STATISTICAL APPROACH TO THE ELASTIC PROPERTY EXTRACTION AND
PLANAR ELASTIC RESPONSE OF POLYCRYSTALLINE THIN-FILMS

DISSertation

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ABSTRACT

A system of numerical methods is proposed to predict planar elastic responses and/or the elastic property (modulus and Poisson’s ratio) of polycrystalline thin-films for Micro-Electro-Mechanical Systems (MEMS) components. A lattice based stochastic model is employed to model the micro structural evolution (nucleation and growth) of thin-film deposition. Given a micrograph, depicting a grain distribution, key kinetics parameters for the deposition process can be extracted by the proposed inverse method within the system.

In order to connect the microstructure evolution simulation and the elastic behavior prediction by the finite element method, a mesh generation algorithm on the lattice based microstructure is developed. The edge matching, essential for the node location on the grain boundaries, is accomplished by a pixel-by-pixel marching scheme. The finite element method with planar anisotropic elasticity model is used to calculate statistically averaged values of Young’s modulus and Poisson’s ratio. The Chi-square test is adopted for the statistical convergence criterion, proven to be statistically admissible and logically reasonable. This criterion is not affected by the appearance order of the variables and well behaved regardless of the variable types considered.
The developed system of models (plane stress/strain), techniques and numerical algorithms are applied to the following polycrystalline materials to calculate statistically scattered effective elastic constants: polysilicon, barium titanate and zircon.

A micro-beam specimen is also analyzed for a statistically scattered elastic response (tip deflection and stress) and compared with classical solutions. The beam is assumed to have a known microstructure (Al₂O₃), in which each grain has a different planar orthotropic axis. The example approach can be applied directly to other MEMS components without bothering to determine statistically averaged elastic constants.

The effects of the polycrystalline specimen size and the anisotropy of the material on the statistical scatter of the effective elastic constants are studied. The larger scatter is observed for a smaller size of specimen and for a material with more severe anisotropy.
Dedicated to my parents, wife Hyangjoo
and my kids; Han Seul, Hangil, and Hanul
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CHAPTER 1

INTRODUCTION

As the role of the polycrystalline thin-films for MEMS and the area of MEMS itself grows, the mechanical performance of the polycrystalline thin-films becomes one of the critical issues. The statistically scattered and size dependent mechanical properties of the polycrystalline thin-films in micro-meter length scale are discovered by intensive experimental and numerical investigations. The deterministic approach can not capture these statistical scatter and the size effects. A statistical approach based on the random stochastic process is proposed herein to reconstruct the microstructure and to compute elastic responses. A phenomenological approach is employed under the following assumptions:

1) A polycrystalline thin-film has a columnar structure, which makes a 2-D simulation on the film surface feasible.

2) The micro structural evolution does not alter the genuine mechanical and crystallographic properties of the constituent single crystals, which helps to ignore the residual stress.

3) The final microstructure is evolved through pure nucleation and growth process, which makes the evolution a pure random stochastic process.
4) A grain is large enough to enclose a very large number of atoms, which allows the use of continuum finite element (FE) to calculate elastic behavior of single crystals in each grain.

5) Any relative motion in grain boundaries under elastic loading can be ignored. Only single phase polycrystalline materials are investigated in two dimensions. The Monte-Carlo method and FE analyses (FEA) on reconstructed microstructures are employed to investigate unknown statistical distribution of elastic responses of single phase polycrystalline thin-films.

All the fundamental assumptions and theories related to the nucleation and growth kinetics, which will be described in Chapter 4, is taken as they are. It is believed that a stable nucleus consisting of a critical number of atoms transform into crystallite from the amorphous state at a constant rate throughout the process. The previously nucleated nuclei grow isotropically with a constant speed until they impinge on each other. The lattice based model is used for an efficient microstructure evolution simulation based on this nucleation and growth mechanism. A pre-defined lattice system is used as a simulation domain. A fast and stable algorithm is developed by employing simple matrix manipulations and the uniformity characteristics of the random numbers.

The kinetics parameters of a deposition process for the polycrystalline thin-films can be extracted by experiments with some assumptions and a priori theories, but it is a rather difficult task at best. A simple inverse method is proposed to extract the kinetics parameters. This inverse method is based on the major quantitative measures of the microstructure, number of grains and the grain size distribution (GSD), and the relations between these measures and the kinetics parameters. GSD is the key factor for the
kinetics parameter extraction method proposed. The previous researches for GSD focused on two extreme cases of the nucleation conditions, i.e., site-saturation and continuous nucleation. All nucleation conditions are covered by adopting the concept of nucleation speed parameter (NSP), $\beta_n$.

While the lattice based microstructure evolution model has an advantage of computational efficiency, there is no explicit algorithm for FE meshing. A 2-D FE meshing technique on the lattice based microstructure is developed. FEM is applied to calculate the equivalent, or effective, elastic constants and the elastic responses of polycrystalline materials.

The statistical convergence criterion, which is essential for the statistical approach, is redefined by using $\chi^2$ (chi-square) test. The validity of this criterion is tested and the feasible confidence interval is derived by investigating the simulation results.

This dissertation is organized in 8 Chapters and 4 Appendices including this introductory chapter. In Chapter 2, previous researches related to the mechanical properties of the polycrystalline materials in the small length scale are reviewed. Chapter 3 summarizes the framework used in this dissertation. The methodologies, theories and algorithms used in this dissertation are explained in Chapters 4 through 6. Chapter 4 describes the theories, the models and the algorithms for the polycrystalline microstructure evolution with additional discussions on the kinetics parameters extraction. Chapter 5 illustrates the algorithms for 2-D FE meshing on the lattice based polycrystalline microstructure, and discusses the adopted orthotropic elastic FE analysis. The statistical convergence criterion, a key factor for the Monte-Carlo method is depicted
in Chapter 6. The framework of the dissertation is validated by the applications exemplified in Chapter 7. And the dissertation is concluded in Chapter 8.

Appendix A explains about the random numbers and their characteristics. Some fundamentals on statistics are reiterated in Appendix B. The reviews on statistics are extended to the goodness of fit test in Appendix C. The Miller indices of the single crystal axes, employed in the dissertation, are briefed in Appendix D.
CHAPTER 2

LITERATURE REVIEW

Experimental and numerical efforts are reviewed here along with a brief historical background of MEMS devices. It is limited to micro-meter scale polycrystalline materials.

2.1 Historical Background

After the discovery of piezo-resistive phenomena [Smith (1952)], the field of MEMS has grown gradually. Since then, the excellence of the silicon as a mechanical component material is found and validated by Petersen (1982). Petersen’s article is one of the big milestones in MEMS area, and it was the turning point of the rapid development in MEMS devices during last few decades [Bryzek (1996)]. Numerous extensive researches on the MEMS and its components have kept pace with the development and the growth of MEMS.

MEMS fabrication methods are categorized by Spearing (2000); (i) the surface micromachining, (ii) the bulk micromachining and (iii) the molding processes.

The surface micromachining is originated from the complementary metal-oxide-semiconductor (CMOS) processes, which is used to fabricate the integrated circuit (IC)
devices [Bustillo et al. (1998)]. The repetitive sequential process steps, the planar process, based on the photolithography are used to create pattern produces the complex geometries in batch process. It is very reliable and precise, up to the feature size of 0.25 – 0.35 µm [Bustillo et al. (1998)]. Figure 2.1 illustrates the process flow of the surface micromachining.

The main purpose of the bulk micromachining is to remove the large amount of the substrate material selectively [Kovacs et al. (1998)]. The use of etching reaction is the major method of the bulk micromachining, e.g., wet etching (isotropic or anisotropic) and dry etching (vapor-phase or plasma-phase). Figure 2.2 illustrates the ‘undercut’ with mask to form a dielectric membrane.

The most widely spread molding process is the LIGA process. The acronym comes from German words; Lithography, Galvanoformung (electroforming) and Abformung (molding) [Spearing (2000)]. The micro-mold in LIGA process is fabricated with deposition and photolithography (x-ray resist), and the actual part is made by electroplating the constituent atom inside this micro-mold. Figure 2.3 shows a typical process flow of the LIGA process.

The deposition process is a major technique in MEMS component construction, perhaps next to the bulk micromachining. This investigation is focused on polycrystalline thin-films by the deposition process. Typical characteristic length scale considered here ranges from several hundred nano-meter to several hundred micro-meter ($10^{-7} \sim 10^{-4}$ m).
2.2 Experimental Methods for Small Specimens

Since the tensile tests for the single crystal silicon by Eisner (1955), numerous experimental techniques have been developed for MEMS components, using their own customized test setups in general.

The testing methods can be categorized into a beam-bending and a direct tension test. The beam-bending test has the advantages in using compact specimens and ease of performing, mainly because the smaller force is needed, compared to the direct-tensile method, and is free from the gripping and alignment problems. However, the direct-tensile testing method is still popular due to simple data interpretation.

2.2.1 Beam-Bending Test Methods

Johansson et al. (1988) measured failure strength of a single-crystal silicon (75 – 240 μm × 8 – 16 μm × 75 – 500 μm) in a scanning electron microscope (SEM) chamber. Reported are results from 77 specimens of different size and two plane normal orientations ([001] and [011]). They used strain gages and load cells, and calibrated with the linear beam theory. Figure 2.4 shows the number of occurrences for measured failure stresses in bending.

Jones et al. (1996) also used the bending test method with the test apparatus shown in Figure 2.5 to measure the failure strain of a polysilicon. The shuttle has three cantilever beams on each side with different length, 3 μm × 1.75 μm × 50, 60 and 70 μm. The load is applied by micromanipulator on the top of the shuttle. The deformation is
analyzed by captured images, and the load is converted from the strain instead of measuring it.

The failure strength of single-crystalline silicon was found by Wilson and Beck (1996) using specimen shown in Figure 2.6 (100 µm × 18 – 30 µm × 300 – 400 µm). To relieve the effect of the surface condition and the stress concentration on the sharp corner, FEM was used to correlate the measured failure strength.

The beam bending test methods need a correlation, either analytical or FEM, to back-calculate the load or strain. A direct interpretation of the test results is impossible.

2.2.2 Direct-Tension Test Methods

Sharpe Jr. et al. (1997) measured strain directly by using interferometric strain/displacement gage (ISDG) for the first time. The schematic of this ISDG is shown in Figure 2.7, and the specimen shape is shown in Figure 2.8 (600 µm × 3.5 µm).

Greek and Johansson (1997) used strain gage for the force measurement and optical encoder to measure displacement. The test was done in a SEM chamber to eliminate the environmental effect. The free end of a specimen was designed as a ring shape to hold the loading pin as shown in Figure 2.9 (10 µm × 10 µm × 250 – 1000 µm).

Ogawa et al. (1997) used CCD (Charge Coupled Device) camera and digital image correlation (DIC) technique to capture the displacement of a specimen (300 µm × 0.5 µm × 1.4 mm). The displacement of a titanium beams was measured.

Tsuchiya et al. (1998) developed a brilliant electrostatic specimen gripping method illustrated in Figure 2.10. By providing an opposite electric current to the specimen and the loading probe, the tensile specimen is securely attached to the loading
end, Figure 2.10(b). After the material failure occurred, the broken specimen is separated from the probe by applying the same current on both sides, Figure 2.10(c). Polysilicon specimens (Figure 2.11, 2 – 5 µm × 2 µm × 30 – 300 µm) were used. The results show that the tensile strength increases as the specimen size decreased. The electrostatic gripping method was also used by Sharpe Jr. et al. (1999).

Chasiotis and Knauss (1998) used atomic-force microscope (AFM) to measure the displacements of polysilicon beams (50 – 80 µm × 2 µm × 400 µm). Two gold marks on the specimen give distinctive topology of the sample so that the displacement is captured by AFM.

2.2.3 Integrated Test

The following examples are the cases of the test apparatus integration, which means that the specimen, the gripping fixture, the loading device and actuators are all integrated by the micromachining process. A comb drive is the commonly used actuator.

Saif and MacDonald (1996) used a sample on the chip device to resolve the problems of the specimen gripping and alignment. A comb drive is integrated with the test specimen. They demonstrated that the micromachined test apparatus can accomplish microscale testing.

Sato et al. (1998) developed specimen structure which is simple as the bending test specimen while the loading the actual specimen experienced is tensile as shown in Figure 2.12. The specimen used was single crystalline silicon with size of 100 µm × 20 – 30 µm × 400 µm.
Ballarini et al. (1999) also developed an integrated test apparatus shown in Figure 2.13 similar to that of Saif and MacDonald (1996). The movement of the comb drive is measured as the displacement and it is converted to the crack propagation by the correlation with the extensive FE calculations.

2.2.4 Notes on the Experimental Results

The experimental efforts described in this section are summarized in Table 2.1 including other results. As described above and shown in Table 2.1, the experimental approaches for the MEMS devices and components are using various test methods and apparatuses. Also, the wide range of results even for the same material was discovered. There is no standard concerning in the test procedure and specimen specifications.

One of the efforts to alleviate this experimental diversity is the round-robin test of polysilicon, proposed by Sharpe Jr. et al. (1998). The specimens were fabricated by the same production run, Multi-User MEMS Processes (MUMPs) #19 and #21 [Cowen et al. (2002), Koester et al. (2003), Miller et al. (2004)]. This round-robin test was conducted by four institutes, University of California at Berkeley (UCB), California Institute of Technology (CalTech), Failure Analysis Associates (FAA) and Johns Hopkins University (Hopkins), by using internally developed testing apparatus at each institute. The results are summarized in Table 2.2 and illustrated in Figure 2.14. Young’s modulus ranges from 122 to 194 GPa and the tensile strength ranges from 1.1 to 3.2 GPa. The Young’s modulus of 194 GPa is the value exceeds the Voigt average (theoretical upper limit [Reddy (1997)]) for polysilicon,191 GPa, with [110] plane normal texture. The Voigt average is calculated by averaging all possible single crystal orientations for each grain of
the polycrystalline material with a large number of grains, which is not the case of the micro-meter length scale specimen. Therefore, the scatter of the elastic properties of the polycrystalline materials in micro-meter length scale is inevitable; in turn it is essential to take this statistical scatter into account to address mechanical performance issues.

2.3 Numerical Methods for Small Specimens

Based on the literature review on experimental results of micro-specimens, it is apparent that micro structural consideration is essential to analyze the mechanical properties in the micro-meter length scale. Microstructure is believed to be directly related to the statistical scatter and the size effect. The length and time scale of the micro structural simulation is summarized. The last portion of review entails multi-scale simulation methods, microstructure evolution models and the mechanical properties extraction methods.

2.3.1 Scale of the Simulation

The micro structural consideration for numerical calculation depends upon the scale of length and time, which can be categorized as *nano-*-, *micro-*-, *meso-* and *macro-*-. The selection of numerical simulation method is also determined by these scales. Raabe (1998) explained and summarized about these scales and the simulation methods in his book. The summary of the microstructure scale and the corresponding simulation methods are compiled in Table 2.3 and Table 2.4, respectively. According to this, the
simulation method of the micro-meso level is needed for the MEMS components, since the concerned length scale of a typical MEMS component is in the range of $10^{-7} \sim 10^{-4}$ m.

The most relevant simulation method for the very small length scale ($<10^{-9}$ m) is the molecular dynamics (MD) simulation. The fastest supercomputer today can solve the MD simulation problem with up to a billion atoms, which correspond to the size of 1 $\mu$m cubic. And the size may increase to 10 $\mu$m in next 15 years even though the computing power is doubled in every 18 months according to Moore (1965)’s law [Liu et al. (2004)].

2.3.2 Multi-Scale Simulations

In order to overcome the computational limit of the atomistic MD simulation, multi-scale simulation methods attract attentions of many researchers in recent years, which allow calculation of two different length scale regimes simultaneously.

Rudd (2001) developed a space multi-scale simulation technique for the micro-resonator by dividing the whole simulation region with MD, continuum FE and handshake regions. For the MD region, with the one-to-one correspondent discritization of the atomic structure of the region, the inter-atomic potential is defined on each atom. The traditional node and element representation are defined on the FE region. The same time interval is used for both regions. In this method, the mesh generation that ensures the one-to-one match on the interface region of MD and FE, and the parallelization of MD equation solving by using the message passing interface (MPI) are critical.

Liu et al. (2004) proposed the atomic FE method (AFEM) by using the multi-body inter-atomic potential based on the energy minimization. The Lennard-Jones type inter-atomic potential is used for up to the second near neighbor atoms. For each atom,
the stiffness matrix is derived by the derivatives of this potential relative to the position vectors of corresponding atoms. This atomistic stiffness matrix is dependent upon the crystallographic structure, e.g., cubic, tetragonal, etc., of the constituent material. They accomplish a smooth transition from atomic to continuum FE region by proposing the transition element that eliminates the “ghost force” on the interface of these two regions. All these multi-scale methods were developed for single crystal materials.

