Determination of Fracture Toughness versus volume fraction behaviour in an aluminium Metal Matrix Composite

"A quest for a stroke of luck", graduate report by A.H.C. Duwel

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ABSTRACT

The metal matrix composites provide an attractive and viable alternative for traditional structural alloys. Ceramic fibre-reinforced MMCs offer a spectrum of advantages in applications where high strength and specific stiffness are of primary concern. However, discontinuously reinforced MMCs, having whiskers, particulates or nodules as the reinforcing phase are preferred for a number of reasons.

In particular, incorporation of discontinuous ceramic reinforcements in an aluminium alloy offers improvement in elastic modulus, wear resistance, strength, reliability and control of physical properties as density and coefficient of thermal expansion. Discontinuous MMCs also provide the advantage of being machinable and workable. The primary disadvantages of MMCs are that they suffer from low strain to failure, low fracture toughness and inferior crack growth and fracture resistance when compared to the conventional metals. Detailed and structural studies of the variation of ductility with microstructural parameters are very few.

In order to extend the knowledge of the effects of microstructural parameters, this work systematically studies the effects of a variation in added volume fraction Al₂O₃ particles on the fracture toughness in a precipitation hardening AA6061 metal matrix. In this study, the powder metallurgy route is chosen, because total control (of composition and structure) over the entire production process is possible. Another advantage of the use of a powder metallurgy (PM) method is that the samples are produced in small batches, allowing the use of different powder compositions for every batch. The effect of a change in the used particle sizes is not studied. To guarantee an accurate determination of the fracture toughness and allow verification of the individual test methods, three different methods (designated ASTM E399, ASTM E1290 and ASTM E1737) have been applied. The validity of the determination of the fracture toughness by these test methods depends upon the establishment of a sharp-crack condition at the tip of the fatigue crack, in a specimen of adequate size.

The aim of this study is to produce MMC materials with different volume fractions of Al₂O₃ particles, and test the variation of fracture toughness. The most important steps in the experimental sequence are the hot compaction of the used powders, the fatigue precracking of the fracture toughness samples and the final fracture toughness testing.

From this research, it can be concluded that the production process is able to produce a 'good' MMC with high density and good bonding between the constituents. The condition that was not satisfied is the conformity with a commercial MMC. This must be kept in mind during comparison of the test results with fracture toughness values reported in literature. The preparation step has to be perfected, especially the fabrication of the chevron notch and the precracking procedure. The used equipment is satisfying, however no clarity exists as to which fatigue loading procedure has to be used in order to obtain a straight and flat crack front. Based on the fracture toughness results obtained it can be undoubtedly concluded that the fracture toughness decreases with increasing volume fraction in MMCs. This is confirmed by two of three test methods. Since the measured fracture toughnesses are high compared to the literature values, it is expected that a structural overestimation is included using this test method.
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NOMENCLATURE

\( a \) = crack length
\( a_0 \) = initial crack length
\( a_i \) = crack length at crack extension event \( i \)
\( a_{\text{surf, max}} \) = maximum surface crack length
\( a_{\text{surf, min}} \) = minimum surface crack length
\( a_s \) = any crack length measurement
\( A_{\text{pl(i)}} \) = surface area under the load/displacement curve

\( b_0 \) = initial length of the unbroken ligament
\( B \) = specimen thickness
\( B_{\text{THI}} \) = Tsai-Halpin coefficient

\( c_2, c_1, c_0 \) = fit constants of crack length - compliance polynome
\( C_1, C_2 \) = power law coefficient used of the power law fitting \( J \) versus crack extension
\( C \) = specimen compliance
\( C_{(i)} \) = uncorrected compliance at crack extension event \( i \)
\( C_{II} \) = specimen compliance, measured from the load line
\( C_{\text{EC(i)}} \) = for rotation corrected specimen compliance at crack extension event \( i \)
\( CMOD \) = crack mouth opening displacement
\( CMOD_{\text{p max}} \) = crack mouth opening displacement at maximum fatigue load
\( CMOD_{\text{p mean}} \) = crack mouth opening displacement at mean fatigue load

\( d \) = particle size
\( D \) = half distance between clip gauge attachment points
NOMENCLATURE

\( E \) = modulus of elasticity

\( E' \) = effective modulus of elasticity

\( E_{//} \) = composite modulus of elasticity in the reinforcement direction

\( E_m \) = matrix modulus of elasticity

\( E_r \) = reinforcement modulus of elasticity

\( f_{\text{curvature}} \) = ratio of curvature

\( f_{\text{oblique}} \) = ratio of obliqueness

\( f_v \) = volume fraction

\( h_{\text{dimple}} \) = dimple height

\( H^* \) = half distance between loading pins

\( J \) = J integral

\( J_{\text{el}} \) = elastic component of J

\( J_{\text{pl}} \) = plastic component of J

\( K \) = stress intensity

\( K_Q \) = unqualified critical stress intensity

\( m_{\text{AA6061}} \) = AA6061 mass added during blending

\( m_{\text{Al2O3}} \) = Al2O3 mass added during blending

\( m_{\text{inair}} \) = mass as measured in air

\( m_{\text{inwater}} \) = mass as measured in water

\( P \) = load

\( P_{\text{max}} \) = maximum fatigue load

\( P_{\text{mean}} \) = mean fatigue load

\( P_Q \) = load at critical crack extension
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<td>$r$</td>
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<tr>
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<tr>
<td>$r_y$</td>
<td>size of crack tip plastic zone</td>
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<td>$R$</td>
<td>rotation length</td>
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<td>$s$</td>
<td>aspect ratio of the reinforcement</td>
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<td>$ds$</td>
<td>increment of the contour path</td>
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<td>$T$</td>
<td>outward traction vector on the contour around a crack</td>
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<tr>
<td>$u$</td>
<td>displacement vector</td>
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<td>$U$</td>
<td>stored energy</td>
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<td>$V_x$</td>
<td>crack opening displacement at location $x$ from the load line</td>
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<td>$w_{dimple}$</td>
<td>dimple width</td>
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<tr>
<td>$W_{strain}$</td>
<td>$\int \sigma_y \varepsilon_y$ is the strain per unit volume due to loading</td>
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<td>$W$</td>
<td>specimen width</td>
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<td>$x, y$</td>
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<td>$\alpha_K$</td>
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\( \gamma_{(i-1)} \) = geometrical parameter

\( \varepsilon_{T,\text{local}} \) = local true strain

\( \eta_{(i-1)} \) = geometrical parameter

\( \lambda_v \) = interparticle spacing

\( \nu \) = Poisson’s ratio

\( \nu_{cm} \) = crack mouth displacement

\( \nu_l \) = load line displacement

\( \nu_{l(l)} \) = load line displacement at crack extension point i

\( \nu_{el(l)} \) = elastic component of load line displacement at crack extension point i

\( \nu_{pl(l)} \) = plastic component of load line displacement at crack extension point i

\( \nu_p \) = plastic component of clip gauge displacement

\( \Theta \) = rotation angle

\( \rho_{\text{Al}_2\text{O}_3} \) = \text{Al}_2\text{O}_3 density

\( \rho_{\text{AA6061}} \) = AA6061 density

\( \rho_{\text{composite}} \) = density of the composite material

\( \rho_{\text{water}} \) = density of the water medium

\( \sigma \) = applied stress

\( \sigma_\infty \) = far field stress

\( \sigma^* \) = critical global stress

\( \sigma_Y \) = yield stress

\( \mathcal{G} \) = energy release rate

\( \zeta_c \) = critical energy release rate
1 INTRODUCTION

The need for lightweight, high performance and wear resistant structural materials to satisfy the demands of aerospace, automotive and consumer-related industries was the trigger for the development and emergence of metal matrix composites (MMCs) [1]. The MMC material class provides an attractive and viable alternative for traditional structural alloys.

Ceramic fibre-reinforced MMCs offer a spectrum of advantages in applications where high strength and specific stiffness are of primary concern. However, discontinuously reinforced MMCs, having whiskers, particulates or nodules as the reinforcing phase are preferred for a number of reasons. In particular, incorporation of discontinuous ceramic reinforcements in an aluminium alloy offers improvement in elastic modulus, wear resistance, strength, reliability and control of physical properties as density and coefficient of thermal expansion, thereby providing improved mechanical performance when compared to the unreinforced metal matrix [2-11]. Specifically, the discontinuously reinforced aluminium matrix composites offer advantages such as a 15-40 % increase in strength and a 30-50% increase in stiffness [12, 13] and low density while generally maintaining the receptivity to processing and characterisation techniques as applied for conventional metals. Discontinuous MMCs provide the additional advantage of being machinable and workable.

The primary disadvantages of MMCs is that they suffer from low strain to failure, low fracture toughness and inferior crack growth and fracture resistance when compared to the conventional metals [14 - 24]. Detailed and structural studies of the variation of ductility with microstructural parameters are very few and complicated.

The yield strength of the matrix is very important in determining the fracture toughness because it determines the size of the plastic zone at the crack tip. Particle size and volume fraction have their influence on the local yield strength and therefore will have an indirect influence on fracture. Besides the change in yield strength, the particle size and volume fraction also have a direct influence on fracture, therefore the influence of MMC parameters is the sum of all influences. To illustrate the complex relations between some parameters and fracture the diagram in figure 1 is included. When experimentally evaluating the dependencies of fracture, it would be best if all parameters are held as constant as possible, however this may not always be possible.

In order to extend the knowledge of the effects of microstructural parameters, this work systematically studies the effects of a variation in added volume fraction Al₂O₃ particles on the fracture toughness in a precipitation hardening AA6061 metal matrix. The effect of a change in the used particle sizes is not studied, because the limited availability of reinforcement powders.
Discontinuous MMCs can be produced by ingot metallurgy, powder metallurgy and mechanical alloying techniques, where each technique results in a composite having different mechanical properties. In this study, the chosen production method is a powder metallurgy route (hot vacuum pressing of the composite powders), because then total control (of composition and structure) over the entire production process is possible. Another advantage of the use of a powder metallurgy (PM) method is that the samples are produced in small batches, allowing the use of different powder compositions for every batch. The hot press method produces tablets that can be machined to satisfy standard disk-shaped compact tension geometry. A drawback of the use of this PM method is the limitation in the size of the samples, the maximum achievable diameter of the samples is 50 mm, whereas conventional fracture toughness test methods use much larger samples.

In order to guarantee an accurate determination of the fracture toughness and allow verification of the test methods, three different methods have been applied. The test methods are described in the annual book of standards [25], published by the American Society for Testing of Materials and are designated ASTM E399, ASTM E1290 and ASTM E1737. The test methods involve testing of notched specimens that have been precracked in fatigue by loading in tension. Load versus displacement across the notch at the specimen edge is recorded. From these load/displacement records, three toughness determinations can be performed sequentially. The validity of the determination of the fracture toughness by these test methods depends upon the establishment of a sharp-crack condition at the tip of the fatigue crack, in a specimen of adequate size.

It is found that the fracture toughness decreases with increasing volume fractions, and that fracture toughness testing of this type of MMCs is complicated. Because of the complications during fracture toughness testing it is hard to identify the actual fracture toughness versus volume fraction behaviour. Based on the discussion of fracture toughness variation with volume fraction of alumina, it can however be concluded that the fracture toughness of an aluminium - MMC composite, where the aluminium is embedded in an MMC network, decreases linearly with an increase in volume fraction.
2 THEORETICAL Backgrounds

2.1 Types of MMCs
The term 'MMC' represent a wide range of metals reinforced with a wide range of reinforcements, with different geometries. Therefore, it is desirable to divide this class of materials into smaller sections that have internal similarity. The clearest division depends on the presence of the reinforcement, either as a continuous fibre, a discontinuous fibre (or whisker) or in the form of particles. This classification is depicted in figure 2.

![Continuous Fibres, Whiskers, Particles](image)

*Figure 2: Schematic representation of the three main reinforcement types [26].*

The continuous fibre reinforced metal matrices provide the best mechanical properties, however they only exhibit excellent mechanical properties in the reinforced direction and are very expensive to produce. The production of discontinuous reinforcements on the other hand is much cheaper, because the particles, short fibres or whiskers can be added in regular metal production processes, without major modifications. After production, discontinuous MMCs can be formed and reshaped, which is impossible for continuously reinforced composites. The main drawback of the application of discontinuous fibres or particles is the lower strengthening efficiency.

2.2 Mechanical properties of MMCs

2.2.1 Load transfer
In order to understand the mechanical properties of composites it is necessary to gain insight into the concept of load sharing between the constituents of the composite. The concept of load sharing is based on the transfer of the load from the metal matrix to the ceramic particle or fibre. In a composite subjected to an externally applied load, the two constituents will not carry the same portion of the load. When the applied loads are low and the deformation of the composite remains elastic, the strains introduced into both constituents are the same. When it is given that the stiffness of the composite is higher than the stiffness of the matrix, the conclusion is easily drawn that the stresses in the reinforcement must be higher than the stresses in the matrix to induce the same strains.

Provided the composite behaves elastically, the distribution of the load will be independent on the magnitude of the externally applied load.
2.2.2 Other strengthening mechanisms

Apart from the load transfer mechanism active in any composite, there are other mechanisms playing a role in the determination of the mechanical behaviour of MMCs. The main reason for the increase in yield and tensile strength of the MMCs over their unreinforced counterparts can be attributed to the following contributions:

**Quench strengthening**

The contribution in the additional strength in MMCs due to quench strengthening finds its origin in the production phase where the composite is cooled from a higher temperature [27]. Upon cooling the differences in thermal coefficient of expansion result in internal stresses, which are relieved by the generation of dislocations through the "punch-out" mechanism or stored as thermal residual stresses.

**Grain size strengthening**

Typically, the grains are smaller in a composite than they are in monolithic material. The amount of strengthening caused by substructure strengthening is dependent on whether the material retains its dislocation structure on annealing. When particulates prevent recrystallisation effectively, the material will retain its dislocation structure [28]. Grain size strengthening increases the yield strength of MMCs [29].

**Constrained plastic flow**

Strengthening also occurs through constraining of plastic flow and the resulting triaxial stress distribution in the matrix. The particles resisting plastic flow generate an average internal stress or back stress in the matrix, thereby lowering the total matrix load [28]. When the total matrix load is reduced, an increase in the ultimate tensile strength of the MMC is expected.

**Inhibition of plastic relaxation**

Plastic relaxation is a mechanism through which local stress concentrations are relieved due to accumulating dislocations. This mechanism also results in the generation of secondary dislocations at the interface [30,31].

**Precipitation hardening**

Precipitation hardening is an additional strengthening mechanism active in aluminium MMCs. For precipitation hardening to occur, the second phase must be soluble at an elevated temperature but must exhibit decreasing solubility at lower temperatures. Precipitation hardening increases grain growth and recrystallisation resistance, the presence of finely dispersed precipitates also complicates dislocation motion.
2.3 Heat treatment of aluminium alloys

Conventional heat treatment of precipitation hardening aluminium alloys, a so-called precipitation treatment consists of two main steps. These two steps can be explained using the quasi-binary phase diagram for Al-Mg-Si alloys included in figure 3:

Step one, indicated by the dot in the phase diagram, is the solutionising step of the heat treatment. In this step, the alloying elements (or precipitating elements) are brought in a solid solution. The alloy composition and the solutionising temperature must chosen such that it is possible to anneal in the single-phase area of the diagram to eliminate the formation of any second phases. It is important that the alloying elements are completely solved in the aluminium phase, therefore the solutionising time must not be underestimated.

During the second step, controlled growth of precipitates in the two-phase area of the diagram takes place. In order to make sure that the maximum hardening capacity is reached, quenching in water is applied to freeze the structure obtained after the solutionising treatment. Because of the quenching, all alloying elements remain in solid solution and the formation of coarse precipitates during the early stages of cooling from the homogenisation temperature is prevented. The sample is sequentially heated to the ageing temperature, which is in the lower temperature region of the phase diagram. During this ageing treatment precipitates form, which enhance the mechanical properties of aluminium alloys. The mechanical properties of the alloy improve with ageing time, until a maximum in mechanical performance is achieved. After this maximum performance peak, the mechanical properties of the alloy will degrade.

The ageing time for which optimum mechanical properties are achieved is called the peak ageing time and is often referred to as the T6 condition. A standard T6 heat treatment for an AA6061 alloy consists of a solutionising step at 530 °C for two hours followed by heating the quenched sample to 175 °C where the alloy is artificially aged for 8 hours.

The optimum heat treatment times for MMCs are expected to be slightly different for MMCs, the same applies for the precipitation kinetics. The precipitation characteristics can be altered by the
differences in energy levels near the reinforcement or chemical reactions between reinforcement and aluminium matrix. Since the reinforcement used here is inert and aluminium-based, it is not expected that any reactions take place at the interface, however modification of the precipitation characteristics by different internal energy distributions is expected.

High dislocation densities at interfaces may substantially lower the activation energy required for precipitation formation. This local lowering of the activation energy introduces preferred sites for precipitation at the interface, which is experimentally verified by Das et al [32]. Das et al also observed that the increased precipitation rates at the interface cause a depletion of precipitating elements in the region directly adjacent to the interface. Because of this depletion, it is expected that a weak layer around the reinforcement is formed.

A structural difference between the heat treatment of MMCs and the heat treatment of aluminium alloys lies in the ductility-recovery on over-ageing. In figure 4, the effect of over-ageing is visualised for three different MMCs. The important difference between MMCs and aluminium alloys is that aluminium alloys recover their toughness upon overageing as opposed to MMCs. This can be explained by the occurrence of deteriorating (chemical) processes at the interface, for example the formation of coarse precipitates.

2.4 Fracture in MMCs

Failure is the separation, or fragmentation, of a solid body into two or more parts under the action of stress.

Fractures can be divided into two general categories, ductile fracture and brittle fracture. Ductile fracture is characterised by serious plastic deformation both before and during the crack propagation and it is characterised by a relatively slow, sometimes stable, crack propagation rate. Brittle fracture in metal is characterised by a rapid crack propagation, having a deformationless fracture mode and a fracture mechanism with low deformation levels.

The difference between fracture mode and fracture mechanism is that the fracture mode is a macroscopic feature whereas the fracture mechanism can only be determined by microscopic examination and is therefore called a microscopic feature of a material. Fracture modes may very well be determined by the loading type and specimen type, fracture mechanisms are primarily determined by the material and therefore may be called material properties, although environmental effects may also affect fracture modes.

Brittle fracture is to be avoided at all cost in engineering applications since it occurs without warning and usually produces disastrous consequences. In MMCs the fracture toughness is
thought to be drastically reduced (with respect to the unreinforced metal) by the addition of a ceramic phase. Therefore a lot of research has been done in order to reduce the brittle nature of fracture in MMCs. Avoiding brittle fracture is a crucial condition for successful application of MMCs in structures.

2.4.1 Failure processes in structural alloys.

The fracture mechanisms in MMCs are closely related to and heavily influenced by the fracture mechanisms of the matrix alloy, therefore a small-scale discussion of fracture in metals is in place. In engineering alloys, fracture is determined by the microstructure of the alloy. As can be seen in figure 5, the nature of these engineering metals is complex and the factors influencing fracture are diverse, varying from the presence of a high melting point secondary phase to the presence of dislocations in the grains or the presence of a grain boundary as an easy fracture propagation path.

![Substitutional Atom Interstitial Atom Edge Dislocation Incoherent Precipitation Coherent Precipitation High Melting Point Secondary Phase Slip Lines Screw Dislocation Intergranular Fracture Layers of Grain Boundary Precipitates Transgranular Fracture Grain Boundary Precipitation](image)

**figure 5:** Factors that have an influence on fracture in structural alloys [33].

Generally, these transgranular and intergranular fracture paths can be associated with the following fracture mechanisms:

**Ductile transgranular fracture by microvoid coalescence.**

Ductile fracture can often be immediately recognised by an examination with the naked eye, because of the extensive deformation occurring before fracture. This plastic deformation results in a strong contraction of the final fracture surface that can easily be recognised.

In MMCs the matrix material is under heavy internal constraint, as described in section 2.2.2. Even if the fracture of the MMC matrix material is ductile by itself, the extensive deformation (and thus the ductile appearance of the fracture surface) will be prohibited by the internal constraint.

When the surfaces of a ductile fracture created by microvoid coalescence are subjected to a microscopical examination however, a dimpled appearance is revealed. The shape of the dimples is directly related to the type of loading as can be seen in figure 6. The size and depth of the dimple are determined by the amount of void growth taking place. In general, an increase in void growth (shown by an increase in void depth and size) increases the fracture toughness of metals.
Brittle transgranular fracture by cleavage

Cleavage is the ultimate brittle fracture mechanism. It takes place by the separation of atoms along well-defined (low-energy) crystal planes. A cleavage fracture surface would have matching planes and will be completely flat and featureless. However, structural alloys are polycrystalline and therefore a cleavage surface is faceted. The crack that propagates through a grain in a favoured direction has to change direction as it crosses a grain boundary.

Pure cleavage is rarely observed in structural metals because of the presence of precipitates that complicate the fracture path. Even in precipitate-free aluminium, cleavage is rarely observed because of the small differences in bond strength between different crystal planes. When these differences are small, it is expected that a propagating crack easily switches to another crystal plane. Despite the rare occurrence, cleavage fracture is not excluded because it might result from the different stress distributions in MMCs.

A typical microstructural feature of cleavage is the existence of river patterns (as depicted in figure 7), that are steps between cleavage on parallel planes. These river patterns always converge in the direction of local crack propagation.
**Intergranular fracture**

Intergranular fracture occurs as a result of sustained load fracture or because of the segregation of embrittling particles/precipitates at the grain boundaries. Intergranular fracture has two appearances caused by presence or absence of void coalescence. The difference in appearance between grain boundary separation with and without microvoid coalescence is depicted in figure 8.

![Intergranular Fracture](image)

**figure 8:** Different appearances of intergranular fracture, depending on the formation of voids at the grain boundaries [33].

### 2.4.2 Failure processes in MMCs

When fracture of MMCs is investigated, it is useful to separate the total event of fracture in several steps. In MMCs the fracture process can be split into three sequential phases, being void initiation, growth and coalescence. The definition of these three steps is possible, because the appearance of the fracture surface of MMCs is generally known. The fracture surface of MMCs has a dimpled appearance characteristic for a matrix ductile rupture mechanism and this mechanism consists typically of the three steps of nucleation, growth and coalescence. The goal of fractographic research done on MMC materials is to determine whether the macroscopically brittle nature of fracture in MMCs is inherent to the class of material, or that changing the material or processing properties may reduce the brittleness. To provide an answer to this question, fundamental knowledge about the different stages of fracture is needed.

