Deformation and fracture of continuous alumina fibre reinforced aluminium composites
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Preface

This thesis was carried out by Benedikt Moser within the frame of a joint effort (interinstitutional collaboration) of EPFL and EMPA. The motivation of EMPA-Thun for its engagement in the present thesis was twofold:

i) Assess the viability of the squeeze casting process technique (currently used in Thun) for the fabrication of high quality continuous fibre reinforced aluminium composites (CFRA) and comparison of the potential of this technique with other techniques such as gas pressure infiltration and ultra-sound assisted continuous infiltration as used at EPFL and 3M, respectively.

ii) Expand the understanding of the influence of matrix composition on the mechanical behaviour and fracture mechanisms in CFRA in the view of developing more performant materials.

The materials investigated in the present thesis have been partially manufactured at EMPA-Thun (squeeze casting) and EPFL (gas pressure infiltration) and provided by the company 3M, USA.

As far as the ultimate tensile strength of CFRA is concerned, no obvious influence of the process technique used is observed, provided the process is well understood and that optimal process parameters are used. The effect of the matrix composition is somewhat masked by the scattering of the results, however, a tendency for better performance with specifically designed high strength binary and ternary matrix alloys is recognized in the experimental results and confirmed by the analytical models used for tensile strength prediction.

Different models and theories proposed in literature have first been critically reviewed with respect to their ability and validity to explain or confirm the experimental results, then refined and/or combined to give suiting analytical descriptions of the phenomena observed. For instance, as a fundamentally new approach, a non-linear elastic behaviour of the Nextel 610 alumina fibre was proposed and implemented to adequately describe the stress strain behaviour of the composites investigated. Moreover, the relevance and applicability of the Weibull statistics and their consideration to describe damage initiation and evolution as well as tensile behaviour for CFRA in combination with existing models is remarkably evidenced. Particular skills for designing and realising experimental set-ups and specifically planned experiments as well as the ability of understanding, questioning and combining complex scientific matters, refining and validating them by the introduction of new aspects demonstrates the scientific capabilities of Benedikt.

In what concerns practical consequences of the results for applied development and fabrication of CFRA, a unique and new method has been developed that allows to quantify fibre damage introduced during composite processing or during service life. It is proposed that this technique could be used for quality control during industrial production and for service life prediction for specified load histories; such a method had not been available before.
In the present context I appreciated this joint EMPA-EPFL thesis as an excellent vehicle to consolidate the collaboration basis between both institutions, with a strong potential for releasing new synergies in Switzerland's scientific community.

Eventually, I would like to acknowledge to the board of directory of EMPA and EPFL for the funding of the thesis and I would also like to thank Prof. F. Eggimann, Mr. W. Muster and Dr L. Rohr for their engagement and support that finally allowed the launching of this joint thesis.

During the whole project I also appreciated the support provided by the whole team of EMPA-Thun, particularly of the metallography team and the personal engagement of Urs Müller and Hans-Rudolf Sieber related to the fabrication of the composite specimens.

I would like to acknowledge Prof. A. Mortensen from EPFL-DMX not only for accepting to participate in this project and for directing Benedikt's thesis but also for the cordial friendship he offered to me during that time. The personal and scientific contact will without doubt persist beyond the scope of this particular project.

Last but not least I appreciated the four years spent with Benedikt, the numerous interesting and stimulating discussions we had together and also the friendship that developed during the project. Benedikt's work enlarged our understanding with respect to the tensile behaviour and fracture mechanisms of continuous fibre reinforced aluminium composites; his work for sure will valorize our current and future R&D-activities in the field of MMCs.