2.3.3 Simulations for Polycrystalline Materials

The studies for the polycrystalline materials started several decades ago. Johnson and Mehl (1939) derived a famous empirical equation, known as the Johnson-Mehl equation, for the time dependent phase transformation fraction of metals, \( f_{tr} \), which can be expressed as:

\[
 f_{tr}(t) = 1 - e^{-At^n} \tag{2.1}
\]

where \( A \) and \( m \) are both the process and dimension dependent constants. The assumptions for this equation are:

1) The phase transformation is processed by the nucleation and growth mechanism.

2) The nucleation continues at a constant rate from the untransformed material.

3) At the same time, the previously nucleated nuclei grow until they impinge on each other.

These assumptions set by Johnson and Mehl (1939) for the phase transformation of metals can be applied to the crystallization of the polycrystalline materials from the amorphous state under the framework of the nucleation and growth mechanism.
On the other hand, Meijering (1953) proposed a simpler model, called *cell model*, by altering above assumptions to:

1) All the nucleation occurs at the initial stage.

2) The growth of these nucleated nuclei continue until they impinge on each other.

This model is also called as Voronoi polygon because of the geometric representation of the model. This model is mathematically well established and many mathematical packages can calculate the geometric representation of this model.

Mullen *et al.* (1997) and Yin (1997) adopted this cell model to calculate the effective Young’s modulus and Poisson’s ratio of polycrystalline materials with a cubic crystallographic structure of a unit dimensional specimen by Monte-Carlo method. Chu (2000) used the same method as Mullen *et al.* (1997) and Yin (1997) by using Johnson-Mehl model on the continuous coordinate system with strong efforts on the Johnson-Mehl tessellation. Both simulations were performed for a unit dimensional simulation domain. They did not consider the specimen size. Instead, the effect of number of grains on the elastic properties is studied. Also the crystallographic structure of a material considered was limited to the cubic structure.

Castro *et al.* (2000) proposed a lattice model for polycrystalline microstructure evolution, based on the crystallization kinetics investigation results from the Johnson-Mehl model [Castro *et al.* (1999)]. A pre-defined lattice system is used for a domain of microstructure evolution simulation, in place of Chu (2000)’s continuous coordinate system. Castro *et al.* (2000) did not investigate the mechanical properties, but only focused on the microstructure evolution and the growth kinetics.
<table>
<thead>
<tr>
<th>Observer, Method, or Coating</th>
<th>Material</th>
<th>Hardness (GPa)</th>
<th>Young's Modulus (GPa)</th>
<th>Friction Coeff.</th>
<th>Residual Stress (GPa)</th>
<th>Failure Strength (GPa)</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>Greek and Johansson (1997)</td>
<td>Polysilicon</td>
<td>167</td>
<td></td>
<td>1.25</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sharpe Jr. et al. (1997)</td>
<td>Polysilicon</td>
<td>168</td>
<td></td>
<td>1.21</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Suwito et al. (1997)</td>
<td>SCS[110]</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<td></td>
</tr>
<tr>
<td>Tsuchiya et al. (1998)</td>
<td>Polysilicon</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sato et al. (1998)</td>
<td>SCS[100]</td>
<td>125</td>
<td></td>
<td></td>
<td></td>
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<td></td>
</tr>
<tr>
<td>SCS[110]</td>
<td>142</td>
<td></td>
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<td>SCS[111]</td>
<td>180</td>
<td></td>
<td></td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>Wilson and Beck (1996)</td>
<td>SCS[100]</td>
<td>130</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SCS[110]</td>
<td>161</td>
<td></td>
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<td></td>
</tr>
<tr>
<td>Brantley (1983)</td>
<td>SCS[100]</td>
<td>130</td>
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<td></td>
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<td></td>
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<tr>
<td>SCS[110]</td>
<td>169</td>
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<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>SCS[111]</td>
<td>188</td>
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<td></td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>Bulk Si</td>
<td></td>
<td>11</td>
<td>220</td>
<td>0.05</td>
<td>0.02</td>
<td></td>
<td>Gupta and Bhushan (1995),</td>
</tr>
<tr>
<td>Cathodic arc carbon</td>
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<td>38</td>
<td>300</td>
<td>0.15</td>
<td>12.5</td>
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<td>Substrate: polished SCS[100]</td>
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<tr>
<td>Ion-beam carbon</td>
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<td>19</td>
<td>150</td>
<td>0.08</td>
<td>1.5</td>
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<td></td>
</tr>
<tr>
<td>PECVD carbon</td>
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<td>140</td>
<td>0.13</td>
<td>0.6</td>
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<tr>
<td>Sputtered carbon</td>
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<td>140</td>
<td>0.13</td>
<td>2</td>
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<td>Sputtered SiC</td>
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<td>255</td>
<td>0.15</td>
<td>1.8</td>
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<tr>
<td>Koinkar and Bhushan (1997)</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>undoped Si[100]</td>
<td></td>
<td>12.6</td>
<td>136</td>
<td>0.05</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>p+ type Si[100]</td>
<td></td>
<td>6.3</td>
<td>73.1</td>
<td>0.05</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>Bulk polysilicon</td>
<td></td>
<td>11</td>
<td>135</td>
<td>0.05</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>n+ type polysilicon</td>
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<td>122</td>
<td>0.07</td>
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</tr>
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</table>

Table 2.1. Summary of previous experimental investigations on various types of silicon.
<table>
<thead>
<tr>
<th></th>
<th>Berkeley – Cantilever bending</th>
<th>Caltech - Tension</th>
<th>FAA – Notched bending</th>
<th>Hopkins - Tension</th>
<th>Hopkins - Tension</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness (µm)</td>
<td>1.9</td>
<td>1.9</td>
<td>2.0</td>
<td>1.5</td>
<td>3.5</td>
</tr>
<tr>
<td>Number tested</td>
<td>90</td>
<td>3</td>
<td>12</td>
<td>19</td>
<td>14</td>
</tr>
<tr>
<td>Young’s Modulus (GPa)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>µ</td>
<td>174</td>
<td>132</td>
<td>137</td>
<td>136</td>
<td>142</td>
</tr>
<tr>
<td>σ</td>
<td>20</td>
<td>–</td>
<td>5</td>
<td>14</td>
<td>25</td>
</tr>
<tr>
<td>COV</td>
<td>11%</td>
<td>–</td>
<td>4%</td>
<td>10%</td>
<td>18%</td>
</tr>
<tr>
<td>Failure Strength (GPa)</td>
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<tr>
<td>µ</td>
<td>2.8</td>
<td>2.7</td>
<td>1.3</td>
<td>1.3</td>
<td></td>
</tr>
<tr>
<td>σ</td>
<td>0.5</td>
<td>–</td>
<td>0.2</td>
<td>0.2</td>
<td>0.1</td>
</tr>
<tr>
<td>COV</td>
<td>18%</td>
<td>–</td>
<td>7%</td>
<td>18%</td>
<td>8%</td>
</tr>
</tbody>
</table>

µ = the mean value.
σ = the standard deviation.
COV = the coefficient of variation.

Table 2.2. Summary of the round-robin test results of polycisicon [from Sharpe Jr. et al. (1998)].
<table>
<thead>
<tr>
<th>Microstructure Scale</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nano-scale</td>
<td>Atomic level</td>
</tr>
<tr>
<td>Micro-scale</td>
<td>Lattice defects ensembles below the grain scale</td>
</tr>
<tr>
<td>Meso-scale</td>
<td>Lattice defects ensembles at grain scale</td>
</tr>
<tr>
<td>Macro-scale</td>
<td>The sample geometry</td>
</tr>
</tbody>
</table>

Table 2.3. Summary of scales for microstructure consideration [Raabe (1998)].
<table>
<thead>
<tr>
<th>Scale (m)</th>
<th>Simulation method</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Nano-Micro Level</strong></td>
<td></td>
</tr>
</tbody>
</table>
| $10^{-10} \sim 10^{-6}$ | - Metropolis Monte-carlo Method  
- Cluster variation method  
- Ising model  
- Bragg-Williams-Gorsky model  
- Molecular field approximation  
- Molecular dynamics                                                 |
| $10^{-12} \sim 10^{-8}$ | • ab-initio molecular dynamics                                                                                                                 |
| **Micro-Meso Level**                                                                                                                                 |
| $10^{-10} \sim 10^{-0}$  | • cellular automata                                                                                                                               |
| $10^{-7} \sim 10^{-2}$  | • spring models  
- vertex models, network models, grain boundary dynamics  
- geometrical, topological and component models |
| $10^{-9} \sim 10^{-4}$  | • dislocation dynamics                                                                                                                           |
| $10^{-9} \sim 10^{-5}$  | • kinetic Ginzburg-Landau type phase field models  
- multistate kinetic Potts models                                                                                       |
| **Meso-Macro Level**                                                                                                                                 |
| $10^{-5} \sim 10^{-0}$  | • large-scale finite element, finite difference, linear iteration, and boundary element methods                                               |
| $10^{-6} \sim 10^{-0}$  | • crystal plasticity finite element models, finite elements with advanced constitutive laws considering microstructure  
- Taylor-Bishop-Hill, relaxed constraints, Sachs, Voigt and Reuss models, Hashin-Shtrikman model, Eshelby and Kr  
ner type self-consistent models |
| $10^{-8} \sim 10^{-0}$  | • cluster models                                                                                                                               |
| $10^{-10} \sim 10^{-0}$ | • percolation models                                                                                                                              |

Table 2.4. Summary of appropriate simulation methods relative to the length scale for microstructure consideration [Raabe (1998)].
Figure 2.1. Typical process flow of surface micromachining for a polysilicon cantilever beam on aluminum base [from Bustillo et al. (1998), Spearing (2000)].
Figure 2.2. Typical example of bulk micromaching. Polysilicon membrane is constructed as a result (from Kovacs et al. (1998), Spearing (2000)).
Figure 2.3. Typical process flow of the LIGA process (from Spearing (2000)) for the construction of a rotating Ni wheel on the metal layers.
Figure 2.4. The number of failures at different stress levels for the beams with (a) [011] and (b) [001] plane normal texture (compiled from Johansson et al. (1988)).
Figure 2.5. Schematic of a bending test apparatus (from Jones et al. (1996)).
Figure 2.6. Schematic of the bending test specimen and the loading directions used by Wilson and Beck (1996).
Figure 2.7. Schematic of the interferometric strain/displacement gage (ISDG) [Sharpe Jr. et al. (1997)].
Figure 2.8. The tensile test specimen of Sharpe Jr. et al. (1997).
Figure 2.9. The tensile test specimen of Greek and Johansson (1997).
Figure 2.10. Schematics of the electrostatic gripping for tensile tests [Tsuchiya et al. (1998)]: (a) initial configuration, (b) electrostatic attraction during the loading stage and (c) electrostatic repulsion after the material failure between the loading probe and the specimen.
Figure 2.11. Tensile test specimen shape and dimension [Tsuchiya et al. (1998)].
Figure 2.12. Schematic of the integrated (gripping and loading tip) tensile specimen [Sato et al. (1998)].
Figure 2.13. Integrated test apparatus for fracture test [Ballarini et al. (1999)].
Figure 2.14. Results from the Round-Robin Test for Polysilicon; (a) Young’s modulus and (b) Strength (compiled from Sharpe Jr. et al. (1998)).
CHAPTER 3

FRAMEWORK FOR THE SIMULATION
OF THE POLYCRYSTALLINE THIN-FILMS

This chapter outlines the framework of the adopted Monte-Carlo simulation method for the prediction of the statistically scattered elastic properties.

The experimental procedure for the mechanical properties measurement starts with the specimen preparation as shown on the top of Figure 3.1. The test apparatus setup and measurement are the next steps. The statistical treatment and analysis of measured quantities complete the experimental procedure. Analogous to this experimental process, the statistical approach employed is illustrated step by step at the bottom of Figure 3.1. The microstructure evolution simulation takes the role of the specimen fabrication in experiments. FE mesh generation on grains and the input file generation correspond to the test apparatus setup stage. The calculation of elastic properties/responses is equivalent to the experimental measurement. The statistical treatment of obtained data is common.

The overall flowchart of the simulation is shown in Figure 3.2, which shows that the simulation is consisted of five major tasks (modules):

(1) kinetics parameter extraction,

(2) microstructure evolution simulation,
(3) mesh generation on grains,
(4) FE analysis and
(5) checking for a statistical convergence.
Steps (2) through (4) form one simulation iteration and give one calculated set of elastic properties for the reconstructed microstructure for the current iteration. After collecting pre-defined number of calculation results (20-30 sets of sampling group), the decision to continue or stop a further iteration is made by the statistical convergence check in step (5). There are two possible iteration loops, one is considering only the random local orientation of each grain for the fixed microstructure (Orientation Trials, inner loop in Figure 3.2), and the other is for the number of microstructures with one set of random local orientations for each microstructure (Microstructure Trials, outer loop in Figure 3.2). The overall flow of the proposed method is briefed in this chapter, followed by detailed discussions on the major tasks in next three chapters.

3.1 Key Inputs/Outputs and Process

The key input(s)/output(s) for each task in Figure 3.2 are shown in Figure 3.3, showing input(s) in the thin solid line box and output(s) from the calculation in the thick solid line box.

When the kinetics parameters for the microstructure evolution are not known but a micrograph is available, the simulation process may start with the parameter extraction module as shown in Figure 3.3(a). For a given micrograph with known dimensions, the horizontal/vertical lengths ($L_H$ and $L_V$) and the thickness ($t$) and the scale factor ($S_c$) the
simulation domain size \((L \text{ and } M)\) is calculated directly. The growth probability, \(p_G\), is fixed to be 0.7 as a result of the simulation of the isolated grain growth. The rest of the parameters, \(p_N\) (the nucleation probability) and \(f_P\) (the fraction of the potential sites), are determined by an inverse method using the master map of the normalized number of grains, \(N'_g\), and the grain size distribution (GSD).

Figure 3.3(b) is for the microstructure reconstruction. With the parameters \((p_N, p_G, f_P, L \text{ and } M)\) are extracted as described above or given as a part of inputs, the microstructure reconstruction is performed based on; the nucleation and growth mechanism by using the characteristics of a random process, the lattice model, and the von-Neumann near neighbor criterion. The output of this microstructure evolution simulation is represented by an integer matrix \(Q\).

As shown in Figure 3.3(c), 2-D FE mesh generation is done with the geometric information in \(Q\) and the global element length parameter \((l_e)\) by using an internally developed mesh generation program by employing a Pixel-by-Pixel marching scheme and Delaunay triangulation algorithm. The nodal coordinates \((x, y)\), the element connectivity \((\text{TRI})\) and the group identity vector \((\text{GID})\) are achieved as a result.

FE input file for FE analysis is composed of; the boundary conditions, the pursuing outputs for the statistical convergence calculation, the random orientation for each grain, and the anisotropic elasticity constants of a constituent single crystal (Figure 3.3(d)). Simple FE analysis code is developed for the integration of the overall calculation process. The material model used is an anisotropic elasticity. A profile solver is used with Sloan (1986)’s profile reduction. The output(s), such as stress, strain, reaction force, displacements and so on, are calculated as a result. This pursuing output
can be a direct variable or a derived variable, such as the effective Young’s modulus under the plane stress condition.

The statistical convergence check is done for the set of outputs by using \textit{Chi-square test} (Figure 3.3(e)). The \textit{Chi-square statistic} ($\chi^2$) is calculated for the set of outputs and compares this with the cumulative probability that gives 50\% confidence interval from \textit{Chi-square distribution}. The output of this process is ‘Yes/No’ decision for the iteration to continue or not.

\textbf{3.2 Controlling the Simulation Flow}

As described at the beginning of this chapter, two basic types of trials are possible; \textit{Orientation Trials} and \textit{Microstructure Trials}, as indicated in Figure 3.2. Then, overall, three types of the simulation are possible with third one being a combination of the first two.

The first one is the simulation flow only with the orientation trials. With the multiple set of the single crystal orientation angles generated randomly, the elastic property of the fixed microstructure is calculated for each set of orientation angles until the results converge statistically. This is useful when the micrograph of a given material is known and when the statistical scatter of the aggregate elastic property is needed. den Toonder \textit{et al.} (1999) used this simulation flow to predict the effective Young’s modulus and Poisson’s ratio of the barium titanate, indium and zircon.

The second simulation flow is applying only microstructure trials. For each reconstructed microstructure, the elastic property of each grain is calculated with one set
of randomly selected single crystal orientation angles. Statistical elastic responses can be computed directly without having to find the aggregate elastic property. Each grain of polycrystalline materials can have one orientation angle, which is unknown and different from each other. Therefore, the second simulation flow, *Microstructure Trials*, seem to be a better choice to address the reliability issues directly.

