Residual Stress in Ceramics and Ceramic Composites

by

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ABSTRACT

Residual stresses in Si$_3$N$_4$ and SiC have been measured with X-ray diffraction after grinding and thermal shock. The produced surface stresses are compressive after both treatments. The stresses show a strong dependence on the quenching temperature up to a certain temperature when cracks relax the stresses.

The influence of the amount of reinforcing phase on the residual stress state in a Al$_2$O$_3$/SiC whisker composite was investigated and correlated to a modified Eshelby model. The agreement is excellent. The composite was quenched in liquid He (4K) and the stress state measured after new no relaxation of stresses, indicating elastic behaviour.

An in situ strain measurement as a function of temperature conducted on a Al$_2$O$_3$/SiC whisker composite and a SiC/TiB$_2$ particle composite show very good agreement with the Eshelby model for the Al$_2$O$_3$/SiC system but no agreement for the SiC/TiB$_2$ system. The reason is believed to be stress relaxation during sample preparation.
PREFACE

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Magnus Odén
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INTRODUCTION

Ceramic materials have been used for several thousand of years, but in the last few years the interest for using structural ceramics in load bearing applications has grown. The benefit of using a ceramic material instead of a metal is the loss of weight, the increase in the working temperature range, and the ability to withstand corrosive environment. The main drawback is that ceramic materials in general exhibit a low tolerance to damage (i.e. have a low fracture toughness).

To increase the fracture toughness, the micro structure can be altered to provide toughening sites which can consists of large grains in a fine matrix or a variety of reinforcements as particles, whiskers, and fibres. These can be added before densification or grown in situ. An additional complication is the fact that several ceramic material are susceptible to sub critical crack growth under the influence environmental contaminates and stress. These are factors that are to be considered when constructing with ceramic components.

The applications today where structural ceramic components are used include heat exchanger, automotive applications such as turbo charge rotors and roller cam followers, power generation components, cutting tools, biomedical implants, and processing equipment used for fabrication a variety of polymer, metal, and ceramic parts.

A major difficulty still to overcome before these material see more widespread use is their cost. Many of the processes are labour intensive and the rejection rate is high, resulting in an unacceptably high cost.
Advanced ceramics like $\text{Si}_3\text{N}_4$ and SiC are very brittle and residual stresses generated during manufacturing and during operating conditions might influence the strength of the component made out of these materials. It is therefore important to know how these stresses are created and the magnitude of them in order to minimise dangerous tensile residual stresses.

Studies of SiC-whisker reinforced ceramics reveal that improvements in fracture toughness and resistance to slow crack growth are achieved by incorporation of a small diameter whisker into ceramic matrices [1,2]. The increase in toughness is caused by several energy absorbing processes, like deflection of a propagating crack, the whiskers form bridges in the crack path, whisker pull out and the formation of microcracks that can deflect cracks. Residual stress play a direct role in some of these processes, particularly whisker pullout, frictional debonding and microcracking.
CHAPTER 1

BACKGROUND

**Compressive Surface Stresses in Ceramics**

The fact that ceramic material are brittle and have an extreme surface sensitivity contributes to early fracture. The flexure strength of a ceramic material may be improved by treating the surface in a fashion that compressive residual stresses are introduced, and it can be lessened by introducing surface tensile residual stresses. During processing of ceramic material, operation as firing, quenching, grinding, coating, and joining, residual stresses will be introduced. Operations like chemical strengthening can also be conducted to improve the strength of the material [3,4].

Surface finishing processes such as grinding which removes material mechanically, introduce damage on the surface of brittle materials. This damage is usually in the form of residual stresses, both compressive and tensile, and cracks in the surface and just below the surface, which will have a major influence on the mechanical properties of the material. The formation of residual stresses due to grinding have been detected using several techniques, besides from X-ray diffraction [5-7], the usage of a curvature technique in Alumina [7], a photo elastic technique in MgO [8], and in indentation technique in glass-ceramics [9]. The studies show a layer near the surface of compressive stresses [6,7] and tensile underneath.

The origin of residual stresses and cracking can be qualitatively understood by regarding the grinding process as a superposition of...
multiple indentations with randomly shaped abrasive grains. An isolated sharp indenter produces a plastically deformed volume in the material that is proportional in size to the indenter impression. When the load is higher than a critical value cracks will form, one lateral parallel to the surface and one radial along the groove normal to the surface. The indenters travels with high speed, which generates heat. The combination of overlapping plastically deformed volume elements and thermal induced stresses will give the zone with compressive stresses.

Eigenmann et al. [6] measured this layer of compressive stresses by using different wavelengths of X-ray radiation and were able to measure the stress at different depth in the material. Their result show a very steep gradient very close to the surface, less than 10 μm. They also show that the zone width depends on the grinding conditions, a heavily ground specimen have a thicker compressive zone than a softly ground specimen of the same material. The direction of grinding will also affect the magnitude of compressive stresses. In the direction of grinding the magnitude will be higher than in the perpendicular direction.

Burman and Ericsson [10] measured the stress in ground SiC and Si₃N₄ with X-ray diffraction and established the full stress tensor. They found the stress field to be biaxial, compressive in the near surface region and tensile deeper inside.

Evidence exist showing that annealing of a ground Alumina sample above 1350° C for two hours completely recovers the stresses [5,6]. The annealing procedure of a ground ceramic will slightly decrease the flexure strength. The formation of crack in the surface due to grinding will have a competing effect with the formation of a compressive residual stress layer on the bending strength. In a paper by Samuel et al [11] they suggest that
crack closure and crack healing during annealing may occur and could be accomplished by diffusion at elevated temperature. They base their assumption on fracture mechanical calculation of the critical crack size.

In several earlier works, suggestions were made to strengthen ceramic material by quenching [12, 13]. An increased flexure strength in Al₂O₃ were observed with increasing quenching temperature. With a curvature technique Gruver et al [12] were able to predict a residual compressive surface stress. Tanaka et al [14] used X-ray diffraction and layer removal methods to determined the depth of the compressive residual stress layer in alumina after quenching. They found the layer to extend more than 500 μm into the material. This is much deeper than what is observed for the layer with compressive stresses caused by grinding.

Eckel et al [15] observed cracking in SiC and Si₃N₄ bars when they sent thermal spikes through them. The fracture occurred when the samples were cooling, indicating thermal induced tensile stresses in the surface.

The origin of a thermally produced compressive stressed layer in the surface can be explained by plastic deformation and cracking in the surface upon rapid cooling. The surface shrinks prior to the interior upon rapid cooling and ends up in tension during the cooling stage, which will produce the damage. When the temperature in the specimen is constant again, at the lower temperature, the surface will be in compression.

Two phase ceramic materials containing fractions of ZrO₂ are known to be able to undergo a phase transformation in the near surface region when ground or due to oxidation [16,17]. It is the transformation of ZrO₂ from tetragonal to monoclinic structure which gives a volume increase of ~3-5% that produce a compressive stress state in the surface. The depth of
the layer was studied in a Al$_2$O$_3$/30-vol% ZrO$_2$ composite and found to be \(-20\ \mu\text{m}\).

**Thermally Induced Residual Stresses in a Ceramic Matrix Composite**

When a two phase material is sintered at an elevated temperature and then cooled to a lower temperature, the two phases will constrain each other so that residual stresses will occur. The reason is the mismatch in thermal expansion, elastic and plastic behaviour, and the coherency and bonding at the interface. In general a ceramic-ceramic composite require weak or non existing chemical bonding at the interface to achieve a high toughness. The friction at the interface should then be the load transferring link between matrix and whisker. The residual stress state at the interface is the controlling parameter of the frictional force.

Extensive work on ceramic composites utilising a neutron diffraction technique has been carried out by Majumdar et al [18-20]. They have looked at ceramic matrix composite systems and measured the strain with neutron diffraction and compared the result with two analytical models. The results from the Eshelby model are very convincing and indicates that an Eshelby model could be a good way of predicting the internal strains in a ceramic matrix composites. For the alumina silicon carbide whisker system, with the whiskers modelled as spheroids with an aspect ratio of 15, good agreement between predicted and measured data were obtained when temperature or volume fraction was changed.

In their paper regarding a WC-base cemented carbide composite, the importance of a good estimate of the aspect ratio is stressed. By changing the aspect ratio from unity to 1.8 the stress inside the inclusion along the a-axis and c-axis will change in magnitude. When the aspect ratio is unity the stress along the a-axis is larger in magnitude than the stress along the
c-axis, but when changed to 1.8 the condition is opposite. The result with a larger stress along the c-axis agrees with experimental results, but it also points out the difficulties associated with predicting the stress state in a composite reinforced with particles. The geometrical shape of the particles are not uniform and an "average" shape has to be assumed.

Predecki et al [21,22] have worked extensively on the Al₂O₃/SiC-whisker system using X-ray diffraction to determine the residual stresses. They have measured the stresses in both phases and separated them into macro- and microstresses. Their results are in agreement with Majumdar et al. The effect of hot pressing versus cold pressing and sintering were also investigated and a significant anisotropy of the stress state were detected in the hot pressed case. They suggest the reason for this being texture developed in the whisker rather than in the matrix. Due to this texture, the matrix is able to relax to a greater extent in the hot pressing direction then in the transverse directions, thereby reducing the residual stress in the hot pressing direction.

Faber et al [23-25] have studied a SiC/TiB₂ particle composite. This system is interesting because the mismatch in coefficient of thermal expansion will produce tensile stresses in the reinforcing phase, here the TiB₂ particles. In the Al₂O₃/SiC-whisker system the whiskers will be in compressive stress state. The SiC/TiB₂ particle composite is also believed to undergo microcracking as a toughening mechanism. They measured the residual stress state before and after bending the samples to fracture and noticed a nearly 60% drop in the microstresses but a slight increase in the SiC standard used as comparison. They contribute this stress relaxation to microcracking. Transmission electron microscopy studies also show an increased density of microcracks close to the fracture surfaces.
Calculated micromechanical internal stresses

Several authors mentioned before in this chapter have attempted to calculate the internal stresses in a composite. Majumdar and Kupperman [19] got good agreement with an Eshelby model and a model proposed by Ledbetter and Austin [25]. Ledbetter and Austin were able to correlate their strain data with a simple sphere-in-hole elastic model for an Al/SiC-particle composite.

The Eshelby model is built on the equivalent inclusion model and has been modified by several authors to be applied to a ceramic composite. The derivation of this model is described in Chapter two and the main contributors are referenced in that section.
CHAPTER 2

THEORY

Bold characters will be used to denote tensor and matrix properties.