Thun, May 2002

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Abstract

Unidirectionally continuous alumina fibre reinforced aluminium matrix composites are produced using two different liquid metal infiltration processes, namely gas pressure infiltration and direct squeeze casting. A fibre winding technique is used to produce net-shape fibre preforms for parallel tensile bars. A fine alumina particle suspension is added to some of the preforms during the winding process to achieve a homogeneous fibre distribution at lower fibre volume fractions. Pure aluminium and simple binary and ternary aluminium alloys are used as matrix material. The fibre volume fraction is determined from the number of fibre tows per preform, counted during the winding process. In addition to these pressure-cast parallel tensile bars produced at the Swiss Federal Laboratories for Materials Testing and Research (EMPA) and at the Swiss Federal Institute of Technology Lausanne (EPFL), commercially available continuous fibre reinforced aluminium composite wires with two different matrices (produced by 3M, St Paul, MN, USA) are tested.

The composites are investigated by transmission optical microscopy (TOM) and mechanically tested in tension parallel to the reinforcing fibres. These tests are conducted between room temperature and 775 °C.

The in-situ metal matrix flow curves are back-calculated from composite stress-strain curves using an improved procedure. This procedure considers and quantifies the strain-dependence of the fibre’s Young’s modulus as well as the deviation from the rule of mixtures due to lateral contraction mismatch between the matrix and the reinforcing phase. The latter effect was examined by finite element analysis of complex unit cells. Resulting in-situ matrix flow curves show significant dislocational matrix hardening, in agreement with previous investigations. Present curves provide more precise measures of the in-situ matrix rate of work hardening, to show that matrix work hardening can in some instances be far higher than in the unreinforced matrix, and is essentially kinematic in nature.

Initial damage in the form of dead ending fibres was detected in the continuous wire composite by TOM. A size effect in the wire fracture strength with molten matrix is found and explained as being a result of fibre damage within the wire. Good agreement is found between the two measurements. Damage evolution as a function of applied stress in the composite wire is monitored using the developed technique of tensile testing above the melting temperature of the matrix. Damage is found to accumulate in composites with single phase matrices of pure aluminium and homogenized Al-2%Cu. Such damage is mainly by uncorrelated fibre fragmentation in accordance with the virgin fibre strength statistics up to composite strengths of 1300 MPa. In a two-phase as-cast Al-2%Cu matrix, the brittle Al2Cu intermetallic is shown to cause a significant increase in the rate of damage accumulation in the composite.

The strength of the continuous composite wire is compared with the prediction of a model based on the stability of a cluster of fibre breaks. Good agreement is found when a simple load-sharing rule is assumed. The strength of the composite decreases with increasing temperature; this is attributed to the decrease in matrix shear yield stress which causes an increase of the volume affected by stress concentration from a broken fibre; this effect is also well explained by the model. It is concluded that the matrix shear yield stress exerts only a negligible
influence on the stress concentrations around a broken fibre over a relatively large range of matrix properties.

The present study provides a quantification of the influence of matrix alloying on the flow stress, and the fracture, of fibre reinforced composites. It is shown that, provided certain conditions are met, a strong matrix will result in improved composite properties.
Zusammenfassung


Die Festigkeit des Verbunddrahtes wird mit einer Vorhersage basierend auf der Stabilität von Gruppen von Faserbrüchen verglichen. Unter der Annahme einer einfachen Lastverteilungs­regel stimmt die Vorhersage gut mit den experimentellen Daten überein. Mit zunehmender Temperatur nimmt die Festigkeit des Drahtes ab. Dies wird mit der Abnahme der Scherfestig­
keit der Aluminiummatrix begründet, die dazu führt, dass die Überlastlänge einer Faser in der Nähe eines Faserbruches zunimmt. Dieser Effekt kann mit dem vorgeschlagenen Modell ebenfalls erklärt werden. Die Daten legen den Schluss nahe, dass die Scherfestigkeit der Matrix über einen weiten Bereich nur einen geringen Einfluss auf die Spannungskonzentration um einen Faserbruch hat.

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A - INTRODUCTION

The production of high specific strength and stiffness materials was, is, and will remain, at the heart of modern technological progress. Unidirectional continuous alumina fibre reinforced aluminium is one answer to this demand for high performance materials. With its stiffness and strength comparable to high strength steel but a density of only about a third that of steel, this material is an attractive candidate for light-weight and structural applications. Compared to polymer matrix composites, these aluminium matrix composites have some advantages, including in particular the fact that the metallic matrix can offer reasonably high strength in directions other than along the fibre orientation.