The extension of simple models developed to describe the ductile fracture of monolithic alloys to discontinuously reinforced MMCs implies that the onset of void nucleation is the parameter controlling ductility in these materials. Because of the apparent importance of the nucleation of voids, this subject has been extensively researched.

Void initiation can either take place in the matrix far away from any particle, or at the interface near a particle. In the following two paragraphs, the stage of void nucleation will be discussed in detail.

**Void nucleation in the matrix**

You et al [34] stated that the initial step of void nucleation occurs in the matrix because of the increased levels of stress and plastic constraint, caused by the addition of the reinforcing particles. The nucleation of voids is then followed by the final stage of decohesion or cracking of the particles. The assumptions made by You et al have been backed up by experimental observations by Roebuck [35].
Void initiation on the matrix/particle interface

When it is assumed that the voids initiate near the particle, there are two possible causes for the nucleation of a void; the first cause is the fracture of the reinforcing particle the second is debonding of the matrix/reinforcement interface. An illustration of the two modes of void nucleation is provided in figure 9.

- Void initiation near the particle
  - Force direction
  - Precipitates or inclusions
  - Complete separation along region of picture, leaving on the two surfaces cusp or dimples
  - Void growth
  - Void coalescence

(a)

- Void initiation near the particle
  - Force direction
  - Precipitates or inclusions
  - Particle fracture
  - Void coalescence
  - Void growth

(b)

*figure 9:* Schematic illustration of void nucleation modes near particles, representing both void nucleation by debonding (mode a) and void nucleation by fracture (mode b) [36].

Both nucleation modes have been experimentally validated (particle fracture by You [34], Lloyd [37] and Roebuck [35], particle decohesion by Manoharan and Lewandowski [38] and Stephens et al [39]). Examples of scanning electron micrographs showing both particle cracking and debonding at the matrix/particle interface are shown in figure 10.

*figure 10:* Examples of scanning electron micrographs showing voids nucleated by particle fracture (a) and matrix/particle decohesion (b) [40].
**Void initiation through interfacial debonding**

Most researchers (Nair [41] being one of them) assume that fracture in discontinuously reinforced MMCs follows the same sequence as in structural alloys, namely nucleation at particles followed by failure in the matrix by void coalescence. Manoharan and Lewandowski [38] observed the straining of an MMC in a SEM and have experimentally isolated and identified the nucleation stage. They concluded that the stage of nucleation takes place at the matrix/reinforcement interface.

The assumption that voiding nucleates near the particle is the most plausible assumption, when stress concentrations are taken into account. Chingsen et al [42] modelled the stress in the area of a crack interacting with a particle, from their results (included in figure 11) it follows that nucleation at the interface is to be expected. Because the stress levels at the interface are comparable to or higher than the stress levels in the matrix, voids will initiate at the interface instead of in the matrix unless the interface is very strong.

![Stress distributions around a crack in interaction with a particle. The values included with the iso-stress curves are the factors with which the externally applied stress must be multiplied. The externally applied stress is 0.6σY (composite).](image)

**Void initiation through fracture of the reinforcing particles**

The third assumption is that voids initiate through the fracture of the reinforcing particles. Particle fracture will occur when a low-strength particle that carries a large portion of the load is applied (this is the case for high-aspect ratio particles). A larger ceramic particle will fail earlier than a small ceramic particle.

Since particle fracture and interfacial debonding are known to occur in the same composite depending on subtle changes in microstructure, it can be concluded that the difference in strength between the interface and the particle is small. Estimates of interfacial strengths by Flom et al [43] and Davidson [44] show that the strength of the interface lies in the range of 1650 - 3000 MPa, SiCₚ fracture stresses are a function of particle size and are likely to vary around 3000 MPa. This confirms the coexistence of and the alternation between two void nucleation modes.
2.4.3 Effect of microstructural variables on fracture

It is certain that void nucleation, growth and coalescence cause fracture in MMCs, as is the case for many unreinforced structural alloys. However, the nucleation of voids is caused by the application of an external load and the stress concentrations that arise at the particle/matrix interface due to load transfer from the matrix to the reinforcement. When trying to quantify the load carried by the reinforcement, the first problem arises, since prediction of the stress levels at the reinforcement is possible, but this stress level will vary for every particle since most reinforcements are irregularly sized and shaped.

Matrix microstructure

The magnitude of the stress that can be supported within the matrix is of great importance when considering failure by void nucleation, as well as the ability of the matrix to flow under heavily constrained conditions. (Flow under constraint is dependent on the combination of yield stress and work hardening rate [26]). Besides these two parameters, many microstructural parameters and microstructure-related features influence fracture. The most important features for structural alloys are summarised by Ewalds & Wanhill [33]:

- Second phases
- Particles and precipitates
- Grain size
- Fibring and texturing owing to mechanical working.

For MMCs these features (except for mechanical working) are expected to be roughly the same, however confirmation is necessary. It is expected that the effects of the finer microstructural parameters such as second phase particles are less noticeable when the influence of the reinforcing particles to the fracture process is strong.

Reinforcement Volume

With increasing volume fraction of reinforcing particles, the local strain in the matrix between the particles becomes higher (provided that the particle is more rigid than the matrix surrounding it), resulting in premature fracture of the matrix. This is caused by the fact that, when strain is the dominating factor for failure, the distance over which this strain should be accomplished, i.e. the interparticle spacing, becomes shorter with increasing particle volume fraction.

Fractographic investigation into the effect of reinforcement volume fractions on fracture mechanisms by Kim et al. [45] showed that in a composite reinforced with 10 vol.% ceramic particles, fracture occurred by the nucleation of voids at large particles, followed by growth until coalescence. On the other hand, fracture in a composite with 30 vol.% ceramic particles was rapid and unstable. Since the constraint imposed on the matrix material determines the plastic zone size, which on its turn influences the overall fracture behaviour, it is assumed that an increase in reinforcement volume fraction leads to a decrease in plastic zone size and thus in fracture toughness.

Interfacial strength

Interfaces in conventional alloys exert an important influence on the fracture behaviour. In MMCs, this influence is expected to be even greater, because of the larger total interface surface, the chemical instabilities of most interfaces and their incoherence. The interfaces play an important role as nucleation sites for precipitation, recrystallisation and fracture and as chemical reaction sites and high speed diffusion paths.
Reinforcement distribution
Fracture properties depend heavily on local conditions. Local conditions can influence the direction in which the crack propagates or the region where the crack nucleates.

It has been reported in literature that the formation of clusters during processing has detrimental effects on the fracture properties of MMCs. Llorca [46] showed by FEM-modelling that the reinforcement distribution has a large impact on reinforcement fracture because the distribution can substantially alter the pattern of plastic flow and the triaxiality levels in the matrix. Because of his modification, the stresses in the reinforcement are higher in regions with high particle densities, leading to premature reinforcement failure. Experimental results [1, 37 and 47] confirm the early occurrence of reinforcement fracture. Srivatsan [1] also concluded from an examination of the fracture surface that fracture occurs early in the particle-rich regions and proceeds quickly. However, if there are particle-rich regions, there must be particle-lean regions. These particle-lean regions aid in the retardation and are used for linkage of the crack.

Lloyd [37] observed that the fracture process is controlled by the large triaxial stress intensification occurring in a cluster. He concluded this because the observation of unique features in the clustered areas, which are not present in structural alloys. The main difference between the stress state in structural alloys and MMCs is the large triaxiality. In a reinforcement cluster, the carried stresses are higher than in the bulk of the composite, this means that void nucleation is enhanced in these regions of high stress.

When the occurrence of clusters is extreme, the structure will resemble an aluminium structure surrounded by an MMC network. In this structure, it expected that the fracture initiates fully in the network, whereas the growth of the voids takes place in the aluminium particles.

Material porosity
Porosity is the permanent presence of voids after production. These voids are very hard to eliminate during PM production and are have deteriorating effects on fracture toughness. The presence of these voids can partially be attributed to an insufficiently optimised production process, however microvoids also exist in commercially produced PM material.

Since voids already exist when the specimen is loaded, the stage of void initiation is skipped and immediate void growth takes place at low stress levels. This void growth takes place at low stresses because of stress concentrations around these voids. The shape of the present voids determines the stress intensities (and thus the stress level necessary for void growth).
2.5 Fracture mechanics

2.5.1 Basic concepts

Fracture mechanics originated in the work of Inglis (1913) who showed that local stresses near an ellipsoidal hole are usually much higher than that in the bulk. He derived the following expression for the stress distribution near a surface crack with length \( a \) (or \( 2a \) in the case of an internal crack) and crack tip radius \( r \):

\[
\sigma = \sigma_\infty \left(1 + 2\sqrt{\frac{a}{r}}\right)
\]

where:
- \( \sigma_\infty \) = far field stress
- \( a \) = crack length
- \( r \) = crack tip radius

This equation provides us with easy to calculate stress concentration factors. When the crack tip radius is decreased, the stress concentration will increase quickly, reaching infinity for a sharp crack. This will automatically result in failure at low stress levels, since the production phase inevitably introduces small cracks in the materials’ surface. Based on the engineering experience at that time, this last consequence can be rejected because most materials performed well under high loads.

Griffith (1920), who suggested that a crack could not propagate unless the total internal energy of the system is decreased, solved this problem. The change in internal energy in tough materials has a complex nature, due to the effective energy absorption at the crack tip by plastic deformation. When considering brittle materials, like glasses, this complexity is strongly reduced as a consequence of the lack of plasticity at the crack tip. In this case, the only energy penalty associated with crack extension is the energy required for the creation of a new surface. Griffith showed that the change in stored energy of loaded plate of unit thickness as result of the introduction of an internal crack of length \( a \) is given by:

\[
\begin{align*}
U &= -\frac{\sigma^2 \pi a^2}{E} \quad \text{(decrease in stored energy)} \\
U &= 4a\gamma \quad \text{(increase in surface energy)}
\end{align*}
\]

where:
- \( \gamma \) = surface energy
- \( \sigma \) = applied stress
- \( E \) = modulus of elasticity
- \( U \) = stored potential energy

When these two contributions are summarised and plotted against the crack length (as is done in figure 12), it is visible that a critical crack length can be defined for which spontaneous crack extension will occur.
The critical crack length can be found by differentiating equations (2a) and (2b) and setting the result to zero.

To encompass tougher materials, Irwin (1948) extended this approach by adding an extra term to the equation of total internal energy, the energy release rate \( \zeta \). Because of the all-embracing nature of the energy release rate, it includes all effects at the crack tip for a given material and geometry. For the global stress required inducing spontaneous fracture in a component, Irwin developed the following equation:

\[
\sigma_* = \sqrt{\frac{\zeta_c E}{\pi a}}
\]

(3)

where: \( \sigma_* \) = critical global stress

\( \zeta_c \) = critical energy release rate

In equation 3 local phenomena are not explicitly included, which makes it simple to use, but of limited applicability. The link between energy approach and stress field approach is provided by the definition of stress intensity factor \( K \).

The measurement of this parameter depends on the material properties and on the type of crack extension taking place (unstable growth, stable growth or fully plastic behaviour). The determination methods also originate from a different field of fracture mechanics, measurement of the stress intensity factor \( K \) can be done using Linear Elastic Fracture Mechanics (LEFM) whereas the J integral is based on Elastic-Plastic Fracture Mechanics (EPFM). In figure 13 a schematic depiction of the ranges of applicability of both EPFM and LEFM is given.

There are several interrelated parameters describing the critical stress intensity for fracture, these will be discussed in the next paragraphs.
In the overlapping range of material properties, both concepts are applicable.

2.5.2 The stress intensity factor

The stress intensity factor $K$ is a parameter that can be used to evaluate the stress field around a flaw in a linear elastic material. If two flaws of the same geometry have the same value of $K$ then the stress distributions around these two cracks are identical. For the general case the stress intensity factor is given by:

$$K = \alpha_K \sigma \sqrt{\pi a} \quad (4)$$

where:

- $K$ = stress intensity
- $\alpha_K$ = geometrical factor

The magnitude of $K$ depends on the geometry of the solid containing the crack, the size and location of the crack and the magnitude and distribution of the load imposed on the solid. The usefulness of the stress intensity factor $K$ is that it can be related to local crack tip parameters. It can be shown that the size of the plastic zone at the crack tip and the crack opening displacement (depicted in figure 14) are given by:

$$2r_y \approx \frac{1}{\pi} \left( \frac{K}{\sigma_y} \right)^2 \quad (5)$$

where:

- $r_y$ = size of crack tip plastic zone
- $\sigma_y$ = yield stress

$$\delta_{col} \approx \left( \frac{K^2}{\sigma_y E} \right) \quad (6)$$

where:

- $\delta_{col}$ = crack opening displacement
These parameters are important since they can be used for conversion between the different measured fracture toughness parameters.

\[ \delta_{\text{COD}} \]

*figure 14: Schematic representation of the crack opening displacement $\delta_{\text{COD}}$.*

When instead of $\sigma$, $\sigma_*$ is used in equation 4 a critical stress intensity factor can be defined corresponding to the case where the associated value of $\zeta$ reaches $\zeta_c$. This critical stress intensity factor $K_c$ is commonly known as the fracture toughness.

In dealing with stress intensities several deformation modes could be applied to the crack, these deformation modes have been categorised and are shown in figure 15.

\[ \text{Mode I, Mode II, Mode III} \]

*figure 15: Illustration of the standardised fracture modes [29].*

The crack opening mode (mode I) corresponds with a tensile stress applied in the y-direction, normal to the faces of the crack. This is the most effective deformation mode to cause fracture and thus fracture toughness values obtained using this deformation mode are lower than fracture toughnesses obtained by loading in mode II or mode III. Since this is the most critical loading mode critical stress intensity factor is mostly determined under mode I loading and is called $K_{\text{IC}}$.

The fracture mode is a very important factor in determining the fracture toughness; ductile fracture leads to higher fracture toughness than brittle fracture. When loading a thin plate the initial crack causes a stress state with a low degree of triaxiality and this leads to a mixed-mode ductile fracture with shear lips. With increasing specimen thickness, the degree of triaxiality increases and the fracture mode becomes more brittle thereby reducing the fracture toughness. Once a critical specimen thickness is reached, the fracture toughness will remain constant, the fracture mode will be pure plane strain and the fracture surfaces will be flat. This trend is visualised in figure 16.
K\text{\textsubscript{\text{lc}}} values can be treated as material properties; a properly determined K\text{\textsubscript{\text{lc}}} represents the fracture toughness of a material independent on crack length, geometry or loading system. However, the fracture toughness is only a basic material property alike the yield strength and it changes with variables as temperature and strain rate. For a given alloy K\text{\textsubscript{\text{lc}}} depends on heat treatment, texture, melting practice, impurities and inclusions.

2.5.3 The J-integral concept

The J integral concept is based on an energy balance approach. The advantage of the use of an energy balance approach is its extended range of validity; the concept can be used (under certain restrictions) when certain levels of plasticity are present.

The underlying assumption of the J integral approach is that the material deformation can be described by the deformation theory of plasticity, where stresses and strains are functions only of the point of measurement and not of the path taken to get there. This is a good assumption for a stationary crack subjected to monotonically increasing load. For growing cracks, where regions of elastic unloading and nonproportional plastic flow can occur, the behaviour is not properly modelled by deformation theory. Despite the lack of theoretical justification, the J integral approach is successfully used in these situations.

Rice [29] showed that the line-integral related to the energy near the crack can be used to solve two-dimensional crack problems in the presence of plastic deformation. This line integral is called the J integral and is defined by:

\[
J = \int_{\Gamma} \left( W_{\text{\text{strain}}} dy - T \frac{\partial u}{\partial x} ds \right)
\]

where:
- \(\Gamma\) = integration path
- \(W_{\text{\text{strain}}} = \int \sigma \epsilon\) is the strain per unit volume due to loading
- \(x, y\) = rectangular co-ordinates
- \(T\) = outward traction vector on the contour around a crack
- \(u\) = displacement vector
- \(ds\) = increment of the contour path
Rice has shown that the J integral is path-independent, which means that the most convenient path can be chosen mostly the specimen boundary. The J integral can be interpreted as the potential energy difference between two identically loaded specimens with slightly different crack lengths, as illustrated in figure 17.

![Figure 17: Physical interpretation of the J integral concept [29].](image)

The heart of the J integral approach is given in equation 8:

\[
J = \frac{\partial U}{\partial a} = \frac{K^2}{E'}
\]

where:
- \( J \) = energy release rate
- \( E' \) = effective modulus of elasticity

Where the J integral is related to the stress intensity factor and the critical energy release rate. The measurement of the critical stress intensity factor is done using a J versus crack extension curve; the critical J integral is then defined as the value of J where a small but definite amount of crack growth is observed.

### 2.6 Reported fracture toughness values

Reports of fracture toughness measurements on MMCs are regrettably few and very wide spread. A way to compare the fracture toughnesses of different materials is to plot the fracture toughnesses versus yield strength behaviour. In common materials, a trend is observed where the fracture toughness decreases with increasing yield strength. For material classes like MMCs containing different composites of with different mechanical properties, this method is especially useful. However, when this a plot is constructed where the fracture toughness values are plotted versus the yield strength for different composites, no such trend is observed, as can be seen in figure 18.
The main reason for the absence of a substantial trend is that MMCs with different heat treatments and reinforcement materials have different mechanical properties and cannot be compared. The production method used is not always included with the test data, which makes it impossible to draw any conclusions about the variation of fracture toughness with volume fraction.

Reported fracture toughness values for AA6061 in T6 condition range from 26.6 to 37.0 MPa√m. Again the same applies as for the fracture toughness reports for MMCs, a large spread and little data.

### 2.7 Fracture toughness models

Though predictions of the fracture toughness in MMCs may not provide us with reasonable estimates of the actual fracture toughness values of MMCs (due to the wide range of performance and the wide spread in results) the predictions may provide us with an insight of how MMC parameters influence the fracture toughness.

In modelling the fracture toughness of MMCs there are three different approaches [1]:

- Crack path models
- Fractography-based models
- Energy-based models

#### 2.7.1 Crack path models

*Crack path models assume that crack extension takes place when the matrix ligament between the existing crack and the particle fails.*

Crack path models provide the most direct and simple route to a prediction of fracture toughness. The crack path model developed by Rice and Johnson [48] is based on precipitation or dispersion hardened aluminium alloys and therefore may very well be applicable to MMCs.
The model is based on the following assumptions:

- Metals fail at strains between 0.2 and 1
- Fracture occurs when the high strain region reaches the size of a critical microstructural dimension.
- A micro-crack is associated with every particle.

Based on these assumptions Hahn and Rosenfield [49] calculated the fracture toughness using $\delta_{\text{COD}} = 0.5\lambda_v$ (where $\lambda_v$ is the interparticle spacing) as the fracture strain criterion. They showed that the stress intensity factor could be related to the volume fraction of reinforcement through relation (9):

$$K_c = \sqrt{\frac{2\sigma_y E}{6} \left(\frac{\pi}{d}\right)^{\frac{3}{2}} f_v^{\frac{1}{2}}}$$

(9)

where: $d$ = particle size  
$f_v$ = volume fraction

When evaluating equation (9) the fracture toughness seems to depend on material properties yield strength, stiffness, particle diameter and volume fraction. One has to be careful drawing conclusions based on this approach because of the interrelated parameters it contains.

There is some confusion as to whether the composite modulus and yield strength must be used or the matrix yield strength and modulus. The answer to this question lies in the size of the plastic zone around the crack tip and the distribution of the reinforcement. When this plastic zone is small and the reinforcing particles are large (large interparticle spacing) it will not contain any reinforcing particles and therefore the reinforcing phase will have little influence on local mechanical properties and the properties will approach the matrix properties.

The existing confusion can easily be removed by evaluation of the size of the plastic zone as a function of crack opening displacement, using equations 5 and 6 on page 139. When the used fracture criterion $\delta_{\text{COD}} = 0.5\lambda_v$ is substituted into equation 5 and 6, the resulting size of the plastic zone will always be greater than the interparticle spacing. In other words, the crack tip plastic zone will always contain reinforcements according to the fracture criterion used.

When the composite properties are used in the crack path model as suggested by Hahn and Rosenfield, an increase in the volume fraction will result in an increase in the predicted fracture toughness. This is due to the stronger effect of the increase in stiffness caused by the extra reinforcement when compared with the increase in volume fraction reinforcement.
2.7.2 Fractography-based models

Fractography based models only have an interpretative function because they use parameters in their equations that can be determined from a fracture surface only.

The definition of the point of fracture (the fracture criterion) is the foundation of the predictions concerning fracture, and therefore a very important parameter. At the same time it is a very complex parameter and it is hard to make theoretical assumptions regarding the fracture criterion. The fractography-based models handle this problem by defining the fracture criterion post-mortem by means of fracture surface analysis.

The critical strain is estimated in terms of local strain, derived from the geometry of the initial void (d) and the coalesced void. The definition of the local true strain can be obtained in two ways, using the strain localisation model and the simple dimple height model. The parameters used in the equations are depicted in figure 19.

![Figure 19: Schematic depiction of crack geometry as used in the fractography-based models predicting the fracture toughness.](image)

If the void grows at the same rate as the imposed strain, the local strain can be coupled to the geometry change of the dimple by means of the following equation:

$$\varepsilon_{T,local} = \ln\left(\frac{h_{dimple}}{d}\right)$$

where:
- $\varepsilon_{T,local}$ = local true strain
- $h_{dimple}$ = dimple height

Based on the geometry of the particles and voids, the fracture strain criterion can now be calculated and used as a better estimate in equation (9), which leads to the prediction of the fracture toughness using the strain localisation model:

$$K_c = \frac{1}{3} \sigma_y E_c d \cdot \frac{\pi^{\frac{3}{2}}}{6} \ln\left(\frac{h_{dimple}}{d}\right)^{\frac{3}{2}} f_v^{\frac{1}{2}}$$

When the simple dimple height model is used, the fracture strain criterion is obtained as the ratio between the dimple height and width:
\[ \varepsilon_{T\text{,local}} = \ln \left( \frac{h_{\text{dimple}}}{w_{\text{dimple}}} \right)^2 \left( \frac{3f_v}{w_{\text{dimple}}} \right) \]

where: \( w_{\text{dimple}} = \text{dimple width} \)

Using equation (12) as the fracture strain criterion, the following relation of fracture toughness with mechanical properties is obtained:

\[ K_c = \frac{\pi}{6} \sigma_{Yc} f_v \cdot \ln \left( \frac{h_{\text{dimple}}}{w_{\text{dimple}}} \right)^2 \left( \frac{3f_v}{w_{\text{dimple}}} \right) \]

As mentioned, the underlying reason for using fractography-based models is that experimental results can be used as parameters for the analysis of the dependencies of materials properties. The use of experimental parameters can increase the accuracy of the model in an early stage of development, whereas the dependencies can provide theoretical background for further research. Parameters in these models can be varied in a limited range to study their effect.