Measurement of Strain and Stress with Diffraction

Diffraction may be used to determine the stress in a crystalline material. The changes in interplanar spacing with tilts of the sample are used to establish a strain tensor. The strain tensors are then together with elastic constants used to determine the stress tensor. In a multi phase material, diffraction will allow determination of the stress tensor in each phase, because they will show different diffraction patterns. This fact makes diffraction a very powerful tool to study materials.

Two Phase Material

The stress distribution in a two phase material will not be uniform, the stress will vary from point to point within the material [26], and this gives rise to macro- and microstresses. Macrostresses are by definition the same in both phases of a two phase material. Macrostresses will occur when, for example, the surface is elongating more than the interior of the sample, see Chapter 1. This can happen when the sample is ground. Microstresses arise because contiguous phases constrain each other microscopically as a result of differences in thermal expansion, elastic and plastic behaviour, and because of the coherency and bonding at the interface. The total stress at any point is the sum of these components.
t, M, and \( \mu \) refer to total, macro- and microstresses respectively. The total stress in a phase is what a diffraction measurement can reveal, but to be able to separate the total stress into macro- and microstresses one need to use the equilibrium conditions.

Noyan [27] showed that from equilibrium conditions for average stresses one can derive the following relationship for a two phase material

\[
(1 - V_f) \langle \mu \sigma_{ij}^a \rangle + V_f \langle \mu \sigma_{ij}^\beta \rangle = 0
\]  

(2.2)

where \( V_f \) is the volume fraction of phase \( \beta \). By determining the total stress in Eq. 2.1 for each phase from measurements and using Eq. 2.2, there are as many equations as unknowns, hence one may separate the macro- and microstresses.

**Determination of the Total Stress in each Phase**

The strain along any given direction in a material is given by the sum of the strains caused by the stresses in that direction.
Figure 1. Definitions of the specimen coordinate system $S$, and the measurement direction $L_3$. 
This can be compactly expressed in the suffix notation [28].

\[ \varepsilon_{\alpha} = \frac{(1 + \nu)}{E} \delta_{\alpha \gamma} - \delta_{\gamma \alpha} \frac{\nu}{E} \sigma_{kk} \quad (2.3) \]

where \( k \) is the dummy suffix and implies summation over all \( k \), and \( \delta_{ij} \) is Kronecker's delta. The X-ray elastic constants \( E \) and \( \nu \) have to be determined for that specific direction.

By using the coordinate system given in Figure 1 and measure the strain in the \( L_3 \) direction one can then transform that strain into the \( S \)-coordinate system, using direction cosines [29]

\[ \left\langle \varepsilon_{\alpha \omega}^a \right\rangle = \left\langle \sigma_{11}^a \right\rangle \frac{S_2^a}{2} \cos^2 \phi \sin^2 \psi + \left\langle \sigma_{22}^a \right\rangle \frac{S_2^a}{2} \sin^2 \phi \sin^2 \psi \\
+ \left\langle \sigma_{33}^a \right\rangle \frac{S_2^a}{2} 2 \cos^2 \phi + \left\langle \sigma_{11}^a \right\rangle + \left\langle \sigma_{22}^a \right\rangle + \left\langle \sigma_{33}^a \right\rangle \right] S_1^a \\
+ \left\langle \sigma_{12}^a \right\rangle \frac{S_2^a}{2} \sin 2\phi sin^2 \psi + \left\langle \sigma_{13}^a \right\rangle \frac{S_2^a}{2} \cos \phi \sin 2\psi \\
+ \left\langle \sigma_{23}^a \right\rangle \frac{S_2^a}{2} \sin \phi sin 2\psi \quad (2.4) \]

The suffix notations refer to the \( S \)-system. The X-ray elastic constants are defined as

\[ \frac{S_2^a}{2} = \frac{1 + \nu^a}{E^a} \quad (2.5a) \]

\[ S_1^a = -\frac{\nu^a}{E^a} \quad (2.5b) \]

By measuring the strain \( \left\langle \varepsilon_{\alpha \omega}^a \right\rangle \) at sufficient number of \( \phi \), and \( \psi \) values the stress tensor may be obtained by a least squares fit solution to Eq. 2.4. By determining the lattice parameter \( d_{\alpha \omega}^a \), using Bragg's law it is easy to obtain the strain by Eq. 2.6.
\[ \langle \varepsilon_{ov}^a \rangle = \frac{d_{ov}^a - d_0^a}{d_0^a} \]  \hspace{1cm} (2.6)

d_0^a \) is the unstrained lattice parameter. An accurate value of \( d_0^a \) is essential in the calculation of the stresses. In this study the unstrained lattice parameter was measured to an accuracy of \( \pm 0.0002 \) Å. A good check of the measurement is to look at the value of \( M\sigma_{33} \) which should be zero according to equilibrium conditions [28].

**Biaxial stress state**

A free surface cannot support a stress normal to it \( (\sigma_{33} = \sigma_{13} = \sigma_{23} = 0) \).

Eq 2.4 will then in for a monolithic material become

\[ \langle \varepsilon_{ov} \rangle = \left[ \langle \sigma_{11} \rangle \cos^2 \phi + \langle \sigma_{22} \rangle \sin^2 \phi + \langle \sigma_{12} \rangle \sin 2\phi \right] \frac{S}{2} \sin^2 \psi \]
\[ + \left[ \langle \sigma_{11} \rangle + \langle \sigma_{22} \rangle \right] S_1 \]  \hspace{1cm} (2.7)

Depending on how the coordinate system is oriented, \( \sigma_{12} \) can either be non-zero or if the coordinate system is rotated in a fashion that it will fall along the principal axes, \( \sigma_{12} \) will be zero. By rewriting Eq. 2.7 and using Eq. 2.6 the well known "d vs sin^2\psi" formula appears

\[ d_{ov} = \frac{S_1}{2} \sin \psi + S_1 d_0 \left[ \sigma_{11} + \sigma_{22} \right] + d_0 \]  \hspace{1cm} (2.8)

\( \sigma_s \) is the stress component along the \( S_s \) direction (Figure 1). The stress along the \( S_s \) direction may be obtained directly from the slope of a least-squares line fitted to experimental data taken at various \( \psi \)-angles. To know the unstressed lattice parameter exactly here is not critical, because it is a multiplier and a small error in \( d_0 \) will not effect the result by much. It is good enough to use the measured value of \( d \) for \( \psi = 0 \).
Analysis of the Diffraction Peak

To measure strains and stresses with X-ray diffraction it is very important to be able to determine the peak position accurately. A stress of 230 MPa will cause a peak shift of 0.1° in 2θ-position of the 146-alumina peak for CuK\(_a\) in the range of \(\psi=0°\) to 45°. The peak position was determined by least square fitting a pseudo-Voigt function including CuK\(_a1\), CuK\(_a2\) to measured data. The fitting parameter for the peak position of the CuK\(_a1\)-peak was taken as the peak position. In the case where overlapping peaks were present both peaks were fitted at once. The background was assumed to be linear, but when the volume fraction of the reinforcing phase was small and the peak was sitting on the tail of a more intense peak, an exponential background was used. The pseudo-Voigt function is the sum of Gaussian and Lorentzian functions and has the form

\[
1 = I_0 (\eta e^{-\frac{4 \log 2 (\theta - \theta_0)^2}{\beta^2}} + \frac{(1 - \eta)}{1 + \frac{4(2\theta - 2\theta_0)^2}{\beta^2}}) \\
+ \frac{1}{2} I_0 (\eta e^{-\frac{4 \log 2 (\theta - \theta_0 - \delta)^2}{\beta^2}} + \frac{(1 - \eta)}{1 + \frac{4(2\theta - 2\theta_0 - \delta)^2}{\beta^2}})
\]  (2.9)
where

\[ I = \text{intensity as a function of } 2\theta \]
\[ I_0 = \text{peak intensity} \]
\[ \eta = \text{fraction Gaussian} \]
\[ 2\theta_0 = \text{peak position} \]
\[ \beta = \text{peak width parameter} \]
\[ \delta = \text{the separation in } 2\theta \text{ of } K_{a1} \text{ and } K_{a2} \text{ peaks} \]

The background functions added were

linear: \[ \text{bg} = A2\theta + B \]
exponential: \[ \text{bg} = Ae^{-B2\theta} + C \]

where A, B, and C are constants, determined by the fitting procedure.

To accurately compare the measured strain and, with the modified Eshelby model, calculated strains it is of great importance to know the volume fraction of whiskers. To measure the volume fraction one can compare the sample of interest with a sample with known compositions of the phase one are interested in. By taking the ratio of the integrated intensity from the unknown sample with the standard sample everything except the volume fraction will cancel \[30\]. To increase the accuracy several peaks were measured and the mean used.

**Errors Associated with Residual Stress Measurement**

The error approximation includes both the instrumental and statistical errors. The instrumental errors associated with variation of the focal point, beam divergence, and sample displacement were taken into account, and by using the error in the fitting parameter of the peak position, it was possible to carry through the statistical errors \[28\]. It should be pointed out that some of the instrumental errors cancel out when two measurements are made with the same set-up. It means that relative changes can be more
accurately determined than what the errors indicate. In this study when measuring residual stress, the statistical errors are small compared with the instrumental ones.

**The Modified Eshelby model**

The matrix notation used below is described by Nye [31]. For more details in the following theory see ref. [32-37].

**Isotropic Inclusion**

An ellipsoidal region is cut from an unstressed elastically homogeneous material, and then imagined to undergo a stress-free shape change (strain $e^{T}$).
Figure 2. Definition of coordinate system for calculating strains in an ellipsoid.
Surface tractions are then applied to return it to its original shape prior to replacing it in the hole from which it was cut. When the inclusion is placed back into the hole and the surface tractions are removed, equilibrium between inclusion and matrix will yield a constrained strain in the inclusion of $e^c$ relative to its original shape before removal. Hooke's law for the elastic strain inside the inclusion can then be expressed as

$$\sigma^I = C^M (e^c - e^T)$$  \hspace{1cm} (2.10)

Where $\sigma^I$ is the stress inside the inclusion. The strain $e^c$ can be written in terms of $e^T$ and the Eshelby tensor $S$, which depends only on the aspect ratio of the inclusion and Poisson's ratio of the matrix.

$$e^c = Se^T$$  \hspace{1cm} (2.11)

**Ellipsoidal Inhomogeneity**

The matrix and inclusion now have different elastic constants. By using a similar argument as above, consider a stress-free transformation strain, $e^T$ (the mismatch between inclusion and matrix), of the ellipsoidal inhomogeneity with respect to the hole it came from on replacing the inclusion it now takes up a constrained shape $e^c$ relative to the original shape of the hole. A second inclusion of the same elastic constants as the matrix can be imagined to undergo a stress-free transformation strain, $e^T^*$ chosen such that when surface tractions are removed and equilibrium is reached with matrix, it will have the same uniform stress state. Thus, the inhomogeneity and the equivalent inclusion can be interchanged without disturbing the matrix. The stress state inside the inclusion in the inhomogeneous case, as given by

$$\sigma^I = C^I (e^c - e^T)$$  \hspace{1cm} (2.12)

and the stress states in the equivalent inclusion and in the homogeneous case are identical.
\[ \sigma^\varepsilon = \mathbf{C}^M (\mathbf{e}^C - \mathbf{e}^{T'}) \] (2.13)

Therefore, by using equation (2.11)
\[ \mathbf{C}^l (\mathbf{Se}^{T'} - \mathbf{e}^T) = \mathbf{C}^M (\mathbf{S} - \mathbf{I}) \mathbf{e}^{T^*} \] (2.14)

where I is the identity tensor. The stress inside the inclusion can then be expressed in terms of \( \mathbf{e}^T \) as
\[ \sigma^T = \mathbf{C}^M (\mathbf{S} - \mathbf{I}) ((\mathbf{C}^l - \mathbf{C}^M) \mathbf{S} + \mathbf{C}^M) \mathbf{e}^T \] (2.15)

It is important to notice that so far the calculation is done for just one inclusion in an infinite large matrix. The strain in the matrix far away from the inclusion is zero. The next sections will address what happens when there is an external stress, a finite volume fraction of inclusions and no orientation of the inclusions.