These materials are not commonly used, largely because of the high price of the fibres needed for compatibility with aluminium. Their main engineering performance parameter, namely their strength and stiffness along the fibre direction, has been the subject of several investigations and much developmental work; however, there is at present no clear rule for optimizing the strength of these materials, particularly as concerns their matrix.

It has been found in several studies that the use of standard high strength matrices is deleterious for the longitudinal properties. Often the fibres are embrittled by alloying elements or brittle matrix second phases, factors which result in lowered composite mechanical performance. A general approach to optimize longitudinal strength of these composites has been to introduce a weak fibre-matrix interface; however, this often requires costly fibre coating and the gain in longitudinal strength is always accompanied by poor transverse performance, thus degrading one of the most attractive features of these composites. It is also known that, even with a strong interface, high strength in the fibre direction can be achieved when a pure aluminium matrix is used. This encourages research towards the development of specifically designed high strength matrices, which avoid the deleterious effects of commercial high strength alloys mentioned above. The use of a high-strength matrix offers the possibility of producing components with high off-axis strength and also high overall strength in unreinforced parts of locally reinforced components.

It is still not well known to what extent and by which mechanisms the strength of the matrix influences the damage evolution and fracture stress of these composites parallel to the reinforcing fibre direction. In addition, the intrinsic behaviour of the matrix material within the composite is important: it can differ significantly from that which it exhibits in the unreinforced condition. Understanding these effects is a prerequisite to engineering the matrix of these composites for improved performance.

The present investigation aims at answering some basic questions on the micromechanics of unidirectionally continuous alumina fibre reinforced aluminium matrix composites. These are centered on the behaviour and influence of their matrix, and can be summarized as follows: (i) how much damage develops before the composite fails? (ii) what is the event that triggers the final macroscopic crack that induces catastrophic failure of the composite? (iii) how does the...
reinforcement influence the in-situ matrix properties? What is the influence of the matrix properties on the composite performance?

To tackle these questions some simple composite systems – mainly pure aluminium reinforced with a high strength alumina fibre – were chosen as model materials. Despite their simplicity some of the systems under investigation are used in commercial products. The in-situ metal matrix properties are determined by an improved back-calculation procedure. Damage development is monitored by measurement of the in-situ fibre bundle strength. Finally an existing model predicting the strength of continuous fibre reinforced composites based on damage development is improved and used to explain the experimental data.

The present thesis comprises a literature review that summarizes the state of the art in the fields relevant to the presented research. Current knowledge about deformation and fracture of continuous fibre reinforced aluminium matrix composites is reviewed, and a special section covers the influence of the matrix on the composite strength. The materials and the experimental procedures are then presented in Chapter C. Chapter D presents the results from the microstructural investigations and from the mechanical testing. Chapters E and F provide a discussion of results, and are respectively focused on the in-situ matrix flow stress and the tensile strength of the composite. Chapter E presents a refined method for calculating the in-situ matrix deformation behaviour and presents in-situ matrix deformation curves for different composite systems. In the second part of the discussion, in Chapter F, a method for measuring the damage evolution is presented and the fracture behaviour and strength of the composites is discussed. The conclusion summarizes what are viewed as the main contributions of this work.
1. **Continuous Fibre Reinforced Aluminium Matrix Composites**

1.1 **General Properties**

Light alloys suffer in structural application from low stiffness, limited fatigue life and in general from low performance at elevated temperatures including poor creep resistance. By the combination of these bulk materials with high strength and high stiffness reinforcements these properties can be greatly improved without losing the advantage of the lower density of the light alloys compared to steel. The use of continuous unidirectional fibre reinforcement yields components with high specific stiffness and strength (strength or stiffness divided by density) parallel to the fibres. Reinforcement with ceramic fibres increases in particular the high temperature strength and creep resistance greatly. The material exhibit highly anisotropic mechanical properties; however, compared to unidirectionally reinforced polymer matrix composites, the transverse performance is attractive, being usually near that of the unreinforced metallic matrix.