2.7.3 Energy-based models

Energy-based models predict the fracture toughness through the energy-release-rate. The energy released on fracture can be predicted by summarising the different energy contributions associated with fracture.

The origin of energy-based models lies in the area of continuously reinforced MMCs. In these materials, damage processes are easier to predict because the damage accumulated by a single fibre is measurable. In MMCs this direct approach of splitting the total fracture energy in several contributions is possible, but the method is entirely different. Davidson [21] divided the energy release upon fracture in three distinct and measurable terms:

- The work done in the plastic zone, which can be measured from the loading curve.
- The mechanical work expended in creating the voided surface, which can be measured from a post-mortem analysis of the void dimensions.
- The surface energy itself, which can be calculated from a geometrical examination.

These three contributions all have a quantitative contribution to the fracture toughness. For example in an Al-4%Mg-15%SiC_p-system the plastic work was calculated as 7.95 MPa√m. The mechanical work for extending the voids was estimated as 0.5 MPa√m and the surface energy was calculated as 0.01 MPa√m. When all these contributions are summed, a fracture toughness around 8.8 MPa√m is found. As can be seen from section 2.6, common fracture toughness values are higher.
2.8 Research assignment

Theoretical studies of the fracture behaviour have been shown in the previous section, however, as can be concluded from 2.6 no experimental verification of the mentioned models exists. Fracture toughnesses have been measured for different volume fractions, however since production methods or material composition are not held constant by all researchers, it is impossible to verify the mentioned models with fracture toughness values found in literature.

Therefore, a dedicated study has to be performed, where the effect of only one parameter is studied, while special attention is paid to the elimination of any other variables.


### 3 EXPERIMENTAL PROCEDURES

#### 3.1 Introduction

In order to measure the fracture toughness variation with ceramic volume fraction, five different series of samples have been produced using the same raw materials. The volume fractions of the samples are 7 %, 10 %, 14 %, 22 % and 30 %. In order to test the methods used and verify the obtained MMC results a verification series was produced from an extruded AA6061 bar. The MMC samples were all lab-scale produced by means of powder metallurgy.

The total production and testing sequence consists of seven sequential steps:

- Powder blending
- Powder cold compaction
- Powder hot compaction
- Sample preparation
- Heat treatment
- Precracking
- Static testing

All steps will be discussed separately in the next sections, thereby identifying critical parameters.

#### 3.2 Powder blending

The available aluminium and ceramic powders in this research are:

- AA6061 - 75 \(\mu\)m, produced by *The Aluminium Powder Co. Ltd.*
- AA6061 - 45 \(\mu\)m, produced by *The Aluminium Powder Co. Ltd.*
- \(\text{Al}_2\text{O}_3\) - 4 \(\mu\)m, (Martoxid\®) produced by *Martinswerk GmbH*.

As can be seen in figure 20 the unblended powders have irregular sizes, and show a large variation in particle sizes. Another important aspect is that the morphology and size of the aluminium powders are very different from the morphology and size of the Martoxid\® powder.

![SEM-photographs](image.png)

*figure 20: SEM-photographs of unblended (a) AA6061 - 45 \(\mu\)m powder, (b) AA6061 - 75 \(\mu\)m powder and \(\text{Al}_2\text{O}_3\) - 4 \(\mu\)m Mart oxid \® powder. The large lump visible in the centre of figure 20c is a conglomerate.*
The large differences in particle sizes used, complicates the blending step by the formation of conglomerates. During blending of the powders, it is very important that conglomerates of ceramic powder are broken and mixed with aluminium powder. If conglomerates survive the blending process, they will remain in the material as defects, locally increasing failure probability.

To prevent this from happening, three different blending methods have been used:

**Blending in a ceramic container using Al₂O₃ balls**

A ceramic container filled with Al₂O₃ balls (having an approximate size of 15 mm diameter) has the advantage of being very effective in destroying the conglomerates. The mixing characteristics of this system however are not excellent, because of the non-reversing, continuous motion of the container.

![Ceramic container blender with Al₂O₃ balls.](image)

**Mechanical alloying using steel balls**

The method of mechanical alloying is very effective in mixing two metal powders, and can therefore be suitable for the mixing of the composite powder. The mechanical alloying process mixes and grinds the powders using small steel balls in a fluid environment. The fluid is used to oxidise the generated free surface and transfer the heat from the blender to the surrounding cooling system.

**Turbula T2C three-dimensional mixer**

The turbula mixer (provided by Tamson, Lamers & Pleuger) is a very effective mixer, because of the different movement directions (rotation, translation and inversion) during mixing. In order to ensure breakdown of the clusters of Al₂O₃, some Al₂O₃ balls are added during mixing.
Mixing times vary from two hours for the container blending method to 40 hours for the turbula mixer. When the powders are weighed and inserted into the blender, the volume fractions of the different series are fixed. They can be determined using equation 14:

\[
 f_v = \frac{1}{\left(\frac{m_{AA6061}}{\rho_{AA6061}} \cdot \rho_{Al_2O_3}\right)} + 1
\]

where:
- \( f_v \) = volume fraction
- \( m_{AA6061} \) = AA6061 mass added during blending
- \( \rho_{Al_2O_3} \) = Al_2O_3 density
- \( \rho_{AA6061} \) = AA6061 density
- \( m_{Al_2O_3} \) = Al2O3 mass added during blending

The morphology of the MMC structure is also determined after mixing of the powders. The only way to modify the morphology of the final material is to change to raw material (use different powders) or to change the mixing or blending method. After blending, the composite powders have the morphology as can be seen in figure 23.
3.3 Powder cold compaction

The cold compaction step is included to compact the powder for the first time, thereby creating solid tablets. These solid tablets are easy to handle and can be used in the hot press, where the final compaction step takes place.

The volume of the samples is determined in this step, so accurate dosage of the powder used is highly important to avoid extra manufacturing steps. The amount of powder used is calculated using the theoretical density and a target sample thickness of 19.5 mm. The target sample thickness is chosen slightly smaller than the official 20.0 mm to allow for low levels of porosity. The target sample diameter is 50 mm.

For cold compaction, a hardened steel die of 50 mm diameter is used in combination with a steel plunger. After insertion of the powder into the die, the die and plunger are placed in an AMSLER press type 500B. The pressure is slowly increased until the required level is reached, then the pressure is held constant 30 seconds. The tablets were squeezed out at high speeds to minimise die damage.

3.4 Powder hot compaction

During the final compaction step (performed under elevated temperatures) the actual bonding between ceramic and aluminium takes place and porosity is removed. The tablets are coated with a boron nitride spray, in order to prevent the MMC sample from attaching itself to the plungers or die. After coating, the tablets are inserted in the hot press, model HP 50 - 60120. This process is the most important step, since it determines the actual material behaviour and structure.

The most important aspects of the process are:

- Reproducibility of the process
- Low contamination of the material
- Good bonding between the two components
- Minimisation of porosity

Reproducibility of the process is, as in any production process, the most important aspect, since a comparison of different materials is only possible if all other parameters are well controlled. A small amount of material contamination is acceptable, provided this amount is the same for all samples and not dominating over the property studied. The reproducibility of the hot compaction process is guaranteed by the use of a control computer, imposing the same temperature-pressure-time program for all samples.

Low contamination of the process is important to minimise the number of microstructural defects in the sample. Contamination is kept low by performing the process under vacuum, preceded by a number of cleaning steps.

Before starting the pressing process, the vacuum chamber is cleaned by subsequently filling the chamber with gas, which is followed by a total drain of the vacuum chamber. In the first cleaning step, nitrogen is used as cleaning gas, in the second step uses argon for cleaning. When this cleaning procedure is completed, the vacuum chamber and its contents are heated to a temperature of 450 °C where it is kept for 30 minutes. After 30 minutes a vacuum of 30-60 mTorr is reached.

Actual process parameters during production are identified and analysed in detail in section 4.1.
3.5 Sample preparation

The hot compacted discs were shaped into DSCT specimens by sequential water jetting and spark erosion steps. The water jetting technique was used to create the notch shape and the loading holes. The use of this technique has proven necessary because of the large wear of any milling tools encountered.

The water jet used had a diameter of 0.8 mm with an average speed of 80 m/sec, which produced a water flow of 2.5 litres per minute. A HP-Barton Granat mesh #150 abrasive addition was used, to increase the abrasive force of the water jet. The abrasive consisted of mainly SiO₂, Al₂O₃ and Fe₂O₃ however, only the specimen surface was contaminated with the abrasive. Despite the technologically advanced process method, the cutting procedure of a single sample lasts between 15 and 20 minutes of process time.

After the sample was shaped using the water jetting technique, a V-shaped or double V-shaped notch was produced using spark erosion techniques. Since spark erosion uses the electrical conduction of the sample, this procedure differs per sample, since not all samples had the same electrical conductivity. (The electrical conductivity is reduced by the addition of alumina, which is an electrical insulator).

3.6 Heat treatment

A circulation hot air furnace (fabricated by Snijstaal B.V.) was used for the heat treatments of all samples. The accuracy of the furnace has been checked. Dummy samples (equipped with calibrated thermocouples) were heated and the response of the thermocouples was measured. Evaluation of the results indicated that the heat treatments were reproducible.

3.7 Characterisation of material and microstructure

After production, the material has to be characterised. Important aspects of the characterisation are the quality of the hot and cold compaction processes and the structure of the MMC produced.

In order to test the quality of the cold and hot compaction processes, density measurements were performed to compare the actual density with the theoretical density of the composite. If the volume fractions are known, the theoretical density of the composite can be calculated by an equation that is related to equation 14, given here:

\[ \rho_{\text{composite}} = \rho_{\text{Al}2\text{O}3}f_v + \rho_{\text{Al}6061}(1-f_v) \]  

where: \( \rho_{\text{composite}} = \text{density of the composite material} \)

The density is measured using a technique where the mass of the sample in air is compared to mass of the sample in water. The mass in water is measured using a pair of scales where the scale holding the sample is immersed in water.
The density of the sample can be related to the density of the water it is measured in using the following equation:

\[
\rho_{\text{composite}} = \rho_{\text{water}} \cdot \frac{m_{\text{air}}}{m_{\text{air}} - m_{\text{inwater}}}
\]

where:
- \( \rho_{\text{water}} \) = density of the water medium
- \( m_{\text{air}} \) = mass as measured in water
- \( m_{\text{inwater}} \) = mass as measured in air

When the measured density approaches the theoretical density of the composite, it can be assumed that the material is well compacted during the compaction process.

Another method to check the quality of the compaction process is to study the interface between matrix and reinforcement using scanning electron microscopy (SEM). Good matrix-reinforcement bonding indicates a satisfying process. The scanning electron microscope used is a JEOL JXA-8900R WD/ED combined micro-analyser, equipped with a Voyager III Sun imaging system software.

The second important aspect is the structure of the material. It is the objective of this research to measure the fracture toughness versus volume fraction behaviour of MMCs. The structure of the produced MMC must be characterised in order to relate the obtained results to obtained structure. For the characterisation of the microstructure, optical microscopy and SEM has been used.

For the characterisation of microstructure, the samples have been polished and ground used the Metalog Method A. Since the aluminium matrix is soft, this method (which is specially developed for soft materials) is chosen. This method was programmed into a polishing machine, type Struers Rotopol-31 and consists of the following steps:

**Step 1:**
- Grinding using SiC Paper
  - Duration: 01:00 minute
  - Force: 25 N/ Sample
  - Lubricant: Water

**Step 2:**
- Polishing using a MD-Largo plate
  - Duration: 05:00 minute
  - Force: 35 N/ Sample
  - Lubricant: DP-Lubricant Red
  - Abrasive: SiC, 9 µm

**Step 3:**
- Polishing using a MD/DP - Dur plate
  - Duration: 04:00 minute
  - Force: 35 N/ Sample
  - Lubricant: DP-Lubricant Red
  - Abrasive: SiC, 6 µm

**Step 4:**
- Polishing using a MD/DP - Mol plate
  - Duration: 03:00 minute
  - Force: 25 N/ Sample
  - Lubricant: DP-Lubricant Red
  - Abrasive: SiC, 3 µm
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Step 5: Chemical Polishing using a MD/OP - Chem plate

Duration: 02:00 minute
Force: 10 N/ Sample
Abrasive: OP-U Suspension **

* The red lubricant is a specially developed lubricant for polishing with diamond abrasive, provided by Struers. The use of this lubricant reduces the entrapping of particles in the surface. The elimination of the inclusion abrasive or (natural) Al₂O₃ particles in the surface of the aluminium is important, since the inclusion of particles during sample preparation will change the particle morphology visualised by the micrographic procedure.

** The OP-U suspension is colloidal silica suspension for final polishing of all materials. The suspension is provided by Struers, as is all equipment used for the sample preparation.

The samples were analysed using an OLYMPUS BX60M optical microscope, equipped with an OLYMPUS DP10 digital camera. The digital photographs were analysed and modified using an analySIS software package.

3.8 Characterisation of the fracture surface

A SEM analysis (for a description of the equipment see 3.7) was used to study the structure of the fracture surface of both the fatigue crack region and the stable crack growth region. To eliminate any malfunctions of the SEM the fracture surfaces were cut from the remainder of the sample, a reduction of the amount of material inserted in the SEM, decreases the contamination risks.

The samples were cleaned using a ultrasonic acetone bath, followed by an ultrasonic propanol bath for removal of the acetone attached to the surface of the sample.

To ensure that only surface information is visualised in the SEM, the acceleration voltages must be kept as low as possible to reduce the penetration dept of the electron rays. Normally an acceleration voltage of 5 kV suffices for fracture surface analysis, however in this research the electrical conduction of the samples is low and higher acceleration voltages of ~ 15 kV have to be used.

3.9 Fracture toughness test methods

When from the characterisation of the material can be concluded that the process performs satisfactory, the mechanical part of this research can commence. When the process does not perform satisfactory and structural levels of porosity are left in the material, measurement of the mechanical properties of the material is irrelevant, since porosity has a very pronounced effect on these mechanical properties.

For the determination of the fracture toughness of metallic materials, three different techniques were employed. These methods are selected from a wider range of ASTM tests [25].

**E399 Plane-Strain Fracture Toughness of Metallic Materials**

The E399 test method involves testing of notched specimens that have been precracked in fatigue by loading either in tension or three-point bending. The load corresponding to a 2% apparent crack extension is established by a specified deviation from the linear portion of the record. The $K_{IC}$ value is from this load by equations based elastic stress analysis of the specimen geometries involved.
**E1290 Fracture Toughness Measurement Crack-Tip Opening Displacement**

The object of the E1290 test method is to determine the value of CTOD at one or more crack extension events. The values of CTOD may correspond to:

- \(\delta_c\): Crack-tip opening displacement at the onset of unstable brittle crack extension with no significant prior slow stable crack extension.
- \(\delta_u\): Crack-tip opening displacement at the onset of unstable brittle crack extension with prior slow stable crack extension.
- \(\delta_m\): Crack-tip opening displacement at the first attainment of a maximum plateau for fully plastic behaviour.

The test involves cross-head or clip gage displacement controlled three-point bending or pin loading of fatigue precracked specimens. The loads and displacements corresponding to the specific events in the crack initiation and extension process are used to determine the values of \(\delta_m, \delta_u\) and \(\delta_c\). The fracture mode during testing (unstable brittle crack extension, slow stable crack extension or fully plastic behaviour) is determined from post-mortem analysis of the fracture surface.

**E1737 J-integral Characterisation of Fracture Toughness**

Tests according to the E1737 test method determine the value of J as a function of crack growth from testing of three-point bending or pin loaded specimens, precracked in fatigue. Three toughness properties can be identified which vary with the amount of crack extension present at test termination. The -- for this research relevant - toughness properties that can be identified are:

- \(J_c\): Instability without prior crack extension
- \(J_{ic}\): The onset of stable crack extension

This method also applies specifically to geometries that contain notches sharpened by precracking.

Determination of the J-integral can be done from multiple specimens containing different crack lengths or from single-specimen tests in which elastic compliance or potential drop techniques are used.

The applicability of the test methods depends largely on the behaviour of the tested material. For example, the E399-test method is used for brittle materials with little or no stable crack growth, the method is based on LEFM, so the limited plasticity is a condition for the results to be valid. Characterisation of \(J_{ic}\) fracture toughness using E1737 (based on EPFM) on the other hand is only possible when the material exhibits certain stable crack extension during testing. Since the behaviour of the investigated material is not known in advance, all three methods have been used.

If the materials' load-displacement curves are known, all three methods can be applied in one single test, using one sample. Sample geometry and preparation methods are similar for all methods, therefore sample geometry and precracking procedure have been developed according to the 'Plane-Strain Fracture Toughness of Metallic Materials' test method.
3.10 Sample geometry for fracture toughness tests

Since the MMC is produced through powder metallurgy by hot pressing in a circular die, the disk-shaped compact tension (DSCT) specimen has to be used for testing. In test methods E399 and E1737 special attention is paid to the testing of DSCT specimens, however in method E1290 no details are given on the subject of testing of DSCT specimens. In order to allow DSCT-specimens to be tested using the method described in E1290, the equations used have to be modified.

For the stress intensity analysis to be valid, an ideal planar crack with essentially zero tip radius needs to be simulated. The validity of fracture toughness measurements depends on the establishment of a (reproducible) sharp-crack condition before testing. This sharp-crack condition depends on both fatigue precracking procedure as on the specimen geometry. The advised geometry to achieve the sharp-crack condition is given in figure 24 for the E399 method.

![Diagram](image1)

**figure 24: Disc Shaped Compact Tension geometry [50].**

To raise stress concentrations in the early stage of precracking and to facilitate the initiation of a fatigue crack, a starter notch is used. The starter notch used in the present work is a chevron-notch, produced by spark-erosion (for the MMC samples) or milling (for the AA6061 samples). The geometry of the starter notch is shown in figure 25.

![Diagram](image2)

**figure 25: Notch geometry according to ASTM E399[50].**
Because of problems with the electrical conduction of the MMC samples, it turned out to be impossible to produce a double V-shaped notch (as shown in figure 26) for all specimens.

![Double V-shape in chevron-notch as advised for maximum stress concentration][50].

If a chevron notch with a rectangular end is used, double initiation of the fatigue precrack is expected on both topside and downside of the starter notch. Post-mortem examination of the fracture surface will then have to point out if a valid precrack was present before testing.

### 3.11 Fatigue precracking

In ASTM E399 [50] the conditions for an ideal planar crack with essentially zero crack tip radius are described. A guideline is given how to achieve this condition of which the most important conditions are summarised here:

- The maximum stress intensity in the final stage of fatigue crack growth is not allowed to exceed 60% of the $K_{\infty}$ value of the material.
- The total crack length of the crack starter configuration and the fatigue crack has to be between 0.45W and 0.55W.
- The crack should be straight.

The first condition for is the easiest one to comply with, the other two conditions are harder to fulfil since the crack itself is invisible during testing.

Crack straightness is one of the biggest problems encountered during testing of AA6061-based material. The nature of these alloys is to exhibit very strong crack tunnelling (higher crack growth rates in the centre of the specimen). A standard fatigue test does not result in a straight crack therefore the applied load during fatigue testing has to be modified in order to obtain a straight crack front.

In order to satisfy the second criterion, a halt point for the fatigue precracking procedure should be defined and an accurate crack length measurement method must be applied (the difference between minimum and maximum crack length is around 4 mm). The crack length measurement method that has been used is the compliance method.
3.11.1 Measurement of the crack length

If a crack grows, the compliance of the specimen will increase, since the CMOD (crack mouth opening displacement) becomes larger under the same applied load. With a clip gauge, it is possible to measure the CMOD during fatigue precracking, which allows the compliance, C, to be calculated:

\[
C = \frac{\Delta \text{CMOD}}{\Delta P} = \frac{\text{CMOD}_{p_{\text{max}}} - \text{CMOD}_{p_{\text{mean}}}}{P_{\text{max}} - P_{\text{mean}}}
\]

where:
- \(C\) = specimen compliance
- \(P_{\text{max}}\) = maximum fatigue load
- \(P_{\text{mean}}\) = mean fatigue load
- \(P\) = load
- \(\text{CMOD}\) = crack mouth opening displacement
- \(\text{CMOD}_{p_{\text{max}}}\) = crack mouth opening displacement at maximum fatigue load
- \(\text{CMOD}_{p_{\text{mean}}}\) = crack mouth opening displacement at mean fatigue load

The values of \(P_{\text{max}}, P_{\text{mean}}, \text{CMOD}_{p_{\text{max}}}\) and \(\text{CMOD}_{p_{\text{mean}}}\) can be calculated from the load and displacement signal.

When measuring the CMOD at the front face of a specimen, Jablonski et al. [51] fitted a compliance function for a disk-shaped compact tension specimen:

\[
\ln \left( \frac{E'V_sB}{P} \right) = \ln [E'CB] = 2.456 - 0.483 \left( \frac{a}{W} \right) + 13.996 \left( \frac{a}{W} \right)^2 - 18.708 \left( \frac{a}{W} \right)^3 + 8.493 \left( \frac{a}{W} \right)^4 + 3.571 \left( \frac{a}{W} \right)^5
\]

where:
- \(V_s\) = crack opening displacement at location \(x\) from the load line
- \(B\) = specimen thickness
- \(W\) = specimen width

This function cannot easily be transformed to calculate the crack length from a given compliance. In order to obtain a useable crack length equation a polynomial expression is fitted on the inverse compliance-crack length curve. This expression has the form of

\[
a = c_2 C^2 + c_1 C + c_0
\]

The constants \(c_2, c_1\) and \(c_0\) are geometry-dependent and for this case given by:

\[
c_2 = -4.7669 \cdot 10^{-25} E + 7.9815 \cdot 10^{-16} - 3.0530 \cdot 10^{-5}
\]
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\[ c_1 = 5.4577 \cdot 10^{-12} E - 4.0691 \cdot 10^{-5} \]

\[ c_0 = 6.1469 \]

The result of the fit-procedures is an equation that relates the crack length to both the compliance and the stiffness of the material.