**Externally Stressed Inclusion**

In the homogeneous system, an externally applied stress \( \sigma^A \) will by superposition give rise to the following stress state inside the inclusion:
\[ \sigma^{inc} = \sigma^T + \sigma^A = \mathbf{C}^M (\mathbf{e}^C - \mathbf{e}^{T*}) + \mathbf{C}^M \mathbf{e}^A = \mathbf{C}^M (\mathbf{e}^C + \mathbf{e}^A - \mathbf{e}^{T*}) \] (2.16)

\( \mathbf{e}^A \) is the extra elastic distortion due to applied stress. By using the same procedure described in the section on ellipsoidal homogeneity and choosing a transformation strain for the homogenous system that will give the same stress state as in the inhomogenous system, the stress state inside the inclusion can then be written as
\[ \sigma^{inc} = \sigma^T + \sigma^A = \mathbf{C}^M (\mathbf{e}^C + \mathbf{e}^A - \mathbf{e}^{T*}) \] (2.17)

for the equivalent inclusion, or for the inhomogeneity
\[ \sigma^{inc} = \mathbf{C}^l (\mathbf{e}^C + \mathbf{e}^A - \mathbf{e}^T) \] (2.18)

\( \mathbf{e}^T \) has two components, one to account for inhomogeneity and one due to different elastic response to the external stress. They are both stress free strains, which means that equation (2.11) still holds.
**Average strain in matrix**

For a material which contains a finite concentration of inclusions, such as a composite material, the stress inside the inclusion can be calculated by a method proposed by Mori and Tanaka [38]. The stress state within the matrix will not be uniform, but we can define an average stress in the matrix as

\[
\langle \sigma^M \rangle = C^M (e^A + \langle e \rangle) \tag{2.19}
\]

where \( \langle e \rangle \) is the average strain in the matrix. Integration of the stress over the entire volume, \( D \), must equal to zero

\[
(1 - V_i)\langle \sigma^M \rangle + V_i \sigma^{inc} = 0 \tag{2.20}
\]

\( V_i \) is the volume fraction of inclusions. By using exactly the same argument as for the external stress for the average stress in the matrix, the stress inside the inclusion can be written as

\[
\sigma^{inc} = \sigma^1 + \sigma^A = C^M (e^C + e^A + \langle e \rangle - e^{T*}) \tag{2.21a}
\]

for the equivalent inclusion, or

\[
\sigma^{inc} = C^I (e^C + e^A + \langle e \rangle - e^{T*}) \tag{2.21b}
\]

for the inhomogeneity.

By substituting equation (2.19) and (2.21) into (2.20) the average strain in the matrix can be determined as

\[
\langle e \rangle = -V_i (e^C - e^{T*}) \tag{2.22}
\]

It must be noted that equation (2.22) implies that all inclusions are aligned along one orientation; this is not always true for a composite. Li [32] calculated for the case of 3-dimensional randomly distributed inclusions using a Voigt approximation

\[
\langle e \rangle = -V_i \langle S \rangle e^{T*} \tag{2.23}
\]

where \( \langle S \rangle \) is the average Eshelby tensor.
Final Expression

Rewriting equation (2.21) and substituting in equation (2.11) and (2.23)

\[ [(C^i - C^M)S + C^M - V_f(C^i - C^M)\langle S \rangle]\mathbf{e}^{T^*} = C^i\mathbf{e}^T - (C^i - C^M)\mathbf{e}^A \] (2.24)

\(\mathbf{e}^{T^*}\) can be solved. By using

\[ \sigma^{bc} = \sigma^A + C^M[S - I - V_f\langle S \rangle]\mathbf{e}^{T^*} \] (2.25a)

\[ \langle \sigma^M \rangle = \sigma^A - V_fC^M\mathbf{e}^{T^*} \] (2.25b)

the stresses in the composite can be calculated. In this study \(\mathbf{e}^A\) was zero and the interest was in strains. The final expression will then be

\[ [(C^i - C^M)S + C^M - V_f(C^i - C^M)\langle S \rangle]\mathbf{e}^{T^*} = C^i\mathbf{e}^T \] (2.26a)

\[ \mathbf{e}^{bc} = [S - I - V_f\langle S \rangle]\mathbf{e}^{T^*} \] (2.26b)

\[ \mathbf{e}^M = -V_f\mathbf{e}^{T^*} \] (2.26c)

The Calculated Strain Along a [hkl]-Direction

When the strains are calculated with equation (2.26), it will yield a strain tensor which has a coordinate system with one axis (\(X_3\)) along the length of the whisker and two in the basal plane (\(X_1, X_2\)), see Figure 2.

Since the SiC whisker is transversely isotropic with respect to the [1 1 1]-direction, there are only two principal microstrains, \(e_{33}\) and \(e_{11}\). The \(e_{33}\) is along the growth direction, [1 1 1], while \(e_{11}\) and \(e_z\) are along the [\(\bar{1} \bar{1} 2\)] and [1 1 0] respectively. The microstrain along any given [hkl]-direction is the given by

\[ e_{[hkl]} = (\alpha_{11}^2 + \alpha_{12}^2) e_{11} + \alpha_{13}^2 e_{33} \] (2.27)

\(\alpha_{ij}\) is the directional cosines with the angle between the [hkl]-direction and the \(X_j\)-axes. The strain measured with X-ray diffraction is an average over all the directions in the \(<hkl>\) family. To be able to compare the strains from the modified Eshelby model and measured strains with X-ray
diffraction, the average strain taken over the \(<hkl>-family\) must be
calculated for the strains from the modified Eshelby model.

\[
\langle e_{(hkl)} \rangle = \frac{\sum e_{(hkl)}}{4}
\]

(2.28)

The summation is over all the different directions, ([hkl], [-h k l], [h -k l],
[h k -l])

Using the principal axes used for SiC, the directional cosines for any
given direction, [hkl], can be expressed as

\[
\alpha_{11} = \frac{(2l - h - k)^2}{3(h^2 + k^2 + l^2)}
\]

\[
\alpha_{12} = \frac{(h - k)^2}{2(h^2 + k^2 + l^2)}
\]

\[
\alpha_{13} = \frac{(h + k + l)^2}{6(h^2 + k^2 + l^2)}
\]

By substituting equation (2.27) and (2.29) into (2.28) and noticing that any
term on the form hk will cancel out in the summation, the expression for
the average strain in any direction will be on the form of

\[
\langle e_{(hkl)} \rangle = \frac{2}{3}e_{11} + \frac{1}{3}e_{33}
\]

(2.30)

**The Stress Field just outside the inclusion**

The stress field in the matrix is more complicated than inside the
inclusion. Equation (2.26) provides a way of determine an average stress,
but it is harder to say something about the distribution. Mura and Cheng [39] have showed that it is possible to determine the stress field just outside
the inclusion

\[
\sigma^{M*} = \sigma^{bc} - C^M B C^M e^T + C^M e^T
\]

(2.31)
B is a matrix which contains the elastic constants of the matrix and the unit vector outward from the inclusion, see Figure 3.

B is defined as

\[ B_{ijkl} = M_{j} n_{k} n_{l} \]

\[ M_{ij} = \frac{1}{\mu M} \delta_{ij} + \frac{n_{i} n_{j}}{2(1 - v M)} \]  \hspace{1cm} (2.32)

For a more explicit expression see Appendix A.
Figure 3. Definition of coordinate system for calculating strains just outside an inclusion.
CHAPTER 3
EXPERIMENTAL METHODS

Materials

**Monolithic Si$_3$N$_4$ and SiC**

Both materials were Hot Isostatic Pressed; Si$_3$N$_4$ from ABB-Cerama and, SiC from Electroschmelzwerk, Kempten, Germany. The specimen were bars with the dimension of 3.5*4.5*10 mm. All specimens were ground in the longitudinal direction and the surface roughness was about 1 μm. The polyform of the materials were identified by X-ray diffraction and correlated with the JCPDS-file. It showed that the Si$_3$N$_4$ was of the hexagonal β-type and the SiC was mainly of the hexagonal α-6H-type.

**Al$_2$O$_3$/SiC-whisker Composite**

Samples were provided from Sandvik Coromant, made of alumina with different contents of silicon carbide whiskers(5-25 weight-%). Small amounts of yttria and magnesia were used as sintering aids (0.05 weight-%). During sintering a single uni axial pressure was applied which results in an orientation of the whiskers in a plane perpendicular to the axial pressure. The diameter of the whiskers were about 0.6 μm and the length after sintering 20-30 μm. The alumina was of α-Al$_2$O$_3$ type and the SiC-whiskers was a mixture of hexagonal and cubic silicon carbide. After sintering the samples were ground which introduced a compressive macrostress in the surface region. When the sample was used for in situ
low temperature strain measurements a thin slice (~1 mm thick) was cut out.

**SiC/TiB$_2$-particle Composite**

The sample was provided by Prof. K Faber, Northwestern University, Evanston, Illinois, USA. The volume fraction of TiB$_2$ was 15% and the average grain size 4.5 $\mu$m. The silicon carbide was a mixture of rhombohedral and hexagonal SiC. The sample were cut to a thickness of about 1 mm using a diamond saw.

**Thermoshock Experiment**

In order to investigate the influence of thermal shock on the residual stress state in ceramics several experiments were performed.