In comparison with polymer matrix composites, metal matrix composites exhibit several important advantages depending of the application, namely: no degradation by UV radiation, higher thermal and electrical conductivity (particularly perpendicular to the fibre direction), no outgassing at elevated temperatures, and no distortion with changing environment humidity. Some disadvantages are present as well: higher production cost, difficulty in producing large parts with complicated shapes, difficult machinability, and a lower specific strength and stiffness.

1.2 **Applications**

Few industrial applications of continuous alumina fibre reinforced aluminium are known to date. The main drawbacks of this material class are the cost of the fibres and of the fabrication process [1]. An additional problem is the availability of easy-to-use design tools that can deal with this highly anisotropic material. There is also still an important lack of understanding of deformation, damage and fracture of these materials.

One potentially high volume application is worth mentioning. The 3M company recently announced the use of alumina fibre reinforced aluminium wires for overhead transmission lines [2]. The composite wire replaces the stainless steel wires in the core of overhead transmission lines. It increases electrical conductivity, and reduces the linear density while maintaining strength. Although the cost per metre for the composite core lines is estimated to be higher than that for steel core lines, the use of the new material can result in overall cost savings as the power transmission capacity of an existing corridor can be increased, due to its higher electrical conductivity, without charging the towers that hold the line. Also, the number of towers can be reduced in new power transmission corridors due to the higher specific stiffness of the composite core lines [1].

3M has a few other, smaller, applications of continuous alumina fibre reinforced aluminium composites such as pushrods for racing cars [3] and a selectively reinforced brake caliper [4].
2. Processing of Continuous Fibre Reinforced Metals

Cost-effective and reliable production is an important factor for materials to be accepted in industry and successful on the market. There are still some burning issues in processing of continuous fibre reinforced aluminium matrix composites, including net-shape production (to avoid the difficult machining) and cost-effective preform production.

2.1 Preform Preparation

In most of the processing routes for continuous fibre reinforced aluminium matrix composites, a fibrous preform must be prepared prior to infiltration or consolidation. Good fibre alignment is critical to avoid processing-induced damage (misaligned fibres can break their neighbours during consolidation). Particularly for high compressive strength – to avoid premature failure due to fibre buckling [5] – good fibre alignment is also crucial. The most widely used process for the preparation of unidirectional fibre preforms is fibre winding. To eliminate unnecessary fibre handling with possible damage, the fibres should be wound directly in the mould for later infiltration or consolidation. Organic or inorganic binders can be used to produce a preform with a certain strength so it can be transferred into another die [6]. This is particularly useful for the production of selectively reinforced parts. Prestraining the fibres during preform preparation before matrix solidification has been proposed as a way to improve the fibre alignment [7]. Additionally, prestrained fibres would influence residual stresses in the composite and as a consequence increase the elastic range of unidirectional composites in tension [8] (but could decreases the tensile strength). To the author's knowledge, these techniques have not been used to date in practice.

2.2 Hybridization

Clustering of fibres during infiltration is known to be a major problem in the production of moderate to low fibre volume fraction composites because the interfibre distance has an important influence on the longitudinal tensile strength via stress concentrations in fibres adjacent to a fibre break [9,10]. An already old approach to solve the problem is hybridisation. Towata and Yamada [11,12] found that the addition of particles to the fibre bundle greatly improves longitudinal and transverse composite strength whereas the addition of whiskers is much less effective. Schaff et al. [13] impregnated carbon fibres with variable amounts of ceramic particles to produce composites with graded fibre volume fraction (25-60 vol.%) for selectively reinforced structures. They found a greatly improved fatigue resistance. Ochiai et al. [14] emphasized that hybridization improves room- and high-temperature strength through strengthening of the matrix leading to a more effective load transfer to the reinforcement. An increase in transverse strength was also reported by Dumant et al. [15].