3.11.2 Modified constant \( K_{\text{max}} \) fatigue procedure

Precracking procedures are extremely important for fracture toughness measurement procedures, since non-standard precracking techniques may result in 'better' crack fronts. The word 'better' in this context means that the crack fronts satisfy the conditions specified in the ASTM standards.

Another aspect of non-standard precracking techniques is that the technique used may affect the obtained fracture toughness results.

Modified precracking techniques are often used in fracture toughness tests performed on welded samples, since the internal residual stresses inhibit the formation of a straight crack front. Two available techniques may be used:

- **Local compression.** Using this technique, internal stresses are removed by the application of a certain amount of plastic deformation. This method is inapplicable to MMCs since it is expected that the local compression significantly alters the fracture behaviour.

- **Stepwise high R-ratio techniques.** The stepwise high R-ratio technique consists of two steps. In the first stage of the precracking procedure a lower R-ratio is used to initiate and grow the crack. During the final stage the R-ratio is increased to propagate the fatigue crack to the desired length [52]. The effect of the stepwise high R-ratio technique depends on specimen size and tested material.

In literature it has been reported that the stepwise high R-ratio significantly decreases the amount of scatter in toughness results [53]. On the other hand, Kocak et al [58] reported that the stepwise high R-ratio technique results in significantly higher CTOD toughness values, when compared to other techniques. Literature also indicated an increased initial compliance because of crack closure effects [52].

Although it is unsure whether the crack front tunnelling in MMCs is caused by internal stresses, modified precracking techniques may reduce the amount of tunnelling and produce a straight crack front.

3.11.3 Quantification of fatigue crack front shapes

To enable comparison of the fatigue crack front shapes a quantification of curvature and obliqueness is needed. The factors are defined according to equation 20 and 21 [54], where the curvature and obliqueness are expressed using the parameters from figure 27.

\[ f_{\text{oblique}} = \left( \frac{a_{\text{surf}, \text{max}} - a_{\text{surf}, \text{min}}}{a_0} \right) \]

where:  
\[ f_{\text{oblique}} \quad = \text{ratio of obliqueness} \]
\[ a_{\text{surf}, \text{max}} \quad = \text{maximum surface crack length} \]
The curvature ratio is determined using the maximum deviation from the average crack length:

\[ f_{\text{curvature}} = \text{abs} \left( \frac{\max(a_x - a_{o})}{a_{o}} \right) \] (21)

where:
- \( f_{\text{curvature}} \) = ratio of curvature
- \( a_x \) = any crack length measurement

Using these parameters, a maximum allowable value of curvature and obliqueness ratios can be defined, making an easy identification of the succeeded crack fronts possible.
3.11.4 Experimental set-up for precracking

In the experimental set-up, consisting of two parts, the first part uses the clip gauge signal and the load signal to determine the actual crack length, the second part regulates the load that is applied during fatigue. The amplitude and offset of the load function are determined from the crack length and the applied values are compared with the measured values and modified if necessary. The clip gauge and load signal are fitted on a sinusoidal function to compensate for crack closure (flattening of the bottom of the wave) and any signal disturbances.

A schematic representation of the experimental set-up has been given in figure 28:

![Schematic diagram of the experimental set-up](image)

**figure 28: Experimental set-up as used for the determination of the crack length and modification of the applied load.**

Using this experimental set-up, a crack length dependent load program can be used to ensure a crack front that satisfies the guidelines given by the ASTM E399.

During design of the load program, the following aspects have to be kept in mind:

- High $\Delta K$-values produce maximum crack growth and reduce test duration.
- To minimise crack front curvature a high $R$-level is advised.
- The applied loads should not exceed a fixed value $K_{\text{max}}$.

Combination of these three statements leads to a load variation with crack length as visualised in figure 29.
In the initial precracking stage, the $\Delta K$ value is kept as high as possible, for maximum crack growth rates. When the crack has emerged from the notch, the $R$-value is increased to flatten the crack front, as visualised in figure 30.

\[ \text{precracking } R \approx 0.8 \]

\[ \text{machined notch} \]

**figure 29:** Variation of the applied fatigue load with crack length.

**figure 30:** Variation of $R$ with crack propagation.
3.12 Fracture toughness tests

The following sections will be dedicated to a detailed description of the fracture toughness test methods, as described in the annual book of ASTM standards.

3.12.1 Plane Strain Fracture Toughness of Metallic Materials, ASTM E399

After precracking, the specimens are loaded until fracture, while load and displacement were recorded. To prevent non-linearity at the beginning of a load-displacement record, the specimens were preloaded and unloaded with the maximum load not producing a stress intensity level exceeding that used in the final stage of fatigue cracking. The rate of the stress intensity factor increase during the $K_c$ test was within 0.55 and 2.75 MPa√m. From these records, of which three types are shown in figure 31, $P_Q$ (which is the load value used to calculate a conditional stress intensity factor, $K_Q$) was determined.

\[ K_Q = \frac{P_Q}{B \sqrt{W}} \cdot f\left(\frac{a}{W}\right) \]  

where:

\begin{align*}
K_Q & = \text{unqualified critical stress intensity} \\
P_Q & = \text{load at critical crack extension}
\end{align*}
For the disk-shaped compact tension specimen used, the geometry factor is given by equation 23:

\[
f\left(\frac{a}{W}\right) = \frac{\left(2 + \frac{a}{W}\right) \left(0.76 + 4.8 \left(\frac{a}{W}\right)^2 - 11.58 \left(\frac{a}{W}\right)^3 + 11.43 \left(\frac{a}{W}\right)^4 - 4.08 \left(\frac{a}{W}\right)^5\right)}{\left(1 - \frac{a}{W}\right)^2}
\]  
(23)

For a \( K_Q \) value to be a valid \( K_{ic} \) value according to this testing method, several requirements must be satisfied [50]:

For all specimen configurations, the specimen thickness and the crack length should exceed \( 2.5(K_Q/\sigma_{ys})^2 \).

The ratio of \( P_{\text{max}}/P_Q \) is not allowed to exceed 1.10.

The crack length after fracture has to be measured at three positions: at the centre of the crack front and midway between the centre of the crack front and the end of the crack front on each surface of the specimen. The average of these three measurements is used to calculate \( K_Q \), whereby the requirements for the fatigue crack front are:

The difference between the average and the measured crack length at any point should not exceed 10%.

For a chevron notch starter, the fatigue crack shall emerge from the chevron on both surfaces of the specimen, neither surface crack length shall differ from the average length by more than 10% and the difference between these two surface measurements shall not exceed 10% of the average crack length.

If and only if all these requirements are met, the calculated \( K_Q \) value is a valid \( K_{ic} \) value for the material.
3.12.2 Fracture Toughness Measurement with critical CTOD, ASTM E1290

For the calculation of the fracture toughness crack-tip opening displacement, the same test data are used as for the E399 test method. Load and displacement are recorded during a displacement-controlled tensile test. The critical CTOD values are defined depending on the shape of the load-displacement records, as visualised in figure 32.

![Diagram](image)

*Figure 32: Determination of the plastic component of the clip gage displacement for critical CTOD calculation for different types of load clip gage displacement records.[55]*

For most types of load-displacement records shown in figure 32, the critical plastic clip gage displacement can be defined as the maximum in load ($P_c$) and displacement ($v_c$). When a pop-in occurs, the part of load-displacement record after the pop-in should be ignored while determining $P_c$ and $v_c$. The main difference between types $a$, $b$ and $c$, $d$ is the amount of non-linearity occurring before $P_c$ and $v_c$. For load-displacement records with little non-linearity (types $a$ and $b$), the E399 test method should be applied, whereas the application of E399 to types $c$, $d$ and $e$ load displacement records will lead to invalid results.

When the critical plastic component of the clip gage displacement is determined, this value should be converted to the relevant value of CTOD using the following relationship for DSCT specimens. This set of equations is comparable to the equations given in ASTM E1290, however geometry dependent functions have been modified to apply to DSCT specimens. (The original method only gives the converting equations for CT specimens.)

$$
\delta_{COD} = \frac{K^2(1 - v^2)}{2\sigma_y E} + \frac{r_p (W - a_0) v_p}{r_p (W - a_0) + a_0 + z} \tag{24}
$$
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\[ K = \frac{YP}{BW^{1/2}} \]  \hspace{1cm} (25)

\[ Y = f\left(\frac{a}{W}\right) = \frac{\left(2 + \frac{a}{W}\right)\left(0.76 + 4.8 \left(\frac{a}{W}\right) - 11.58 \left(\frac{a}{W}\right)^2 + 11.43 \left(\frac{a}{W}\right)^3 - 4.08 \left(\frac{a}{W}\right)^4\right)}{\left(1 - \frac{a}{W}\right)^{3/2}} \]  \hspace{1cm} (26)

where:

\[ P = \text{Load corresponding to } P_{\alpha}, P_{\nu}, \text{ or } P_m \text{ in figure 32} \]
\[ \nu_p = \text{Plastic component of clip gage opening displacement corresponding to } V_{\alpha}, V_{\nu}, \text{ or } V_m \text{ in figure 32.} \]
\[ \nu = \text{Poisson's ratio} \]
\[ r_p = \text{plastic rotation correction factor} \]
\[ a_0 = \text{initial crack length} \]
\[ z = \text{distance between clip gage attachment point and the load line} \]

The geometrical factor \( Y \) used in equation 24 here is essentially the same as the one given by equation 23.

The nature of the pin loading test as performed in this research is in fact very similar to a bend test, only the bending point is situated somewhere in the ligament between \( a_0 \) and \( W \). Because of the nature of the test, the correction factor for plastic rotation \( r_p \) has been used. The correction factor is defined by equation 27.

\[ r_p = 0.4 \left(1 + \alpha_r\right) \]  \hspace{1cm} (27)

\[ \alpha = 2 \sqrt{\frac{a_0}{b_0}} + \frac{a_0}{b_0} + \frac{1}{2} - 2 \left(\frac{a_0}{b_0} + \frac{1}{2}\right) \]  \hspace{1cm} (28)

where:

\[ \alpha_r = \text{geometrical factor for calculation of plastic rotation correction factor} \]
\[ b_0 = \text{initial length of the unbroken ligament between } a_0 \text{ and } W \]

The obtained CTOD values are valid if the following conditions apply:

The values of CTOD should be equal to or less than the measurement capacity of the specimen. The measurement capacity of the specimen can be determined from \( \delta_m \).

After measurement of the fatigue crack length at nine equally spaced positions along the crack front, the difference between the maximum and minimum of all 9 fatigue crack length measurements does not exceed 10% of the original fatigue crack length \( a_0 \).

No part of the fatigue crack front is closer to the machined notch than the lesser of 0.025W or 1.3 mm.

The plane of the fatigue crack does not exceed an angle of 10° from the plane of the notch. The fatigue crack front is not multi-planar or branched.
3.12.3 J-integral Characterisation of Fracture Toughness, ASTM E1737

The core of the J-integral characterisation of fracture toughness by ASTM E1737 [56] is the determination of the value of the J integral as a function of crack extension. For J_{lc} determination, a least squares power law is fitted to approximate the J versus crack growth behaviour. Subsequently a construction line representing 0.2 mm stable crack growth and crack stretch effects is drawn. The intersection point of the construction line with the power law (J_{q}) represents the value of J_{lc} if the specified test requirements are satisfied.

To obtain the J versus crack extension behaviour both the value of the J integral and the actual crack length have to be known. J integral values can be calculated from the plastic component of the load-line displacement signal and the load signal, whereas the crack length can be estimated from specimen compliance, as is done during precracking. To measure the specimen compliance, the standard tensile test has to be interrupted by unloadings.

Using the test data obtained by the standard experimental set-up and specimen geometry, the crack mouth displacement (as measured with the clip gage from the front face of the specimen) has to be converted to load-line displacement for calculation of J values. This conversion is based on the principle that the pin loading of a compact tension specimen is in fact a bending test, as can be seen from figure 33. However, the rotation point depends on both specimen geometry and crack length, and is defined by equations 27, 28 and 29 [55]:

\[ R = r_p(W - a) \]  (29)

where: \( R \) = rotation length

\[ \frac{V_{II}}{a + r_p(W - a)} = \frac{V_{cm}}{a + r_p(W - a) + z} = \tan \Theta \]  (30)

where: \( V_{II} \) = load line displacement \( V_{cm} \) = crack mouth displacement \( \Theta \) = rotation angle

*figure 33: Conversion from crack mouth displacement to load-line displacement.*

When the rotation point is known, the similarity of the triangles leads to equation 30:
The load-line displacement can then be calculated from equation 31:

\[ \nu_{p} = \frac{\nu_{cm}(a + r_p(W - a))}{a + r_p(W - a) + z} \]

After the data has been converted from crack mouth displacement to load-line displacement, the crack lengths have to be calculated for determination of the J versus crack extension curve. As mentioned crack lengths are calculated from unloading interrupts during the standard tensile test, examples of a load-displacement and loading curves are given in figure 34 and figure 35.

**Figure 34:** Example of a load-displacement curve used for the determination of the J integral.

**Figure 35:** Example of a displacement-time curve for determination of the unloading intervals.
As can be seen in figure 34 the load-displacement curve consists of a continuous load-displacement signal where the unloadings determine the crack lengths at the marked locations. For an accurate determination of the compliance from the unloadings, the intervals can be identified from the load/displacement versus time plot. Start time and end time are identified and the compliance on this isolated interval is calculated according to equation 32:

\[
C'_W = \frac{dv'_W}{dP} \tag{32}
\]

where: \( C'_W \) = specimen compliance, measured from the load line

From the compliance values obtained from the unloading intervals a first estimate of the crack growth is calculated, using equation 33 and 34 [56]:

\[
a_i = \frac{0.998193 - 3.88087 \cdot U_x + 0.187106 \cdot U_x^2}{W} + 20.3714 \cdot U_x^3 - 45.2125 \cdot U_x^4 + 44.5270 \cdot U_x^5
\]

\[
U_x = \frac{1}{\sqrt{BE'C'_W + 1}} \tag{34}
\]

where: \( a_i \) = crack length at crack extension event \( i \)

The compliance determined according to equation 32 not only contains the effect of crack opening and extension, but also has an error introduced by rotation of the specimen. In order to correct for this error, the correction method depicted in figure 36 can be applied.

*figure 36: Correction of the elastic compliance for specimen rotation [56].*
The resulting correction factor is given in equation 35:

\[
C_{ic(i)} = \frac{C_{(i)}}{ \left[ \frac{H^*}{R} \sin\Theta - \cos\Theta \right] \left[ \frac{D}{R} \sin\Theta - \cos\Theta \right]}
\]  

(35)

where:  
\(C_{(i)}\) = uncorrected compliance at crack extension event \(i\)  
\(C_{ic(i)}\) = for rotation corrected specimen compliance at crack extension event \(i\)  
\(H^*\) = half distance between loading pins  
\(D\) = half distance between clip gauge attachment points

After correction, the crack lengths are calculated according to equations 33 and 34. The initial crack length is then defined as the crack length measured from the first unloading, in order to determine the amount of crack extension. Since the Young's modulus of the tested material is unknown, an estimate of the modulus has to be made for crack length calculation. The Young's modulus is estimated by fitting the initial crack length measured from unloading compliance to the actual initial crack length as measured from the fracture surface. When the compliance measured from the first unloading is unreliable, another measurement point for the compliance is allowed, on condition that there is no crack extension present at the measurement point. If the stiffness of the tested material is known and the procedure proves unreliable, the initial crack length determined from the fracture surface can be used.
The estimated Young's moduli can sequentially be verified with a validated model predicting the stiffness of particle reinforced MMCs as a function of volume fraction. A model fit for this material is the Tsai-Halpin model [57]. The Tsai-Halpin model predicts the stiffness of MMCs as a function of the moduli of both constituents and the particle aspect ratio, as shown in equations 36 and 37:

\[
E_{ll} = E_m \frac{1 + 2sB_{TH}f_v}{1 - B_{TH}f_v} \tag{36}
\]

and:

\[
B = \frac{E_r}{E_m} - 1 \div \frac{E_r}{E_m} + 2s \tag{37}
\]

where:
- \(E_{ll}\) = composite modulus of elasticity in the reinforcement direction
- \(E_m\) = matrix modulus of elasticity
- \(s\) = aspect ratio of the reinforcement
- \(B_{TH}\) = Tsai-Halpin coefficient
- \(E_r\) = reinforcement modulus of elasticity

If the estimated moduli fit the model adequately, it can be assumed that the estimate may be used for determination of the crack length.

The final preparation step for the calculation of \(J\) is the separation of the load-line displacement in an elastic component and a plastic component. This is done according to equations 38 and 39:

\[
v_{ll}(i) = v_{ll,el}(i) + v_{ll,pl}(i) \tag{38}
\]

\[
v_{ll,pl}(i) = v_{ll}(i) - P_iC_{ll}(i) \tag{39}
\]

where:
- \(v_{ll}(i)\) = load line displacement at crack extension point \(i\)
- \(v_{ll,el}(i)\) = elastic component of load line displacement at crack extension point \(i\)
- \(v_{ll,pl}(i)\) = plastic component of load line displacement at crack extension point \(i\)

Now the values of load, plastic component of clip gage displacement and crack length are available in the form of table 1:

<table>
<thead>
<tr>
<th>Unloading</th>
<th>Load</th>
<th>Plastic clip gage displacement</th>
<th>Crack Extension</th>
</tr>
</thead>
<tbody>
<tr>
<td>[ # ]</td>
<td>[ N ]</td>
<td>[ mm ]</td>
<td>[ mm ]</td>
</tr>
<tr>
<td>0</td>
<td>(P_0)</td>
<td>(v_{ll,0})</td>
<td>(a_0)</td>
</tr>
<tr>
<td>...</td>
<td>...</td>
<td>...</td>
<td>...</td>
</tr>
<tr>
<td>...</td>
<td>...</td>
<td>...</td>
<td>...</td>
</tr>
<tr>
<td>(i)</td>
<td>(P_i)</td>
<td>(v_{ll,i})</td>
<td>(a_i)</td>
</tr>
</tbody>
</table>

With this data, the actual \(J\) versus crack extension curve can be determined from equations 40, 41, 42, 43, 44, 45 and 46 [56].
The first step is the separation of $J$ in two components, an elastic component and a plastic component:

$$J = J_{el} + J_{pl}$$  \hspace{1cm} (40)

where:

- $J_{el} = \text{elastic component of } J$
- $J_{pl} = \text{plastic component of } J$

The elastic component of $J$ is formed by the stress intensity at the load-displacement point $(i)$, and is determined by the crack length and applied load only.

$$J_{el(i)} = \frac{K_{(i)}^2(1 - v^2)}{E}$$  \hspace{1cm} (41)

$K$ is the stress intensity factor in point $(i)$ and is given by:

$$K_{(i)} = \frac{P_{(i)}}{\sqrt{BB\lambda W}} f\left(\frac{a_i}{W}\right)$$  \hspace{1cm} (42)

The geometrical factor $f\left(\frac{a_i}{W}\right)$ is determined from the geometry of the tested sample and for DSCT-specimens given by:

$$f\left(\frac{a_i}{W}\right) = \frac{\left(2 + \frac{a_i}{W}\right)\left(0.76 + 4.8\left(\frac{a_i}{W}\right) - 11.58\left(\frac{a_i}{W}\right)^2 + 11.43\left(\frac{a_i}{W}\right)^3 - 4.08\left(\frac{a_i}{W}\right)^4\right)}{\sqrt{\left(1 - \frac{a_i}{W}\right)\left(1 - \frac{a_i}{W}\right)}}$$  \hspace{1cm} (43)
Now the elastic component is known, and the focus is switched to the plastic component calculation. The plastic component of J is the integrated part and given by:

\[ J_{pl(i)} = \left[ J_{pl(i-1)} + \left( \eta_{(i-1)} \right) \frac{A_{pl(i)} - A_{pl(i-1)}}{b_{(i-1)}} \right] 1 - \gamma_{(i-1)} \frac{a_i - a_{i-1}}{b_{i-1}} \]  

(44)

where:  
\( \eta_{(i-1)} \) = geometrical parameter  
\( \gamma_{(i-1)} \) = geometrical parameter  
\( A_{pl(i)} \) = surface under load-displacement curve

where \( \gamma, \eta \) and \( A_{pl(i)} \) are given by:

\[ \eta_{(i)} = 2.0 + 0.522 \frac{b_{(i)}}{W} \]  

(45)

\[ \gamma_{(i)} = 1.0 + 0.76 \frac{b_{(i)}}{W} \]  

(46)

\[ A_{pl(i)} = A_{pl(i-1)} + \frac{\left( P_i + P_{i-1} \right) \left( \nu_{ii,pl(i)} - \nu_{ii,pl(i-1)} \right)}{2} \]  

(47)

Now the calculation of J is complete and the following steps have to be executed for proper calculation.

**Verification of the initial compliance measurement**

The initial compliance has a very large influence on the value of J. Overestimation of the initial crack length will lead to an underestimation of the crack growth. Since the J versus crack extension curve has a steep slope at small crack extensions, a small overestimation of the initial compliance may lead to large errors in J.

**Verification of the used stiffness**

Since the actual stiffness of the material is unknown, the stiffness must be determined from the first unloading compliance and initial crack length. The, from the first unloading compliance measurements, calculated initial crack length changes when the e-modulus used in equation 34 is changed. When the initial crack length is known, in this case from post-mortem analysis of the fracture surface, the stiffness can be varied until the calculated crack length equals the measured crack length.

The J versus crack extension curve is then approximated by a least square fit of the power law given in equation 48, using the fit coefficients \( C_1 \) and \( C_2 \):

\[ J = C_1 (\Delta a)^{C_2} \]  

(48)
The determination of the fracture toughness as characterised by the J integral consists of the following steps.