To study this effect, in the $\text{Si}_3\text{N}_4$ and SiC samples, a tube furnace was used. The specimens were slowly heated to different elevated temperatures, they stayed at the elevated (600-1200° C) temperature for 30 min and were then rapidly cooled in water or in air to 20° C.

To see if thermal shock in the $\text{Al}_2\text{O}_3$/SiC-whisker composite would introduce any additional residual stresses different temperature ranges were chosen. Both heating to 1200° C and cooling in air, and quenching from room temperature to a lower temperature.

The heating experiment was done with a tube furnace and the samples stayed in the furnace for 5 and 20 min and were then cooled to room temperature in air. This was done with samples containing 20 weight-% SiC whisker.
To perform the quenching to a lower temperature, the samples were immersed in liquid nitrogen (77 K) or liquid helium (4.2 K) and then brought back up to room temperature. This was carried out for samples containing 5 weight-% and 25 weight-% SiC whiskers.

In all the experiments involving thermal treatments the residual stress was measured both before and after the sample was thermally treated. In the monolithic samples a biaxial stress state was assumed, but in the composite material a full triaxial stress measurement was carried out.

**Diffraction Measurements**

**Residual Stress Measurements**

To measure the stress in the Si$_3$N$_4$ and SiC samples a JEOL diffractometer with an $\Omega$-goniometer, a position sensitive detector, and Cr$_{K\alpha}$-radiation was used. The crystal planes studied were the 411 for Si$_3$N$_4$ and the 116 for SiC. A biaxial stress state was assumed and thus only the stress along the grinding direction of the sample was measured. The elastic constants were taken from the literature [40], see Table 1.

*Table 1. X-ray elastic constants for Si$_3$N$_4$ and SiC*

<table>
<thead>
<tr>
<th>Sample</th>
<th>Plane</th>
<th>$E$ [GPa]</th>
<th>$\nu$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si$_3$N$_4$</td>
<td>411</td>
<td>310</td>
<td>0.26</td>
</tr>
<tr>
<td>SiC</td>
<td>116</td>
<td>408</td>
<td>0.30</td>
</tr>
</tbody>
</table>

To measure the triaxial state in an Al$_2$O$_3$/SiC-whisker composite is a necessity if one attempts to understand the stress state. The phase $\alpha$-Al$_2$O$_3$ has a rhombohedral structure which will give rise to a 146 diffraction peak at ~136° 2θ for Cu$_{K\alpha}$-radiation and $\beta$-SiC (cubic 3C) gives
rise to a peak at $-134^\circ \theta$ for the $511+333$-reflection. These peaks overlap considerably and that has to be taken into account when determining the peak positions.

To align the sample correctly several procedures were employed:

i. The sample surface was made parallel to the incident X-ray beam $(\theta, \psi=0)$, by cutting the beam in half for $\theta=0$ and then rocking the sample around the $\psi$-axis until maximum was obtained.

ii. To get the sample surface perpendicular to the $\phi$-axis, a laser beam was used. By adjusting the sample until the reflection spot from the laser did not move when the sample was rotated around the $\phi$-axis good alignment was obtained.

iii. To position the sample in the centre of the diffractometer, a very thin layer of Si-powder was put on the sample surface and then the Nelson-Riley [30] method for determining the sample displacement was applied. The sample displacement was less than 30 $\mu$m in all experiments.

A General Electric diffractometer with a quarter-circle goniometer was used in these diffraction measurements. The generator was run at 50 kV and 14 mA. For optical details see Figure 4. Filter was not used.

The samples was mounted on the diffractometer such that the grinding direction corresponds to the $S_1$-direction (see Figure 1). The peaks were point counted, corrected for Lorentz-polarisation and absorption, and fitted with pseudo-Voigt function (see Equation 2.9).

Figure 5 shows two examples of the relative intensities of the $\text{Al}_2\text{O}_3$-peak and the $\text{SiC}$-peak at $\phi$ and $\psi$ equal to zero. The counting time at each point varied between different samples depending on the content of $\text{SiC}$-whiskers. For the 5 weight-% samples, each point in the $\text{SiC}$-peak was counted for 300 seconds, and for the 25 weight-% samples for 40 seconds.
The Al₂O₃-peaks were counted for 10 seconds at each point for all the samples.

The Al₂O₃-peaks were fitted to a pseudo-Voigt function assuming a linear background plus a pseudo-Voigt function for the SiC peak.
Figure 4. Optical details for diffraction measurements in ceramic composites
Figure 5. Diffraction pattern of Al$_2$O$_3$/SiC with a pseudo-Voigt profile fit; a. The fit for determining the Al$_2$O$_3$-peak position in the case of 25 weight-% whiskers. b. The SiC-peak position of the sample with 5 weight-% whiskers.
For the determination of the SiC-peaks the background was assumed to be an exponential function plus a constant, because the SiC-peak sits on the tail of the Al$_2$O$_3$-peak. The peak positions were than obtained from the functional parameters. Measurements were done for $\psi$ equal to $0^\circ$, $\pm18.43^\circ$, $\pm26.57^\circ$, $\pm33.21^\circ$, $\pm39.23^\circ$, $\pm45^\circ$ for $\phi$ equal to $0^\circ$, $60^\circ$, $120^\circ$.

To transform measured d-values into stresses the following elastic constants were used:

*Table 2. Elastic Constants used to determine the stress in the Al$_2$O$_3$/SiC-whisker composite*

<table>
<thead>
<tr>
<th>Phase</th>
<th>Plane</th>
<th>E [GPa]</th>
<th>$\nu$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al$_2$O$_3$</td>
<td>146</td>
<td>407[41]</td>
<td>0.23[41]</td>
</tr>
<tr>
<td>SiC</td>
<td>511+333</td>
<td>352[42]</td>
<td>0.21[41]</td>
</tr>
</tbody>
</table>

In situ Strain Measurements at Low Temperature

In order to measure the strain in a ceramic composite a cooling device mounted on a diffractometer was used. The cooling device consisted of two systems, a vacuum system to prevent the formation of ice and suppress heat convection, and a cooling system to control the temperature.

The sample was placed inside a Beryllium-dome, see Figure 6. A vacuum of $-10^5$ Torr was achieved inside the dome by using a mechanical pump and a turbo pump.
Figure 6. The set-up for in situ strain measurements
The sample holder consisted of a cold finger of a Dispelled Closed-Cycle Refrigeration System which was mounted on the quarter circle/goniometer attachment for a diffractometer. This system was equipped with a heater element controlled with an Air-products APD-E temperature controller. The temperature during each measurement was controlled to within ±0.5 K and measured with a chromel versus gold-0.07 atomic-% iron thermocouple attached the cooling stage. The set-up allowed strain measurements in the temperature range of 45-295 K.

To correct for shrinkage of the cold finger and the sample holder, the d-spacing of a Si-powder at different temperatures using several peaks at each temperature was measured, and by using Nelson-Riley method a calibration curve of the shrinkage versus temperature for the sample holder was obtained. This curve was used throughout the experiment as correction for shrinkage.

The samples used for the experiment were Al$_2$O$_3$/SiC whisker composite described above, powder of the Al$_2$O$_3$ and loosely packed SiC-whiskers in free form. The SiC/TiB$_2$ system was also used for this experiment but, as will be shown in the result and discussion sections, it did not yield a good result.

The measurements were done with a General Electric diffractometer and operating condition are shown in Table 3.
Table 3. Operating condition for in situ diffraction strain measurements

<table>
<thead>
<tr>
<th></th>
<th>Al₂O₃/SiC</th>
<th>SiC/TiB₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>Power</td>
<td>35kV/14mA</td>
<td>35kV/19mA</td>
</tr>
<tr>
<td>Radiation</td>
<td>Cu Kα</td>
<td>Cr Kα</td>
</tr>
<tr>
<td>Slits: Incident</td>
<td>0.4°</td>
<td>1°</td>
</tr>
<tr>
<td>Receiving</td>
<td>0.1°</td>
<td>0.2°</td>
</tr>
<tr>
<td>Filter</td>
<td>none</td>
<td>Vanadium</td>
</tr>
</tbody>
</table>

To eliminate any effects from thermal cycling, all the data for each specimen was taken in one run, it means that the temperature was never increased during the experiment and the specimen was never heated between the strain measurements at different temperatures. The measurements were done on the way down in temperature and to make sure that a temperature equilibrium was reached at the sample surface, the sample were held at the temperature of interest for 30 min before the measurement of the lattice parameter started.

Eshelby Calculations

Numerical Calculations

To solve equation (2.26, 2.27, and 2.31) a computer program written in Turbo Pascal was constructed and implemented on a personal computer machine. It meant that the strains in the matrix and in the inclusion can be calculated. The calculation requires that the material constants are known. In this study most of these were collected from the literature. In the case of
unstressed d-spacings and coefficient of thermal expansion for Al₂O₃ and SiC, measured values in this study were used.

**Elastic Constants**

The fact that elastic constants are slightly temperature dependent have been ignored and room temperature values have been used.

*Table 4. Elastic constants for Eshelby calculations*

<table>
<thead>
<tr>
<th>Single Crystal</th>
<th>C₁₁ (GPa)</th>
<th>C₁₂ (GPa)</th>
<th>C₁₃ (GPa)</th>
<th>C₄₄ (GPa)</th>
<th>lit ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiC (3C)</td>
<td>352</td>
<td>140</td>
<td>-</td>
<td>23</td>
<td>[43]</td>
</tr>
<tr>
<td>TiB₂</td>
<td>69</td>
<td>410</td>
<td>320</td>
<td>250</td>
<td>[44]</td>
</tr>
<tr>
<td>Polycrystalline</td>
<td>E (GPa)</td>
<td>v</td>
<td></td>
<td></td>
<td>lit ref.</td>
</tr>
<tr>
<td>average</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>402</td>
<td>0.23</td>
<td></td>
<td></td>
<td>[32]</td>
</tr>
<tr>
<td>SiC</td>
<td>401</td>
<td>0.18</td>
<td></td>
<td></td>
<td>[32]</td>
</tr>
</tbody>
</table>

**Coefficient of Thermal Expansion**

The coefficient of thermal expansion is strongly dependent on the temperature. For the materials used in this study, no measurements of the coefficient of thermal expansion at low temperatures were found in the literature. Several authors have measured the coefficient of thermal expansion for high temperatures (25°C-1000°C).
Table 5. Coefficient of thermal expansion data collected from literature

<table>
<thead>
<tr>
<th></th>
<th>$\alpha(\times 10^{-6}/K)$ at 25°C</th>
<th>$\alpha(\times 10^{-6}/K)$ at 25°C</th>
<th>polycrystalline average</th>
<th>lit ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\text{Al}_2\text{O}_3$</td>
<td>7.69</td>
<td>5.4</td>
<td>Yes</td>
<td>[45]</td>
</tr>
<tr>
<td>$\text{SiC (3C)}$</td>
<td>4.45</td>
<td>3.2</td>
<td>No</td>
<td>[46]</td>
</tr>
<tr>
<td>$\text{SiC (6H)}$</td>
<td>4.35</td>
<td>3.2</td>
<td>Yes</td>
<td>[47]</td>
</tr>
<tr>
<td>$\text{TiB}_2$</td>
<td>7.70</td>
<td>5.2</td>
<td>Yes</td>
<td>[48]</td>
</tr>
</tbody>
</table>

By using the values given in Table 5 and the measured d-spacings for a stress free powder, see Appendix B, it is possible to get a good estimate of the average thermal expansion between two temperatures.