2.3 Solid State Consolidation

Hot isostatic pressing is mainly used for the production of SiC monofilament reinforced titanium. A recent review on this topic can be found in reference [16]. Several procedures are used to combine fibre and matrix before consolidation: fibre/foil layup, fibre/matrix-wire combination, matrix-coated fibres, powder/fibre combination and plasma sprayed matrix. The
method was also successfully used for the production of aluminium matrix composites reinforced with monofilaments [8]; however, it did not gain wide application with fine ceramic fibres.

2.4 Liquid Metal Infiltration
Liquid metal infiltration is definitely the most widely applied means to combine bundled ceramic fibres with aluminium. In this method, the liquid matrix is made to penetrate a fibrous preform and solidifies to form the composite. Since alumina fibres are essentially stable in molten aluminium alloys (with the exception of certain alloying elements, such as Mg or Li), the liquid metal route is the most gentle way of combining fibres and matrix. Solidification determines the microstructure of the matrix (see Section B - 2.5) and has as a consequence an important influence on subsequent composite performance (see Section B - 4.2). The governing phenomena of infiltration processing of fibrous preforms in general are (i) capillary phenomena, (ii) transport phenomena and (iii) the mechanics of potential fibre preform deformation. These were summarized in a recent article by Michaud and Mortensen [17].

Infiltration of an alumina fibre preform by liquid aluminium is not spontaneous because of the non-wetting behaviour of this system. Chemically induced wetting can be promoted by fibre coating or alloying elements in the matrix to allow pressureless infiltration processes to be used. However the resulting composites usually suffer from poor mechanical properties due to reaction products at the fibre matrix interface.

In most of the processes the liquid metal is forced to penetrate the preform by the application of pressure in the range of 0.1 to several hundreds of MPa. These methods differ in the amount of pressure and the way how it is applied.

The different liquid metal infiltration processes (with and without pressure) are reviewed by Mortensen [18]. Some of these processes are briefly presented in the following.

Ultrasonic Infiltration
High frequency pressure waves improve the wettability in the system alumina/aluminium greatly [19]. It has been proposed that this is due to the hysteresis between the advancing and receding contact angle combined with the high frequency, causing the infiltration threshold pressure to drop to zero. The process has been used by the Nippon Carbon and 3M company for continuous infiltration of an alumina fibre reinforced aluminium wire [20,21].

Gas Pressure Infiltration
Gas pressure is another way to force the liquid aluminium to penetrate the ceramic fibre preform. Usually pressure in the range of 1 to 10 MPa is applied to force the melt into the preferably pre-evacuated preform. The versatility of this process is outlined by San Marchi et al. [22]. A number of different methods of gas pressure infiltration exist. In some, the liquid metal and the preform have the same [22] or different [23] temperatures, while other methods are especially designed for near net shape production of MMC parts [24,25]. Experimental measurements and theoretical work on the flow behaviour of aluminium in a short fibre preform under low pressures can be found in [26-28].

Direct Squeeze Casting
Squeeze casting is a process where solidification is promoted under high pressure within a reusable die. Used for the production of various high quality metallic parts it is nowadays one
of the most popular fabrication routes for metal matrix composites. For a review of the squeeze casting process in a general sense, the reader is referred to Ghomashchi [29] or Yue and Chadwick [30]. The production of metal matrix composites by squeeze casting generally consists of pouring the molten metal in a preheated die cavity that holds the preform. Subsequently high pressure (up to 100 MPa) is applied to force the liquid metal to penetrate the preform. Pressure is maintained until the composite is fully solidified. The process is generally controlled by the ram displacement speed and the initial melt, preform and die temperatures. Extensive literature, mainly on the infiltration of short fibre preforms, is available [31,32]. Experimental studies and theoretical considerations about the melt flow behaviour during the infiltration [17,18,26,28] can give some guidelines for process optimization.

There are finally a few other infiltration processes including reactive infiltration [18,33], infiltration by centrifugal force [34], Lorentz force driven infiltration [35] or a modified investment casting technique [25] that have been demonstrated for aluminium matrix composites.