**Construction of a blunting line**

The blunting line determines the critical value of crack extension. This line is needed because the crack-tip plastic zone influences the crack extension. In the ASTM E399 method crack-tip plasticity is neglected because this method is intended for brittle materials, where the effect of crack tip plasticity is small. The blunting line is defined based on the assumption that the crack advance is equal to one-half at the crack opening displacement.

**Determination of J_q**

J_q is now determined as the intersection between the blunting line and the power law. J_q is qualified as J_c if the data meets the requirements.

**Qualification of data**

**Fatigue Crack Length**

- The fatigue crack is measured at nine equally spaced positions along each fracture surface. From the two surface measurements, the average surface crack length is determined. The total crack length is then defined as the average of the seven internal measurements and the average surface length. None of the measurements should differ more than 5% from the total crack length.

**Crack Extension**

- None of the crack extension measurements shall differ more than 50% from the average crack extension.

- The total crack extension is determined according to the same procedure described above. Again, none of the measurements may differ more than 5% from the average.

- The total crack extension determined by unloading compliance measurements shall not differ more than 0.15 Δ_a, for crack extensions under 0.2b_0. For larger crack extensions the difference shall not exceed 0.03 b_0.

**J versus crack extension curve**

- The power coefficient c_2 shall not exceed 1.0

- The number of points in the data set used for determination of the power law coefficients shall be larger than or equal to 5.

- The correlation of the least squares fit of the power law shall be greater than 0.96.

- All data that fall outside the region of qualified data visualised in figure 37 must be eliminated from the least square fit procedure.
EXPERIMENTAL PROCEDURES

Sample geometry requirements

- The sample thickness should exceed $25 \frac{J_q}{\sigma_Y}$.
- The length of the initial ligament should exceed $25 \frac{J_q}{\sigma_Y}$.

If these requirements are met, the value of $J_q$ can be qualified valid and when stable crack extension is observed, this will lead to the conversion from $J_q$ to $J_{IC}$.

3.12.4 Experimental set-up for tensile testing

For the tensile tests an Instron 4505 testing machine equipped with a 100 kN loadcell was used. This machine was equipped with a programmable controller in which 20 loading or unloading steps can be programmed. For measurement of the crack mouth displacement an Instron clip gauge was used with an extension of $12.5 \pm 5$ mm. This clip gauge is modified to reduce the measurement opening from 12.5 to 6 mm, analogous to the modification visualised in figure 38:

To prevent the clip gauge from damaging during specimen fracture, the clip gauge was not fixated on the specimen, but clamped in the mouth of the sample using a spring.

The data was recorded from both load cell and clip gauge using the "Series IX Automated Materials Test System" version 7.51.00 as provided by the Instron Corporation.
3.13 Visualisation of the stable crack extension

The crack extension can be visualised by interrupting the tensile test and using a fatigue load to extend the present crack to total fracture. Another possibility is to heat tint the fracture surface after testing by heating the sample in an oxidising environment for several hours, followed by a statically induced total fracture. Using the contrast between fresh and oxidised fracture surface the amount of stable crack extension can be measured.
4 EVALUATION OF TEST RESULTS

In this chapter, the results obtained will be discussed. Four different series of materials have been produced, given in table 2, included their production method and composition.

*Table 2: Overview of all produced samples, blending methods, used powders and processes.*

<table>
<thead>
<tr>
<th>Sample Code</th>
<th>Volume Fraction</th>
<th>Cold Compaction Phase</th>
<th>Hot Compaction Phase</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rotation blended powders</td>
<td>Original process</td>
<td>Application of compaction pressure after sintering</td>
<td>&lt; 75 μm powder</td>
</tr>
<tr>
<td>9</td>
<td>10</td>
<td>1990</td>
<td>30</td>
</tr>
<tr>
<td>Rotation blended powders</td>
<td>Refined process</td>
<td>Application of compaction pressure after sintering</td>
<td>&lt; 75 μm powder</td>
</tr>
<tr>
<td>10 -12</td>
<td>10</td>
<td>1990</td>
<td>30</td>
</tr>
<tr>
<td>13 - 16</td>
<td>30</td>
<td>1990</td>
<td>30</td>
</tr>
<tr>
<td>Rotation blended powders</td>
<td>Refined process</td>
<td>Application of compaction pressure during sintering</td>
<td>&lt; 75 μm powder</td>
</tr>
<tr>
<td>17 - 18</td>
<td>10</td>
<td>1990</td>
<td>30</td>
</tr>
<tr>
<td>23 - 25, 27</td>
<td>10</td>
<td>1990</td>
<td>30</td>
</tr>
<tr>
<td>26, 28</td>
<td>10</td>
<td>1990</td>
<td>30</td>
</tr>
<tr>
<td>29</td>
<td>30</td>
<td>1990</td>
<td>30</td>
</tr>
<tr>
<td>30 - 31</td>
<td>30</td>
<td>1990</td>
<td>30</td>
</tr>
<tr>
<td>32</td>
<td>30</td>
<td>1990</td>
<td>60</td>
</tr>
</tbody>
</table>
The production processes used have been designated "original process", "refined process", "early load-application process" and "definitive process". Two series of materials have been produced and actually tested, being sample codes 9-32 and the "final" series marked series 07 - series 30. The "final" series have been marked using the volume fraction as the identifier (the first two digits), followed by a sequential code representing the sample number in its own batch after the digital point. Of these two tested series, only the second series have been produced by an optimised process. Since mechanical properties of the non-optimised material are irrelevant, the first series will only be used for the description of the search for an optimised production process.

4.1 Structures obtained from different powders and blending methods

During the search for the optimum process, blending method and powders, the following sample have been produced, using different powders, process methods and blending methods.

4.1.1 Rotational blending method

For the first series of samples, aluminium powder type AA6061-75 was used. This powder has a particle size below 75 micron. The powders were blended using the rotating ceramic container method. The powders were blended until no macroscopic conglomerates remained visible a feature that was verified using examination by an optical stereo microscope at a 40 x magnification.

The blending method does not produce a conglomerate-free composite powder as can be seen in figure 39. This can be attributed to the fact that the powder sticks to the walls of the ceramic.
container. When the powder is removed from the container, these conglomerates will come loose from the walls and settle as defects in the material during cold pressing.

**figure 39: Macroscopic photograph of the presence of conglomerates in the fracture surface.**

The powder was cold pressed at a pressure of 1990 bar and the pressure was held constant for 30 seconds. The samples were then sintered at 600 °C, the compaction pressure was manually or computer-controlled applied during sintering and cooling. A schematic representation of the process is given in figure 40:

**figure 40: Representation of the "original process" used for the first sample.**

During the drying and sintering phases, the upper plunger has no contact with the specimen. When these stages of the hot compaction process are completed, the pressure is applied by making contact between the plungers and the sample. The movement of the plungers is force-controlled,
which results in the generation of a peak load when the samples touch. This peak load cannot be
measured by the control system of the hot vacuum press, based on the displacement in this short
period, it is expected that these peak load are much higher than visible in figure 41. Since a very
large part of the compaction (approximately 70 %) of the sample takes place during this peak load,
the reproducibility of the process is in danger.

This reproducibility problem can be solved by manually applying the load and handling the control
over to the computer system before the specimen is heated to a higher temperature. In the drying
and sintering phases a small pre-pressure (0.5 bar) is thus programmed to compensate for thermal
expansion of the specimen. In this way, the controlling system is already stabilised at lower
temperatures, and the application of the pressure will be more gradually and reproducible. The
effect of the computer-controlled application of the load can be seen in figure 42.
The computer controlled application of the pressure does not compact the specimen more efficiently, but it provides better control over the process.

The evacuation of porosity is one of the main challenges left in the search for an optimum material. In order to reduce porosity, the "refined process" is modified. Subject of modification is the moment when the compaction pressure is applied. In the first two sets of samples the pressure was applied during cooling after sintering, in the "early load-application process", the compaction pressure is applied when the sintering temperature is reached. The sample is then compacted and sintered during a certain amount of time.

The densities of the samples produced by the rotational blending method have been measured. The results are summarised in table 3.

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>09 07</td>
<td>39.23</td>
<td>23.95</td>
<td>2797.52</td>
<td>2568.27</td>
<td>91.81%</td>
<td></td>
</tr>
<tr>
<td>10 07</td>
<td>35.58</td>
<td>22.50</td>
<td>2797.52</td>
<td>2720.09</td>
<td>97.23%</td>
<td></td>
</tr>
<tr>
<td>11 07</td>
<td>42.06</td>
<td>26.54</td>
<td>2797.52</td>
<td>2710.65</td>
<td>96.89%</td>
<td></td>
</tr>
<tr>
<td>12 07</td>
<td>43.93</td>
<td>27.95</td>
<td>2797.52</td>
<td>2750.12</td>
<td>98.31%</td>
<td></td>
</tr>
<tr>
<td>13 22</td>
<td>35.66</td>
<td>22.25</td>
<td>2985.01</td>
<td>2668.73</td>
<td>89.07%</td>
<td></td>
</tr>
<tr>
<td>14 22</td>
<td>33.57</td>
<td>20.79</td>
<td>2985.01</td>
<td>2628.25</td>
<td>88.05%</td>
<td></td>
</tr>
<tr>
<td>15 22</td>
<td>40.92</td>
<td>25.94</td>
<td>2985.01</td>
<td>2730.67</td>
<td>91.48%</td>
<td></td>
</tr>
<tr>
<td>16 22</td>
<td>44.01</td>
<td>28.11</td>
<td>2797.52</td>
<td>2769.04</td>
<td>98.98%</td>
<td></td>
</tr>
<tr>
<td>17 07</td>
<td>44.22</td>
<td>28.26</td>
<td>2797.52</td>
<td>2770.78</td>
<td>99.04%</td>
<td></td>
</tr>
<tr>
<td>18 07</td>
<td>43.45</td>
<td>27.89</td>
<td>2797.52</td>
<td>2792.20</td>
<td>99.81%</td>
<td></td>
</tr>
<tr>
<td>19 07</td>
<td>41.26</td>
<td>26.44</td>
<td>2797.52</td>
<td>2783.39</td>
<td>99.50%</td>
<td></td>
</tr>
<tr>
<td>20 07</td>
<td>98.71</td>
<td>63.35</td>
<td>2797.52</td>
<td>2791.36</td>
<td>99.78%</td>
<td></td>
</tr>
<tr>
<td>21 07</td>
<td>43.68</td>
<td>28.01</td>
<td>2797.52</td>
<td>2788.04</td>
<td>99.66%</td>
<td></td>
</tr>
<tr>
<td>22 07</td>
<td>44.83</td>
<td>28.77</td>
<td>2797.52</td>
<td>2791.79</td>
<td>99.80%</td>
<td></td>
</tr>
<tr>
<td>23 22</td>
<td>42.83</td>
<td>28.40</td>
<td>2985.01</td>
<td>2968.98</td>
<td>99.47%</td>
<td></td>
</tr>
<tr>
<td>24 22</td>
<td>44.35</td>
<td>29.35</td>
<td>2985.01</td>
<td>2955.97</td>
<td>99.03%</td>
<td></td>
</tr>
<tr>
<td>25 22</td>
<td>88.42</td>
<td>58.61</td>
<td>2985.01</td>
<td>2966.57</td>
<td>99.39%</td>
<td></td>
</tr>
</tbody>
</table>

The measured densities of the samples coded 9, 14, 15, and 16 are overestimated, because of 'open' porosity. This means that the pores in the material are connected, allowing the water in which the measurement is done to penetrate deeply into the material. This penetration of the measurement fluid results in an overestimation of the density and can be identified by an ever increasing mass of the sample when it is immersed in the measurement medium.

For comprehensibility, these results have been graphically presented in figure 43. Based on the obtained densities it can be concluded that the application of the compaction pressure during sintering results in higher densities when compared the process where the compaction pressure is applied during cooling after sintering.
applied after sintering. When sintering times and compaction times are reduced, no structural effect on the composite density is observed.

![Graph showing relative density and compaction times](image)

**Figure 43**: Overview of the obtained densities, using different sintering and compaction times.

To verify whether the ceramic and aluminium particles are well-bonded, scanning electron microscopy (SEM) is applied to study the fracture surface of broken samples. From observations of the fracture surface the quality of the matrix/reinforcement bond can be estimated. The results of this study for sample 10 are shown in figure 44. From these figures, it can clearly be seen that the two constituents have not bonded together at a micro-level. The application of the compaction step during sintering resulted in an increased quality of the matrix/reinforcement bond strength.
Based on the density measurements and the SEM investigation, it can be concluded that the process where the compaction step is applied during sintering performs satisfactory. No significant effect of sintering and compaction temperature was observed, however since no interfacial reactions are expected there is no reason to shorten the sintering and compaction times. The final process from these series will be applied during hot compaction of all the other samples, and the process parameters used in this definitive process are summarised in table 4:

<table>
<thead>
<tr>
<th>Process Step</th>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Drying Step</td>
<td>Drying Time</td>
<td>30</td>
</tr>
<tr>
<td></td>
<td>Drying Pressure</td>
<td>0.5 bar</td>
</tr>
<tr>
<td></td>
<td>Drying Temperature</td>
<td>450 °C</td>
</tr>
<tr>
<td>Sintering Step</td>
<td>Sinter Temperature</td>
<td>615 °C</td>
</tr>
<tr>
<td></td>
<td>Sinter Time</td>
<td>120 minutes</td>
</tr>
<tr>
<td></td>
<td>Sinter Pressure</td>
<td>2040 bar</td>
</tr>
<tr>
<td>Cooling Down Cycle Step</td>
<td>Cooling Rate</td>
<td>Natural Cooling</td>
</tr>
<tr>
<td></td>
<td>Cooling Pressure</td>
<td>2040 bar</td>
</tr>
</tbody>
</table>

The microstructure of the material produced should resemble the structure of a traditional discontinuously reinforced MMC, where the reinforcement is evenly distributed throughout the matrix. In order to check if the process as described above produces this structure, a microstructural examination was performed using light microscopy with magnifications of 400x. In...
figure 45 the results of this microscopic analysis are given, and it is clear that the ceramic particles are distributed as a network throughout the matrix.

![Microscopic Analysis](image)

*figure 45: Light microscopy photographs showing the microstructure of (a) 22 vol. % Al₂O₃ MMC and (b) commercially produced Duralcan material.*

From the micrographs, it is clear that the aluminium particles have remained more or less intact during the entire process and that the ceramic particles (that are much smaller) have settled around the aluminium particles as visualised in figure 46.

![Suggested Morphology](image)

*figure 46: Suggested morphology of blended powders before any compaction step.*

From these observations, it can be concluded that the combination of large AA6061 particles with small Al₂O₃ particles, in combination with the container blending method leads to an unfavourable structure where the ceramic particles are weakly bonded to the aluminium particles. During sintering, some penetration of aluminium in the 'ceramic' network takes place, however, the network structure obtained after powder blending remains and the penetration of the aluminium in the 'ceramic' network is not sufficient.
4.1.2 Mechanical alloying blending method

In order to modify the powder morphology, some kind of milling has to be applied. When this milling is combined with the actual blending, a well-blended powder is expected. When a mechanical alloying procedure is applied, the steel balls crush the powders and the average size of the powders is decreased. Because the aluminium powder is much weaker than the alumina powder, the decrease in size of the aluminium powder is expected to be larger than that of the alumina powder. A side effect of the powder crushing is an increase in powder surface area and an oxidisation of the aluminium particle surface but since oxide layers on aluminium are only a few nm thick, it is not expected that the composition of the powder alters significantly. A milling fluid has to be used during this process to ensure that the friction heat generated during crushing is evacuated. In this case alcohol is used as the milling fluid. The use of alcohol also eliminates any explosion dangers since all free surface that is generated will be directly oxidised by the alcohol.

In order to investigate the effect of mechanical alloying on the powders, an amount of unblended composite powder is inserted in the mechanical alloying mill and samples were taken from the mill every hour. The result after six hours is a collection of powders with blending times varying from 0 hours (the as fabricated, de-mixed condition) till 6 hours. These powders were then compacted into thin discs, an assembled to a layered structure in the hot vacuum press as shown in figure 47. An aluminium interlayer alternated the layers to separate the different powders. This layered sample was then hot compacted using the process described in table 4, with a prolonged drying stage to remove any alcohol left in the sample.

![Layered Structure](image)

**figure 47:** Schematic depiction of the layered structure used to examine the effect of mechanical alloying time on microstructure.

The result of this process is a layered sample where different powder morphologies have been compacted under the same circumstances. The difference between the aluminium interlayers and the MMC powders is very clear, as can be seen in figure 48:
The layered sample was subjected to a microscopic investigation to identify the milling time that results in the wanted, homogeneous MMC structure. The results of the microscopic examination are given in figure 49 - figure 55, this structure is processed using the definitive process, as described in table 4.

**figure 48:** Surface photograph of the layered structure, clearly showing the aluminium interlayers.

**figure 49:** Microstructure of hot compacted powder, as fabricated condition.

**figure 50:** Microstructure of hot compacted powder, milled for 1 hour.
figure 51: Microstructure of hot compacted powder, milled for 2 hours.

figure 52: Microstructure of hot compacted powder, milled for 3 hours.

figure 53: Microstructure of hot compacted powder, milled for 4 hours.

figure 54: Microstructure of hot compacted powder, milled for 5 hours.
EVALUATION OF TEST RESULTS

As can be seen from these pictures, using the mechanical alloying mill a homogeneous microstructure, with the Al2O3 particles well embedded in the aluminium matrix, is obtained after 6 hours of milling. Another observation is that the particles are, especially for short milling times, flattened, which results in the observed layered structure.

The powder that remained in the mechanical alloying mill after this test sequence was dried and cold compressed into three tablets with the correct volume to produce the fracture toughness samples (19.5 mm thick and 50 mm diameter). These samples were hot compacted according to the process mentioned in table 4, however after the first sample was hot compacted, it was clear that, during milling, the structure of the material was significantly altered. The sintering/compaction temperature was increased up to 800 °C in the following two samples, while a compaction pressure of 200 bar was maintained. The fact that an 'aluminium MMC' does not deform at 800 °C under a pressure of 200 bar is a good reason to assume that the milled powders cannot produce a significant MMC. Besides the fact that there was probably not much metal left in the sample, there was also the danger of explosion due to severe oxidisation of the sample. When the sample was sliced and a new surface was created, this triggered an oxidation reaction in the sample. Because of this oxidation, a lot of heat is produced and in some cases the material exploded because of the thermal expansion in the inside. Clearly, this is no construction material.

Thus, despite the homogenous material, the milling method is not used any further in this research.

4.1.3 Turbula Blending Method

In order to obtain a better MMC structure, without making use of the milling method, the AA6061-45 type powder was used, with particle sizes under 45 μm. For an optimum structure, a powder with an average particle size of 4.5 μm is needed, but this type was not available at the time.

The powders were blended in the turbula-type mixer for at least 48 hours, to ensure full and permanent blending. Again the powder was analysed using a stereo microscope with a magnification of 40x. Four series of powders were blended, having volume fractions Al2O3 of 7, 10, 14, 22 and 30 vol. %.

The powders were cold compacted at a pressure of 2040 bar, which resulted in the semi-compact structure visualised in figure 56. An eye-catching feature of the cold compacted structure is the absence of high levels of porosity. From the density measurements it can be deducted that there must be a high level of porosity, however it is not visible at this magnification. Higher magnifications using light microscopy are not possible because of the lack of depth-of-focus.
EVALUATION OF TEST RESULTS

Figure 56: Example of cold pressed tablet (a) and the cold compacted structure at a magnification of 25x (b).

Hot compaction of this series of samples is performed according to the method described in table 4 and this process was highly reproducible, as can be seen from figure 56 and figure 58, where the temperatures and applied compaction pressures are shown for five different samples.

Figure 57: Temperature versus temperature for 5 hot compaction steps based on the same program, showing good reproducibility.
In order to determine the compaction level of the samples produced with the "definitive process", all samples produced were subjected to a density measurement, the results are given in table 5.