The third flow of analysis, a combination of the first two, is more interesting. In that, one can obtain both the deterministic elastic responses by employing the averaged aggregate (effective) elastic property and statistically scattered elastic responses directly at the same time. Some interesting comparisons are given later in Chapter 7.
Figure 3.1. Analogy between experiments and the proposed statistical approach.
Figure 3.2. Flowchart of the Monte-Carlo simulation process

\( N_1^0 = N_2^0 = 20 \) in general.

39
Figure 3.3. Key inputs/outputs and process descriptions for the each module in Figure 3.2; (a) kinetics parameter extraction, (b) microstructure evolution, (c) mesh on grains, (d) FE analysis, and (e) convergence decision.
Pixel-by-Pixel Marching Scheme
Delaunay Triangulation

Output(s)

Chi-square statistic
\[ \chi^2 = \sum_{i=1}^{n} \left( \frac{f_i - f_i^*}{f_i^*} \right)^2 \] Equation (6.3)

Compare \( \chi^2 \) to ‘a’ where
\[ \Pr(\chi^2_{n-2} < a) = 0.5 \] Equation (6.5)

\( \chi^2 < a \) : stop
or
\( \chi^2 \geq a \) : continue
CHAPTER 4

MICROSTRUCTURE EVOLUTION SIMULATION
WITH KINETICS PARAMETER EXTRACTION BY AN INVERSE METHOD

4.1 Nucleation and Growth Mechanism

A schematic of LPCVD (Low Pressure Chemical Vapor Deposition) process is shown in Figure 4.1. Single crystal substrates are located in the heated (> 600°C) tube, and the gas contains the preferred atoms flow inside this tube. The interaction between the substrate and the atom cluster with more than the critical number of atoms overcomes the activation energy barrier for the nucleation [Christian (1970), Germain et al. (1977), Germain et al. (1979), Zellama et al. (1979)], then it becomes a stable nucleus (Nucleation). The temperature determines the plane normal direction of this nucleus [Kamins (1980)], while the in-plane orientation is arbitrary. The subsequent atom cluster among the gas with more than the growth barrier energy (usually less than the nucleation energy) sticks to the previously nucleated nucleus with the same orientation (Growth). The repetition of these processes, nucleation and growth, constructs a polycrystalline thin-film.
The polycrystalline microstructure evolution mechanism described above is called *Nucleation and Growth Mechanism*. Since the nucleation and growth mechanism well explain the polycrystalline microstructure evolution, especially for the deposition process, the most of the microstructure evolution simulation techniques used this mechanism [Johnson and Mehl (1939), Meijering (1953), Hillert (1965), Louat (1974), Getis and Boots (1978), Anderson et al. (1984), Atkinson (1988), Price (1990), Erukhimovitch and Baram (1994), Erukhimovitch and Baram (1995), Fanfoni and Tomellini (1996), Van Siclen (1996), Doherty (1997), Castro et al. (1999), Pineda and Crespo (1999), Castro et al. (2000), Choi et al. (2004), Choi and Lee (2004)]. In this mechanism, two key parameters are the nucleation rate and the growth rate. The nucleation rate, $\alpha$, is expressed as following compact form [Castro et al. (2000)];

$$\alpha \propto e^{-E_n/(kT)} \quad (4.1)$$

where $E_n$ is the nucleation activation energy, which is also called Gibbs energy for the migration, $k$ is the Stephan-Boltzmann constant and $T$ is the absolute temperature. The nucleation activation energy is represented with the enthalpy ($\Delta h_m$) and entropy ($\Delta S_m$) of the nucleation as [Christian (1970)];

$$E_n = \Delta h_m - T\Delta S_m. \quad (4.2)$$

The critical number of atoms of a stable nucleus, $N_{cr}$, is defined by [Zellama et al. (1979)];

$$N_{cr} = \left( \frac{2\Delta G_c}{\Delta g'} \right)^{2/3} \quad (4.3)$$
where $\Delta G_c$ and $\Delta g'$ are the Gibbs energies of the crystallite with the critical size and for crystallization, respectively. The general equation of the critical size, $D_{cr}$, can be expressed as:

$$D_{cr} = \sqrt[3]{\frac{N_{cr}}{N_0}} V_0$$

(4.4)

where $N_0$ is the number of lattices per unit cell and $V_0$ is the volume of the unit cell of a single crystal. Table 4.1 summarizes these relations for various crystallographic structures.

The growth rate, $G$, is depend on the growth barrier energy ($E_g$) as [Castro et al. (2000)];

$$G \propto e^{-\frac{E_g}{kT}}.$$  

(4.5)

The cessation of the growth, or impingement, produces the grain boundary.

The location of new nucleation site is chosen according to the acceptance/reject condition as shown in Figure 4.2. If the candidate location of the new nucleation is inside the radius of the previously nucleated grain growth, then the new nucleation is rejected, otherwise it is accepted.

4.2 The Lattice Model

The embodiment of the nucleation and growth mechanism for the microstructure evolution simulation is done by using the lattice based model [Castro et al. (2000)] in this report. There are two types of the lattice system with respect to the crystallographic structure of the concerning material, i.e., the triangular and rectangular lattice systems.
The materials with the monoclinic, hexagonal and trigonal crystallographic structure can be modeled by the triangular lattice system, and the materials with the cubic, tetragonal and orthorhombic crystallographic structure can be modeled by the rectangular lattice system. However, since both lattice systems give the same microstructure by means of two major quantitative measures of the microstructure [Castro et al. (2000)], the number of grains and the grain size distribution (GSD), only the rectangular lattice system is used in this report.

A simulation domain can be defined by $L \times M$ uniform rectangular lattices and $L \times M$ pixels. Each pixel contains one or more stable nuclei, of which size is defined by the critical number of atoms given in equation (4.3). The actual size of one pixel, $L_p$, is defined in terms of a scale factor as $S_c = \frac{L_p}{D_{cr}}$. In turn, the size of an actual micrograph is;

$$
L_H = L_p L = S_c D_{cr} L
$$
$$
L_V = L_p M = S_c D_{cr} M
$$

where $L_H$ and $L_V$ are the horizontal and vertical length, respectively, of a micrograph to be simulated. The kinetics parameters for the lattice model are;

- the fraction of the potential sites, $f_P$ ($0 < f_P \leq 1.0$)
- the nucleation probability, $p_N$ ($0 < p_N \leq 1.0$)
- the growth probability, $p_G$. ($0 < p_G \leq 1.0$)

The fraction of the potential sites ($f_P$) represents the fraction of possible nuclei in the whole domain. For an example, if $f_P = 1.0$, then the whole domain pixels are
considered to be possible sites for nucleation, i.e., a homogeneous nucleation. Otherwise, it is a heterogeneous nucleation condition, which is closer to the real situation.

The parameters ($\alpha$ and $G$) in equations (4.1) and (4.5) are assumed to be constant, and can be combined as:

$$\alpha' = \alpha / G \approx e^{-(E_a - E_f)/(kT)}, \quad (4.7)$$

and $G$ is set to be 1.0, i.e. existing nuclei grow by one unit, one pixel in the lattice model, at a simulation time step ($\tau$). In the lattice model, $\alpha'$, in equation (4.7), is replaced with $p_N$ and $f_P$ as;

$$p_N = \frac{\alpha'}{f_PLM}. \quad (4.8)$$

Zellama et al. (1979) showed that $\alpha$ and $G$, which are related to $p_N$ and $f_P$, can be determined by experiments with some assumptions and a priori theories for germanium and silicon. However, this experimental approach is not trivial due to the difficulties associated with the in-situ measurement and the long time duration of the deposition process. In order to relieve the difficulties and time drag of this experimental procedure of the kinetics parameters extraction, an inverse method of this extraction is proposed based on the quantitative measures of the given microstructure.

4.3 Kinetics parameters Extraction – Inverse Method

The key parameters for the microstructure simulation are the domain size ($L, M$), $p_N$ and $f_P$. The domain size is determined by the specimen size and the resolution of the simulation. The fraction of the potential sites is the property of the substrate. Since $p_N$ and
\( f_p \) is related to \( \alpha' \) (\( = \alpha/G \)) as shown in equation (4.8), \( \alpha \) and \( G \) should be determined prior to set up the parameters for the microstructure simulation.

The proposed inverse method to extract \( p_N \) and \( f_p \) is based on major quantitative measures of a microstructure as schematically illustrated in Figure 4.3. For a given micrograph of a microstructure with known dimensions, two major quantitative measures of the microstructure, the number of grains \( (N_g) \) and GSD, are extracted by an image processing technique. Then, the kinetics parameters, \( p_N \) and \( f_p \), extraction is possible by using the mapping methods for these two measures, \( N_g \) and GSD, as discussed below.

The number of grains, \( N_g \), is directly related to the kinetics parameters and for the continuous nucleation case, it is given by [Getis and Boots (1978)];

\[
N_g = c_{g1}(\alpha')^{2/3}
\]  

(4.9)

where \( c_{g1} \) is a constant. Equations (4.8) and (4.9) give;

\[
N_g = c_{g2} \left( p_N f_p L M \right)^{2/3}
\]  

(4.10)

where \( c_{g2} \) is a constant related to the growth probability \( (p_G) \). However, the number of grains calculated by equation (4.10) does not give correct estimation for all nucleation and growth conditions. Therefore, a parametric study of the number of grains with respect to \( p_N \) and \( f_p \) is accomplished to obtain a relationship between the number of grains and the kinetics parameters. A master map for the number of grains is achieved as a result and shown in Figure 4.4.

GSD is a key factor for the proposed method. Previous researchers focused on two extreme cases for the nucleation conditions; site-saturation and continuous nucleation.
The empirical equation of GSD for the site-saturation case is given by [Weaire et al. (1986)];

$$P(A') = \frac{\beta_n^\beta_n}{\Gamma(\beta_n)} A'^{\beta_n-1} e^{-\beta_n A'},$$  \hspace{1cm} (4.11)

where $A'$ is the reduced grain size, $\Gamma$ is the gamma function and $\beta_n = 3.65$. GSD for the continuous nucleation is [Mulheran (1994)];

$$P(A') = e^{-A'}.$$  \hspace{1cm} (4.12)

It is noted that equation (4.12) is a special case of equation (4.11) when $\beta_n=1.0$. Hence, $\beta_n$ is taken as “Nucleation Speed Parameter (NSP).” In the viewpoint of the nucleation speed, the site-saturation is the fastest and the continuous nucleation is the slowest nucleation condition. The parameter $\beta_n$ has the range of 3.65~1.0 in this investigation. In Figure 4.5, the thick solid line represents GSD of equation (4.11) with $\beta_n=3.65$ and as $\beta_n$ decreases the graph approaches to the distribution of equation (4.12).

With the fact that $\beta_n=3.65$ when $p_N=1.0$, and $\beta_n=1.0$ when $f_P=1.0$ [Weaire et al. (1986), Mulheran (1994)], the quadratic mapping is assumed between the normalized length of the iso-number of grains curve from the point of $f_P=1.0$ and the target value of $\beta_n$ by employing a length function between two points, $Len()$, which is expressed as;

$$Len(P_1, P_s) = Len(P_f, P_s) \left[ 1.0 + \sqrt{\frac{3.65 - \beta_1}{2.65}} \right]$$  \hspace{1cm} (4.13)

where $\beta_1$ is the target $\beta_n$, $P_1$ is the seeking point on the iso-number of grains curve, $P_f$ is the point on the iso-number of grains curve where the nucleation is the fastest, i.e., $p_N=1.0$, and $P_s$ is the point where the nucleation is the slowest, i.e., $f_P=1.0$. Figure 4.6
shows the relation between the normalized distance from the point where \( f_p = 1.0 \) and \( \beta_n \).
The solid curve represents the equation (4.13) and the filled circles are data points of the mean values of \( \beta_n \) as a result of 30 microstructure evolution simulations. The assumed quadratic mapping is reasonable.

The kinetics parameters extraction procedure is summarized as follows;

1. Calculate the normalized target number of grains as;
   \[
   N'_g = \frac{N_g}{LM} .
   \]  (4.14)
2. Pull out the iso-number of grains curve by using the master map (Figure 4.4).
3. Calculate the total length of the iso-number of grain curve and select one pair which satisfies equation (4.13).

4.4 Microstructure Evolution Algorithm

The microstructure evolution simulation is summarized as follows;

(Step 1) Set up the simulation domain.

(Step 2) Initialize the matrices for the potential sites (P) and the microstructure domain (Q).

(Step 3) Update P and Q at every simulation time step (τ) until the transformation fraction reaches the expected value.

Each step is further illustrated below.
4.4.1 Step 1: Set up the simulation domain

The domain size, the integer values of $L$ and $M$ are determined by the size of a specimen $L_H$ and $L_V$, and one domain pixel, $L_p$. From equations (4.4) and (4.6);

\[
L = \text{int} \left( \frac{L_H}{D_1} \right) \\
M = \text{int} \left( \frac{L_V}{D_1} \right)
\]

(4.15)

where \text{int}( ) is the integer function which converts the real value into integer, and

\[
D_1 = S_c \sqrt[3]{N_{cr} V_0 / N_0}.
\]

(4.16)

4.4.2 Step 2: Initialize $P$ and $Q$

The dimensions of two matrices, $P$ and $Q$, are now both $L$ by $M$. Initially $Q$ is zero-matrix and $P$ is the matrix with $f_p \times L \times M$ non-zero elements. The selection of the non-zero elements of $P$ is done by using the characteristic of the random numbers (see Appendix A), i.e., the distribution uniformity which can be expressed as;

\[
\Pr(x, x \leq c; \ c \in [0,1]) = c \\
\Pr(x, c_1 \leq x \leq c_2; \ c_1, c_2 \in [0,1] \text{ and } c_1 < c_2) = c_2 - c_1
\]

(4.17)

when $x$ is a set of the random numbers in the range of $[0, 1]$, $\Pr(x)$ is the probability function and $c$, $c_1$ and $c_2$ are real numbers in $[0, 1]$.

The selection of a fraction of elements by using equation (4.17) is $O(N)$ computation complexity as shown in Figure 4.7. So, the selection method proposed in this report is very efficient relative to the other method, such as the method based on the element sorting.
4.4.3 Step 3: Update P and Q

At each simulation time step, \( \tau \), two matrices \( P \) and \( Q \) are updated by the nucleation and growth mechanism. The following three tasks in ‘Step 3” are repeated until the fraction of the crystallization reaches the expected value. Figure 4.8 illustrates the sequence of the microstructure evolution simulation described in this section.

4.4.3.1 Selection of the nucleation sites

This process is consisted of two tasks; one is to pick up \( p_N \) portion of lattice sites from non-zero elements of \( P \), and the other is to impose the cumulatively successive grain number on each nucleation site and assign the number to the corresponding element of the matrix \( Q \).

The pick up is done by using the equation (4.17), and can be represented by the operations as:

\[
\begin{align*}
\text{Step 1} & : P_{\text{temp}} = \text{random}(L, M) \\
\text{Step 2} & : P_{\text{temp}} = P_{\text{temp}} \otimes P \\
\text{Step 3} & : P_{\text{temp}} = P_{\text{temp}} \le p_N
\end{align*}
\]

where \( P_{\text{temp}} \) is temporary \( L \) by \( M \) matrix with the random numbers generated by operation ①. In the operation ②, the symbol ‘\( \otimes \)’ means the element by element multiplication of two matrices. With the operation ③, \( P_{\text{temp}} \) now has the value 1 when the original value is equal to or less than \( p_N \) at a particular location and other elements are set to zeros.

Since the updated \( P \) matrix, discussed in 4.4.3.3, has non-zero elements on the locations where the values of the current \( Q \) matrix are zeros, the acceptance/reject
condition, in 4.1, of the nucleation is inherently satisfied. After the pick up, the corresponding lattice sites of \( P \) are set to be zero.

4.4.3.2 Calculation of the grain growth

The tasks involved in the calculation of the grain growth are; (i) calculation of the periphery lattice sites for the current \( Q \) matrix, (ii) selection of the growth lattice sites, and (iii) updating \( Q \) matrix. Finding the periphery lattice sites for the current \( Q \) matrix is completed with 15 simple matrix manipulations, which are based on von-Neumann neighbor criterion. The selection of the growth lattice sites is also performed by operation \( \odot \) of (4.18) by substituting \( Q_{\text{peri}} \), the matrix of the periphery lattice sites, and \( p_G \) in places of \( P_{\text{temp}} \) and \( p_N \), respectively.