SiC (6H) is believed to behave very similar to SiC (3C) in the temperature region of interest (45-293 K). In the absence of SiC (6H) powder data, data from SiC (3C) was used instead.

To get an estimate of how the d-spacing changes for a TiB$_2$-powder with temperature the data from Table 5 and data from JCPDS-file was used. This estimate is not very accurate since it yields a linear behaviour which is probably not the case. This is a source of uncertainty in the calculation but it is the best estimate one can produce when data is lacking.

Crystallographic Orientations and Shapes

The matrix in both composites are assumed to be isotropic and thus has polycrystalline averages been used as the material constants for the matrix phase. SiC whiskers grow in the [111]-direction, the two perpendicular directions are $[\bar{1} \bar{1} 2]$ and $[1 \bar{1} 0]$. The aspect ratio (L/d) has
been shown to have a small effect on the strains when it exceeds 10. In the Al$_2$O$_3$/SiC samples used in this study, the whisker dimensions will give an aspect ratio of about 25. In the calculations an aspect ratio of 10 was chosen. The TiB$_2$-particles were modelled as spheres.
CHAPTER 4

RESULTS

Residual Stresses in Si$_3$N$_4$ and SiC Caused by Grinding and Thermal Shock

The result from as received (ground) specimen and after thermal shock are presented first in the form of tables (Table 6-9) and then in figures (Figure 7-12). The first two figures (Figure 7-8) are showing the distribution for several samples of the residual stress after grinding in the two materials. The next four figures (Figure 9-12) show the influence of the quenching temperature on the residual stress. It is worth pointing out that, what is marked in the figures as "Ground specimen" (open circles) are the residual stress values before heat treatment. They are just shown for comparison.

All the results presented in this chapter are later discussed in Chapter 5, Discussion.
Table 6. Residual stress in $\text{Si}_3\text{N}_4$ as received and quenched in water

<table>
<thead>
<tr>
<th>Specimen no</th>
<th>Residual stress after grinding (MPa)</th>
<th>Quenching temperature (°C)</th>
<th>Residual stress after quenching (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SN1</td>
<td>-184±30</td>
<td>612</td>
<td>-231±36</td>
</tr>
<tr>
<td>SN2</td>
<td>-337±26</td>
<td>606</td>
<td>-369±53</td>
</tr>
<tr>
<td>SN3</td>
<td>-173±32</td>
<td>708</td>
<td>-267±26</td>
</tr>
<tr>
<td>SN4</td>
<td>-170±31</td>
<td>710</td>
<td>-283±51</td>
</tr>
<tr>
<td>SN5</td>
<td>-195±23</td>
<td>800</td>
<td>-412±46</td>
</tr>
<tr>
<td>SN6</td>
<td>-320±40</td>
<td>817</td>
<td>-453±59</td>
</tr>
<tr>
<td>SN7</td>
<td>-89±37</td>
<td>910</td>
<td>-349±79</td>
</tr>
<tr>
<td>SN8</td>
<td>-186±31</td>
<td>908</td>
<td>-341±41</td>
</tr>
<tr>
<td>SN9</td>
<td>-102±31</td>
<td>1000</td>
<td>362±31</td>
</tr>
<tr>
<td>SN10</td>
<td>-100±32</td>
<td>1000</td>
<td>-395±36</td>
</tr>
</tbody>
</table>

One $\text{Si}_3\text{N}_4$ specimen that was quenched from 1100°C cracked when it hit the water.
Table 7. Residual stress in Si$_3$N$_4$ as received and cooled in air

<table>
<thead>
<tr>
<th>Specimen no</th>
<th>Residual stress after grinding (MPa)</th>
<th>Cooling temperature (°C)</th>
<th>Residual stress after cooling (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SN12</td>
<td>-153±38</td>
<td>700</td>
<td>-192±12</td>
</tr>
<tr>
<td>SN13</td>
<td>-154±37</td>
<td>700</td>
<td>-149±23</td>
</tr>
<tr>
<td>SN14</td>
<td>-204±24</td>
<td>800</td>
<td>-260±19</td>
</tr>
<tr>
<td>SN15</td>
<td>-202±23</td>
<td>800</td>
<td>-278±56</td>
</tr>
<tr>
<td>SN16</td>
<td>-89±37</td>
<td>910</td>
<td>-163±38</td>
</tr>
<tr>
<td>SN17</td>
<td>-128±40</td>
<td>910</td>
<td>-356±47</td>
</tr>
<tr>
<td>SN18</td>
<td>-224±30</td>
<td>1000</td>
<td>-355±49</td>
</tr>
<tr>
<td>SN19</td>
<td>-101±31</td>
<td>1000</td>
<td>-430±58</td>
</tr>
<tr>
<td>SN20</td>
<td>-149±14</td>
<td>1100</td>
<td>-342±44</td>
</tr>
<tr>
<td>SN21</td>
<td>-224±30</td>
<td>1100</td>
<td>-304±53</td>
</tr>
<tr>
<td>SN22</td>
<td>-87±21</td>
<td>1200</td>
<td>-124±19</td>
</tr>
<tr>
<td>SN23</td>
<td>-87±21</td>
<td>1200</td>
<td>-48±26</td>
</tr>
</tbody>
</table>
Table 8. Residual stress in SiC as received and quenched in water

<table>
<thead>
<tr>
<th>Specimen no</th>
<th>Residual stress after grinding (MPa)</th>
<th>Quenching temperature (°C)</th>
<th>Residual stress after quenching (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SC1</td>
<td>-349±39</td>
<td>606</td>
<td>-372±44</td>
</tr>
<tr>
<td>SC2</td>
<td>-349±39</td>
<td>606</td>
<td>-349±57</td>
</tr>
<tr>
<td>SC3</td>
<td>-316±39</td>
<td>707</td>
<td>-402±60</td>
</tr>
<tr>
<td>SC4</td>
<td>-316±39</td>
<td>707</td>
<td>-441±37</td>
</tr>
<tr>
<td>SC5</td>
<td>-228±32</td>
<td>817</td>
<td>-433±62</td>
</tr>
<tr>
<td>SC6</td>
<td>-228±32</td>
<td>817</td>
<td>-462±29</td>
</tr>
<tr>
<td>SC7</td>
<td>-52±44</td>
<td>908</td>
<td>-472±44</td>
</tr>
</tbody>
</table>

One SiC specimen that was quenched from 900°C cracked when it hit the water.
Table 9. Residual stress in SiC as received and cooled in air

<table>
<thead>
<tr>
<th>Specimen no</th>
<th>Residual stress after grinding (MPa)</th>
<th>Cooling temperature (°C)</th>
<th>Residual stress after cooling (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SNC9</td>
<td>-343±39</td>
<td>800</td>
<td>-389±56</td>
</tr>
<tr>
<td>SNC10</td>
<td>-228±32</td>
<td>810</td>
<td>-248±34</td>
</tr>
<tr>
<td>SC11</td>
<td>-316±65</td>
<td>909</td>
<td>-409±54</td>
</tr>
<tr>
<td>SC12</td>
<td>-52±44</td>
<td>915</td>
<td>-394±39</td>
</tr>
<tr>
<td>SC13</td>
<td>-389±69</td>
<td>1000</td>
<td>-457±56</td>
</tr>
<tr>
<td>SC14</td>
<td>-389±69</td>
<td>1000</td>
<td>-429±49</td>
</tr>
<tr>
<td>SC15</td>
<td>-389±69</td>
<td>1100</td>
<td>-234±28</td>
</tr>
<tr>
<td>SC16</td>
<td>-190±29</td>
<td>1100</td>
<td>-222±27</td>
</tr>
<tr>
<td>SC17</td>
<td>-190±29</td>
<td>1200</td>
<td>-101±30</td>
</tr>
</tbody>
</table>
Figure 7. The distribution of residual stress over several samples after grinding in Si$_3$N$_4$. 
Figure 8. The distribution of residual stress over several samples after grinding in SiC
Figure 9. The residual stress induced after quenching Si$_3$N$_4$ in water as a function of quenching temperature. (The values for ground specimen indicates the stress in the sample before quenching)
Figure 10. The residual stress induced after quenching SiC in water as a function of quenching temperature. (The values for ground specimen indicate the stress in the sample before quenching.)
Figure 11. The residual stress induced after cooling Si$_3$N$_4$ in air as a function of cooling temperature. (The values for ground specimen indicates the stress in the sample before cooling.)
Figure 12. The residual stress induced after cooling SiC in air as a function of cooling temperature. (The values for ground specimen indicates the stress in the sample before cooling.)
Residual Stresses in Al$_2$O$_3$/SiC-whisker Composite after Different Thermal Treatments

The result shown below in Table 10-12 and in Figure 13-16 are experimentally derived according to Chapter 3 where triaxial stress measurements are described. All data shown here are separated into macro- and microstresses according to Chapter 2. First stress tensors from as received samples are shown and then the stress tensors measured after different thermal treatments. Data are then presented in form of figures, some of the figures will reoccur under the next heading, Eshelby Calculations, where measured data and calculated data are compared. A more extensive comparison but from a slightly different angle is presented under heading In situ strain measurements as a function of temperature.
Table 10. Stress tensors for Al₂O₃/SiC-whisker composite, as received. The values in parentheses are the errors (one standard deviation)