**table 5: Measured versus theoretical densities for the samples produced with the definitive process.**

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>MMC 07.1</td>
<td>6.86 %</td>
<td>48.979</td>
<td>31.346</td>
<td>2795.77</td>
<td>2803.12</td>
<td>99.31 %</td>
</tr>
<tr>
<td>MMC 07.2</td>
<td>6.86 %</td>
<td>42.648</td>
<td>27.383</td>
<td>2795.77</td>
<td>2793.67</td>
<td>99.93 %</td>
</tr>
<tr>
<td>MMC 07.3</td>
<td>6.86 %</td>
<td>46.160</td>
<td>29.538</td>
<td>2795.77</td>
<td>2776.87</td>
<td>99.32 %</td>
</tr>
<tr>
<td>MMC 07.5</td>
<td>6.86 %</td>
<td>43.887</td>
<td>28.134</td>
<td>2795.77</td>
<td>2785.78</td>
<td>99.64 %</td>
</tr>
<tr>
<td>MMC 10.1</td>
<td>9.98 %</td>
<td>44.687</td>
<td>28.915</td>
<td>2834.71</td>
<td>2833.14</td>
<td>99.94 %</td>
</tr>
<tr>
<td>MMC 10.2</td>
<td>9.98 %</td>
<td>45.580</td>
<td>29.489</td>
<td>2834.71</td>
<td>2832.47</td>
<td>99.92 %</td>
</tr>
<tr>
<td>MMC 10.3</td>
<td>9.98 %</td>
<td>43.334</td>
<td>28.040</td>
<td>2834.71</td>
<td>2833.23</td>
<td>99.95 %</td>
</tr>
<tr>
<td>MMC 14.1</td>
<td>14.16 %</td>
<td>43.577</td>
<td>28.428</td>
<td>2866.99</td>
<td>2876.39</td>
<td>99.63 %</td>
</tr>
<tr>
<td>MMC 14.3</td>
<td>14.16 %</td>
<td>47.547</td>
<td>30.904</td>
<td>2886.99</td>
<td>2856.70</td>
<td>98.95 %</td>
</tr>
<tr>
<td>MMC 14.4</td>
<td>14.16 %</td>
<td>43.380</td>
<td>28.246</td>
<td>2886.99</td>
<td>2866.22</td>
<td>99.28 %</td>
</tr>
<tr>
<td>MMC 14.5</td>
<td>14.16 %</td>
<td>46.317</td>
<td>30.148</td>
<td>2886.99</td>
<td>2864.38</td>
<td>99.22 %</td>
</tr>
<tr>
<td>MMC 14.6</td>
<td>14.16 %</td>
<td>45.933</td>
<td>30.019</td>
<td>2886.99</td>
<td>2868.15</td>
<td>99.97 %</td>
</tr>
<tr>
<td>MMC 22.1</td>
<td>21.95 %</td>
<td>38.317</td>
<td>25.431</td>
<td>2984.41</td>
<td>2973.36</td>
<td>99.63 %</td>
</tr>
<tr>
<td>MMC 22.2</td>
<td>21.95 %</td>
<td>42.114</td>
<td>27.889</td>
<td>2984.41</td>
<td>2960.38</td>
<td>99.20 %</td>
</tr>
<tr>
<td>MMC 22.3</td>
<td>21.95 %</td>
<td>44.577</td>
<td>29.556</td>
<td>2984.41</td>
<td>2967.47</td>
<td>99.43 %</td>
</tr>
<tr>
<td>MMC 22.4</td>
<td>21.95 %</td>
<td>40.923</td>
<td>27.182</td>
<td>2984.41</td>
<td>2977.99</td>
<td>99.78 %</td>
</tr>
<tr>
<td>MMC 22.5</td>
<td>21.95 %</td>
<td>42.927</td>
<td>28.480</td>
<td>2984.41</td>
<td>2971.16</td>
<td>99.56 %</td>
</tr>
<tr>
<td>MMC 22.6</td>
<td>21.95 %</td>
<td>40.480</td>
<td>26.885</td>
<td>2984.41</td>
<td>2977.39</td>
<td>99.76 %</td>
</tr>
<tr>
<td>MMC 22.7</td>
<td>21.95 %</td>
<td>41.565</td>
<td>27.569</td>
<td>2984.41</td>
<td>2969.60</td>
<td>99.50 %</td>
</tr>
<tr>
<td>MMC 30.1</td>
<td>29.83 %</td>
<td>47.056</td>
<td>31.621</td>
<td>3082.88</td>
<td>3048.47</td>
<td>98.88 %</td>
</tr>
<tr>
<td>MMC 30.2</td>
<td>29.83 %</td>
<td>47.626</td>
<td>31.947</td>
<td>3082.88</td>
<td>3037.38</td>
<td>98.52 %</td>
</tr>
<tr>
<td>MMC 30.3</td>
<td>29.83 %</td>
<td>95.947</td>
<td>64.376</td>
<td>3082.88</td>
<td>3038.90</td>
<td>98.57 %</td>
</tr>
</tbody>
</table>

The large majority of sample has densities of over 99% of the theoretical density. This is the proof that the optimised process is also applicable to the new powder type. The samples with 30 vol. %
alumina have the lowest relative densities. The data of table 5 is graphically represented in figure 59.

![Graph showing relative densities for the samples produced with the definitive process.](image)

**figure 59:** Relative densities for the samples produced with the definitive process.

For this series of samples, scanning electron microscopy was used for two purposes, first a SEM study gives a clear contrast between the aluminium matrix (which is dark, because of the good electrical conduction) and the alumina particles (light coloured, non-conducting). The second reason is that SEM allows for larger magnification, which are necessary to study the interface between matrix and reinforcement. Thus, SEM can be successfully applied to study both the structure of the material and the bonding between the components.

![SEM images showing the structure of the material.](image)

(a) 10 vol. %  
(b) 14 vol. %
Apart from the improved wetting of the 'ceramic' network, the structure still resembles the structure as obtained by the container blending method, and is still different from the reference structure of the Duralcan material. This structure is best characterised as an AA6061-MMC composite, where the MMC forms the network around the aluminium grains. This MMC network has much higher volume fractions of alumina than the nominal values. Based on an image analysis, where the white areas are defined as alumina and the dark areas are defined as aluminium, the local volume fraction in the MMC network can be estimated. Therefore, a "white area fraction" is defined as the fraction of the total area being white.

Application of this procedure to the overall sample as shown in figure 60d results in an overall "white area fraction" of 21%. When the analysed area is narrowed to an area entirely filled with the MMC network, a "white area fraction" of 69% is measured. This indicates that the local alumina volume fraction in the MMC network is up to three times higher than the nominal volume fraction. When these results are translated to real volume fractions, a local volume fraction of 90 volume percent alumina is found for the MMC network.

As can be seen from figure 61, the network phase is fully wetted with aluminium, which indicates good bonding.
The shadows that are visible in figure 61(b) are caused by the penetration of electrons in the material. Because of this penetration an image of the not only the surface layer, but also the interior of the material at the surface is obtained.

Based on the overall characterisation of the material as described above, it can be concluded that this material is qualified for a determination of the mechanical properties. The results of the mechanical tests will be described in the next section.

4.2 Sample preparation

All samples produced with the "definitive process" have been prepared for fracture toughness testing. All samples were shaped by water jetting and the detailed machining of the Chevron-notch was done using spark erosion. The double V-notch production succeeded for all samples with volume fractions under 22 vol. %. For the higher volume fractions, the electrical conduction proved insufficient and a Chevron notch ending in a rectangular front had to be machined.

For verification of the fracture toughness measurement method, a series of metal samples was produced from an extruded AA6061 cylinder by milling.

All samples (including the AA6061 samples) received a solutionising heat treatment at 530 °C followed by quenching in water. After natural ageing for 15 hours at –20 °C the samples were artificially aged for 8 hours at 175 °C again followed by quenching in water.

4.3 Results of precracking procedure

Following heat treatment, the samples were precracked at a frequency of 35 Hz using the constant \( K_{\text{max}} \) technique and online compliance measurement. When the desired crack length was reached, the load was automatically lowered to zero and the test was terminated.

The required stress intensities during the precracking procedure were determined by stepwise increases of the maximum stress intensity until the crack growth initiated. The applied stress intensities for all samples are included in figure 62:

![Graph](image_url)  

*figure 62: Applied minimum and maximum stress intensities during precracking.*
In figure 62 only the results of successful precracking procedures are included. Because of the highly experimental nature of the experimental set-up used, some results and some samples were lost during precracking. In table 6 an overview is given of the lost samples.

**Table 6: Overview of the samples lost during precracking, and the events that lead to the loss.**

<table>
<thead>
<tr>
<th>Sample Code</th>
<th>Event causing loss</th>
</tr>
</thead>
<tbody>
<tr>
<td>AA6061.3</td>
<td>Equipment Failure</td>
</tr>
<tr>
<td>MMC 07.2</td>
<td>Equipment Failure</td>
</tr>
<tr>
<td>MMC 07.3</td>
<td>Operator Error</td>
</tr>
<tr>
<td>MMC 14.3</td>
<td>Power Failure</td>
</tr>
<tr>
<td>MMC 14.4</td>
<td>Power Failure</td>
</tr>
<tr>
<td>MMC 22.2</td>
<td>Equipment Error</td>
</tr>
<tr>
<td>MMC 22.3</td>
<td>Operator Error</td>
</tr>
<tr>
<td>MMC 30.1</td>
<td>Static Fracture</td>
</tr>
<tr>
<td>MMC 30.2</td>
<td>Power Failure</td>
</tr>
<tr>
<td>MMC 30.3</td>
<td>Power Failure</td>
</tr>
</tbody>
</table>

All other samples were successfully precracked using the stress intensity and R-ratio versus crack length procedures as visualised in figure 63 and figure 64, which have been measured for a typical sample.

**Figure 63:** Stress intensity versus crack length for the applied precracking procedure (measured for sample MMC 07.5).
Application of this procedure leads to a crack growth curve as depicted in figure 65, where the instantaneous crack lengths has been calculated using the compliance method.

In figure 65 an increase in the error of the crack length is visible. This increase in spread can be easily explained since the measurement of the compliance is based on the difference between minimum and maximum values of both load and displacement. If this difference becomes smaller (which is the case when the R-ratio is increased with constant $K_{\text{max}}$) the uncertainty will increase. Because both differences (load and displacement) decrease, a strong decrease in measurement sensitivity is observed.
Since the compliance can only be related to a crack length using the Young’s modulus, this value has to be estimated before the start of the precracking procedure. After precracking, the end crack length was measured from a post-mortem analysis, and the apparent Young’s modulus was determined by backward calculation from the last compliance measured.

The results of this backward calculation of the apparent Young’s modulus have been included in table 7 and figure 66:

**Table 7: Results of the backward calculation of the Young’s modulus.**

<table>
<thead>
<tr>
<th>Sample Code</th>
<th>Last specimen compliance [μm/kN]</th>
<th>Backwards calculated apparent Young’s Modulus [GPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>AA6061.1</td>
<td>36.61</td>
<td>87.30</td>
</tr>
<tr>
<td>AA6061.2</td>
<td>34.17</td>
<td>80.69</td>
</tr>
<tr>
<td>AA6061.3</td>
<td>33.81</td>
<td>97.01</td>
</tr>
<tr>
<td>MMC 07.5</td>
<td>41.55</td>
<td>61.33</td>
</tr>
<tr>
<td>MMC 10.1</td>
<td>38.54</td>
<td>58.85</td>
</tr>
<tr>
<td>MMC 10.2</td>
<td>46.25</td>
<td>51.05</td>
</tr>
<tr>
<td>MMC 10.3</td>
<td>49.50</td>
<td>55.93</td>
</tr>
<tr>
<td>MMC 14.1</td>
<td>33.44</td>
<td>71.81</td>
</tr>
<tr>
<td>MMC 14.4</td>
<td>42.06</td>
<td>62.82</td>
</tr>
<tr>
<td>MMC 14.6</td>
<td>39.82</td>
<td>75.08</td>
</tr>
<tr>
<td>MMC 22.2</td>
<td>34.66</td>
<td>91.78</td>
</tr>
<tr>
<td>MMC 22.4</td>
<td>35.26</td>
<td>84.63</td>
</tr>
<tr>
<td>MMC 22.5</td>
<td>32.49</td>
<td>109.99</td>
</tr>
<tr>
<td>MMC 22.6</td>
<td>28.77</td>
<td>112.59</td>
</tr>
<tr>
<td>MMC 22.7</td>
<td>31.26</td>
<td>88.91</td>
</tr>
</tbody>
</table>

The results are graphically represented in figure 65.
It must be noted that this backwards calculated apparent Young's modulus includes other precracking effects and in fact is used as a control parameter for crack growth. This value cannot be used as the actual Young's modulus of the composite.

Whether the precracking procedure satisfies the demands imposed by the fracture toughness measurement methods, can be seen after post-mortem analysis of the fracture surface. As can be seen from figure 67, the difference between fatigue crack and stable crack extension during tensile testing can easily be made. The results of fatigue crack length measurements are included in table 8 and figure 68.
The fatigue crack fronts are only useable for fracture toughness determination if they are sufficiently straight, therefore it is useful to quantify the crack curvature (caused by tunneling) and crack obliqueness (caused by differences in crack growths across the specimen) using equation 20 and 21, the results are given in table 8.

### Table 8: Results of the characterisation of the fatigue crack front

<table>
<thead>
<tr>
<th>Sample Code</th>
<th>Average Crack Length [mm]</th>
<th>Crack Curvature [%]</th>
<th>Crack Obliqueness [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>AA6061.1</td>
<td>19.41</td>
<td>5.93%</td>
<td>12.18%</td>
</tr>
<tr>
<td>AA6061.2</td>
<td>18.19</td>
<td>4.57%</td>
<td>7.48%</td>
</tr>
<tr>
<td>AA6061.3</td>
<td>15.41</td>
<td>18.95%</td>
<td>9.80%</td>
</tr>
<tr>
<td>AA6061.4</td>
<td>19.80</td>
<td>2.73%</td>
<td>8.61%</td>
</tr>
<tr>
<td>AA6061.5</td>
<td>15.11</td>
<td>15.88%</td>
<td>7.13%</td>
</tr>
<tr>
<td>MMC 07.1</td>
<td>17.76</td>
<td>6.78%</td>
<td>4.59%</td>
</tr>
<tr>
<td>MMC 07.5</td>
<td>17.45</td>
<td>13.69%</td>
<td>6.80%</td>
</tr>
<tr>
<td>MMC 10.1</td>
<td>18.30</td>
<td>7.72%</td>
<td>10.25%</td>
</tr>
<tr>
<td>MMC 10.2</td>
<td>17.30</td>
<td>5.76%</td>
<td>4.58%</td>
</tr>
<tr>
<td>MMC 10.3</td>
<td>18.67</td>
<td>15.47%</td>
<td>6.27%</td>
</tr>
<tr>
<td>MMC 14.1</td>
<td>18.60</td>
<td>10.63%</td>
<td>8.31%</td>
</tr>
<tr>
<td>MMC 14.3</td>
<td>22.32</td>
<td>14.42%</td>
<td>5.41%</td>
</tr>
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<td>MMC 14.4</td>
<td>18.63</td>
<td>9.72%</td>
<td>6.21%</td>
</tr>
<tr>
<td>MMC 14.5</td>
<td>18.72</td>
<td>15.46%</td>
<td>8.40%</td>
</tr>
<tr>
<td>MMC 14.6</td>
<td>19.48</td>
<td>15.12%</td>
<td>6.74%</td>
</tr>
<tr>
<td>MMC 22.2</td>
<td>18.46</td>
<td>4.44%</td>
<td>9.31%</td>
</tr>
<tr>
<td>MMC 22.4</td>
<td>17.64</td>
<td>23.87%</td>
<td>9.59%</td>
</tr>
<tr>
<td>MMC 22.5</td>
<td>19.12</td>
<td>26.57%</td>
<td>12.05%</td>
</tr>
<tr>
<td>MMC 22.6</td>
<td>18.32</td>
<td>28.59%</td>
<td>12.46%</td>
</tr>
<tr>
<td>MMC 22.7</td>
<td>17.14</td>
<td>5.43%</td>
<td>4.11%</td>
</tr>
</tbody>
</table>
From figure 68 it can be concluded that the experimental set-up used is sufficiently accurate in stopping the fatigue precracking process in time. Only two tests falls outside the region of allowed fatigue crack lengths, and these could be ascribed to an equipment failure (for sample AA6061.3) and a power loss causing a severe overload (for sample MMC 14.3).

The quantification of the fatigue crack front shapes cannot strictly identify the samples having an invalid crack front according to the conditions imposed by the fracture toughness measurement methods. In the fracture toughness measurement methods multiple conditions are combined for the curvature of the fatigue crack front, these conditions cannot easily be expressed as curvature ratios. However, when both the curvature and the obliqueness ratios are small, the fatigue crack front are valid or close to valid according to the fracture toughness test methods.
EVALUATION OF TEST RESULTS

Precracking of the samples containing 30 volume percent alumina proved very difficult. A very small overload causes brittle failure and crack propagation rates are very low. For illustration, the sample MMC 30.3 has been precracked for 63 hours, which corresponds to approximately eight million stress variations. The procedure was ended by a power failure, which caused brittle failure of the tested sample. After failure, only a very small amount of crack growth (~1 mm) was observed.

4.4 Fracture toughness measurements

After precracking, the samples were loaded until fracture. During loading, load and clip gauge displacement were recorded, resulting in a load-displacement plot as included in figure 70:

![figure 70: Example of a load displacement plot containing nine partial unloadings.]

This type of data collection can be used for the three methods for determination of fracture toughness, as described in section 3. The unloading curves shown in figure 70 can only be programmed when the shape and position of the load/displacement curve is known. Therefore, load-displacement curves containing unloadings are not available for all tested samples. For all samples, the plain strain fracture toughness could be calculated according to ASTM E399, and ASTM E1290. The J-integral characterisation of fracture toughness could be performed for all samples except the samples coded AA6061.1, MMC 10.1, MMC 14.1 - MMC 14.6.

4.4.1 E399 Fracture toughness results

An example of the determination of the $P_q$ value according to ASTM E399 is included in figure 71. To aid in the identification of the linear part of the load-displacement section, which is used for the calculation of the slope, a local slope is calculated. The region in which the local slope remains constant is the region in which the load versus displacement behaviour is linear. An example of the local slope versus displacement curve is included in figure 72.

![figure 71: Example of the determination of the $P_q$, using the 95% slope procedure.]

---

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EVALUATION OF TEST RESULTS

The load/displacement curve may be, based on what is visible in figure 71 and figure 72, divided into four sections:

<table>
<thead>
<tr>
<th>Table 9: Characterisation of the load/displacement curves by the identification of four sections.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Section I</td>
</tr>
<tr>
<td>Section II</td>
</tr>
<tr>
<td>Section III</td>
</tr>
<tr>
<td>Section IV</td>
</tr>
</tbody>
</table>

Section 1 is not used for determination of the fracture toughness, in accordance with the ASTM E399 method [50]. Section 2 is similar to the linear behaviour normally observed during plain strain fracture toughness tests. Sections 3 and 4 heavily influence the determination of plain strain fracture toughness according to ASTM E399, since they determine the intersection point of the load/displacement curve with the 95% slope construction line.
The results of the plain strain fracture toughness determination according to ASTM E399 are summarised in table 10:

**Table 10: Overview of the measured plain strain fracture toughness values and their validity.**

<table>
<thead>
<tr>
<th>Sample Code</th>
<th>$K_I$ or $K_{IC}$</th>
<th>Crack Length</th>
<th>Crack Curvature</th>
<th>Crack Obliqueness</th>
<th>Load Rate</th>
<th>$P_k/P_{max}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>AA6061.1</td>
<td>40.39</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
</tr>
<tr>
<td>AA6061.2</td>
<td>41.22</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
</tr>
<tr>
<td>AA6061.3</td>
<td>36.98</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
</tr>
<tr>
<td>AA6061.4</td>
<td>40.75</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
</tr>
<tr>
<td>AA6061.5</td>
<td>42.06</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
</tr>
<tr>
<td>MMC 07.1</td>
<td>13.34</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
</tr>
<tr>
<td>MMC 07.5</td>
<td>12.17</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
</tr>
<tr>
<td>MMC 10.1</td>
<td>13.16</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
</tr>
<tr>
<td>MMC 10.2</td>
<td>12.49</td>
<td></td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
</tr>
<tr>
<td>MMC 10.3</td>
<td>13.28</td>
<td></td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
</tr>
<tr>
<td>MMC 14.1</td>
<td>13.34</td>
<td></td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
</tr>
<tr>
<td>MMC 14.5</td>
<td>15.17</td>
<td></td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
</tr>
<tr>
<td>MMC 14.6</td>
<td>14.06</td>
<td></td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
</tr>
<tr>
<td>MMC 22.2</td>
<td>13.50</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
</tr>
<tr>
<td>MMC 22.4</td>
<td>12.74</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
</tr>
<tr>
<td>MMC 22.5</td>
<td>13.90</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
</tr>
<tr>
<td>MMC 22.6</td>
<td>13.81</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
</tr>
<tr>
<td>MMC 22.7</td>
<td>11.83</td>
<td></td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
<td>INVALID</td>
</tr>
</tbody>
</table>

Altogether, one measured fracture toughness value has been qualified "valid according to ASTM E399". It can be seen from table 10 that it is possible to produce a valid crack length, and a valid crack front. In four cases a valid crack front curvature, length and obliqueness was obtained, however the $P_k/P_{max} < 1.10$ condition is harder to satisfy. Only two samples, both containing 22 vol. \% $\text{Al}_2\text{O}_3$ satisfy this condition.
The fracture toughness values obtained are graphically represented in figure 73:

Figure 73: Plane strain fracture toughness versus volume fraction, according to ASTM E399. Circles are used to accentuate the fracture toughness values that have been qualified "valid according to ASTM E399".
EVALUATION OF TEST RESULTS

Fracture toughness results

Before the J-integral can be calculated, a number of preparations steps have to be carried out. First, the measured crack mouth displacement has to be converted to the load-line displacement. This correction results in a scaling of the entire load/displacement curve as shown in figure 74.

![Scaling of load/displacement curve](image)

Figure 74: Scaling of the load/displacement curve caused by the geometrical conversion from crack mouth displacement to load-line displacement, load/displacement curves obtained from sample MMC 10.2.

From the load versus load-line displacement data, the unloading compliances are calculated for the individual unloadings. Using the compliance from the first unloading (or from another area where the crack extension is known to be zero) an estimate of the Young's modulus can be calculated.

The second correction is the compliance correction for specimen rotation. Because the measured crack mouth displacements are very small, the corrected compliance value differs less than 1.5% from the uncorrected compliance. Although the effect of the correction is small, it has been included in the calculations for all samples.

When the calculation of the specimen compliance is finished, the fit procedure is applied to estimate the Young's modulus of the composite. The modulus is modified using steps of 0.01 GPa until the calculated initial crack length equalled the measured initial crack length. The estimates of the modulus are reported in table 11 and figure 75. The crack effective modulus needed for calculation of the crack length was calculated using a Poisson's ratio of 0.33.
table 11: Estimated modulus value by way of fitting the calculated crack length on the measured crack length.

<table>
<thead>
<tr>
<th>Sample Code</th>
<th>Estimated Modulus [GPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>MMC 07.1</td>
<td>65.03</td>
</tr>
<tr>
<td>MMC 07.5</td>
<td>69.25</td>
</tr>
<tr>
<td>MMC 10.2</td>
<td>74.91</td>
</tr>
<tr>
<td>MMC 10.3</td>
<td>73.54</td>
</tr>
<tr>
<td>MMC 22.2</td>
<td>100.5</td>
</tr>
<tr>
<td>MMC 22.4</td>
<td>87.50</td>
</tr>
<tr>
<td>MMC 22.5</td>
<td>94.37</td>
</tr>
<tr>
<td>MMC 22.6</td>
<td>89.53</td>
</tr>
<tr>
<td>MMC 22.7</td>
<td>96.06</td>
</tr>
</tbody>
</table>

In figure 75 the estimated values are compared to the predicted values according to the model suggested by Tsai-Halpin [57].

figure 75: Predicted and estimated Young's moduli obtained from the initial crack length fit procedure.