4.4.3.3 Update of the matrix \( P \)

The potential sites can be covered by the growth of the previously nucleated grains. So, the matrix \( P \) should be updated right after the grain growth calculation is completed. This process is done by the logical NOT operation and the element by element multiplication of two matrices as;

\[
P = P \otimes (\sim Q)
\]  

(4.19)

where the symbol ‘\( \sim \)’ represents the logical NOT operation, which converts the non-zero elements to zeros and the zero elements to ones simultaneously.
4.5 Microstructure Evolution Simulation

The microstructure reconstruction is performed to validate the microstructure simulation algorithm and the proposed kinetics parameter extraction for given micrographs shown in Figure 4.9(a), Figure 4.10(a) and Figure 4.11(a). The main purpose of the microstructure reconstruction is to extract kinetics parameters in a statistically equivalent way.

The microstructure shown in Figure 4.9(a), Figure 4.10(a) and Figure 4.11(a) has 57 grains in 546 × 410 µm [den Toonder et al. (1999)], 183 grains in 538×315 µm [Ilzhofer et al. (1997)] and 108 grains in 100×100 µm [Mahin et al. (1980)], respectively. By assuming that the microstructure is for the polysilicon thin-film and following the procedure described in Section 4.3, the simulation parameters summarized in Table 4.2 are retrieved.

Table 4.3 summarizes the results, found to be statistically equivalent, from samples of 100 microstructure evolution simulations. The number of grains for each micrograph by the simulation shows a very good agreement with each micrograph given. The values of $\beta_n$ are well correlated, thus, the microstructure simulation model and algorithm for the kinetics parameters extraction proposed in this report are verified. Typical examples of the reconstructed microstructure by the simulation are shown in Figure 4.9(b), Figure 4.10(b) and Figure 4.11(b).

The computation efficiency, one of the most important factors for the statistical simulation approach, is discussed next. The computation time against the domain size (with the same number of grains) and against the number of grains (with the same
domain size) are considered. Four domain sizes, 100×100, 200×200, 300×300 and 400×400 with 10 grains are used for the domain size effect. And 10, 20, 30, 40 and 50 grains in 100×100 domain are used for the number of grains effect. The results are shown in Figure 4.12. While the computation time increases as the total number of pixels in a domain increases with \( O(N^2) \) where \( N \) is the number of pixels in a domain, the increase of the number of grains requires less computation time. The less computation time for the increased grains is another advantage of the lattice based microstructure simulation.
<table>
<thead>
<tr>
<th>Lattice Type</th>
<th>Lattice per Unit Volume ($N_o$)</th>
<th>Unit Volume ($V_o$)</th>
<th>$D_c$, formula (equation (4.4))</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Cubic</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$a = b = c$</td>
<td>I</td>
<td>2</td>
<td>$a^3$</td>
</tr>
<tr>
<td>$\alpha = \beta = \gamma = 90^\circ$</td>
<td>I</td>
<td>2</td>
<td>$\sqrt[3]{0.5N_c a^3}$</td>
</tr>
<tr>
<td>$a = b = c$</td>
<td>F</td>
<td>4</td>
<td>$a^3$</td>
</tr>
<tr>
<td>$\alpha = \beta = \gamma = 90^\circ$</td>
<td>F</td>
<td>4</td>
<td>$\sqrt[3]{0.25N_c a^3}$</td>
</tr>
<tr>
<td><strong>Tetragonal</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$a = b \neq c$</td>
<td>I</td>
<td>2</td>
<td>$a^2 c$</td>
</tr>
<tr>
<td>$\alpha = \beta = \gamma = 90^\circ$</td>
<td>I</td>
<td>2</td>
<td>$\sqrt[3]{0.5N_c a^2 c}$</td>
</tr>
<tr>
<td>$a = b \neq c$</td>
<td>F</td>
<td>4</td>
<td>$abc$</td>
</tr>
<tr>
<td>$\alpha = \beta = \gamma = 90^\circ$</td>
<td>F</td>
<td>4</td>
<td>$\sqrt[3]{0.25N_c abc}$</td>
</tr>
<tr>
<td><strong>Orthorhombic</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$a \neq b \neq c$</td>
<td>I</td>
<td>2</td>
<td>$abc$</td>
</tr>
<tr>
<td>$\alpha = \beta = \gamma = 90^\circ$</td>
<td>I</td>
<td>2</td>
<td>$\sqrt[3]{0.5N_c abc}$</td>
</tr>
<tr>
<td><strong>Monoclinic</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$a \neq b \neq c$</td>
<td>P</td>
<td>1</td>
<td>$abc \sin \beta$</td>
</tr>
<tr>
<td>$\alpha = \gamma = 90^\circ, \beta \neq 120^\circ$</td>
<td>C</td>
<td>2</td>
<td>$\sqrt[3]{0.25N_c abc \sin \beta}$</td>
</tr>
<tr>
<td><strong>Hexagonal</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$a = b \neq c$</td>
<td>P</td>
<td>1</td>
<td>$\frac{1}{2} \sqrt{3} a^2 c$</td>
</tr>
<tr>
<td>$\alpha = \beta = 90^\circ, \gamma = 120^\circ$</td>
<td>P</td>
<td>1</td>
<td>$\sqrt[3]{N_c \frac{1}{2} \sqrt{3} a^2 c}$</td>
</tr>
<tr>
<td><strong>Trigonal</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$a = b = c$</td>
<td>P</td>
<td>1</td>
<td>$a^3 \sin \beta \sin \gamma$</td>
</tr>
<tr>
<td>$\alpha = \beta = \gamma \neq 90^\circ$</td>
<td>P</td>
<td>1</td>
<td>$\sqrt[3]{N_c a^3 \sin \beta \sin \gamma}$</td>
</tr>
</tbody>
</table>

Table 4.1. Summary of the crystallographic structure and their characteristics.
Table 4.2. Summary of extracted simulation parameters for given microstructures by the inverse method [Choi et al. (2004), Choi and Lee (2004)]. Microstructure #1, #2 and #3 are from den Toonder et al. (1999), Ilzhofer et al. (1997) and Mahin et al. (1980), respectively.

<table>
<thead>
<tr>
<th>Simulation Domain</th>
<th>Microstructure #1 (Al₂O₃) [den Toonder et al. (1999)]</th>
<th>Microstructure #2 (Nickel) [Ilzhofer et al. (1997)]</th>
<th>Microstructure #3 (Fe-Si) [Mahin et al. (1980)]</th>
</tr>
</thead>
<tbody>
<tr>
<td>$L \times M$</td>
<td>306×230</td>
<td>477×280</td>
<td>300×300</td>
</tr>
<tr>
<td>$N_{cr}$</td>
<td></td>
<td>100 [Zellama et al. (1979)]</td>
<td></td>
</tr>
<tr>
<td>$D_{cr}$</td>
<td></td>
<td>1.5877 nm (equation 4.4)</td>
<td></td>
</tr>
<tr>
<td>$S_{c}$</td>
<td></td>
<td>1127</td>
<td>711.88</td>
</tr>
<tr>
<td>Kinetics Parameters ($p_G=0.7$)</td>
<td>$p_N$</td>
<td>3.4085×10⁻⁵</td>
<td>5.7705×10⁻⁵</td>
</tr>
<tr>
<td></td>
<td>$f_{P}$</td>
<td>0.9358</td>
<td>0.9491</td>
</tr>
</tbody>
</table>

Table 4.3. Summary of the microstructure simulation results for the reconstruction of given microstructures.

<table>
<thead>
<tr>
<th>Microstructure #1</th>
<th>Microstructure #2</th>
<th>Microstructure #3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Micrograph</td>
<td>Simulation</td>
<td>Micrograph</td>
</tr>
<tr>
<td>$N_G'$</td>
<td>$1.826\times10^3$ (57 grains)</td>
<td>$1.820\times10^3$ (56.8 grains)</td>
</tr>
<tr>
<td>$\beta_n$</td>
<td>1.143</td>
<td>1.146</td>
</tr>
</tbody>
</table>
Figure 4.1. Schematic of Low Pressure Chemical Vapor Deposition (LPCVD) process.
Figure 4.2. Schematic of the condition to accept or reject a new nucleation.
Figure 4.3. Schematic of the proposed inverse method for kinetics parameter extraction.
Figure 4.4. Master map of the number of grains with respect to $p_N$ and $f_P$. Numbers on the contour are the normalized number of grains calculated by equation (4.14).
Figure 4.5. The grain size distribution plots for various $\beta_n$ in equation (4.11).
Figure 4.6. The illustration of the quadratic mapping between the nucleation speed parameter, $\beta_n$, and the normalized distance from the slowest nucleation point of the iso-number of grain curve.
Figure 4.7. Computation time for 10% random pick relative to the total number of pixels.
Figure 4.8. Microstructure evolution process; (a) at initial stage, and when (b) 30%, (c) 60% and (d) 90% of domain is crystallized. The gray background represents the region not crystallized yet.
Figure 4.9. The binary image of (a) the given microstructure #1 (Al₂O₃) [from den Toonder et al. (1999)] and (b) the reconstructed microstructure by the microstructure evolution simulation.
Figure 4.10. The binary image of (a) the given microstructure #2 (Nickel) [Ilzhofer et al. (1997)] and (b) the reconstructed microstructure by the microstructure evolution simulation.
Figure 4.11. The binary image of (a) the given microstructure #3 (Fe-Si) [Mahin et al. (1980)] and (b) the reconstructed microstructure by the microstructure evolution simulation.
Figure 4.12. The computation time versus (a) the domain size with 10 grains and (b) the number of grains in 100×100 domain.
CHAPTER 5

FINITE ELEMENT MESH GENERATION
AND FINITE ELEMENT ANALYSIS

It is desirable to use a computationally reconstructed microstructure directly to
determine elastic responses or/and effective elastic property. Coupling the proposed
microstructure simulation with the FE analysis is exposed in this chapter. A method to
generate a quasi-uniform FE mesh on a simulated micrograph is developed.

5.1 Finite Element Meshing Process

One of the results from the simulation is a matrix, \( Q \), containing integer values
corresponding to the grain numbers as shown in Figure 5.1. In order to generate a FE
mesh on grains, vertex points and the grain boundaries must be recognized first to make
sure that an element does not cross a grain boundary.

5.1.1 Extraction of the Vertices and Grain Boundaries

Grain boundaries and vertex points where two or more grain boundaries meet
must be defined geometrically first. A vertex may belong to; (1) the domain corners, (2)
the domain boundaries and (3) the open simulation domain. The domain corners are natural vertices and trivial to identify. Vertex points on the domain boundaries can be found by searching pixels whose adjacent pixels on the domain boundary have different integer numbers, belonging to different grains. Then a vertex point is located on the middle of these two pixels. A vertex inside the domain is the middle point which has three or more different values within 2x2 pixel search windows surrounding the particular pixel in question, as illustrated in Figure 5.2.

The domain boundaries are the natural grain boundaries. A grain boundary inside the domain can be located by identifying the pixels that have different grain numbers from the adjacent pixels. The solid lines in Figure 5.1 indicate the identified grain boundaries. When all the grain boundaries are defined by pixels, nodes can be placed in a quasi-uniform fashion over the simulation domain and must be grouped by each grain.

5.1.2 Locating Nodes

There are 3 types of nodes to be located; (1) inside a grain, (2) on the grain boundaries, and (3) on the vertices. The location of the nodes on the vertices is simply done by imposing nodal coordinate information on the vertices, which are calculated as described in previous section. The equidistant positioning of nodes inside the grains accomplishes the node location inside each grain. While the nodes on the vertices can be shared by up to four grains, the nodes inside a grain belong to only one grain.

In order to get the nodes on the grain boundaries, the grain boundaries must be recognized first. A grain boundary consists of piecewisely linear edges with two vertices. All edges must be ordered in a consistent way for each grain for correct element grouping.
The best way of doing this is arranging the vertices of each grain in counter-clockwise or clockwise, relative to a reference point, e.g., center of a grain. The counter-clockwise numbering scheme is employed to be in line with the usual FE node numbering scheme. Two vertices can define one or two edges as shown in Figure 5.3. The grain with only two vertices, as shown in Figure 5.3 (b), is called *lens*.

The first step of this edge finding procedure is to extract the grain boundary pixels and order those pixels in the counter-clockwise direction. The grain boundary extraction is done by using the neighborhood criteria; von Neumann’s or Moore’s neighborhood criteria [Weisstein (2004, vonNeumann)] (see Figure 5.4). Since the Moore’s neighborhood criterion gives redundant pixels for the grain boundary extraction as shown in Figure 5.5 (c), von Neumann’s neighborhood criterion is used in this dissertation. The ordering of the extracted grain boundary pixels is done by using a *Pixel-by-Pixel Marching Scheme* as follows.

With a starting pixel located closest to the first vertex of the grain, the next pixel is located to be the closest one to the starting pixel in clockwise direction, relative to the vector from the first vertex to the starting pixel. The subsequent pixels are selected by searching pixel by pixel as shown in Figure 5.6. There are eight possible cases for the current and previous pixels configurations. Initially, the starting pixel is the ‘previous’ and the second pixel is the ‘current’ pixel. In each case, the number in the box is the priorities of the selection of the next pixel, i.e., if one grain boundary pixel exists on the position labeled with “1”, then that pixel is selected as the next one, otherwise proceed to check next priority. The correct order of the grain boundary pixels is achieved by repeating this until all grain boundary pixels for each grain are processed. The grain
boundary pixels for each edge can be found by ordering the vertices for each grain and finding the closest grain boundary pixels to each vertex.

The preliminary arrangement of the vertices is done by sorting the angles of each vertex relative to the reference line, $\theta_i$, as shown in Figure 5.7, and can be represented as:

$$\theta_i = \text{sgn}(v_i \cdot v_i + v_i, v_i) \cos^{-1}
\begin{pmatrix}
\frac{v_i \cdot v_i}{|v_i||v_i|}
\end{pmatrix}
$$

where

- $\text{sgn}$: sign function
- $v_i$: the reference vector
- $v_i$: the $i$th vector (vector from the center of grain to the $i$th vertex)
- $v_i, v_i, v_i, v_i$: $x$ and $y$ components of the reference and the $i$th vectors
- $n_v$: the number of vertices

Rearrangement is needed only when the preliminary arrangement has any problem. Since a grain is not necessarily a convex polygon nor star-shaped [Getis and Boots (1978), Preparata and Shamos (1985)], this preliminary arrangement becomes wrong under certain circumstances. For example, in the case of Figure 5.8, the preliminary arrangement gives “1-2-3-4-5-6-7” as the proper order of vertices based on equation (5.1). However, this arrangement provides wrong information about the edges of the grain, e.g., the edges 2-3 and 7-1 are not actual edges. Instead, 7-2, 2-1 and 1-3 are actual edges. To check this systematically, a normalized vector from $\text{fore}$- to $\text{aft}$-vertices from the preliminary ordering would be necessary;
\[ \hat{v}_{re} = \frac{v_{re}}{\left| v_{re} \right|}, \]

\[ v_{re} = \begin{bmatrix} x_{v_{af}} \\ y_{v_{af}} \\ 0 \end{bmatrix} - \begin{bmatrix} x_{v_{fore}} \\ y_{v_{fore}} \\ 0 \end{bmatrix} \]  \tag{5.2}

where \( x_{v_{af}}, y_{v_{af}}, x_{v_{fore}}, y_{v_{fore}} \) are \( x \) and \( y \) coordinates of \( fore \)- and \( aft \)-vertices, respectively.

Then check whether one of four left-hand side (four right-hand side) lattice sites for each \( fore \)- (and \( aft \)-) vertices. Big circles in box (1) and (2) of Figure 5.8 (b), belong to the grain in consideration. These four check-up points are selected by:

\[ x_1 = \text{int} \left( x_{v_{af}} - y_{v_{af}} \right), \quad y_1 = \text{int} \left( y_{v_{af}} + x_{v_{af}} \right) \]

\[ x_2 = x_1, \quad y_2 = y_1 + 1 \]

\[ x_3 = x_1 + 1, \quad y_3 = y_1 \]

\[ x_4 = x_1 + 1, \quad y_4 = y_1 + 1 \]  \tag{5.3}

for \( fore \)-vertex, and:

\[ x_1 = \text{int} \left( x_{v_{af}} - y_{v_{af}} \right), \quad y_1 = \text{int} \left( y_{v_{af}} + x_{v_{af}} \right) \]

\[ x_2 = x_1, \quad y_2 = y_1 + 1 \]

\[ x_3 = x_1 + 1, \quad y_3 = y_1 \]

\[ x_4 = x_1 + 1, \quad y_4 = y_1 + 1 \]  \tag{5.4}

for \( aft \)-vertex. \((x_1, y_1)\) in equation (5.3) gives the integer valued position of \( v_{re} \) rotated 90° counter-clockwise about \( fore \)-vertex, and \((x_1, y_1)\) in equation (5.4) gives the integer valued position of \( v_{re} \) rotated 90° clockwise about \( aft \)-vertex. The box (1) in Figure 5.8 (b) shows that all check-up points lie on the out-side of the considering grain, in turn a wrong arrangement which should be switched, while the box (2) shows that the right arrangement of vertices. Therefore, the final arrangement of vertices for this grain is “1-3-4-5-6-7-2.”
The closest grain boundary pixel for each vertex becomes the end points of the grain edges. The nodes location on the grain boundaries can be accomplished by positioning equidistant points on the grain boundary pixels between these end points of each edge. Every node on the grain boundaries belongs to two grains, which share the corresponding grain boundary, except for the grain boundaries on the simulation domain.