<table>
<thead>
<tr>
<th>Vₜ</th>
<th>As Received Samples (MPa)</th>
<th>Microstress</th>
<th>Microstress Al₂O₃</th>
<th>Microstress SiC</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.06</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>-160(73)</td>
<td>-4(22)</td>
<td>-1231(73)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>-160(73)</td>
<td>-10(8)</td>
<td>-65(23)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>-5(38)</td>
<td></td>
<td>5(0)</td>
</tr>
<tr>
<td></td>
<td>Microstress</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>79(73)</td>
<td>4(22)</td>
<td>0(8)</td>
<td>-1231(73)</td>
<td>-65(23)</td>
</tr>
<tr>
<td>73(73)</td>
<td>2(8)</td>
<td>-1143(73)</td>
<td>-37(9)</td>
<td></td>
</tr>
<tr>
<td>77(39)</td>
<td></td>
<td>-1205(36)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.12</td>
<td></td>
<td>-283(60)</td>
<td>0(18)</td>
<td>-1127(69)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>-198(60)</td>
<td>-8(7)</td>
<td>7.7(22)</td>
</tr>
<tr>
<td></td>
<td>Microstress</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>152(56)</td>
<td>1(14)</td>
<td>2(6)</td>
<td>-1127(69)</td>
<td>-7(22)</td>
</tr>
<tr>
<td>156(56)</td>
<td>1(6)</td>
<td></td>
<td>-1151(69)</td>
<td>-5(8)</td>
</tr>
<tr>
<td>171(34)</td>
<td></td>
<td>-1256(69)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.18</td>
<td></td>
<td>-32(82)</td>
<td>-7.27</td>
<td>4(10)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>-34(82)</td>
<td>5(10)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Microstress</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>226(83)</td>
<td>-3(27)</td>
<td>-2(10)</td>
<td>-1120(81)</td>
<td>14(27)</td>
</tr>
<tr>
<td>245(83)</td>
<td>-3(10)</td>
<td></td>
<td>-1116(81)</td>
<td>12(10)</td>
</tr>
<tr>
<td>243(36)</td>
<td></td>
<td>-970(34)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.24</td>
<td></td>
<td>-153(73)</td>
<td>-15(22)</td>
<td>3(8)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>-138(73)</td>
<td>-5(8)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Microstress</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>296(73)</td>
<td>2(12)</td>
<td>2(8)</td>
<td>-939(73)</td>
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</tr>
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<td>305(73)</td>
<td>0(8)</td>
<td></td>
<td>-968(73)</td>
<td>1(0)</td>
</tr>
<tr>
<td>273(39)</td>
<td></td>
<td>-902(37)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.30</td>
<td></td>
<td>-79(68)</td>
<td>-20(14)</td>
<td>4(8)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>-91(68)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Microstress</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>381(66)</td>
<td>9(13)</td>
<td>0(8)</td>
<td>-889(70)</td>
<td>-20(14)</td>
</tr>
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<td>380(66)</td>
<td>0(8)</td>
<td></td>
<td>-887(70)</td>
<td>7(10)</td>
</tr>
<tr>
<td>387(32)</td>
<td></td>
<td>-902(34)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Table 11. Measured stress tensors for Al$_2$O$_3$/SiC-whisker composite before and after quenching to liq. Nitrogen (77 K) and liq. Helium (4 K) from room temperature (295 K). The values in parentheses are the errors (one standard deviation)

<table>
<thead>
<tr>
<th>Stress component</th>
<th>Microstress</th>
<th>Microstress Al$_2$O$_3$</th>
<th>Microstress SiC</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>as received</td>
<td>quenched in liq</td>
<td>quenched in liq</td>
</tr>
<tr>
<td></td>
<td>N</td>
<td>He</td>
<td>N</td>
</tr>
<tr>
<td>$\sigma_{11}$</td>
<td>-160</td>
<td>-107</td>
<td>183</td>
</tr>
<tr>
<td></td>
<td>(73)</td>
<td>(81)</td>
<td>(82)</td>
</tr>
<tr>
<td>$\sigma_{22}$</td>
<td>-160</td>
<td>-101</td>
<td>-193</td>
</tr>
<tr>
<td></td>
<td>(73)</td>
<td>(81)</td>
<td>(82)</td>
</tr>
<tr>
<td>$\sigma_{33}$</td>
<td>-5</td>
<td>-22</td>
<td>-35</td>
</tr>
<tr>
<td></td>
<td>(38)</td>
<td>(40)</td>
<td>(39)</td>
</tr>
<tr>
<td>$\sigma_{12}$</td>
<td>-4</td>
<td>2</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td>(22)</td>
<td>(24)</td>
<td>(25)</td>
</tr>
<tr>
<td>$\sigma_{13}$</td>
<td>1</td>
<td>-13</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>(8)</td>
<td>(10)</td>
<td>(10)</td>
</tr>
<tr>
<td>$\sigma_{23}$</td>
<td>10</td>
<td>3</td>
<td>-3</td>
</tr>
<tr>
<td></td>
<td>(8)</td>
<td>(10)</td>
<td>(10)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>$V_{p=6%}$</th>
<th>Microstress</th>
<th>Microstress Al$_2$O$_3$</th>
<th>Microstress SiC</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>as received</td>
<td>quenched in liq</td>
<td>quenched in liq</td>
</tr>
<tr>
<td></td>
<td>N</td>
<td>He</td>
<td>N</td>
</tr>
<tr>
<td>$\sigma_{11}$</td>
<td>-79</td>
<td>-126</td>
<td>-127</td>
</tr>
<tr>
<td></td>
<td>(68)</td>
<td>(60)</td>
<td>(46)</td>
</tr>
<tr>
<td>$\sigma_{22}$</td>
<td>-91</td>
<td>-113</td>
<td>-151</td>
</tr>
<tr>
<td></td>
<td>(68)</td>
<td>(60)</td>
<td>(46)</td>
</tr>
<tr>
<td>$\sigma_{33}$</td>
<td>15</td>
<td>22</td>
<td>50</td>
</tr>
<tr>
<td></td>
<td>(32)</td>
<td>(35)</td>
<td>(31)</td>
</tr>
<tr>
<td>$\sigma_{12}$</td>
<td>10</td>
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<td>-8</td>
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<td>(17)</td>
<td>(10)</td>
</tr>
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<td>$\sigma_{13}$</td>
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<td></td>
<td>(8)</td>
<td>(6)</td>
<td>(4)</td>
</tr>
<tr>
<td>$\sigma_{23}$</td>
<td>-14</td>
<td>10</td>
<td>-4</td>
</tr>
<tr>
<td></td>
<td>(8)</td>
<td>(6)</td>
<td>(4)</td>
</tr>
</tbody>
</table>
Table 12. Measured stress tensors for Al$_2$O$_3$/SiC-whisker composite before and after cooling down in air to room temperature from 1200° C. The sample were at the elevated temperature for 5 minutes and for 20 minutes. The values in parentheses are the errors (one standard deviation)

<table>
<thead>
<tr>
<th>$V_c$=24%</th>
<th>Macrostress</th>
<th>Macrostress Al$_2$O$_3$</th>
<th>Macrostress SiC</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Cooled from 1200°C to RT</td>
<td>Cooled from 1200°C to RT</td>
<td>Cooled from 1200°C to RT</td>
</tr>
<tr>
<td>Stress component</td>
<td>as received</td>
<td>5</td>
<td>20</td>
</tr>
<tr>
<td>$\sigma_{11}$</td>
<td>-153 (73)</td>
<td>.73 (37)</td>
<td>-.76 (37)</td>
</tr>
<tr>
<td>$\sigma_{22}$</td>
<td>-138 (73)</td>
<td>.77 (37)</td>
<td>-.84 (37)</td>
</tr>
<tr>
<td>$\sigma_{33}$</td>
<td>-27 (38)</td>
<td>.6 (30)</td>
<td>4 (31)</td>
</tr>
<tr>
<td>$\sigma_{12}$</td>
<td>-15 (22)</td>
<td>4 (8)</td>
<td>5 (9)</td>
</tr>
<tr>
<td>$\sigma_{13}$</td>
<td>3 (8)</td>
<td>2 (5)</td>
<td>-2 (4)</td>
</tr>
<tr>
<td>$\sigma_{23}$</td>
<td>-5 (8)</td>
<td>2 (3)</td>
<td>1 (4)</td>
</tr>
</tbody>
</table>
Al₂O₃/SiC-whisker Composite

Figure 13. Average principal macrostress versus volume fraction for as received Al₂O₃/SiC-whisker composite
Figure 14. Average principal microstress in the two phases versus volume fraction for as received Al₂O₃/SiC-whisker composite
Figure 15. Average principal microstress in Al₂O₃/SiC-whisker composite after quenching to liq. Nitrogen and liq. Helium.
Figure 16. Average principal micro stress in Al$_2$O$_3$/SiC-whisker composite after cooling in air to room temperature from 1200° C, were the samples had been for 5 and 20 minutes.
Eshelby Calculations

The calculation results from equation (2.26) are presented in forms of figures. The parameters that have been changed are the volume fraction, $V_f$, and the temperature. The reason for choosing these parameters is that measurements are done for comparison. First Figure 17-19 show the influence of the volume fraction and of the temperature on the stress in the composite. In Figure 20 equation (2.30) has been applied and the measured values are also plotted.
Figure 17. Calculated stress in the matrix and inside the inclusion in a Al₂O₃/SiC-whisker composite as a function of volume fraction. The aspect ratio of the inclusion is 10, and the temperature is 295 K
Figure 18. Calculated stress in the matrix and inside the inclusion in a $\text{Al}_2\text{O}_3/\text{SiC}$-whisker composite as a function of temperature. The aspect ratio of the inclusion is 10, and the volume fraction is 25%
Figure 19. Calculated stress in the matrix and inside the inclusion in a SiC/TiB$_2$ composite as a function of temperature. The aspect ratio of the inclusion is 1 (spheres), and the volume fraction is 15.2\%
Figure 20. Calculated average stress in the matrix and average stress inside the inclusion and measured average principal stresses in a $\text{Al}_2\text{O}_3$/SiC-whisker composite as a function of volume fraction. The aspect ratio of the inclusion is 10, and the temperature is 295 K.
In situ Strain Measurements as a Function of Temperature

All the results shown here are in the form of figures with d-spacing versus temperature. To be able to compare measured d-values with calculated ones, the calculated strains were converted into d-spacings using the best fit curve for the loosely packed powder and equation (2.6). Figure 21-24 presents both calculated and measured data. The calculated data are the average according to equation (2.28).

