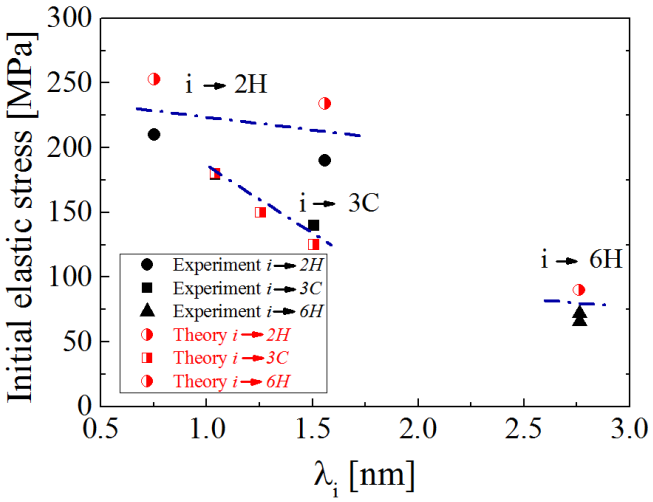

Fig. 5. Polytype phase transitions in dependence on the shortest translation period λ_i and the initial elastic sheer stress.

Summary

External mass, entropy and energy fluxes are generally able to destabilize the polytype structure. The governing reason is the formation of stacking faults and instabilities of partial dislocations associated

with them. A new ansatz is proposed to describe the structural instabilities using nonequilibrium thermodynamics and the entropy production. A criterial form for polytype transitions is proposed. The criterial form is applied to describe observed structural instabilities occurring under different external actions.

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