5.1.3 Triangular Element Generation with Delaunay Triangulation

The triangular element generation is performed by using Delaunay triangulation algorithm with the located nodes as described in 5.1.2. For the Delaunay triangulation, the algorithm implemented in MATLAB™, which is based on the qhull algorithm (http://www.thesa.com/software/hull), is used in this dissertation.

Since the Delaunay triangulation is related to the circumcircles of the nearest neighbor and the element edge should not cross the grain boundary, the distance between two nodes on the grain boundaries should be less than the minimum distance between two nodes, one node on the grain boundary and the other, which is not on the grain boundary. This caution is described again in 5.1.5 for the mesh refinement.

5.1.4 Grouping Nodes and Elements

The located nodes by the process described in 5.1.2 have the information about the corresponding grains. Author defines this grain information for each node as group ID of nodes. The nodes inside each grain and on the domain corners belong to only one grain, so that they have only one group ID. The nodes on the grain boundaries have two
group IDs except grain boundaries on the domain boundary, which have one group ID. And the nodes on the vertex points can have up to four group IDs.

Each element consists of three nodes, of which the types are described in 5.1.2. Those are (1) nodes inside the grain, (2) nodes on the grain boundary and (3) nodes on the vertices. Each element can be composed of any combinations of these three types of nodes. The number of cases of these combinations is the same as the problem of choosing $k$ entities from $n$ different symbols by allowing repetition without order significance, in other words “$k$ Multichoose $n$.” The number of choices can be calculated by [Weisstein (2004, MultiChoose)];

$$\binom{n}{k} = \binom{n+k-1}{k} = (n-1,k)!$$  \hspace{1cm} (5.5)

where

$$\binom{n}{k} \equiv \frac{n!}{(n-k)!k!}$$  \hspace{1cm} (5.6)

is a binomial coefficient and $(n-1,k)!$ is a multinomial coefficient.

Since each element chooses 3 nodes from 3 types of nodes, $n = k = 3$ in this case. There are total 10 cases of the combinations possible as follows;

Case 1: Element with nodes of types (1)(1)(1)
Case 2: Element with nodes of types (1)(1)(2)
Case 3: Element with nodes of types (1)(1)(3)
Case 4: Element with nodes of types (1)(2)(2)
Case 5: Element with nodes of types (1)(2)(3)
Case 6: Element with nodes of types (1)(3)(3)
Case 7: Element with nodes of types (2)(2)(2)
Case 8: Element with nodes of types (2)(2)(3)
Case 9: Element with nodes of types (3)(3)(3)
Case 10: Element with nodes of types (3)(3)(3)

All the cases are illustrated in Figure 5.9. The number in the middle of each element in Figure 5.9 corresponds to the Case number above of that element.

If the element is in Case 1, the group ID of any connected node is the group ID for the element. In other cases, the common group ID of three constituent nodes for each element is found and that common group ID is designated as the group ID of the element.

5.1.5 Cautions in the Finite Element Meshing

The constructed mesh system has uniform and regular shapes inside each grain, but not near the grain boundaries because of the irregularity of the grain boundaries. So, the mesh refinement is needed for more accurate FE calculation. The generally accepted criteria, area and the smallest interior angle of an element, are used for the factors of the mesh refinement. The area criterion, \( A_{cr} \), used is defined as:

\[
A_{cr} = 1.2A_{norm} = \frac{1.2}{2} l_e^2
\]

(5.7)

where \( A_{norm} \) is the area of a normal element, which can be defined as the half of the global element length squared, and \( l_e \) is defined global element length pre-defined. The interior angle criterion used is 10 degree.
This mesh refinement based on the area of the element and the interior angle near the grain boundaries enhances the numerical accuracy. However, there is the restriction, i.e., the element edge should share the grain boundary for the elements attached to the grain boundaries. For example, if the nodal positions inside a grain nearest to a grain boundary are configured as shown in Figure 5.10 (a), then the common element edge shares the grain boundary. However, if the nodal positions are as shown in Figure 5.10 (b), then the element edge crosses the grain boundary. Since the Delaunay triangulation used in this dissertation is the dual to the Voronoi tessellation and is based on the smallest circumcircle of three nearest points, the criterion for the good configuration is:

\[
\begin{align*}
R_{134} &< R_{123} \quad \text{and} \quad R_{134} < R_{124} \\
R_{234} &< R_{123} \quad \text{and} \quad R_{234} < R_{124}
\end{align*}
\]  

(5.8)

where \(R_{123}\) is the radius of a circle for the triangle 1-2-3 and defined by [Pedoe (1995)];

\[
R_{123} = \frac{\sqrt{b_x^2 + b_y^2 - 4ac}}{2|a|}
\]  

(5.9)

where

\[
a = \begin{vmatrix}
x_1 & y_1 & 1 \\
x_2 & y_2 & 1 \\
x_3 & y_3 & 1
\end{vmatrix},
\]

(5.10)

\[
b_x = \begin{vmatrix}
x_1^2 + y_1^2 & y_1 & 1 \\
x_2^2 + y_2^2 & y_2 & 1 \\
x_3^2 + y_3^2 & y_3 & 1
\end{vmatrix},
\]

\[
b_y = -\begin{vmatrix}
x_1^2 + y_1^2 & x_1 & 1 \\
x_2^2 + y_2^2 & x_2 & 1 \\
x_3^2 + y_3^2 & x_3 & 1
\end{vmatrix},
\]

(5.11)

\[
c = -\begin{vmatrix}
x_1^2 + y_1^2 & x_1 & y_1 \\
x_2^2 + y_2^2 & x_2 & y_2 \\
x_3^2 + y_3^2 & x_3 & y_3
\end{vmatrix},
\]

(5.12)
and \((x_1, y_1)\), \((x_2, y_2)\) and \((x_3, y_3)\) are coordinates of each point labeled as shown in Figure 5.10. The other radii are defined in the similar way. If the mesh refinement nodes, nodes 1 and 2 in Figure 5.10, are located on the positions which violates the criteria represented by equation (5.8), then that refinement is ignored. Figure 5.11 illustrates the example of the mesh refinement. The mesh system without the mesh refinement is shown in Figure 5.11 (a), and with the mesh refinement is shown in Figure 5.11 (b), respectively. In Figure 5.11 (b), the thick solid line and the large hollow circle represent the original mesh and the additionally generated nodes by mesh refinement, respectively.

5.1.6 Computation Complexity of the Mesh Generation

The computation efficiency is discussed here. First, the effect of the number of grains is considered with 100×100 domain as shown in Figure 5.12 (a). Each point in the graph represents the average CPU time in seconds of 100 mesh generations. The computation time for the mesh generation is linearly increased with respect to the number of grains, while the computation time for the microstructure evolution simulation is exponentially decreased with respect to the number of grains [Choi and Lee (2004)]. So, this \(O(N)\) computation complexity is advantageous for the multi-simulation. Then, the computation times relative to the domain size and the global element length are also investigated. As shown in Figure 5.12 (b), there is \(O(N^2)\) computation complexity with respect to the domain size. The computation time decreases exponentially as the global element length increasing as shown in Figure 5.12 (c). All data points in Figure 5.12 (b) and Figure 5.12 (c) are also the average value of the 100 simulations.
5.2 Finite Element Analysis

The background for FE analysis used in this dissertation is described in this section. The anisotropic elasticity material model for the single crystals is used. The local material orientation due to the plane normal texture is transformed via Euler angle representation and the in-plane orientation is transformed by general transformation operator method for the fourth order tensor. The profile solver with Sloan (1986)’s profile reduction algorithm is adopted. The constructed FE code is compared with the analytic solution and the result of commercial FE analysis package ABAQUS™.

5.2.1 Anisotropic Elasticity

The stress-strain relation of the generalized Hooke’s law is expressed as [Reddy (1997)];

\[
\sigma = C \varepsilon
\]

in tensor notation where \( \sigma \) and \( \varepsilon \) are the second order tensor of the Cauchy stress and infinitesimal strain respectively, and \( C \) is the fourth order tensor which has 81 components. Equation (5.13) can be converted to index notation as;

\[
\sigma_{ij} = C_{ijkl} \varepsilon_{kl} \quad i, j, k, l = 1, 2, 3
\]

or

\[
\sigma_i = C_{ij} \varepsilon_j \quad i, j = 1, 2, ..., 6
\]

with Kelvin-Voigt notation by the index substitutions as \( 11 \rightarrow 1, 22 \rightarrow 2, 33 \rightarrow 3, 23/32 \rightarrow 4, 13/31 \rightarrow 5 \) and \( 12/21 \rightarrow 6 \).
The number of independent components in \( \mathbf{C} \) is reduced to 21 due to the symmetry of stress, strain and \( \mathbf{C} \) itself [Kittel (1996), Reddy (1997)]. Therefore, for the case of fully anisotropic material, all 21 components are supposed to be defined. However, due to the symmetry from the crystallographic structure of a material, the number of independent constants in \( \mathbf{C} \) reduced again to 3~13, which the number is dependent upon the crystallographic structure of a material. The number of the independent components in \( \mathbf{C} \) is summarized in Table 5.1 [Kittel (1996)]. For example, there are only 3 independent components in \( \mathbf{C} \) for the cubic material, \( C_{1111}, C_{1122} \) and \( C_{2323} \) (\( C_{11}, C_{12} \) and \( C_{44} \) with Kelvin-Voigt notation).

For 2-D analysis of this dissertation, i.e. the plane stress or plane strain condition, the coordinate transformation of this \( \mathbf{C} \) is needed relative to the plane normal direction. This transformation of \( \mathbf{C} \) can be expressed as;

\[
C_{p_q,r_s} = C_{o_p,o_q} l_p l_q l_r l_s \tag{5.16}
\]

where \( l_p, l_q, l_r \) and \( l_s \) are direction cosines defined by the Euler angle representation as shown in Figure 5.13. For the plane normal vector \( V_n = (a, b, c) \), first rotate the old coordinate system through the angle \( \varphi \) around \( z \)-axis, then rotate this intermediate coordinate system \((x', y', z')\) through the angle \( \phi \) around \( x' \)-axis. The angles \( \varphi \) and \( \phi \) are given by:

\[
\varphi = \frac{\pi}{2} + \text{acos}\left(\frac{a}{\sqrt{a^2 + b^2}}\right),
\]

\[
\phi = \text{acos}\left(\frac{c}{\sqrt{a^2 + b^2 + c^2}}\right). \tag{5.17}
\]
Hence, the rotation matrix is given by [Reddy (1997)];

\[ \mathbf{R} = \mathbf{R}_2 \mathbf{R}_1 \]  

(5.18)

where

\[
\mathbf{R}_1 = \begin{bmatrix}
\cos(\phi) & \sin(\phi) & 0 \\
-sin(\phi) & \cos(\phi) & 0 \\
0 & 0 & 1
\end{bmatrix}, \quad \mathbf{R}_2 = \begin{bmatrix}
1 & 0 & 0 \\
0 & \cos(\phi) & \sin(\phi) \\
0 & -\sin(\phi) & \cos(\phi)
\end{bmatrix}.
\]  

(5.19)

Therefore, the direction cosines in equation (5.16) are expressed as;

\[
\mathbf{L} = \begin{bmatrix}
l_{11} & l_{12} & l_{13} \\
l_{21} & l_{22} & l_{23} \\
l_{31} & l_{32} & l_{33}
\end{bmatrix} = \begin{bmatrix}
\cos \phi & \sin \phi & 0 \\
-sin \phi \cos \phi & \cos \phi \sin \phi & \sin \phi \\
\sin \phi \sin \phi & -\cos \phi \sin \phi & \cos \phi
\end{bmatrix}.
\]  

(5.20)

Also the local orientation angle of each grain is different from one another. So, the similar local coordinate transformation should be applied. However, in this case only the rotation around the out of plane axis, \(\varepsilon''\)-axis in Figure 5.13, is considered.

The constant strain triangular (CST) element is used with the anisotropic elasticity material model described above.

5.2.2 Profile Solver

The profile solver, which is also called as the skyline solver, is adopted for the fast solution. In order to use the profile solver, the storage of the global stiffness should be carefully accomplished. The first step of this storing is the evaluation of the profile height for each diagonal location of the global stiffness matrix. Then, only the active columns of the stiffness matrix are stored. The pointers for diagonal element locations for each column are separately stored.
For example, let’s assume that the global stiffness matrix is a 9×9 matrix as:

\[
K = \begin{bmatrix}
    k_{11} & k_{12} & 0 & 0 & k_{15} & 0 & 0 & 0 & 0 \\
    k_{22} & k_{23} & k_{24} & 0 & 0 & 0 & 0 & 0 & 0 \\
    0 & k_{35} & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\
    k_{44} & k_{45} & k_{46} & k_{47} & 0 & 0 & 0 & 0 & 0 \\
    k_{55} & k_{56} & k_{57} & 0 & 0 & 0 & 0 & 0 & 0 \\
    k_{66} & k_{67} & k_{68} & 0 & 0 & 0 & 0 & 0 & 0 \\
    k_{77} & k_{78} & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\
    k_{88} & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\
    k_{99} & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0
\end{bmatrix}.
\] (5.21)

Then, the profile heights for each column are 1, 2, 2, 3, 5, 3, 4, 3 and 1. The number of elements to be stored is the sum of these profile heights, which is 24 in this example. If the order of storage is \(k_{11} \rightarrow k_{12} \rightarrow k_{22} \rightarrow k_{23} \rightarrow k_{33} \rightarrow \ldots\), then the profile stiffness vector, \(K_{\text{profile}}\), and the diagonal pointer vector, \(ID\), for this global stiffness matrix can be represented as:

\[
K_{\text{profile}} = [k_{11} \quad k_{12} \quad k_{22} \quad k_{23} \quad k_{33} \quad k_{24} \quad 0 \quad k_{44} \quad k_{15} \quad \ldots \quad k_{99}]^T\quad \text{and}\quad
ID = [1 \quad 3 \quad 5 \quad 8 \quad 13 \quad 16 \quad 20 \quad 23 \quad 24]^T.
\] (5.22)

The profile solver explained is used with Sloan (1986)’s profile reduction algorithm to maximize the computation efficiency [Sloan (1986)]. The penalty method [Chandrupatla and Belegundu (1997)] is used for the boundary conditions treatment with the penalty parameter of:

\[
P_{\text{penalty}} = \max |K| \times 10^4.
\] (5.23)
5.2.3 Verification of Finite Element Analysis Code

The simple problem shown in Figure 5.14 is solved to verify FE analysis program coded. Silicon is used as a material, and the matrix representation of $C_{ij}$ is shown in equation (7.4). The results and the element stiffness matrix components are compared with the hand-calculation of equation (D-21) in Appendix D and ABAQUS™ result. The element stiffness matrix for these two elements calculated by the developed code, the hand-calculation and ABAQUS™ are identically same as;

\[
k_1^e = \begin{bmatrix}
44.53 & 11.36 & 14.36 & -44.53 & -58.89 & 30.17 \\
76.54 & -12.10 & -14.36 & -2.264 & -62.17 \\
76.54 & -14.36 & -90.90 & 26.46 \\
44.53 & 58.89 & -30.17 \\
149.8 & -56.63 & 92.34 
\end{bmatrix}
\] (5.24)

for element #1, and

\[
k_2^e = \begin{bmatrix}
76.54 & -14.36 & -90.90 & 26.46 & 14.36 & -12.10 \\
44.53 & 58.89 & -30.17 & -44.53 & -15.36 \\
149.8 & -56.63 & -58.89 & -2.264 \\
92.34 & 30.17 & -62.17 \\
44.53 & 14.36 \\
76.54 
\end{bmatrix}
\] (5.25)

for element #2.