### 2.5 Microstructure of Melt Processed Composites

Usually the metallic matrix solidifies in a relatively unperturbed manner for processing conditions under which the resulting microstructure of the matrix is significantly finer than the interfibre spaces in the composite [36]. In most cases the reinforcing phase modifies solidification and therefore the microstructure of the matrix alloy. In binary aluminium-copper alloys it was found that the solidification front between fibres is curved and as the primary phase avoids the fibres, second phases being therefore preferentially found at the fibre-matrix interface [37].

A comprehensive review on the fundamental principles of solidification phenomena in MMC can be found in [38]. The influence of the matrix properties on the composite performance is reviewed in Section B - 4.2.

### 2.6 Processing Induced Damage

The producers of continuous fibre reinforced composites are always concerned about processing induced damage like fibre fracture. Warren et al. [39] investigated the problem of fibre fragmentation during solid state consolidation of metal coated fibres and found that the origin of most of the damage is the misalignment of fibres. Gorey et al. [40] found that fibre fragmentation during hot pressing can occur even in the absence of bending through matrix flow in the fibre axial direction.

In liquid metal infiltration processing fibre fragmentation can occur when the infiltration speed is too high or the preform temperature is too low [32,41]. Damage can also be introduced later in the processing route. Ertürk et al. [42] investigated damage in long fibre metal matrix composite during forging and elaborated forming limit criteria.

Metal matrix composites are known to be difficult to machine. Not only tool wear but also surface quality and sub-surface damage can be encountered. Most of the studies concerning machining of MMCs are focused on particle reinforced metals [43,44] where electrical discharge machining turned out to be a very useful tool [45].

Curtin and Zhou [46] summarized the influence of processing damage on the performance of fibre reinforced composites.
3. Deformation and Fracture

3.1 Elastic Deformation

It is experimentally and intuitively evident that the elastic composite properties parallel to the fibre direction are dominated by the fibre properties [47-49]. Figure B.1 shows some data from the literature for the fibre parallel Young’s modulus. The dependence on the fibre volume fraction is clearly shown.

The stress-strain curve of fibre reinforced metals is generally referred to as bilinear [50] with fibre and matrix both deforming in a purely elastic manner in the initial part, while the matrix plastifies in the second. Some authors did not observe any initial linear elastic deformation in their experiments [51]. Residual stresses, which depend on processing and thermal treatment, are the likely reason for these differences in behaviour. The initial elastic deformation is usually described by the composite elastic modulus $E_c$ according to a simple rule of mixtures (ROM) that can be found in basic mechanics textbooks [52]

$$E_c = V_f E_f + V_m E_m$$  \hspace{1cm} (B.1)

where $V$ is the phase volume fraction and $E$ the phase Young’s modulus with the subscripts $f$ and $m$ denoting fibre and matrix respectively. For engineering purposes, the ROM gives a reasonably precise value for the composite Young’s modulus.

Deviations from the ROM are caused by the lateral contraction of the two phases, which usually differ by a non negligible amount. Therefore additional stresses build up in the two
phases, and the simple ROM is no more an exact solution of the problem. There are a number of different approaches to deal with this problem.

**Direct Approach**
This approach deals with a simplified geometry like a composite cylinder assemblage (CCA) as proposed by Hashin [53]. The result is an approximation for the longitudinal composite modulus:

\[
E_{\text{CCA}} = E_f V_f + E_m V_m + \frac{4 \cdot V_f V_m \cdot (v_f - v_m)^2}{V_m + \frac{V_f}{k_f} + \frac{1}{k_m} G_m}
\] (B.2)

where \( v \) is the phases Poisson’s ratio, \( k \) the phases bulk modulus and \( G \) the shear modulus with \( f \) and \( m \) denoting fibre and matrix respectively.