Figure 23 and 24 shows the strain in the SiC/TiB$_2$ composite. The predicted and measured data do not agree. To see if the specimen was broken during sample preparation a biaxial stress measurement at room temperature was performed. The stresses are very close to zero in both phases (-69±45 for SiC and -11±13 for TiB$_2$).
Figure 21. d-spacing as a function of temperature in the Al₂O₃ matrix in a Al₂O₃/SiC-whisker composite
Figure 22. d-spacing as a function of temperature in the SiC whisker in a $\text{Al}_2\text{O}_3/\text{SiC}$-whisker composite
SiC/TiB$_2$-particle Composite

The SiC Phase, Peak (202)

- SiC/TiB$_2$-particle composite
- The Modified Eshelby model
- Estimated stress free d-spacings

Figure 23. d-spacing as a function of temperature in the SiC matrix in a SiC/TiB$_2$ composite
SiC/TiB₂-particle Composite

The TiB₂ Phase, Peak (201)

---

Figure 24. d-spacing as a function of temperature in the TiB₂ particle in a SiC/TiB₂ composite
CHAPTER 5
DISCUSSION

Residual Stress in Monolithic Si$_3$N$_4$ and SiC

**Ground Si$_3$N$_4$ and SiC**

Measurement results of the residual stress after grinding are presented in Table 6-9 and Figure 7-8. The distribution of stresses is more concentrated in the Si$_3$N$_4$ case than in the SiC case, and have a peak around 200 MPa. For SiC the number of specimens are limited but the range of stresses is broadened.

**Thermal shocked Si$_3$N$_4$ and SiC**

Buessem and Grover [50] performed a calculation of the thermally induced residual stress in quenched Al$_2$O$_3$. Their model is based on linear plasticity (vacancy creep) which is described by the Nabarro-Herring equation and a transient creep before steady state. They conclude that there are three factors determining plastic deformation during quenching, (1) the difference between the average temperature in the sample and the local temperature, (2) the deformability, $1/(3\eta)$ where $\eta$ is the viscosity, which is an exponential function of temperature, (3) the time period when the body experience temperature gradients within the range of plastic temperatures. Factor (1) will vary linear by the quenching temperature difference for a given rate of heat transfer. Factor (2) is a material factor which changes with temperature and factor (3) depends on the rate of heat
transfer, the quenching temperature difference, and the starting temperature.

The result from quenching Si$_3$N$_4$ and SiC in water and in air are presented in Table 6-9 and Figure 9-12. The straight lines in Figure 9-12 are drawn to indicate trends. They all show a trend of increasing compressive residual stress with increasing quenching temperature for low to intermediate temperatures. This increase seems to be linear up to a certain temperature for the samples cooled in air, but around 1000°C this behaviour become reversed and as the temperature is increased further the surface residual stresses approach zero. For samples quenched in water this phenomenon is not observed, as the quenching temperature is increased the compressive stresses increase until the sample cracks. The only difference between the two experiments is the cooling rate (i.e. rate of heat transfer). The rate of heat transfer is higher for quenching in a water bath than when cooling in air. This would indicate that factors (1) and (3) are the dominating contributors in this experiment, but as long as the deformability is an unknown function of temperature a certain answer can not be given.

The two materials, Si$_3$N$_4$ and SiC, behave very similar when thermally treated, but the magnitude of the stresses for SiC are slightly higher than for Si$_3$N$_4$. The reason for a decrease in the magnitude of the stresses at high temperatures when cooled in air are not known. It could be a relaxation due to cracking but not as severe as in the water quenched case. Scanning electron microscopy studies have been performed but the results were inconclusive.

Si$_3$N$_4$ cracked completely when quenched in water from 1100°C and the same thing happened for SiC when quenched from 900°C. The higher
thermal shock resistance for Si$_3$N$_4$ follows the calculated values of the thermal shock parameters [51].

A simple superposition of residual stresses introduced during grinding and during thermal conditioning can not be done. The stresses due to grinding are relaxed to some extent during the heating process, and the amount of relaxation is determined by time and temperature.

**Residual Stress in a Al$_2$O$_3$/SiC-Whisker Composites**

Table 10 and Figure 13 and 14 show the measured stress in as received samples. The macrostresses are probably due to cutting and grinding. The influence of amount reinforcing phase seems to be non existing, but the errors in the experiment are too big to completely rule out a dependence. Studies of ground monolithic Al$_2$O$_3$ [5] and monolithic SiC [49] show that grinding of SiC will produce higher compressive stresses than Al$_2$O$_3$, but no simple rule of mixture will apply here.

The microstress state (Table 10) is pure hydrostatic in both phases. These samples were hot isostatic pressed and the penetration depth of Cu-radiation in Al$_2$O$_3$ exceeds 40 μm, which means that surface effect will have a limited influence, and a hydrostatic stress state is expected.

The microstress in the carbide phase and in the alumina phase varies with volume fraction as is shown in Figure 14. An increase of reinforcing phase will increase the tensile stress in the matrix and decrease the compressive magnitude of the stresses in the whiskers. This is what is expected when the two phases have to counter balance each other. The stress in the alumina phase varies from about 50 MPa to 400 MPa and in the carbide phase from -1.3 GPa to -900 MPa. This is not the maximum
stresses in the composite, as will be shown in the next section, some
 crystallographic direction will experience even higher stresses. These
 results are very close to what is reported in the literature from both X-ray [21] and neutron [18,19] diffraction experiment.

In Figure 15 the principal stresses \((\sigma_{11}+\sigma_{22})/2\) for macrostresses and
\((\sigma_{11}+\sigma_{22}+\sigma_{33})/3\) for microstresses, before and after quenching from room
 temperature to 77 K and 4 K respectively are presented. No significant
 changes in the stresses are observed and the probable reason is elastic
 behaviour of both phases through out the experiment. An additional
 indication of elastic behaviour is that no changes of peak shapes were
 observed.

The experiment where the sample was heated to 1200°C and then
 cooled in 25°C air show a decrease of the magnitude of compressive
 macrostress. They decrease by a factor of two, and no significant
 difference between having the sample in the furnace for 5 minutes or for
 20 minutes was observed. The reason for this change in macrostress is not
 clear, but annealing of alumina has been observed by several authors [5, 6].

The macrostress after thermal treatment is the same for both times at
 elevated temperature, 5 minutes and 20 minutes. An explanation could be
 that the stresses are produced during cooling, and 5 minutes at 1200°C
 was sufficient to anneal the sample completely.

Eshelby Calculations

To exemplify some of the parameters that influence the stress in a
 ceramic composite, calculations were done when the volume fraction and
 the temperature were changed. The result are presented in Figure 17-19. In
Figure 17 the stresses in a Al₂O₃/SiC-whisker composite along the x₁-, x₂- and x₃-axis (see Figure 2) inside the inclusion are shown as a function of volume fraction. The reason for the much larger magnitude of the compressive stress along the x₃-axis is the fact that it is the same direction as the crystallographic direction [111] which have the highest stiffness. It is also along the long direction of the whisker. Figure 17 also clearly points out that the whiskers will be in compression and the matrix in tension and an increase of reinforcing phase will increase the stress in matrix. The model predicts this relationship to be linear.

Figure 18 and 19 compare the stresses in Al₂O₃/SiC-whisker composite and in a SiC/TiB₂-particle composite as a function of temperature. The particles are modelled as sphere with the x₁-, x₂- and x₃-axis representing [100], [010] and [001] respectively. Due to the fact that TiB₂ has a hexagonal structure, the stiffness along the x₃-axis will be the highest and hence the stress. The trends are similar, a decrease of temperature will increase the magnitude of all stresses. The difference between the two composite system is the mismatch in coefficient of thermal expansion. In the Al₂O₃/SiC-whisker composite the matrix phase and Al₂O₃ has a larger coefficient of thermal expansion than SiC, but in the SiC/TiB₂-particle composite the particles coefficient of thermal expansion is the highest. This will produce internal stresses in the two matrices of opposite sign, tensile in Al₂O₃ and compressive in the SiC matrix.

The magnitude of all the calculated stresses in Figure 17-18 show very high compressive stresses in the whiskers along the x₃-direction. For quite normal condition, a volume fraction of 15% at room temperature will give rise to a stress about -1.7 GPa.
A problem associated with calculating the stress in the SiC/TiB\textsubscript{2}-system was the lack of data for the coefficient of thermal expansion at low temperature for TiB\textsubscript{2}. To estimate the stresses the value at room temperature was used and it should be an overestimate. In the case of using measured data at low temperature for cubic SiC as input for calculating the stresses in hexagonal SiC the errors are believed to be marginal. The reason being the fact that the difference between hexagonal and cubic SiC is just a stacking fault and that whiskers used for measuring the d-spacing in cubic SiC will contain hexagonal parts.

There are four parameters that will alter the internal stress in a composite by Equation (2.26), they are the thermal mismatch, \((\alpha^1 - \alpha^M)\), the elastic mismatch, \((C^1 - C^M)\), the aspect ratio of the inclusion, \((L/d)\), and the volume fraction, \(V_f\). These should all be considered when one wants to understand the stress state in a ceramic composite.

Fig 20 shows the calculated stresses as a function of volume fraction, when the average is taken for the SiC-phase according to Equation (2.30), plotted together with measured microstresses in the two phases. The calculated values seem to predict the stress very well when the volume fraction is changed. Majumdar et al [19] have reported a similar plot for neutron diffraction experiments and using a simpler Eshelby model.

**Strain as a Function of Temperature in a Ceramic Composite**

The results from calculations and measurements are presented in Figure 21-24. It can clearly be seen that good agreement for the Al\textsubscript{2}O\textsubscript{3}/SiC composite is achieved (Figure 21,22). In this system both the same experimental set-up and diffraction line for measuring the stress-free
lattice-spacing and the stressed d-spacing in the composite was used. It is of vital importance to know the relative position of the unstressed lattice parameter at all temperatures and by using the same experimental procedure systematic errors will play a less important part. By knowing the coefficient of thermal expansion (see Appendix B) at all temperatures, the numerical calculation could be performed to exactly fit this system. The result that a $\text{Al}_2\text{O}_3/\text{SiC}$ composite behave elastic when quenched from room temperature to 4 K, can here easily be seen.

In the SiC/TiB$_2$ case, Figure 23 and 24, the measured values and the predicted ones do not agree. The result is hard to evaluate. The d-spacings in the composite were the only measured, and since no powder of the specimen was available the stress-free d-spacings had to be estimated. For SiC the measured values for SiC-whiskers used in the $\text{Al}_2\text{O}_3/\text{SiC}$ system were used. This whisker had a cubic polyform and the SiC in the SiC/TiB$_2$ composite was hexagonal, but this is not believed to be major problem. For TiB$_2$, data for thermal expansion or d-spacings at low temperature do not exist, instead the coefficient of thermal expansion at room temperature was used for the whole temperature range. This will give the calculated strains a linear relationship with temperature which is not likely to be the case. The unstressed lattice spacing at 295 K was collected from the JCPDS file and recalculated to suit this crystallographic direction. A systematic error of, for example, the 2$\theta_0$-position in the experimental set-up will produce uncertainties which are hard to estimate. The usage of data from literature is not optimal.