The von-Mises stress results of the above three calculations are also identically same as $\hat{\sigma}_1 = 15.10$, $\hat{\sigma}_2 = 13.04$. Therefore, the accuracy of the developed FE analysis code is verified.
<table>
<thead>
<tr>
<th>Crystal Structure</th>
<th>Possible Configuration</th>
<th>Independent Components in $\mathbf{C}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cubic</td>
<td>$P^<em>$, $I^{<strong>}$, $F^{</strong></em>}$</td>
<td>$C_{11}, C_{12}, C_{44}$</td>
</tr>
<tr>
<td>Tetragonal</td>
<td>$P$, $I$</td>
<td>$C_{11}, C_{12}, C_{13}, C_{33}, C_{44}, C_{66}$</td>
</tr>
<tr>
<td>Orthorhombic</td>
<td>$P$, $I$, $F$, $C^{****}$</td>
<td>$C_{11}, C_{12}, C_{13}, C_{22}, C_{23}, C_{33}, C_{44}, C_{55}, C_{66}$</td>
</tr>
<tr>
<td>Monoclinic</td>
<td>$P$, $C$</td>
<td>$C_{11}, C_{12}, C_{13}, C_{16}, C_{22}, C_{23}, C_{26}, C_{33}$, $C_{36}, C_{44}, C_{45}, C_{55}, C_{66}$</td>
</tr>
<tr>
<td>Hexagonal</td>
<td>$P$</td>
<td>$C_{11}, C_{12}, C_{13}, C_{33}, C_{55}$</td>
</tr>
<tr>
<td>Trigonal</td>
<td>$P$</td>
<td>$C_{11}, C_{12}, C_{13}, C_{14}, C_{33}, C_{44}$</td>
</tr>
</tbody>
</table>

*P: Primitive, **I: Body-centered, ***F: Face-centered, ****C: Side-centered

Table 5.1. The relation between the crystallographic structure and the independent components in $\mathbf{C}$ [Kittel (1996)].
Figure 5.1. Example grain boundaries and edges from the microstructure evolution simulation (30×30 lattice system, $p_N = 0.005$ and $f_P = 1.0$, 21 grains)
Figure 5.2. Illustration of the vertex points locations.
Figure 5.3. Illustration of the grain boundaries defined by two vertex points; (a) one grain boundary and (b) two grain boundaries.
Figure 5.4. Two criteria for the near neighbor problem, 4-connected neighbor (von-Neumann) and 8-connected (Moore) neighbor.

Figure 5.5. Illustration of the simple grain boundary extraction; (a) the original grain shape, (b) the grain boundary extracted based on 4-connected neighbor criterion, and (c) the grain boundary extracted based on 8-connected neighbor criterion.
Figure 5.6. The illustration of the Pixel-by-Pixel Marching scheme.
Figure 5.7. Illustration of the preliminary arrangement of the vertices.

Figure 5.8. Rearrangement of the vertices; (a) original arrangement and (b) rearrangement schematics.
Figure 5.9. Element connectivity with 3 types of nodes.
Figure 5.10. Circumcircles for the Delaunay triangulations. (a) acceptable and (b) unacceptable element connection.
Figure 5.11. Examples of the mesh system (a) without mesh refinement and (b) with mesh refinement.
Figure 5.12. CPU time relative to (a) the number of grains, (b) the domain size and (c) the global element size.
Figure 5.13. Euler angles for local coordinate transformation.
Figure 5.14. Two element model for the patch test.
CHAPTER 6

STATISTICAL CONVERGENCE CRITERION

The Monte-Carlo method is the statistical approach based on the random trial for governing parameters. The governing parameters for this dissertation are the nucleation probability, the fraction of the potential sites, the growth probability, and a set of random orientation angles. Therefore, a statistical test is essential to decide whether or not the number of random trials is sufficient to cover all the possible range.

den Toonder, et al. (1999) tried 30 sets of the random orientation angle for each grain, total 57 grains, of the actual microstructure to calculate the effective Young’s modulus and Poisson’s ratio of BaTiO$_3$, In and ZrSiO$_4$. Mullen, et al. (1997) and Chu (2000) attempted 100 and 1000 microstructure evolution simulations, respectively, to get the effective elastic constants. While the former did not make any comments about the validity of the 30 trials, the later tried to setup the number of sufficient trials by adopting the concept of the coefficient of variation (COV) and the standard deviation (SD), which are defined by:

\[
COV = \frac{1}{(N-1)E_{avg}^2} \sum_{i=1}^{N} \left[ \frac{\hat{E}_i^2 - \hat{E}_{avg}^2}{E_{avg}} \right] = \frac{SD}{E_{avg}},
\]

\[
SD = \frac{1}{(N-1)} \sum_{i=1}^{N} \left[ \frac{\hat{E}_i^2 - \hat{E}_{avg}^2}{E_{avg}} \right],
\] (6.1)
where \( N \) is the number of trials, \( \bar{E}_{\text{avg}} \) is the average of the effective Young’s modulus and \( \bar{E}_i \) is the effective Young’s modulus of \( i^{\text{th}} \) trial. As the number of trials increasing, \( \text{COV} \) and \( SD \) converge to a certain value eventually.

However, the absolute values of the convergent \( \text{COV} \) and \( SD \) are dependent upon the order of the constituent outputs, which means that it is impossible to set the convergent value explicitly prior to the simulation. Therefore, author established the clear and definite convergent criterion based on the hypothesis testing, specifically goodness of fit test.

6.1 Revisit of the Statistics

The important statistical terms are revisited and their relationship to this dissertation is described in this section.

The population is the term specifies the complete group, while the sample is the selected finite number of elements, also called outcomes, from the population [Downing and Clark (1997)]. Selecting one set of sample is called event. Within the focus of this dissertation, the population can be interpreted as all possible elastic properties of the given microstructure, which is not possible to achieve thoroughly, and the sample is the set of calculated elastic properties in a certain number of trials. Hence, each outcome corresponds to the result of a single iteration.

When the sample is selected randomly, it is called the random sample (or variable). A set of random variables is discrete if the value of each random variable is integer, and is continuous if the value of each random variable is real [Downing and
Clark (1997)]. The most famous example of the discrete random variable is the coin toss problem, since the value of its result is zero (head) or one (tail), or vice versa. The results of the current simulation are the effective elastic constants and/or elastic responses (deflection, strains, stresses, etc.) that vary continuously within a certain range, which makes them continuous random variables.

The *estimate* is an educated guess for an unknown outcome based on known information [Weisstein (2004, Estimate)], i.e., the sample. And the *estimator* is a rule of this estimate, e.g., the sample mean is an estimator for the population mean. The *expectation value* is the sum, for discrete random variable(s), or the integration, for continuous random variable(s), of the product of the outcome value and its probability. Hence, *the maximum likelihood* expectation values for the population with the normal distribution, see Appendix B, is the *mean* and the *standard deviation* of the sample [Weisstein (2004, MaximumLikelihood)].

For $N$ numbers of the random trials, if the events in each trial is independent, then the process is called *Poisson process* [Rice (1995)]. Since the random process events of the proposed simulation do not affect each other, it is a *Poisson process*.

For $N$ results, the *sample*, of the Monte-Carlo simulations ($X$), we can calculate the expectation $E(X)$ and the standard deviation of $X$ as:

$$
E(X) = \mu = \frac{1}{N} \sum_{i=1}^{N} x_i
$$

$$
\sigma = \sqrt{\frac{\sum_{i=1}^{N} (x_i^2 - \mu^2)}{N - 1}}
$$

(6.2)
where $x_i$ is $i^{th}$ result. Note that in equation (6.2) the denominator for the standard deviation is $(N-1)$, not $N$, since it is the sample deviation, not the population deviation [Downing and Clark (1997)]. If this sample of size $N$ is the representative one for the population, the expectation and the standard deviation are close to those of the population and the sample distribution is also close to the population distribution [Downing and Clark (1997)]. Rephrasing it, once the sample’s distribution is thought to resemble the distribution of the population, the sampling can be stopped.

The sample can be interpreted as a set of calculated results by some number of iterations. The population can be the distribution of the characteristics of the investigating specimen, which is unknown. The sampling is the iterative solution process of the simulation in this case. Therefore, when the distribution of the trial calculations is close to the distribution of that specimen’s characteristic, the iteration can be stopped. A statistical convergence criterion can be used to make the decision.

An estimation method for the resemblance of two distributions is needed while the expected distribution of the specimen’s behavior can be assumed. The most relevant method of estimation in statistics is the hypothesis test. The goodness of fit test, briefed in Appendix C, is used in this dissertation. It is assumed that the expected distribution of the specimen’s behavior is the normal distribution, because the distribution as a result of random sampling converges to the normal distribution [Fry (1965), Downing and Clark (1997)].
6.2 Convergence Criterion by using Chi-square Test

In order to use the Chi-square test as the convergence criterion, the confidence interval (CI) and the degree of freedom of the Chi-square distribution should be determined. For \( N \) results from \( N \) iterations, the Chi-square statistic, \( \chi^2 \), can be calculated, once the number of frequency histogram interval (\( m \)) is determined, as:

\[
\chi^2 = \sum_{i=1}^{m} \left( \frac{f_i - f_i^*}{f_i^*} \right)^2
\]

where \( f_i \) is the \( i \)th frequency of \( N \) results and \( f_i^* \) is the \( i \)th frequency of the normal distribution, which are calculated by the process described in Appendix C. Since the value of \( \chi^2 \) is strongly dependent upon \( m \), it should be determined carefully. To insure the convergence, the minimum value of \( \chi^2 \) with \( m \) ranges from 7 to 15 is used in this dissertation. Then the degree of freedom, \( n_f \), for the Chi-square distribution is calculated as:

\[
n_f = m - 1 - n_0
\]

where \( n_0 \) is the number of data used for determining the expected distribution, which is the normal distribution in this dissertation. \( n_0 = 2 \), since the mean and the standard deviation are needed to define the normal distribution.

The convergence criterion is calculated by finding the value ‘\( a \)’ from:

\[
\Pr(\chi_{n_f}^2 < a) = CI
\]

where \( CI \) is the confidence interval, and the Chi-square distribution of \( n_f \) degree of freedom shown in Figure B.3.
To determine the value of CI, the parametric study about the effect of CI on the convergence is performed. Total 60 different simulations with different settings, the simulation domain and kinetics parameters, were done. When the value of CI is set to be greater than 0.5, all 60 simulations were converged successfully with less than 50 iterations. 59 of 60 simulations were converged when CI = 0.5 with average iteration of 64.1. If CI is less than 0.5, the simulation is rarely converged. Therefore, CI = 0.5 is used throughout.

Figure 6.1 illustrates the validity of the convergence criterion described in this chapter. The convergent effective Young’s modulus of the barium titanate (BaTiO$_3$) (546×410 µm with 57 grains) is achieved with 140 iterations. The convergence criterion used is the Chi-square test with CI = 0.5. In Figure 6.1, the horizontal axis serves two roles. One is to show the microstructure number, which corresponds to the iteration of the simulation, and the other is to show the frequency of the frequency histogram, of which the actual scale is not shown. The results of all microstructure numbers are shown on one position, ‘All’, in this case. One hollow circle in Figure 6.1 corresponds to one iteration result. The bars in Figure 6.1 represents the frequency of the calculation results for the given frequency histogram intervals. The thick solid-line curve shows the normal distribution with the mean (170.9 GPa) and standard deviation (5.7) of the calculated results. As shown, the assumption that the elastic properties will distribute by the normal distribution is reasonable. The validity of using CI = 0.5 is also verified.
Figure 6.1. The frequency histogram of the calculated effective Young’s modulus (bars) compared with Gaussian distribution (−). The convergent effective Young’s modulus of BaTiO$_3$ (tetragonal, 57 grains in 546x410 µm) is calculated with 140 iterations.
In this chapter, applications of the developed Monte-Carlo simulation package are exemplified. First, the effective elastic constants, Young’s modulus and Poisson’s ratio, extractions for BaTiO$_3$ (barium titanate), ZrSiO$_4$ (zircon) and polysilicon are demonstrated. The results of polysilicon are compared with a round-robin test data. Then, the elastic response prediction of a polysilicon micro-beam is examined. Also discussed are the effects of specimen size, global element length of FE mesh and the levels of anisotropy.

7.1 The Effective Elastic Constants Extraction

7.1.1 Fundamentals of the Effective Elastic Constants Extraction

The effective elastic constants, Young’s modulus and Poisson’s ratio, are calculated with the model shown in Figure 7.1. From the boundary conditions of Figure 7.1 and equation (5.13), the effective elastic constants are calculated by [Mullen, et al. (1997), Yin (1997), den Toonder, et al. (1999), Chu (2000)];
Plane Stress:

\[ \nu_{eff}^\sigma = \frac{\sigma_{22}}{\sigma_{11}} \]  
\[ E_{eff}^\sigma = \frac{\sigma_{11}^2 - \sigma_{22}^2}{\sigma_{11} \sigma_{22}} \]  

(7.1)

Plane Strain:

\[ \nu_{eff}^e = \frac{\sigma_{22}}{\sigma_{11} + \sigma_{22}} \]  
\[ E_{eff}^e = \frac{(\sigma_{11} + 2\sigma_{22})(\sigma_{11} - \sigma_{22})}{\sigma_{11} (\sigma_{11} + \sigma_{22})} \]  

(7.2)

where:

\[ \nu_{eff}^\sigma \] : the effective Poisson's ratio for plain-stress condition
\[ \nu_{eff}^e \] : the effective Poisson's ratio for plain-strain condition
\[ E_{eff}^\sigma \] : the effective Young's modulus for plain-stress condition
\[ E_{eff}^e \] : the effective Young's modulus for plain-strain condition.
\[ \sigma_{11} \] : the nominal stress for the loading direction
\[ \sigma_{22} \] : the nominal stress for the transverse direction
\[ \overline{\epsilon}_{11} \] : the nominal strain for the loading direction

The nominal stress and strain are given by:

\[ \sigma_{11} = \frac{F_x}{L_y}, \quad \sigma_{22} = \frac{F_y}{L_y} \]  
\[ \overline{\epsilon}_{11} = \frac{\delta}{L_H} \]  

(7.3)

with unit thickness assumption, where \( F_x \) is the sum of reaction force on the left vertical side, \( F_y \) is the sum of reaction force on the bottom or top horizontal side and \( \delta \) is the displacement on right vertical side, which are all shown in Figure 7.1.
7.1.2 Comparison with the Round-Robin Test

The effective Young’s moduli of the polysilicon with assumed microstructure and size are calculated by using developed Monte-Carlo simulation package. The assumed size of a specimen is 100×100 \( \mu \text{m}^2 \) with 20 grains, and the simulation domain is 100×100 pixels. Plane stress and strain conditions are investigated for the fast, \( p_N = 1.0 \) and \( f_P = 0.002 \), and slow, \( p_N = 0.00018676 \) and \( f_P = 1.0 \), nucleation conditions, and [100] and [110] plane normal textures are also considered. The total simulation cases are:

(i) \( \text{PE}_{[100]} \) Fast: Plane strain, [100] plane normal and Fast nucleation
(ii) \( \text{PE}_{[100]} \) Slow: Plane strain, [100] plane normal and Fast nucleation
(iii) \( \text{PS}_{[100]} \) Fast: Plane stress, [100] plane normal and Fast nucleation
(iv) \( \text{PS}_{[100]} \) Slow: Plane stress, [100] plane normal and Fast nucleation
(v) \( \text{PE}_{[110]} \) Fast: Plane strain, [110] plane normal and Fast nucleation
(vi) \( \text{PE}_{[110]} \) Slow: Plane strain, [110] plane normal and Fast nucleation
(vii) \( \text{PE}_{[110]} \) Fast: Plane strain, [110] plane normal and Fast nucleation
(viii) \( \text{PE}_{[110]} \) Slow: Plane strain, [110] plane normal and Fast nucleation

The results are summarized in Figure 7.2, which also shows a comparison with the round-robin test reported by Sharpe Jr. et al. (1998). The horizontal index starts with the round-robin test result, and is followed by each simulation case listed above. The vertical axis indicates the effective Young’s modulus in GPa unit.

The average values of the calculated effective Young’s moduli for fast and slow nucleation conditions are similar. But the scatter under the slow nucleation condition is larger than the fast nucleation case due to the irregularity in the grain size distribution for the slow nucleation condition. Young’s modulus of [110] plane normal texture is higher.
than that of [100] plane normal texture, which is due to the higher principal in-plane orthotropic elastic constants, $C_{11}$, $C_{12}$ and $C_{44}$.

### 7.1.3 Comparison with Previous Researches

The effective elastic properties for BaTiO$_3$ (cubic and tetragonal structures) and ZrSiO$_4$ (tetragonal structure) are computed by employing the microstructure shown in Figure 4.9. The BaTiO$_3$ possesses two different crystallographic structures depending on the temperature; a cubic structure above its Curie temperature ($T_c = 130^\circ$C) and a tetragonal structure below $T_c$ [den Toonder, et al. (1999)]. The results are compared with the previous work of den Toonder, et al. (1999). The single crystal properties of each material used are summarized in Table 7.1 [Berlincourt and Jaffe (1958), Hellwege (1979)].