After determination of the plastic component of the load-line displacement, the value of J for several crack extension events is calculated. The obtained data points of J versus crack extension are fitted on a power law, and the result of the calculation and fit procedure is shown in figure 76. In order to determine the critical J value, a blunting line is drawn using a yield strength of 295 MPa for the aluminium samples and 400 MPa for the MMC samples. This yield strength (obtained from literature values [26]) was used for all MMC samples since the actual yield strength is unknown and because the effect on J of a variation in yield strength is small.
From mathematical equations of the blunting line and the power law representing the J versus crack extension behaviour, the intersection point is calculated and identified as J_q. This J value is then converted to critical stress intensity for convenience. The results of the J integral characterisation of fracture toughness are given in figure 77.

<table>
<thead>
<tr>
<th>Sample Code</th>
<th>Initial Crack Length</th>
<th>Final Crack Length</th>
<th>Initial Crack Length</th>
<th>Final Crack Length</th>
<th>J_q [J/m²]</th>
<th>K_j [MPa√m]</th>
</tr>
</thead>
<tbody>
<tr>
<td>AA6061.2</td>
<td>18.19</td>
<td>20.59</td>
<td>18.19</td>
<td>-</td>
<td>5.08×10⁴</td>
<td>53.89</td>
</tr>
<tr>
<td>AA6061.3</td>
<td>15.41</td>
<td>18.64</td>
<td>15.41</td>
<td>21.78</td>
<td>4.96×10⁴</td>
<td>41.41</td>
</tr>
<tr>
<td>AA6061.4</td>
<td>19.81</td>
<td>20.63</td>
<td>19.81</td>
<td>20.38</td>
<td>5.82×10⁴</td>
<td>43.60</td>
</tr>
<tr>
<td>MMC 07.1</td>
<td>17.76</td>
<td>18.25</td>
<td>17.76</td>
<td>-</td>
<td>1.69×10⁴</td>
<td>35.18</td>
</tr>
<tr>
<td>MMC 07.5</td>
<td>17.45</td>
<td>18.2</td>
<td>17.45</td>
<td>18.62</td>
<td>1.99×10⁴</td>
<td>39.35</td>
</tr>
<tr>
<td>MMC 10.2</td>
<td>17.30</td>
<td>20.94</td>
<td>17.30</td>
<td>-</td>
<td>1.10×10⁴</td>
<td>30.44</td>
</tr>
<tr>
<td>MMC 10.3</td>
<td>18.67</td>
<td>21.33</td>
<td>18.67</td>
<td>21.69</td>
<td>1.38×10⁴</td>
<td>33.75</td>
</tr>
<tr>
<td>MMC 22.2</td>
<td>18.61</td>
<td>21.31</td>
<td>18.46</td>
<td>-</td>
<td>3.40×10⁴</td>
<td>19.68</td>
</tr>
<tr>
<td>MMC 22.4</td>
<td>17.64</td>
<td>21.69</td>
<td>17.64</td>
<td>22.75</td>
<td>3.62×10⁴</td>
<td>18.86</td>
</tr>
<tr>
<td>MMC 22.5</td>
<td>19.12</td>
<td>20.26</td>
<td>19.12</td>
<td>20.40</td>
<td>3.17×10⁴</td>
<td>18.32</td>
</tr>
<tr>
<td>MMC 22.6</td>
<td>18.32</td>
<td>21.77</td>
<td>18.32</td>
<td>22.55</td>
<td>3.53×10⁴</td>
<td>18.85</td>
</tr>
<tr>
<td>MMC 22.7</td>
<td>17.14</td>
<td>21.56</td>
<td>17.14</td>
<td>23.19</td>
<td>1.72×10⁴</td>
<td>13.56</td>
</tr>
</tbody>
</table>

These results are graphically represented in figure 77:
To check the validity of the tests carried out, the conditions imposed by the ASTM E1737 [56] standard are checked for validity. The results will be individually discussed for each imposed condition.

**Condition 1:**

None of the measurements should differ more than 5% from the total crack length.

This fatigue crack front condition is more rigorous than the conditions imposed by the ASTM E399 method [50]. Therefore, only one sample satisfies the conditions, being sample MMC 22.7.

**Condition 2:**

None of the crack extension measurements shall differ more than 50% from the average crack extension.

Despite extensive tunnelling during stable crack extension, this condition is satisfied by sample MMC 22.7.

**Condition 3:**

The total crack extension is determined. Again, none of the measurements may differ more than 5% from the average.

Due to the extensive tunnelling during stable crack extension, this condition cannot be satisfied.

**Condition 4:**

The total crack extension determined by unloading compliance measurements shall not differ more than 0.15 $\Delta a_p$ for crack extensions under 0.2$b_o$. For larger crack extensions the difference shall not exceed 0.03 $b_o$. 

---

*figure 77: Fracture toughness versus volume fraction as characterised by ASTM E1737.*
The total measured and calculated crack extensions are given in table 12. A visual representation of the values is included in figure 78, where the calculated values of final crack extension are compared to the measured final crack length.

![Graph showing measured and calculated crack extensions](image)

**figure 78:** Measured versus calculated crack extensions for all samples.

As can be seen, only for samples AA6061.4 and MMC 22.5 the calculated final crack lengths approach the measured final crack length. All other measurements are invalid according to this condition.

**Condition 5:**

*The number of points in the data set used for determination of the power law coefficients shall be larger than or equal to 5.*

The maximum number of useable data points in this series of tests is four. All tests are invalid according to this condition.

**Condition 6:**

*The sample thickness should exceed 25 J_q/\sigma_y. The length of the initial ligament should exceed 25 J_q/\sigma_y.*

These conditions are satisfied for the combination of maximum J_q and minimum yield strength. Therefore, this condition is valid for all samples.

In general, it can be concluded that the test results do not satisfy the conditions. Especially the conditions regarding crack front shape and calculation are invalid. However, since the fracture toughness is characterised and not measured according to ASTM E1737, the results may still be used for evaluation.
4.4.3 E1290 Fracture toughness results

For a fracture toughness characterisation using the ASTM E1290 method, there are two important aspects:

The curves should have similar shapes. If the differences between the shapes of the curves are large, the definition of the critical load and displacement for calculation of the critical crack tip opening displacement (CTOD) introduces large measurement errors.

The crack growth behaviour must be the same for all tested samples. If this is not the case, different types of critical CTOD values will be determined and a comparison of the results will be impossible.

Fracture toughness measurements using method ASTM E399 have already shown that a substantial amount of stable crack extension takes place. If this were not the case, all samples would fracture in a brittle mode, having a load/displacement curve that suddenly drops towards zero load after the maximum load is exceeded. The similarity of the load/displacement curves is demonstrated by joining all data in a single graph. Because of the large difference in applied loads between the different series of samples, the data is compared in separate plots.

![Figure 79: Load/displacement curves for the aluminium samples, the lower the curve is positioned, the higher the initial crack length (compare figure 68).](image)

When figure 79 is studied, it can be seen that the curves having a higher maximum load, have a smaller initial crack length. When the stress intensity plots are calculated, this difference in height of the curves will be compensated.
EVALUATION OF TEST RESULTS

From Figure 79, Figure 80 and Figure 81 it can be concluded that the load/displacement plots are similarly shaped. Combination of the similar shape and the universally observed stable crack extension after maximum load allows the fracture toughness to be characterised using the method described in ASTM E1290. In all cases, the critical load and displacement values are defined as respectively the maximum load and the corresponding displacement.

The plastic component of the critical crack mouth opening displacement is measured using the initial slope obtained from the ASTM E399 method. The Young’s modulus is obtained from the backward calculation as done during J-integral characterisation of fracture toughness.

Figure 80: Load/displacement curves for the samples containing 7-14 vol. %, Al₂O₃.

Figure 81: Load/displacement curves for the samples containing 22 vol. %, Al₂O₃.
An overview of the used data and critical CTOD values is given in table 13, critical CTOD values have been converted to stress intensities for convenience.

**Table 13: Overview of the calculated critical CTOD and critical stress intensities.**

<table>
<thead>
<tr>
<th>Sample Code</th>
<th>Initial Slope [kN/mm]</th>
<th>Critical Displacement [mm]</th>
<th>Critical Load [kN]</th>
<th>δc [mm]</th>
<th>KCTOD [MPa/\text{m}]</th>
</tr>
</thead>
<tbody>
<tr>
<td>AA6061.1</td>
<td>20.01</td>
<td>0.824</td>
<td>15.919</td>
<td>4.79×10^{-2}</td>
<td>42.65</td>
</tr>
<tr>
<td>AA6061.2</td>
<td>26.11</td>
<td>0.845</td>
<td>18.745</td>
<td>7.76×10^{-2}</td>
<td>42.97</td>
</tr>
<tr>
<td>AA6061.3</td>
<td>36.11</td>
<td>0.750</td>
<td>22.595</td>
<td>8.08×10^{-2}</td>
<td>43.83</td>
</tr>
<tr>
<td>AA6061.4</td>
<td>20.52</td>
<td>0.919</td>
<td>15.040</td>
<td>8.00×10^{-2}</td>
<td>43.61</td>
</tr>
<tr>
<td>AA6061.5</td>
<td>38.95</td>
<td>0.779</td>
<td>24.680</td>
<td>9.43×10^{-2}</td>
<td>47.36</td>
</tr>
<tr>
<td>MMC 07.5</td>
<td>33.32</td>
<td>0.764</td>
<td>8.756</td>
<td>1.36×10^{-1}</td>
<td>60.44</td>
</tr>
<tr>
<td>MMC 10.1</td>
<td>32.00</td>
<td>0.575</td>
<td>7.971</td>
<td>8.50×10^{-2}</td>
<td>49.68</td>
</tr>
<tr>
<td>MMC 10.2</td>
<td>38.05</td>
<td>0.537</td>
<td>8.606</td>
<td>8.71×10^{-2}</td>
<td>50.28</td>
</tr>
<tr>
<td>MMC 10.3</td>
<td>29.87</td>
<td>0.545</td>
<td>7.392</td>
<td>7.58×10^{-2}</td>
<td>46.92</td>
</tr>
<tr>
<td>MMC 14.1</td>
<td>40.86</td>
<td>0.300</td>
<td>7.434</td>
<td>3.37×10^{-2}</td>
<td>32.92</td>
</tr>
<tr>
<td>MMC 14.5</td>
<td>33.68</td>
<td>0.314</td>
<td>6.835</td>
<td>3.08×10^{-2}</td>
<td>31.48</td>
</tr>
<tr>
<td>MMC 14.6</td>
<td>32.68</td>
<td>0.318</td>
<td>6.409</td>
<td>3.18×10^{-2}</td>
<td>31.97</td>
</tr>
<tr>
<td>MMC 22.2</td>
<td>34.00</td>
<td>0.163</td>
<td>4.919</td>
<td>7.20×10^{-3}</td>
<td>16.41</td>
</tr>
<tr>
<td>MMC 22.4</td>
<td>37.21</td>
<td>0.168</td>
<td>5.169</td>
<td>1.00×10^{-2}</td>
<td>19.37</td>
</tr>
<tr>
<td>MMC 22.5</td>
<td>31.08</td>
<td>0.169</td>
<td>4.584</td>
<td>7.66×10^{-3}</td>
<td>16.93</td>
</tr>
<tr>
<td>MMC 22.6</td>
<td>32.42</td>
<td>0.168</td>
<td>4.940</td>
<td>6.52×10^{-3}</td>
<td>15.62</td>
</tr>
<tr>
<td>MMC 22.7</td>
<td>42.87</td>
<td>0.137</td>
<td>4.570</td>
<td>9.87×10^{-3}</td>
<td>19.22</td>
</tr>
</tbody>
</table>

The $K_{\text{CTOD}}$ values are graphically represented in figure 82.

**Figure 82: Fracture toughness characterisations calculated by way of the critical CTOD, results qualified "valid according to ASTM E1290" are accentuated with a circle.**
EVALUATION OF TEST RESULTS

To check the validity of the tests, the conditions imposed by the ASTM E1290 [55] standard are checked for validity. The results will be individually discussed for each imposed condition.

**Condition 1:**

*The values of CTOD should be equal to or less than the measurement capacity of the specimen. The measurement capacity of the specimen can be determined from $\delta_m$.*

Since all measured critical CTOD values are of the $\delta_m$ type, no measured value exceeds the capacity of the specimen.

**Condition 2:**

*After measurement of the fatigue crack length at nine equally spaced positions along the crack front, the difference between the maximum and minimum of all 9 fatigue crack length measurements does not exceed 10% of the original fatigue crack length $a_0$.*

The conditions imposed by ASTM E1290 [55] are comparable to the fatigue crack front conditions as mentioned in ASTM E399 [50]. Due to small differences in the phrasing of the conditions, the MMC 14.6 crack front, which is qualified invalid by the ASTM E399 test method, is qualified valid according to ASTM E1290. The other sample (MMC 22.7) qualified valid by the ASTM E399 method is also qualified valid by the ASTM E1290 test method. In summary, two crack fronts are qualified valid, however in the results no structural difference can be seen between fracture toughness values obtained from the valid and invalid fatigue crack fronts.

**Condition 3:**

*No part of the fatigue crack front is closer to the machined notch than the lesser of 0.025W or 1.3 mm.*

For the used DSCT geometry, the smallest distance between the machined notch and the fatigue crack front should be $0.025W = 0.925$ mm. Application of this restriction to the tested samples excluded the samples coded AA6061.4, AA6061.5, MMC 07.5, MMC 10.1, MMC 14.1, MMC 14.5 and MMC 22.4.

**Condition 4:**

*The plane of the fatigue crack does not exceed an angle of 10° from the plane of the notch.*

In all cases, the fatigue crack front did not show a deviation from the plane of the notch.

**Condition 4:**

*The fatigue crack front is not multi-planar or branched.*

Machining difficulties prohibited the production of a double V-shaped notch for the samples contain 22 vol. % and more $\text{Al}_2\text{O}_3$. When the Chevron notch ends in with a large surface, the possibility exists that the fatigue crack front becomes multi-planar or branched. Examination of the fracture surfaces indicated that this was only the case for sample 22.4.

When all the conditions are superimposed, it can be concluded that only the results obtained from samples MMC 10.2, MMC 14.6 and MMC 22.7 can be qualified valid according to this test method. From figure 82, it can be concluded that there is no large difference between the valid and invalid results.

A noticeable difference between the ASTM E1290 results presented in figure 82 and the ASTM E399 presented in figure 73 on page 91 is that the $K_{\text{CTOD}}$ values of the MMC samples with low volume fractions of $\text{Al}_2\text{O}_3$ are higher than the $K_{\text{CTOD}}$ values obtained for the aluminium samples.
which is not observed during fracture toughness testing according to ASTM E399. The expected decrease in fracture toughness is however observed in the ASTM E1290 test results.

4.5 Characterisation of the fracture surface

A SEM analysis of the fracture surface is carried out, where special attention has been paid to the differences between the samples and the differences between fatigue crack growth and stable crack extension in a single sample.

All photographs have been taken with an acceleration voltage of 15 kV and a working distance of 22 mm.

4.5.1 Characterisation of the region of fatigue crack growth

The fatigue crack front is characterised by a stepped structure, caused by the preferred crack growth directions in the aluminium. It seems that the fatigue crack propagates solely through the aluminium matrix, based on the observation of little or no particles on the fracture surface.

In some cases crack growth in a direction perpendicular to the fracture surface can be observed, as shown in figure 83.

![Figure 83: Cracks growing perpendicular to the fracture surface, obtained from MMC 07 at a magnification of 10,000x.](image)

In the following sequence of figures, the region of fatigue crack growth is visualised. The figures show that the disturbances in the crack growth increase with increasing volume fraction of alumina. Another important feature can be seen from the fatigue crack front of sample MMC 30, where particle shapes can be identified, these particles have approximately the same size as the original aluminium particles. It is assumed that the fatigue fracture proceeds through the MMC matrix for the MMC 30 samples. This assumption is backed up by the observation that the fatigue crack growth proceeds extremely slowly for this series (section 4.3).

The small amounts of particles on the fracture surface may be explained by the cleaning procedures (the sample has been cleaned in an ultrasonic bath for 5 minutes) and the testing
procedures (particle fall-out is expected both during precracking and toughness testing). It is also expected that particles are loosely bonded (the local volume fractions approach 90%). So, the absence of particles on the interface does not rule out that the fracture proceeds through the MMC network.

Figure 84: General features of the fatigue crack growth region for all samples. The photographs have been taken at magnification of 1,000x.

The transition from fatigue crack to stable crack growth is very clear for the samples containing lower volume fraction alumina, see figure 85. For the higher volume fractions, the transition cannot be found.
Characterisation of the region of stable crack growth

The surface of the stable crack extension region is characterised by the presence of dimples. From figure 86 it is concluded that the voids initiate by particle fracture for MMC 07. The conclusion is based on two observations: the particles are situated at the centre of the void (as can be seen in figure 86a) and the particles are not debonded from the matrix. Since void initiation is assumed to take place either through debonding or particle fracture, particle fracture is held responsible for the initiation of the voids. For other samples, the identification of the void initiation mode is complicated or even impossible.

The picture sequence on the next page shows that a voided fracture surface structure is maintained for volume fractions under 30 volume percent. The MMC 30 material behaves different.
from the other samples in the sense that fracture is believed to occur through the matrix (similar to intergranular fracture). This fracture surface is characteristic of a very brittle fracture, although brittle fracture mostly shows a featureless fracture surface. The roughness of the fracture surface can be explained by figure 87.

![Figure 87: Illustration of the crack propagation through the MMC network, causing the rough appearance of the fracture surface.](image)

When the fracture proceeds by the dotted line and it is assumed that the particles fall out of the fracture surface, a rough structure as visualised in figure 87b is obtained. In the following picture sequence, the regions of stable crack growth are shown for all samples.

![Picture sequence showing regions of stable crack growth for AA 6061, MMC 07, MMC 10, and MMC 14.](image)
The assumption that the voids initiate at the particles is partially confirmed for the MMC 07 material and contradicted for higher volume fractions. In figure 89 it is visible that the particles have initiated the void for the MMC 07 sample, however the MMC 22 material shows that particles remained in the surface of the large void and did not initiate a void themselves.

It is assumed that the particles visible in figure 89b do not fracture because of their smaller size and that they do not cause enough stress concentrations to initiate a void.

figure 88: General features of the (stable) crack growth region for all samples. The photographs have been taken at magnification of 1,000x.

figure 89: The presence of particles on the fracture surface for the MMC 07 and MMC 22 material.
5 DISCUSSION

Production process

The production process is able to produce a 'good' MMC where the porosity is low and the bonding between the constituents is good. This means that the results obtained from any tests on this material are actually material properties and not a result of porosity or imperfect bonding. Therefore, the discussion of the results will be limited to the samples produced with the "definitive process" and the AA6061 <45 μm powder. These samples are coded MMC 07, MMC 10, MMC 14, MMC 22 and MMC 30.

The obtained structure can be characterised as a composite consisting of large aluminium particles (~45 μm) enclosed in a MMC network. The local Al₂O₃ volume fractions in this material range from zero in the aluminium phase to 90 % in the MMC network.

Precracking

The fabrication of a fatigue precrack satisfying the demands by the various ASTM test methods is very difficult. Development of an experimental set-up dedicated to the fatigue precracking of this material did not result in a high success-ratio for the tested material. The failure of the experimental set-up is two-sided, the crack length measured during testing did not always turn out to be the actual crack length and the shape of the fatigue crack front is unsatisfactory. The main reasons for the low success-ratio of the experimental set-up are summarised here:

Stiffness as a parameter for the compliance method

When the crack length is calculated using the compliance method, the stiffness of the material is used as a parameter. In this research, the stiffness of the tested material is unknown (for the MMC specimens), therefore the stiffness had to be estimated before the fatigue procedure was started. This estimate leads to inaccurate crack length predictions for the first tested specimen of a single series. After the first specimen was tested, the stiffness can be calculated backward from the crack length measured from the fracture surface, from this measurement a second estimate of the crack length can be made, leading to a more accurate measurement of the crack length during precracking.

As can be seen from the results of the precracking procedure, represented in figure 66 the stiffness of the AA6061 samples is higher than the stiffness of most MMC samples. In reality, it is expected that this is not the case, which is confirmed by the stiffnesses calculated during "J-integral characterisation of fracture toughness" (see section 0). The overestimation of the stiffness of the aluminium samples, using the backward calculation based on the precracking results, is probably caused by the extreme surface roughness of the aluminium specimens. This surface roughness may well influence the displacement signal for when small displacements are involved.

In other words, the stiffness used in the live-compliance method should not be the stiffness of the material as represented by its young's modulus, but it should represent every phenomenon influencing the load-displacement behaviour during precracking. This parameter can easily be calculated from the last measured compliance and the optically measured crack length, but this procedure should be repeated for every tested material.
**Accuracy of the equipment used**

When a material like the MMCs tested here is tested, very accurate measurement devices have to be used. The 100 kN servo-hydraulic fatigue machine used was far too heavy for these specific tests, since the applied loads were all lower than 10% of the maximum output of the machine. This low output level implies that the error in the load-signal is relatively large, in order to reduce the measurement error, a pre-amplifier has been used to amplify the load signal. However, this amplifier can only reduce the measurement error and not the output-error introduced by the fatigue machine.

The clip gauge introduces the second and largest inaccuracy during measurement of the compliance. The amplitude of the displacement signal is in the order of magnitude of 25 microns, because of the high stiffness of the tested material and the low applied loads. In order to measure these small displacements during dynamic testing; a very sensitive clip gauge has to be used, of which the vibrations do not influence the displacement signal. This imposes a controversial demand on the clip gauge, since most sensitive clip gauges are very light, thereby disturbing the displacement signal with their own vibrations. Identification of this error is difficult, because the disturbances have the same frequency as the measured displacement and may originate from the clip gauge amplifier used, which is set to operate at maximum sensitivity.

The sensitivity of the measurement method becomes more important for low $\Delta K$-fatigue (or high R-ratio). The error in the calculation of the compliance (based on mean and maximum values of both load and displacement) becomes relatively larger when the differences between minimum and maximum decrease.

**Notch placement**

The position of the notch must be exactly in the centre of the specimen, especially when small crack growth distances are involved (which is the case for the chosen specimen geometry). If the notch is positioned slightly out of the centre of the specimen, the crack growth distances from the notch to the surfaces of the sample differ. The compensation of this difference will become harder if the total crack growth distance is small, because of the large differences in crack growth rates needed to compensate the out-of-centre initiation position.