In spite of all the errors associated with the calculated data, the reason for poor agreement between calculated and measured data is believed to be some sort of stress relaxation during sample preparation.
prior to cooling. The sample is cut to a width of about 1 mm to accommodate easy heat transfer, and during the cutting procedure cracks could have been introduced, which the zero stress state indicates.
CHAPTER 6

SUMMARY

Residual Stress in Monolithic Si$_3$N$_4$ and SiC

1. The distribution of residual stresses after grinding is broader in the SiC case than in the Si$_3$N$_4$.

2. Quenching and rapid cooling of Si$_3$N$_4$ and SiC will introduce an increasing compressive surface residual stress with increasing quenching temperature.

3. The increase of compressive stresses is almost linear up to certain point, when in the water quenching case the sample cracks and in the cooling in air case the magnitude of the stresses decrease.

4. A simple superposition of residual stress introduced during grinding and during thermal conditioning can not be done, some of the stresses will relax during heating.

Residual Stress in Al$_2$O$_3$/SiC-Whisker Composite

1. X-ray diffraction permits the determination of the residual stress state in a alumina/silicon carbide (whisker) system
2. Measured values of microstresses show hydrostatic stress states in both phases.

3. The microstresses in the whiskers are quite high in magnitude (~1 GPa). The microstresses in the Al₂O₃-phase and in the SiC-phase show a both strong dependence on the amount of reinforcing phase.

4. The composite acts elastically.

5. A short hold time (less than 5 minutes) at an elevated temperature of 1200°C followed by cooling in air reveals some of the macrostresses.

Comparison of Measured and Predicted Stresses/Strains in a Ceramic Composite

1. The modified Eshelby model is a powerful tool to estimate the stresses in a ceramic composite.

2. To be able to compare measured and calculated data a good knowledge of the material parameters are very important. The influence of the temperature dependent coefficients of thermal expansion need to be known.

3. For the Al₂O₃/SiC-whisker system excellent agreement was found between measured and calculated values with increasing volume fraction, and the holds for increasing temperature.

4. For the SiC/TiB₂ system the comparison between calculated and measured data show no correlation, and the reason is believed to be stress relaxation during sample preparation.
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APPENDIX A

The Matrix Components for Stress and Strain Calculation

Eigenstrain or the stress free strain $e^{T*}$,

$$e^{T*} = \begin{pmatrix} e_{11}^{T*} \\ e_{22}^{T*} \\ e_{33}^{T*} \\ 2e_{23}^{T*} \\ 2e_{13}^{T*} \\ 2e_{12}^{T*} \end{pmatrix} \quad (A1)$$

Applied Stress $\sigma^A$,

$$\sigma^A = \begin{pmatrix} \sigma_{11}^A \\ \sigma_{22}^A \\ \sigma_{33}^A \\ \sigma_{23}^A \\ \sigma_{13}^A \\ \sigma_{12}^A \end{pmatrix} \quad (A2)$$
Thermal Strain $\mathbf{e}^T,$

$$\mathbf{e}^T = \begin{bmatrix} e_{11}^T \\ e_{22}^T \\ e_{33}^T \\ 2e_{23}^T \\ 2e_{13}^T \\ 2e_{12}^T \end{bmatrix} = (T_1 - T_2) \begin{bmatrix} \alpha_{11}^I - \alpha_{11}^M \\ \alpha_{22}^I - \alpha_{22}^M \\ \alpha_{33}^I - \alpha_{33}^M \\ 0 \\ 0 \\ 0 \end{bmatrix}$$ (A3)

Elastic Stiffness $\mathbf{C}$

$$\mathbf{C} = \begin{bmatrix} C_{11} & C_{12} & C_{13} & C_{14} & 0 & 0 \\ C_{12} & C_{22} & C_{23} & C_{24} & 0 & 0 \\ C_{13} & C_{23} & C_{33} & 0 & 0 & 0 \\ C_{14} & C_{24} & 0 & C_{44} & 0 & 0 \\ 0 & 0 & 0 & 0 & C_{55} & C_{56} \\ 0 & 0 & 0 & 0 & C_{56} & C_{66} \end{bmatrix}$$ (A4)

Simplifications can be done depending on which crystallographic system the elastic constant refers to.
The Eshelby Tensor $S$,

\[
S = \begin{bmatrix}
S_{1111} & S_{1122} & S_{1133} & 0 & 0 & 0 \\
S_{2211} & S_{2222} & S_{2233} & 0 & 0 & 0 \\
S_{3311} & S_{3322} & S_{3333} & 0 & 0 & 0 \\
0 & 0 & 0 & 2S_{2323} & 0 & 0 \\
0 & 0 & 0 & 0 & 2S_{1313} & 0 \\
0 & 0 & 0 & 0 & 0 & 2S_{1212}
\end{bmatrix}
\]  

(A5)

The different elements $S_{ijkl}$ are described in ref. [33].

The average Eshelby tensor $\langle S \rangle$

\[
\langle S \rangle = \begin{bmatrix}
\langle S \rangle_{11} & \langle S \rangle_{12} & \langle S \rangle_{13} & 0 & 0 & 0 \\
\langle S \rangle_{11} & \langle S \rangle_{12} & \langle S \rangle_{13} & 0 & 0 & 0 \\
\langle S \rangle_{11} & \langle S \rangle_{12} & \langle S \rangle_{13} & 0 & 0 & 0 \\
0 & 0 & 0 & 0 & 0 & 0 \\
0 & 0 & 0 & 0 & 0 & 0 \\
0 & 0 & 0 & 0 & 0 & 0
\end{bmatrix}
\]  

(A6)

where

\[
\langle S \rangle_{11} = \frac{(S_{1111} + S_{2211} + S_{3311} - 1)}{3} \quad \text{(A7a)}
\]

\[
\langle S \rangle_{12} = \frac{(S_{1122} + S_{2222} + S_{3322} - 1)}{3} \quad \text{(A7b)}
\]

\[
\langle S \rangle_{13} = \frac{(S_{1133} + S_{2233} + S_{3333} - 1)}{3} \quad \text{(A7c)}
\]
The matrix for calculating the stress field just outside the inclusion, \( \mathbf{B} \).

\[
\begin{bmatrix}
B_{11} & B_{12} & B_{13} & B_{14} & B_{15} & B_{16} \\
B_{12} & B_{22} & B_{23} & B_{24} & B_{25} & B_{26} \\
B_{13} & B_{23} & B_{33} & B_{34} & B_{35} & B_{36} \\
B_{14} & B_{24} & B_{34} & B_{44} & B_{45} & B_{46} \\
B_{15} & B_{25} & B_{35} & B_{45} & B_{55} & B_{56} \\
B_{16} & B_{26} & B_{36} & B_{46} & B_{56} & B_{66}
\end{bmatrix}
\]

\[
\mathbf{B} = \frac{1}{\mu} \begin{bmatrix}
B_{11} & B_{12} & B_{13} & B_{14} & B_{15} & B_{16} \\
B_{12} & B_{22} & B_{23} & B_{24} & B_{25} & B_{26} \\
B_{13} & B_{23} & B_{33} & B_{34} & B_{35} & B_{36} \\
B_{14} & B_{24} & B_{34} & B_{44} & B_{45} & B_{46} \\
B_{15} & B_{25} & B_{35} & B_{45} & B_{55} & B_{56} \\
B_{16} & B_{26} & B_{36} & B_{46} & B_{56} & B_{66}
\end{bmatrix}
\]

\text{(A8)}

where

\[
B_{ii} = n_i^2 - n_i^4 / (2(1 - v^M)) \quad \text{i=1,2,3}
\]

\[
B_{ij} = -n_i^2 n_j^2 / (2(1 - v^M)) \quad \text{i\neq j; i,j=1,2,3}
\]

\[
B_{14} = -n_1^2 n_2 n_3 / (2(1 - v^M))
\]

\[
B_{16} = n_1 n_2 - n_1^3 n_2 / (1 - v^M)
\]

\[
B_{25} = -n_2^2 n_3 n_1 / (1 - v^M)
\]

\[
B_{34} = n_3 n_2 - n_3^3 n_2 / (1 - v^M)
\]

\[
B_{36} = -n_3^2 n_1 n_2 / (1 - v^M)
\]

\[
B_{55} = n_1 n_2 - 2n_1^2 n_2 / (1 - v^M)
\]

\[
B_{55} = n_1 n_2 - 2n_1^2 n_2 / (1 - v^M)
\]

\[
B_{66} = n_1 n_2 - 2n_1^2 n_2 n_3 / (1 - v^M)
\]
APPENDIX B

The coefficient of thermal expansion of Al₂O₃ powder and SiC whisker

The coefficient of thermal expansion can be calculated using the following expression

\[ \alpha(T) = \frac{1}{d} \frac{\delta d}{\delta T} \]  \hspace{1cm} (B1)

Using Equation B1 for the best fit curves for Al₂O₃ powder and SiC-whisker the coefficient of thermal expansion as a function of temperature is presented in Table B1 and Table B2.

The best fits are:

**Al₂O₃ powder**

\[ d = 0.83032 + 1.0728 \times 10^{-6} \times T + 5.1472 \times 10^{-6} \times T^2 \]  \hspace{1cm} (B2)

**SiC-whisker**

\[ d = 0.83855 + 2.3359 \times 10^{-6} \times T + 2.0252 \times 10^{-6} \times T^2 \]  \hspace{1cm} (B3)

Table B1. The coefficient of thermal expansion of Al₂O₃ powder

<table>
<thead>
<tr>
<th>Temperature (K)</th>
<th>Measured ( \alpha ) (*10⁻⁶/K)</th>
<th>From literature ( \alpha ) (*10⁻⁶/K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>295</td>
<td>4.94</td>
<td>5.4 [45]</td>
</tr>
<tr>
<td>200</td>
<td>3.78</td>
<td></td>
</tr>
<tr>
<td>100</td>
<td>2.53</td>
<td></td>
</tr>
</tbody>
</table>
Residual stresses in Si₃N₄ and SiC have been measured with X-ray diffraction after grinding and thermal shock. The produced surface stresses are compressive after both treatments. The stresses show a strong dependence on the quenching temperature up to a certain temperature when cracks relax the stresses.

The influence of the amount of reinforcing phase on the residual stress state in a Al₂O₃/SiC whisker composite was investigated and correlated to a modified Eshelby model. The agreement is excellent. The composite was quenched in liquid He (4K) and the stress state measured after show no relaxation of stresses, indicating elastic behaviour.

A in situ strain measurement as a function of temperature conducted on a Al₂O₃/SiC whisker composite and a SiC/TiB₂ particle composite show very good agreement with the Eshelby model for the Al₂O₃/SiC system but no agreement for the SiC/TiB₂ system. The reason is believed to be stress relaxation during sample preparation.