*den Toonder, et al. (1999)* calculated the effective elastic constants by using the FE mesh on the given microstructure with 30 different sets of orientation angles for the grains. The current work extracts the effective elastic constants by using the reconstructed microstructures based on the kinetics parameters extracted for the micrograph of Figure 4.9(a). The results are summarized in Table 7.2 and Figure 7.3. In Figure 7.3, the filled rectangles represent the average value of current work and the filled circles represent the average value by den Toonder, et al. (1999). Indices on the horizontal axis indicate the plane normal of a specimen. Figure 7.3 (a, b and c) are the effective elastic properties for BaTiO$_3$ (cubic), BaTiO$_3$ (tetragonal) and ZrSiO$_4$ (tetragonal), respectively.
The results of current work are within the range of the previous work, and the scatter of current work is larger than that of the previous work. The ‘Microstructure Trials only’ simulation flow of a current work causes the larger scatter of the elastic properties. It is found that the effective elastic constants are strongly dependent upon the plane normal direction.

7.1.4 The Effective Elastic Constants of the Polysilicon

Also calculated are the effective elastic constants of the polysilicon with the same microstructure reported by Figure 4.9 Since the silicon is a cubic material, there are only 3 independent terms in the modulus matrix $C$. Namely they are $C_{11}$, $C_{12}$ and $C_{44}$. The values are given as [Simmons and Wang (1971)], for [001] plane normal texture;

\[
C = \begin{bmatrix}
165.7 & 63.9 & 63.9 & 0 & 0 & 0 \\
63.9 & 165.7 & 63.9 & 0 & 0 & 0 \\
63.9 & 63.9 & 165.7 & 0 & 0 & 0 \\
0 & 0 & 0 & 79.6 & 0 & 0 \\
0 & 0 & 0 & 0 & 79.6 & 0 \\
0 & 0 & 0 & 0 & 0 & 79.6
\end{bmatrix} \text{ (GPa)} \tag{7.4}
\]

and for [110] plane normal texture;

\[
C = \begin{bmatrix}
194.4 & 63.9 & 35.2 & 0 & 0 & 0 \\
63.9 & 165.7 & 63.9 & 0 & 0 & 0 \\
35.2 & 63.9 & 194.4 & 0 & 0 & 0 \\
0 & 0 & 0 & 79.6 & 0 & 0 \\
0 & 0 & 0 & 0 & 50.9 & 0 \\
0 & 0 & 0 & 0 & 0 & 79.6
\end{bmatrix} \text{ (GPa)} \tag{7.5}
\]

They must be transformed according to equation (5.18).
The results are shown in Table 7.3 and Figure 7.4. The average effective Young’s modulus of 147.97 GPa with 5.92 standard deviation and the average effective Poisson’s ratio of 0.1792 with 0.0328 standard deviation are extracted for [001] plane normal texture. For [110] plane normal texture, the effective Young’s modulus and Poisson’s ratio are 168.67 GPa with 2.70 standard deviation and 0.2487 with 0.0098 standard deviation, respectively. As can be seen, the plane normal direction significantly influences the calculation of effective elastic constants. The scatter of the constants for [001] texture is larger than that for [110] texture due to the severe anisotropy, which is further discussed in 7.3.3.

7.2 Elastic Response of a Micro-Beam Specimen

Elastic responses of a polysilicon micro-beam specimen for in-plane bending are examined next (cf. Figure 7.5). The beam shape mechanical components are very common in MEMS devices. The beam component shown in Figure 7.5 is assumed to be fabricated under the same process condition as the microstructure of Figure 4.9(a). The average number of grain in this micro-specimen can be calculated from the pro-rated equation;

\[ N_{\delta}^{\text{beam}} = N_{\delta}^{\text{micrograph}} \frac{A_{\text{beam}}}{A_{\text{micrograph}}} \]  

(7.6)

where \( N_{\delta} \) and \( A \) are the number of grains and the area respectively, ‘\( ||_{\text{beam}} \)’ is for the beam component, and ‘\( ||_{\text{micrograph}} \)’ is for the micrograph of Figure 4.9(a). From equation (7.6), the average number of grains in the beam specimen is 6.37, and the nucleation
speed parameter is assumed to be the same, $\beta_n = 1.143$. With these two quantitative measures of a microstructure and the simulation domain of 400×40 lattices, the kinetics parameters for this beam specimen are extracted to be $p_N = 0.00007137$ and $f_P = 0.8807979$. Examples of reconstructed microstructures are shown in Figure 7.6 with FE mesh on them.

Analytic solutions by beam bending theories (Euler-Bernoulli and Timoshenko) and the isotropic plane stress FE solution by using ABAQUS® are also calculated for the comparison. The effective Young’s modulus and Poisson’s ratio are needed for the isotropic deterministic calculations by theories and by ABAQUS®. The effective elastic constants calculated in 7.1.4 can be used, but recomputed specifically for the micro-beam specimen for a confirmation. As shown in Figure 7.7, the mean values of the effective elastic constants are almost same for both calculations, but the scatter of the micro-beam’s effective constants are much wider than that of the micrograph shown in Figure 4.9(a). Combinations of the effective Young’s modulus and Poisson’s ratio are used for the deterministic calculations of the minimum and maximum deflections of the beam.

The analytic solution for the beam in bending as shown in Figure 7.5 can be expressed as;

Euler-Bernoulli Theory (Pure Bending)

$$\delta_{\text{max}} = \frac{PL^3}{3EI}, \quad \sigma_{\text{max}} = \frac{PLz}{I}$$  \hspace{1cm} (7.7)

and

Timoshenko Theory (with Shear Effect)

$$\delta_{\text{max}} = \frac{PL^3}{3EI} \left[ 1 + \frac{3E}{\kappa^2G} \frac{I}{AL^2} \right], \quad \sigma_{\text{max}} = \frac{PLz}{I}$$  \hspace{1cm} (7.8)
where;

\[ P: \] the loading at the tip
\[ L: \] the length of a beam
\[ I: \] the 2nd moment of internia of a beam section.
\[ z: \] the maximum height from the neutral axis
\[ \kappa: \] the shear factor (=1.2 for the rectangle)

The results are compared in Table 7.4 and Figure 7.8. In figure 7.8, the horizontal axis indicates the deflection at the loading tip, and the vertical axis shows the frequency of each frequency histogram interval. The minimum and maximum values of the analytical and ABAQUS™ solutions are calculated with the corresponding combination of the elastic properties extracted. The analytical and ABAQUS™ solutions are close to the current work for [001] plane normal texture. For [110] plane normal texture, the isotropic solutions show deviation from the current work. The possible reason for this deviation is explained below;

\[ C_{11} = C_{22} \] in case of [001] plane normal texture, while \( C_{11} \neq C_{22} \) for [110] texture, see equations (7.4) and (7.5). This difference of elastic constants with respect to the principal directions causes the deviation of the calculated deflection. Since \( C_{11} \) of [110] texture is 1.17 times higher, the possible minimum deflection is smaller than that of isotropic calculation.

### 7.3 Discussion

The effects of the specimen size, the global element length of FE mesh and the anisotropy level of a material on the effective elastic constants are studied.
7.3.1 Effects of the Specimen Size on the Effective Elastic Constants

The specimens with three different sizes are considered to investigate the effect of the specimen size on the effective elastic constants. The first specimen is 100×100 µm square with 40 grains. The second specimen is 50×50 µm. From equation (7.6), the number of grains for this specimen is 10. The third specimen is 20×20 µm with 2 grains. All three cases are under the plane stress condition with the same model micrograph and [100] texture.

The results of the effective Young’s modulus are compared in Figure 7.9. The horizontal axis shows the specimen size. Each small hollow rectangle on graph indicates one simulation result and the filled rectangles show the average values. The average value of the effective Young’s modulus does not change with respect to the specimen size. The statistical scatter, however, becomes larger as the specimen size decreases.

7.3.2 Effects of the Global Element Length

Numerical effects of the global element length of the FE mesh on the effective elastic constants are investigated. The simulation domain of 100×100 pixels is used. Three global element length settings (2, 4 and 6 pixels) are used for two different numbers of grains (10 and 50 grains) with the same microstructures.

The results are compared in Figure 7.10 and Figure 7.11, which show no significant differences in the mean values but the scatter increases slightly as the mesh size increases. Therefore, it can be concluded that the global element length affects the accuracy of the individual FE analysis only but not the effective elastic constants.
7.3.3 Effects of the Anisotropy Levels of a Material

The main reasons for the statistical scatter are the small number of grains and the anisotropy of the constituent single crystals. The effects of the anisotropy levels on the scatter of the elastic properties for various materials are investigated in this section.

The anisotropy of a material is defined by the levels of anisotropy, $A, \nu$ and $\lambda$, which are defined by [Reddy (1997)];

$$A = \frac{2C_{44}}{C_{11} - C_{12}}, \quad (7.9)$$

$$\nu = \frac{C_{12}}{C_{11} + C_{12}} \quad (7.10)$$

$$\lambda = \frac{C_{22}}{C_{11}} \quad (7.11)$$

The material is isotropic if $A = 1$ and $\lambda = 1$, and is cubic if $\lambda = 1$.

By using previously calculated effective elastic properties of two materials (BaTiO$_3$ (cubic and tetragonal) and ZrSiO$_4$), the levels of anisotropy and the statistical characteristics of the calculated effective elastic properties are summarized in Table 7.5. The last column of Table 7.5, the relative scatter, $S'$, is calculated by;

$$S'_{E_{\text{eff}}} = \frac{SD_{E_{\text{eff}}}}{\mu_{E_{\text{eff}}}}$$

where

$$SD_{E_{\text{eff}}} = \sqrt{\frac{1}{N-1} \sum_{j=1}^{N} \left( E_{\text{eff}}^{\sigma} |_{j} - \mu_{E_{\text{eff}}}^{\sigma} \right)^{2}} \quad (7.12)$$

$$\mu_{E_{\text{eff}}}^{\sigma} = \frac{\sum_{j=1}^{N} E_{\text{eff}}^{\sigma} |_{j}}{N}$$
for $N$ effective Young’s modulus results. Similarly,

$$S'_{\nu_{\text{eff}}} = \frac{SD_{\nu_{\text{eff}}}}{\mu_{\nu_{\text{eff}}}}$$

where

$$SD_{\nu_{\text{eff}}} = \sqrt{\frac{1}{N-1} \sum_{i=1}^{N} (\nu_{\text{eff}}^i - \mu_{\nu_{\text{eff}}})^2}$$

$$\mu_{\nu_{\text{eff}}} = \frac{\sum_{i=1}^{N} \nu_{\text{eff}}^i}{N}$$

for effective Poisson’s ratio. From equations (7.12) and (7.13), $S'$ is the standard deviation of the elastic properties normalized by the mean values.

By defining the relative anisotropy, $A'$, as;

$$A' = |A - 1|,$$  \hspace{1cm} (7.14)

which can be interpreted as the distance of $A$ from the value 1, the relation between $S'$ and $A'$ is retrieved as shown in Figure 7.12. The horizontal axis shows the value of $A'$ and the vertical axis indicates the relative scatter, $S'$, of the calculated effective elastic properties.

As shown, there is a tendency of larger scatter of the effective elastic constants as the anisotropy of a material becomes severe. So, the difference of scatter in the effective elastic properties of a polysilicon, which is described in 7.1.4, is now explained. The severe anisotropy level of [100] plane normal ($A' = 0.5639$) leads larger scatter of elastic properties than [110] plane normal case ($A' = 0.2199$).
<table>
<thead>
<tr>
<th></th>
<th>BaTiO$_3$ cubic</th>
<th>BaTiO$_3$ tetragonal</th>
<th>ZrSiO$_4$ tetragonal</th>
</tr>
</thead>
<tbody>
<tr>
<td>$C_{11}$</td>
<td>173.0</td>
<td>275.1</td>
<td>424.0</td>
</tr>
<tr>
<td>$C_{12}$</td>
<td>82.0</td>
<td>179.0</td>
<td>70.0</td>
</tr>
<tr>
<td>$C_{44}$</td>
<td>108.0</td>
<td>54.3</td>
<td>113.0</td>
</tr>
<tr>
<td>$C_{33}$</td>
<td>164.9</td>
<td>490.0</td>
<td></td>
</tr>
<tr>
<td>$C_{13}$</td>
<td>151.6</td>
<td>150.0</td>
<td></td>
</tr>
<tr>
<td>$C_{66}$</td>
<td>113.1</td>
<td>48.5</td>
<td></td>
</tr>
</tbody>
</table>

Table 7.1. Single Crystal Elastic Constants (GPa) [Berlincourt and Jaffe (1958), Hellwege (1979)].

<table>
<thead>
<tr>
<th></th>
<th>BaTiO$_3$ cubic</th>
<th>BaTiO$_3$ tetragonal</th>
<th>ZrSiO$_4$ tetragonal</th>
</tr>
</thead>
<tbody>
<tr>
<td>$E_{eff}^\mu$</td>
<td>174.0</td>
<td>154.7 [001] 195.5 [110]</td>
<td>145.1</td>
</tr>
<tr>
<td>$\sigma$</td>
<td>7.0 [001] 8.0 [100] 7.4 [110]</td>
<td>9.2</td>
<td>7.8 [001] 3.6 [100] 11.5 [110]</td>
</tr>
<tr>
<td>$V_{eff}^\mu$</td>
<td>0.221</td>
<td>0.127 [001] 0.276 [110]</td>
<td>0.322</td>
</tr>
<tr>
<td>$\sigma$</td>
<td>0.025</td>
<td>0.046 [001] 0.023 [110]</td>
<td>0.030</td>
</tr>
</tbody>
</table>

$\mu$: the mean value.
$\sigma$: the standard deviation.

Table 7.2. Summary and the comparison of the effective elastic constants extraction.
Table 7.3. Effective elastic constants of the polysilicon with the microstructure of Figure 4.9(a).

<table>
<thead>
<tr>
<th>Plane Normal</th>
<th>[001]</th>
<th>[110]</th>
</tr>
</thead>
<tbody>
<tr>
<td>$E_{\text{eff}}$</td>
<td>147.97 (5.92)$^*$</td>
<td>168.67 (2.70)</td>
</tr>
<tr>
<td>$\nu_{\text{eff}}$</td>
<td>0.1792 (0.0328)</td>
<td>0.2487 (0.0098)</td>
</tr>
</tbody>
</table>

* Numbers in the parentheses are the standard deviations.

Table 7.4. Comparison of the tip deflection and the maximum stress for a micro scale cantilever beam.

<table>
<thead>
<tr>
<th></th>
<th>Theory</th>
<th>ABAQUS</th>
<th>Current Work</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Euler-Bernoulli</td>
<td>Timoshenko</td>
<td></td>
</tr>
<tr>
<td>$\delta_{\text{max}}$ (mm)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>[001]</td>
<td>1.352 (1.215–1.487)</td>
<td>1.357 (1.220–1.494)</td>
<td>1.371 (1.228–1.512)</td>
</tr>
<tr>
<td>[110]</td>
<td>1.186 (1.113–1.246)</td>
<td>1.191 (1.117–1.252)</td>
<td>1.212 (1.126–1.254)</td>
</tr>
<tr>
<td>$\sigma_{\text{max}}$ (GPa)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>[001]</td>
<td>0.060</td>
<td></td>
<td>0.564</td>
</tr>
<tr>
<td>[110]</td>
<td></td>
<td></td>
<td>0.569</td>
</tr>
<tr>
<td>Material</td>
<td>Plane normal</td>
<td>$A$</td>
<td>$\overline{v}$</td>
</tr>
<tr>
<td>----------</td>
<td>--------------</td>
<td>------</td>
<td>---------------</td>
</tr>
<tr>
<td>Si</td>
<td>[0 0 1]</td>
<td>1.5639</td>
<td>0.2783</td>
</tr>
<tr>
<td></td>
<td>[1 1 0]</td>
<td>1.2199</td>
<td>0.2474</td>
</tr>
<tr>
<td>BaTiO$_3$ cubic</td>
<td>[0 0 1]</td>
<td>2.3736</td>
<td>0.3216</td>
</tr>
<tr>
<td></td>
<td>[1 1 0]</td>
<td>1.4072</td>
<td>0.2583</td>
</tr>
<tr>
<td>BaTiO$_3$ tetragonal</td>
<td>[0 0 1]</td>
<td>2.3538</td>
<td>0.3942</td>
</tr>
<tr>
<td></td>
<td>[1 0 0]</td>
<td>0.8794</td>
<td>0.3553</td>
</tr>
<tr>
<td></td>
<td>[1 1 0]</td>
<td>0.5760</td>
<td>0.3083</td>
</tr>
<tr>
<td>ZrSiO$_4$</td>
<td>[0 0 1]</td>
<td>0.2740</td>
<td>0.1417</td>
</tr>
<tr>
<td></td>
<td>[1 0 0]</td>
<td>0.8248</td>
<td>0.2613</td>
</tr>
<tr>
<td></td>
<td>[1 1 0]</td>
<td>1.5533</td>
<td>0.3367</td>
</tr>
</tbody>
</table>

Table 7.5. Summary of the anisotropy of the materials and the statistical characteristics.
Figure 7.1. Constant strain model for the effective elastic constants extraction.
Figure 7.2. Effective Young's moduli for the plane stress (PS) and strain (PE) conditions, [100] and [110] plane normal textures, under slow and fast nucleation conditions. The simulation results are compared with the Round-Robin Test [Sharpe Jr., et al. (1998)].
Figure 7.3. The calculated effective Young's modulus of (a) BaTiO3 cubic, (b) BaTiO3 tetragonal and (c) ZrSiO4. The comparison with den Toonder, et al. (1999)’s work is also given. The mean (■, ●) and the minimum and maximum (−) values are shown.