Although much attention has been paid to the position of the notch, some samples had notch positions out of the centre as a results of errors introduced during spark erosion. During this preparation step, it is assumed that the conducting metal used to generate the sparks does not erode however this proved not to be the case for the spark erosion of the MMC samples.

**Crack front shape**

The used program to reduce the tunnelling of the fatigue crack front does not result in a straight crack front; it only reduces the crack curvature. The effectiveness of this step can be seen on some fracture surfaces where the increase of the R-ratio is visible. An example of such a fracture surface is included in figure 90.
From this figure it can be seen that there is an effect of R-ratio on the fatigue crack front, however the size of this effect is small, since only the shape of the crack front near the surface is different. An increase of the final R-ratio may have a stronger effect, however due to limitations in the sensitivity of the crack length measurement method, is proved impossible to combine a high R-ratio fatigue procedure with an accurate crack length measurement.

In summary the precracking procedure may assist in the growth of a straight crack front, however other tools have to be used in combination to obtain a sufficiently straight crack front.

As a last remark on the subject of precracking, it is important to accentuate the effect of the shape of the crack front on the load/displacement records during the tensile testing. To illustrate this effect, the load/displacement records of three specimens all containing 22 vol. % alumina particles are included in figure 92. The average crack lengths of the specimens are comparable, being respectively 18.46, 18.32 and 17.14 mm for the samples MMC 22.2, MMC 22.6 and MMC 22.7. The shape of the fatigue crack fronts is very different, being straight but heavily tunnelled for MMC 22.2, oblique and tunnelled for MMC 22.6 and straight and slightly tunnelled for MMC 22.7, as can be seen in figure 91:

![Fracture surfaces of the samples MMC 22.2(a), MMC 22.6(b) and MMC 22.7(c), contrast has been enhanced to visualise the fatigue precrack area.](image-url)
It is clear from figure 92 that the curves are similar but different. The main differences between the load/displacement curves are:

- **Position of the peak load**
  The peak load is positioned towards larger crack mouth opening displacements for the samples having invalid crack lengths (MMC 22.2 and MMC 22.6).

- **Value of the peak load**
  The peak loads for the samples MMC 22.2 and MMC 22.6 are higher than the peak load applied during testing of the MMC 22.7 sample.

- **Slope of the linear section**
  The slope of the initial section of the load/displacement curve is higher for the MMC 22.7 sample. Another aspect of the slopes is that the slope of the MMC 22.7 curve remains constant during a larger interval, whereas the other two curves show non-linearities both at the beginning and at the end of the linear section.

- **Slope of the initial part of the load/displacement curve**
  Most load/displacement curves obtained during static tensile testing have a decreasing slope in the initial section of the linear part of load/displacement curve as can be seen in figure 93.
DISCUSSION

Fatigue crack front curvature and/or obliqueness may very well cause this variation in slope, as can be explained using figure 94:

When the flanks of the cracks are loaded elastically, the specimen will behave as if it has a crack length marked as "initial crack length". When the load is increased, the flanks of the crack front will deform plastically and the apparent crack length will be increased towards the final crack length. It must be mentioned however that figure 94 is intended to illustrate...
DISCUSSION

and not to give an accurate picture of the real situation. An increase in crack length in the specimen will decrease the stiffness of the specimen, which is exactly what happens during the first section of the loading curve. A lower slope of the load/displacement curve indicates a lower stiffness.

All observations mentioned above confirm that an imperfect shape of the crack front causes extra barriers for crack extension and thus an overestimate of the critical stress intensity needed for crack extension, the fracture toughness. Observed irregularities in the linear section can also be related to the shape of the crack front.

The growth of a fatigue precrack has shown possible for samples with volume fraction lower than 30 volume percent. The MMC 30 material could not be precracked successfully.

**E399 fracture toughness**

In the ASTM E399 regulations, it is not mentioned that any nonlinearity in the beginning of the load/displacement curve should be neglected. If the procedure described is followed conscientiously, this will lead to improbably low fracture toughness values, because of the shape of the initial portion of the loading curve. As explained in the previous section however, the initial curvature in the load/displacement records can be related to the shape of the fatigue crack front. Therefore, the initial section of the load/displacement curve will be neglected during determination of the initial slope.

Based on the discussion of the precracking procedure in the previous section, it can also be concluded that a valid crack front may lead to the elimination of irregularities in the linear section of the load/displacement curve. Irregularities in the load/displacement curve, especially a decrease in slope, have a very pronounced effect on the fracture toughness as determined by ASTM E399. It can thus be concluded that when a valid crack front is available, the ASTM E399 standard may lead to reliable fracture toughness values, however when small irregularities are introduced by fatigue crack front curvature or obliqueness, the results of the ASTM E399 fracture toughness tests may be called useless. This is very effectively visualised in figure 95, where the load/displacement curves of the samples (MMC 07.5 and MMC 22.7) are compared.

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figure 95: Comparison of the load/displacement curves of samples MMC 07.5 and MMC 22.7, sample MMC 22.7 has been qualified valid according to ASTM E399.

The samples (MMC 07.5 and MMC 22.7) have similar initial crack lengths (17.45 and 17.14 mm) and similar fracture toughness (12.17 and 11.83 MPa\(\text{m}\)) values according to ASTM E399, however the load/displacement curves are entirely different.

Since most fatigue crack fronts must be qualified invalid in this research, the ASTM E399 method is unusable for the determination of the variation of fracture toughness versus volume fraction alumina.

5.4 E1290 fracture toughness

The fatigue crack front conditions imposed by the ASTM E1290 method are similar to the ASTM E399 conditions, therefore most fatigue crack fronts have also been qualified invalid by this method. However, the effects of the shape of the fatigue crack front are not as pronounced as they are for the ASTM E399 method.

When the two samples that have been compared in the previous section are compared again using the fracture toughnesses obtained using method E1290, a difference in fracture toughness is observed (60.44 MPa\(\text{m}\) for MMC 07.5 and 19.22 MPa\(\text{m}\) for MMC 22.7).

Since fracture toughness characterisation depends heavily on the shape of the load/displacement curve and the position of the maximum load, problems can arise when different classes of materials are compared.
In figure 96 two load displacement curves are compared, the curve depicted in figure 96a is a typical load displacement curve for this MMC material. This curve shows a short linear section, followed by a drop in slope, after which the load and displacement steadily rise until the maximum load is reached. For the AA6061 sample depicted in figure 96b, the load versus displacement is different, showing a long linear section, followed by a drop in slope just before the maximum load is reached. After the drop, the slope remains steady for a certain displacement interval, showing a plateau for maximum load.

When (for the MMC material) the first drop in slope is caused by crack extension, the critical crack opening displacement obtained from the maximum load and the corresponding displacement may not be reported as fracture toughness. Fracture toughness is defined as the critical stress needed for the first crack extension, so it is important that the nonlinearity in the curve positioned before the maximum load is identified.

The results of the unloading compliance measurements obtained during characterisation of fracture toughness using method E1737 show that the first unloading visible in figure 96a corresponds to a crack extension of 0.114 mm, which is 0.6% of the initial crack length. Since in the ASTM E399 the critical amount of crack extension is defined as 2% of the original crack length, the conclusion should be that no crack extension takes place before the maximum load is reached. What causes the decrease in slope is hard to define based on the currently available data. However it is reasonable to assume that the non-linearity of the load/displacement curve can be attributed to the growth of the plastic zone and the redistribution of stresses around the reinforcements in the plastic zone.

This hypothesis can be partially confirmed by the comparison of two load/displacement curves, as is done in figure 97:
When it is kept in mind that the maximum applied precracking loads are around 4 kN for both samples, it may be concluded that the effect plays its role when the precracking load is exceeded. In the case of sample MMC 07.5, the exceeding the precracking load does deflect the load/displacement curve. In the other case, this deflection cannot be distinguished because occurs just before the achievement of the maximum load.

Another confirmation of the role of the plastic zone can be found in the case where a power failure lead to a severe overload at the end of the precracking procedure. An example to illustrate this is included in figure 98:
figure 98: Comparison of load/displacement curves of samples MMC 14.1 (a) and MMC 14.4 (b).

During precracking of sample MMC 14.4 an overload occurred after which the precracking procedure was stopped, during this overload crack extension took place, which can be seen on the fracture surface of the sample. Because of this crack extension the initial crack length of MMC 14.4 (22.32 mm) is much higher than the initial crack length for MMC 14.1 (18.60 mm). This difference in crack length complicates the comparison, and lowers the entire load/displacement curve.

However, the MMC 14.4 curve shows that the decrease in slope, as seen in MMC 14.1 and other samples, is absent. Theoretically, this would be the case when the observed non-linearity is caused by the increase in size of the plastic zone, because there is no increase in plastic zone size for MMC 14.4. The presence of a non-linear zone results in a very high value of plastic clip gauge displacement, thereby measuring a high fracture toughness. This fracture toughness value is likely to be overestimated.

The conclusion of the discussion of the results obtained by the ASTM E1290 method must be that the fracture toughness values obtained by this method may be used to compare the fracture toughnesses of materials showing the same load/displacement behaviour. Realistic measurement of the fracture toughness using this method is excluded when a non-standard material is tested, because the observed non-linearity causes a large overestimation of the fracture toughness.

5.5 E1737 fracture toughness

The E1737 fracture toughness method seems the most reliable of the three methods used in this research. This can be concluded based on the discussion in the previous paragraphs of the results obtained using methods E1290 and E399, where it turned out that the results are unreliable because of irregularities in the initial section of the load/displacement record. Because the initial portion of the load/displacement curve is not used for determination of J, this method usually provides better results.

This independence of the initial part of the load/displacement records is not true in this case, because the actual stiffness of the tested MMC material is unknown and has to be determined from the first unloading in the load displacement record, where zero crack extension is assumed. The first unloading is performed after the application of the prestraining load, and the load is reduced
from 3kN to 100 N. Although the stress levels during unloading do not exceed the precracking loads, it is assumed that the first part of the unloading is not influenced by the precracking stress. This assumption is confirmed by figure 99, where it can be seen that the first part of the unloading follows the extrapolation line (grey) of the linear section of the load/displacement curve.

To verify the reliability of this method, the test results have been compared with predicted stiffnesses according to the Tsai-Halpin model. The variation of modulus with volume fraction follows the model, however the spread in the modulus of elasticity is substantial so it can be assumed that this method predicts the right trend but is not very accurate.

While the stiffness of the AA6061 alloy is roughly known, the backward calculation of the modulus is not strictly necessary. However, the same procedure is applied for the aluminium samples. The determination of the modulus from the first unloading is more complex in the case of the aluminium samples. In figure 100, it is visible that a large part of the unloading deviates from the linear behaviour of the rest of the load/displacement curve.

This stronger deviation, when compared with the MMC samples, can be explained by the fracture surface of the fatigue precrack. This fracture surface is for the aluminium samples very rough, whereas the MMC samples have a flat and featureless fatigue fracture surface. Together with the in figure 93 observed curving of the load/displacement record due to crack front tunnelling and obliqueness, it is expected that the roughness and shape of the fracture surface cause this strong deviation.
Due to the strong deviation from the linear behaviour, the first unloading compliance cannot be used for determination of the stiffness. The second unloading is, in most cases situated near the maximum load and at that position, the assumption that no crack growth has taken place is invalid. The compliance needs to be determined from the linear section above the precrack load, where the applied loads are high enough to compensate the effect of the fracture surface and low enough to ensure the absence of crack extension. As can be seen from figure 101, the slope of an unloading in the linear section is different from the local slope of the load/displacement record. Therefore, the slope of the load/displacement cannot be used as a replacement for the unloading compliance.

Since the effective modulus cannot be determined from the first unloading, the crack length as measured from the fracture surface has to be used in combination with the known stiffness of the
AA6061 alloy. This may be done when the crack length as determined from the unloading compliance corresponds with the crack length as measured from the fracture surface. When the crack length that is determined from the second unloading is comparable or slightly higher, it may be assumed that the unloading compliance crack length measurement method corresponds with the actual crack length. This is the case for all samples, so the optically measured initial crack length is used for the calculation of \( J \).

The use of a different method for the \( J \)-characterization of the fracture toughness implies that a comparison between the values obtained for MMC and AA6061 may not be compared. This comparison was already doubtful since the production method for both material types is very different. Despite the inaccurate measurement method, the fracture toughness values obtained for AA6061 correspond with the literature values, though they are relatively high. Relatively high fracture toughness values are found for all materials tested here.

5.6 Characterisation of the fracture surface

From the characterisation of the fracture surface, it can be concluded that the fracture is heavily influenced by the volume fraction of alumina and by the fracture behaviour of the matrix alloy. The fracture behaviour of the MMC materials with low volume fractions resembles the fracture behaviour of aluminium alloys, with increasing volume fraction, the resemblance is reduced.

The fatigue crack is assumed to propagate through the aluminium matrix for low volume fractions, since only occasionally particles are observed on the fatigue fracture surface. For the higher volume fractions, this is unsure because of the microscopic roughness of the fracture surface. It is assumed that this roughness is caused by the fall-out of the reinforcing particles during the testing and preparation steps. In other words, for the higher volume fractions (material MMC 30), it is expected that the fatigue fracture propagates through the MMC network.

A region of stable crack growth is identified for the MMC 07, MMC 10 and MMC 22 samples, and it is assumed that stable crack propagation also occurs in the MMC 14 materials. The shape of the load/displacement curve, showing a gradual decrease in load after the peak load is passed confirms this assumption. The fracture surface contains voids, also indicating a ductile fracture mechanism. For the MMC 30 material, no stable crack growth has been identified and no voids are present based on the fracture surface.

The initiator of the fracture is thought to be particle fracture for the MMC material containing low alumina levels. Although voids are observed for all MMC samples (except for the MMC 30 samples), in most cases the initiation mechanism cannot be identified. In samples with high volume fractions, small particles were observed in the surface of the voids. These particles did not initiate a void, which can be caused by the fact that the stresses around the particles are too small to cause initiation of void, either by particle debonding or by particle fracture. The difference in observed particle sizes on the fracture surfaces of MMC 07 and MMC 22 may be explained by the assumption that larger particles are more susceptible to particle fall-out, and the weaker particle bond in the MMC 22 material. As can be seen from the analysis of the microstructure, the alumina particle sizes are similar for all materials produced.

5.7 Discussion of the obtained fracture toughness values

After discussion of the fracture toughness measurement methods, the obtained results of the fracture toughness tests will be discussed. The results of all three methods are included in table 13.
In section 2.7, three models were suggested to predict the fracture toughness of MMC as a function of particle size and volume fraction. Two models (the fractography-based and energy-based models) are based on measurement of the void size on the fracture surface. The third model (the crack path model) model is based in only on mechanical properties of the MMC.

In order to check the applicability of the models, reasonable mechanical properties have been substituted in the model equation. The yield strength has been chosen between 295 and 400 MPa varying linearly with the volume fraction alumina, the stiffness has also been chosen to vary linearly between 70 GPa and 100 GPa. In the crack path model suggested by Hahn and Rosenfield dependence is shown, as is exhibited by the results obtained using the E399 method, see figure 102. The model predicts the fracture toughness to drop at low volume fractions, after which the fracture toughness remains more or less constant.
The absolute values predicted by the model are inaccurate, so is the difference in fracture toughness between the AA6061 and the MMC, but if the test results obtained by ASTM E399 would be less doubtful, the predicted trend could be qualified as being able to predict a correct trend. The results obtained by ASTM E1290 and ASTM E1737 both show a gradual increase in fracture toughness, this trend is not predicted by this crack path model.

Verification of the two other models (the fractography and energy-based models) is harder, because the height of the dimples is not determined in this research.
In order to determine which predicted trend is the most accurate trend, all fracture toughness values have been visualised in

![Graph showing fracture toughness vs volume fraction](image)

**Figure 103: Overview of all measured fracture toughness data.**

Three different trends can be visualised:

- The first trend measured using E399 shows a drop in fracture toughness for low volume fractions, followed by a plateau, where the fracture toughness is only slightly influenced by the volume fraction of alumina.

- The second trend (by E1737) shows a gradual decrease in fracture toughness.

- The third trend shows a decrease in fracture toughness for increasing volume fractions. However, according to this trend the fracture toughness increases when an aluminium alloy is reinforced with low volume fractions of alumina. Again, this is caused by the non-linearity of the load/displacement records for MMC material having low volume fractions.

The fracture toughness values as measured for the MMC 22 material seem to correspond for all materials. As can be seen in figure 104, the spread in results is substantial for methods E1290 and E1737 also a structural difference between the three methods can be observed.
It can be assumed that the trend predicted by ASTM E1737 method is the most accurate representation of the variation of fracture toughness with volume fraction of alumina. This assumption is based on the following observations:

Because of the structural non-linearity in the initial portion of load/displacement records of the MMC samples with low volume fractions, the fracture toughness is severely underestimated using the E399 method. It is on the other hand expected that the E399 fracture toughnesses of the material containing higher volume fractions of alumina and exhibiting less non-linearity in the load/displacement record are more reliable.

The same non-linearity that influences the E399 method, also influences the E1290 method, albeit in another way. Because of the non-linearity the plastic component of the clip gauge displacement used for calculation of the E1290 fracture toughness, is structurally overestimated, resulting in a structural increase in the fracture toughness values measured by this method.

Since the E399-method underestimates the fracture toughness and the E1290-method overestimates the fracture toughness of the MMC material, it is reasonable to believe that the actual trend is in between both trends.

Based on the discussion of fracture toughness variation with volume fraction of alumina, it can be concluded that the fracture toughness of an aluminium - MMC composite, where the aluminium is embedded in an MMC network, decreases linearly with an increase in volume fraction.

\[
\frac{\rho_{\text{P}}}{\rho_{\text{eff}}} \approx \frac{\rho_{\text{P}}}{\rho_{\text{eff}}} \quad \text{and} \quad P \cdot V \quad \text{curve}
\]
6 CONCLUSIONS

6.1 Production process
The production process is able to produce a 'good' MMC with high density and good bonding between the constituents. The only condition that was not satisfied is the conformity with a commercial MMC. This must be kept in mind during comparison of the test results with fracture toughness values reported in literature.

6.2 Sample preparation
The preparation step has to be perfected, especially the fabrication of the chevron notch and the precracking procedure. The used equipment is satisfying, however no clarity exists as to which fatigue loading procedure has to be used in order to obtain a straight and flat crack front.

The crack fronts obtained using the stepwise R-ratio technique as proposed in section 3 are mostly invalid according to the methods described in the ASTM handbook for testing and evaluation of materials.

Precracking of MMCs, especially when higher volume fractions of reinforcement are present, is complex. Fatigue crack growth rates are low and the stress concentrations needed to grow a fatigue crack are relatively close to the critical stress intensity (fracture toughness). In general, it can be concluded that sophisticated equipment is needed for the precracking procedure to succeed.

6.3 Fracture toughness
The ASTM E399 method cannot be used for the characterisation of the fracture toughness before the specimen preparation is perfected and straight crack fronts are produced. Low levels of obliqueness and curvature have a drastic effect on the test results obtained.

The ASTM E1290 method is by far the easiest method to characterise fracture toughness. The main drawback of this method is the 'subjective' determination of the crack extension event, causing the method to be suitable for comparison of fracture toughnesses between series of samples when the overall behaviour is comparable. Since non-standard (MMC) load/displacement records are not included in the description of the method, no standardised fracture toughness can be measured.

ASTM E1737 is the most useable method applied in this research since precracking effects can be eliminated from the test results. It is on the other hand the most labour intensive and complex method and since this research was not focussed on the J integral characterisation, the accuracy of the J-integral characterisation as described here is too low. A specialised study should provide better results.

Based on the results obtained it can be undoubtedly concluded that the fracture toughness decreases with increasing volume fraction in MMCs. This is confirmed by two of three test methods. Whether the fracture toughness of an aluminium alloy decreases when it is reinforced with ceramic particles cannot directly be concluded from this research. Since the measured fracture toughnesses are high compared to the literature values, it is expected that a structural overestimation is included using this test method.
7 RESEARCH RECOMMENDATIONS

In order to increase the success rate during determination of the fracture toughness of MMCs, the following modifications of the experimental procedure are suggested:

7.1 Specimen geometry

The sample geometry has to be modified in three ways; first, the specimen diameter has to be increased, second the fixations for the clip gauge must be positioned at the load line and finally it is recommendable to make use of side-grooves. An increase in specimen diameter has two large advantages, eliminating the biggest problems in the sample preparation step:

**Increase of the maximum allowable crack growth**

An increase in the maximum allowable crack growth from the notch to the maximum crack length as dictated by the ASTM methods will likely result in less curvature of the fatigue crack fronts. This can be illustrated by figure 105:

![Figure 105: Illustration of the suggested increase in maximum allowable crack growth.](a)

When crack growth is started in the centre of the specimen, the crack growth will tend to be the same in every direction. This leads, for the present case, to the crack front visualised in figure 105a, where the distance from any point on the crack front to the initiation point is constant. If the crack growth lengths are increased (figure 105b) the same crack front construction will show less curvature. Second, it is expected that any obliqueness will level out during crack growth.

**Increase of the live-compliance method accuracy during precracking**

When the sample diameter is increased, the displacements recorded during precracking will also be increased. Measurement of these, currently very small displacements, introduces the largest inaccuracy during precracking.

![Figure 105: Illustration of the suggested increase in maximum allowable crack growth.](b)
The second modification of the sample geometry beholds the movement of the clip gauge fixation points from the crack mouth to the load line. This modification eliminates the conversion from crack mouth displacement to load-line displacement during determination of the J integral.

The use of side-grooves will increase the fatigue crack growth rates at the surfaces of the sample, since they act as stress-raisers. This increased growth rate results in a flatter crack front.

7.2 Precracking procedure

The most important modification of the precracking procedure is the decrease of the testing frequency. A decrease in frequency will increase the preparation times, but heavily influence the accuracy of the crack length measurement, since vibrations of the clip gauge are diminished.

7.3 Fracture toughness testing

The fracture toughness test method has to be modified to allow for accurate determination of the fracture toughness using all three methods. Especially the J integral characterisation test has to contain more unloadings and these unloadings have to be positioned in the elastic region of the load/displacement record. This enables an accurate determination of the initial crack length and stiffness. Another advantage of the use of many unloadings is the large increase in accuracy.
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