(continue)
Figure 7.4. Effective (a) Young’s modulus and (b) Poisson’s ratio of a polysilicon (546×410 μm with 57 grains) for [001] and [110] plane normal texture. The mean (■) and the minimum and maximum (–) values are shown for both elastic properties.
Figure 7.5. The micro-beam specimen for in-plane bending.
Figure 7.6. Examples of the micro-beam specimen microstructure and the corresponding FE mesh for the first 9 iterations.
Figure 7.7. Comparison of the effective elastic constants; (a) Young's modulus and (b) Poisson's ratio. The results are calculated for the micrograph in Figure 4.9(a) and the micro-beam specimen in Figure 7.5. The mean (■, ) and the minimum and maximum (−) values are shown.
Figure 7.8. Distribution of the tip deflection for the micro-beam specimen with in-plane bending compared with analytical solutions (---: Euler-Bernoulli, -: Timoshenko) and isotropic FEM (--: ABAQUS); (a) [001] plane normal texture and (b) [110] plane normal texture.
Figure 7.9. Specimen size effect on the effective Young’s modulus.

Polysilicon (Plane Stress)
Figure 7.10. Effects of the global element size for FE mesh. 100x100 simulation domain with 10 grains.
Figure 7.11. Effects of the global element size for FE mesh. 100x100 simulation domain with 50 grains.
Figure 7.12. Effects of the anisotropy on the effective (a) Young’s modulus and (b) Poisson’s ratio.
CHAPTER 8

CONCLUDING REMARKS

An integrated system for Monte-Carlo analysis and FE analysis is developed in this dissertation to calculate the scattered elastic properties of the polycrystalline materials in micro-meter length scale in 2 dimensions. The system includes modules for; simulation of microstructure evolution, FE meshing on simulated grain distribution, the planar elastic FE analysis, and the calculation of statistical characteristics. As a by-product, given a micrograph, scattered elastic responses can be computed directly for actual MEMS devices without determining effective constants.

The developed numerical algorithm appears to be stable and efficient to capture and simulate microstructure evolution on a pre-defined lattice system. An inverse method for the extraction of key kinetics parameters of the microstructure evolution is also proposed and verified through several applications. The reconstruction of the microstructure by the combination of statistical simulation of microstructure evolution and FEM seems to be realistic.

It is now possible to build a possible grain distribution numerically for a given set of kinetics parameters related to the microstructure evolution. Or, one can extract the parameters from a given micrograph showing a grain distribution in micro-meter scale.
Either way, statistically distributed elastic constants can be computed by the proposed mesh generation on grains and the FEM. The developed mesh generation algorithm closes the gap between the microstructure simulation and the FE analysis.

The adopted statistical convergence criterion (Chi-square test) is numerically shown to be; statistically admissible, logically reasonable, stable in terms of the appearance order of the considered parameters, and the variable types considered.

Numerical investigation reveals that a higher anisotropy yields a wider scatter of the elastic properties, which can be a major concern for the selection of MEMS component materials.

Effects not considered in this investigation entail the grain boundary density, phase change and the residual stress, among others, and are left for a future research. The present investigation is restricted to orthotropic elasticity in 2D, but each module is flexible enough to be extended to fracture, plasticity, and so on. These extensions could pave a way for numerical evaluation of MEMS design, manufacturing process and reliability. Extending the current work to 3D would be a challenging task.
APPENDIX A

RANDOM NUMBERS

A.1 Definition

The informal definition of the random number is the sequence of real numbers in the given range, e.g. the unit interval [0, 1], which does not have any pattern in the sequence [Cheney and Kincaid (1994)].

A.2 Random Number Generator

The computer generated random numbers are usually called *pseudo-random numbers* to distinguish from the truly random numbers generated by a random physical process, such as radioactive decay. The simplest and the most widely used random number generation algorithm is the *linear congruence method* using the linear recurrence relation that is represented as [Weisstein (2004, LinearCongruenceMethod)];

\[ x_{n+1} = ax_n + c \pmod{m} \]  \hspace{1cm} (A.1)

where \(a\), \(c\) and \(m\) are generator specific constants and \(x_n\) is the sequence of random values. The initial value \(x_0\) is called the *seed* and is selected as any integer between 1 and the
Mersenne prime number, which is one less than a power of two $2^k - 1$ [Cheney and Kincaid (1994)].

A.3 Characteristic of Random Numbers

If the numbers are monotonically increasing or decreasing, or there are any dependencies between the numbers, then the sequence is not random. So, the random numbers are independent [Fry (1965), Cheney and Kincaid (1994)].

The distribution of the truly random numbers is uniform, which can be expressed as;

$$
\Pr(x, x \leq c; c \in [0,1]) = c
$$

$$
\Pr(x, c_1 \leq x \leq c_2; c_1, c_2 \in [0,1] \text{ and } c_1 < c_2) = c_2 - c_1
$$

where $c$, $c_1$ and $c_2$ are constants. And the probability density function of this kind is shown in Figure B.1.
CONTINUOUS DISTRIBUTION FUNCTIONS

There are several statistical distribution functions for the continuous random variables, e.g. the normal distribution, the Chi-square distribution, the $t$ distribution and the exponential distribution. The major three continuous distributions, which were used extensively in this dissertation, are the uniform distribution, the normal distribution and the Chi-square distribution.

B.1 Uniform Distribution

A uniform distribution, also called a rectangular distribution, is a distribution that has constant probability. An example of the probability density of a uniform distribution in case of the range $[a \ b]$ is shown in Figure B.1 and its probability density function is expressed as:

$$
Pr(x) = \begin{cases} 
0 & \text{for } x < a \\
\frac{1}{b-a} & \text{for } a < x < b \\
0 & \text{for } x > b
\end{cases} 
\quad \quad \text{(B.1)}
$$
B.2 Normal Distribution

The most popular and frequently used distribution function in statistics is the normal distribution. It is not easy to find practical example of obeying this distribution exactly. However the normal distribution has its importance because that almost other distributions approach to the normal distribution as a limiting case [Fry (1965)]. Downing and Clark (1997) listed some examples of which distributions are close to it as; (1) the IQ of a randomly selected person, (2) the result of measurement of a physical quantity, such as the molecular weight of a chemical, (3) the velocities of molecules in a gas, etc.

The function form of the normal distribution is expressed as;

\[ f(x) = \frac{1}{\sigma \sqrt{2\pi}} e^{-\frac{1}{2} \left(\frac{x - \mu}{\sigma}\right)^2} \]  \hspace{1cm} (B.2)

where \( f(x) \) is the probability density of value \( x \), \( \mu \) is the mean value and \( \sigma \) is the standard deviation. For the standard normal random variable, i.e. \( \mu = 0 \) and \( \sigma = 1 \), equation (B.2) becomes;

\[ f(x) = \frac{1}{\sqrt{2\pi}} e^{-\frac{1}{2} x^2} \]  \hspace{1cm} (B.3)

Figure B.2 illustrates the probability density function for the standard normal distribution.
B.3 Chi-square Distribution

Let $Z$ be a standard normal random variable, and define:

$$Y = Z^2$$  \hspace{1cm} \text{(B.4)}

then $Y$ is also a continuous random variable, and it is called a Chi-square ($\chi^2$) random variable with one degree of freedom. Its probability density function is expressed as [Fry (1965), Downing and Clark (1997)];

$$f(y) = (2\pi)^{-1/2} y^{-1/2} e^{-y/2} \quad \text{for } y \geq 0$$  \hspace{1cm} \text{(B.5)}

For $n$ independent random variables, $Z_1, Z_2, \ldots, Z_n$, define:

$$Y_n = Z_1 + Z_2 + \cdots + Z_n$$  \hspace{1cm} \text{(B.6)}

then $Y_n$ is called a $\chi^2$ variable with $n$ degrees of freedom, and is depicted as $\chi^2_n$. The probability density function for $\chi^2_n$ is given by [Downing and Clark (1997)];

$$f(y) = \frac{1}{c} y^{n/2-1} e^{-y/2}$$  \hspace{1cm} \text{(B.7)}

where $c$ is a coefficient so that the integration of $f(y)$ for the whole possible interval becomes one, i.e. the probability of all possible equals to one. So,

$$c = \int_0^\infty y^{n/2-1} e^{-y/2} dy.$$  \hspace{1cm} \text{(B.8)}

Figure B.3 illustrates the probability density functions of $f(y)$ in equation (B.7) for $n = 1, 2, 3, 4$ and 5. As $n$ becomes larger, the $\chi^2$ distribution is contiguous to the normal distribution.
Figure B.1. Probability density function for a uniform distribution in the range of \([a \ b]\).

Figure B.2. The probability density function for the standard normal distribution.
Figure B.3. The probability density functions of $\chi_n^2$ distribution for $n = 1, 2, 3, 4$ and 5.
APPENDIX C

GOODNESS OF FIT TEST

(Chi-square test)

Whether the statistical characteristics of the population are known or not, it is possible to check the sample’s fitness for the population. This kind of test is called goodness of fit test, which is also called Chi-square test and one of the hypothesis testing methods [Fry (1965), Downing and Clark (1997)]. Author will demonstrate the procedure of the goodness of fit test for the continuous random variables in this chapter.

Let’s assume that there are $N$ observed data, i.e. the sample of the continuous random variables $x_i, i = 1, 2, \ldots, N$. The frequency histogram for this observation is achieved by;

(i) Divide the range of the observed data, i.e. from minimum to maximum value, into $m$ equally space intervals.

(ii) Count the number of data belong to each interval, $f_j, j = 1, 2, \ldots, m$.

With the assumption that the observed data will converges to the normal distribution eventually [Fry (1965)], calculate the mean value and the standard deviation of the sample as [Downing and Clark (1997)];

$$\mu = \frac{1}{N} \sum_{i=1}^{N} x_i,$$

(C.1)
respectively. Then the expected frequency, \( f_j^* \), \( j = 1, 2, \ldots, m \), for each interval when the sample is assumed to follow the normal distribution is calculated as:

\[
f_j^* = \frac{N\Delta}{\sigma\sqrt{2\pi}} e^{-\frac{1}{2} \left( \frac{x_j - \mu}{\sigma} \right)^2}
\]

\[
x_j = X_{\min} + \frac{j\Delta}{2}
\]

where \( X_{\min} = \min(x_i) \), \( X_{\max} = \max(x_i) \) and \( \Delta = (X_{\max} - X_{\min})/m \).

The Chi-square statistic can be calculated as [Downing and Clark (1997)]:

\[
\chi^2 = \sum_{j=1}^{m} \frac{(f_j - f_j^*)^2}{f_j^*}
\]

By using equations (B.7) and (B.8), the value \( a \) can be calculated by;

\[
\Pr(\chi_n^2 < a) = CI
\]

with \( CI \) confidence interval. If \( \chi^2 < a \), then the observed data is considered to fit well with the expected distribution.
APPENDIX D

CRYSTAL AXES AND MILLER INDICES

The fundamental of the single crystal crystallography is summarized in this appendix. The crystal axes for a single crystal are defined, and then fourteen Bravais lattice types are illustrated. There are fourteen Bravais lattice types according to the symmetry groups [Kittel (1996)]. The Miller indices of the crystallographic plane and vector are then defined.

D.1 Crystal Axes and Fourteen Bravais Lattice Types

The crystal axes $a$, $b$ and $c$ are defined as shown in Figure D.1(a) with angles $\alpha$, $\beta$ and $\gamma$. The axes of $a$, $b$ and $c$ does not have to be the same length nor unit vectors. They are or are not orthogonal each other. For one general single crystal unit cell shown in Figure D.1(b), illustrated with dotted line and filled circle, the crystal axes are attached as shown. $a \neq b \neq c$ and $\alpha \neq \beta \neq \gamma \neq 90^\circ$ in this case.

There are fourteen possible configurations of a single crystal unit cell as shown in Figure D.2, one general (triclinic) and thirteen special cases. There are three lattice types (P: Primitive, I: Body centered and F: Face centered) for cubic, two types (P and I) for
tetragonal, (P and C: Side centered) for monoclinic, four types (P, I, F and C) for orthorhombic, and one type (P) for trigonal, hexagonal and triclinic crystal systems. The only difference between cubic and tetragonal crystals, which are the major concern in this dissertation, is that one of the lengths $a$, $b$ and $c$ is different.

**D.2 Miller Indices for Position and Orientation of Crystal Planes**

The position and orientation of a single crystal plane can be specified by any three points on the plane, which are not collinear. Miller indices are widely used plane orientation definition method. The construction of Miller indices is summarized as [Kittel (1996)];

1. Find the intercepts on the axes $a$, $b$ and $c$ in terms of the lattice constants.
2. Take the reciprocals of the intercepts.
3. Find the smallest integer have the same ratio with the reciprocals, e.g. $h$, $k$ and $l$.

4. Then $(hkl)$ are Miller indices for this plane.

For example, in case of Figure D.3(a), the intercepts on the axes $a$, $b$ and $c$ in terms of the lattice constants are 1, 1 and 1, respectively. The reciprocals are 1, 1 and 1, so Miller indices for this plane are (111). Here the intercepts in terms of the global coordinate system shown are 3, 2 and 2. Hence, in case of Figure D.3(b), $3,2,2 \rightarrow 1/3,1/2,1/2 \rightarrow 2,3,3 \rightarrow (233)$ are Miller indices.

In case of the cubic crystals, the vector denoted by $[hkl]$ is the plane normal to the plane $(hkl)$ in terms of Miller indices, e.g. $[233]$ is normal to (233) in case of Figure
D.3(b). Here, the notation ‘[ ]’ is for the vector, and ‘( )’ is for the plane. Other than the cubic crystals, Miller indices for the plane normal are calculated as follows;

As an example, for the tetragonal single crystal \((a = b \neq c, c = 2a = 2b)\) shown in Figure D.4, Miller indices for the plane in the figure are \((111)\). The vector [111] is also shown in the figure, which is not normal to the plane \((111)\). To get Miller indices for the plane normal, first find the equation of a plane in terms of the global coordinate system;

\[
\frac{x}{a'} + \frac{y}{b'} + \frac{z}{c'} = 1
\]  

(D.1)

where \(a', b'\) and \(c'\) are the intercepts with respect to the global coordinate system. In this example, \(a' = (1,0,0), b' = (0,1,0)\) and \(c' = (0,0,2)\). So, the equation of the plane in terms of the global coordinate system is;

\[
2x + 2y + z - 2 = 0.
\]  

(D.2)

The normal vector to the plane of equation (D.2) is given by;

\[
V_n = \begin{bmatrix} 2 \\ 2 \\ 1 \end{bmatrix}
\]  

(D.3)

By following the process of Miller indices construction \((2,2,1 \rightarrow 1/2,1/2,1 \rightarrow 1,1,2)\), Miller indices for the plane normal vector to the plane shown in Figure D.4 are [112]. This plane normal calculation process becomes very complicated, if the crystal axes are not orthogonal.
Figure D.1. The crystal axes (a) $a$, $b$ and $c$ for a single crystal unit cell and (b) an example of the crystal axes definition for a given single crystal unit cell.
Figure D.2. Fourteen Bravais lattice types of a single crystal unit cell [Kittel (1996)].
Figure D.3. Two planes identical in global coordinate system; with respect to the crystal axes $a$, $b$ and $c$ have the ratio of (a) 3:2:2 and (b) 1:1:1
Figure D.4. Miller indices for the plane and its normal in case of the tetragonal crystal.


Choi, J. and Lee, J. K., *Lattice based microstructure evolution simulation with kinetics parameters extraction for the polycrystalline thin-film*, to be published, 2004


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