**Self-Consistent-Field Method**
This method is based on the work of Eshelby [54,55] and usually considers an inclusion embedded in a medium that has the mean composite properties. The original work is based on an ellipsoidal inclusion. Hill [56] included a single continuous fibre in an unbounded homogeneous composite medium. Self consistent approaches usually yield reliable results for low fibre volume fractions; however, they become imprecise for higher volume fractions and high stiffness ratios between fibre and matrix.

**Variational Calculation**
This method does not aim at a direct prediction of the properties under consideration but on bounding them. Hashin and Rosen [57] used the principle of minimum potential energy and minimum complementary energy to predict bounds for the elastic moduli of fibre reinforced materials. They determined bounds for a hexagonal fibre array and an approximative direct prediction for a random fibre array. Hill [58] investigated the problem based on a theoretical mechanics approach. A single cylindrical fibre with a cylindrical matrix shell yields the lower bound. By inverting the materials properties ("matrix" cylinder surrounded by "fibre" shell) Hill determined the upper bound. These bounds are valid for all continuous fibre reinforced composites regardless of the transverse geometry, and are the closest possible as long as one does not further specify the geometry of spatial fibre distribution in the composite.

\[
\frac{4 \cdot V_f V_m \cdot (v_f - v_m)^2}{V_m + \frac{V_f}{k_f} + \frac{1}{k_m} G_m} \leq E_c - E_f V_f - E_m V_m \leq \frac{4 \cdot V_f V_m \cdot (v_f - v_m)^2}{V_m + \frac{V_f}{k_f} + \frac{1}{k_m} G_m}
\] (B.3)

It is noted that Hill’s lower bound is identical with the direct approach by Hashin for a composite cylinder assemblage, Equation (B.2), as can be expected.
Numerical Techniques
Using finite element modelling techniques the effective elastic properties of the composite can also be determined. Hexagonal and square fibre array are often employed because of their small unit cell which greatly reduces computational time; larger and more complex unit cells can also be used.

All the models presented so far usually deal with linear elasticity as described by Hooke’s law. However it is well established that Hooke’s law is nothing more but a very good approximation to reality. Most engineering materials undergo plastic deformation well before any elastic nonlinearity can be observed. Some materials, like whiskers, show large elastic deformations: in these materials, while the deformation is still purely elastic and hence reversible, a noticeable nonlinearity can be observed in the stress-strain curve, as shown in Figure B.2 [59].

![Figure B.2: Stress-strain relationship for a high strength silicon whisker (from [59]). Although the behaviour is purely reversible and therefore elastic the nonlinear deformation at higher strains is clearly visible.](image)

This nonlinearity is a direct consequence of the nonlinear behaviour of the interatomic forces at larger interatomic distances. This phenomenon is shown in Figure B.3.
Nonlinearity in ceramics is well documented for whiskers and other single crystals. Data for pressure derivatives and higher order elasticity can be found in Simmons and Wang [61] and Landolt-Börnstein [62], respectively. Voigt and Reuss bounds for the elasticity constants of polycrystalline alumina can be readily derived. These values correspond very well to values derived from tests on polycrystalline alumina [63] if the latter are corrected for porosity [64]. Porosity was shown to decrease both Young’s modulus and Poisson’s ratio [64].

Thus if one looks for the most precise estimation of the composite Young’s modulus over a larger strain range the nonlinear elastic behaviour of the phases and possible porosity in the phases have to be considered.

3.2 Elastic-Plastic Deformation

When the stress-strain curve is described as bi-linear, then usually the slope of the second part is approximated as being equal to $E/V_f$ [50]. This implies that the fibres are linear elastic and the matrix is behaving as a linear elastic–perfectly plastic material, and assumes the validity of the rule of mixture. When the matrix hardens, the slope of the composite stress-strain curve just gradually decreases and approaches the value $E/V_f$. The ROM can then still be used just by using an instantaneous modulus for the matrix, equal to the strain-hardening rate of the matrix stress-strain curve.

As for the purely elastic case, the ROM is only exact for equal lateral contraction of both constituent phases. More so than for the elastic case, this is not true in the elastic-plastic case since the apparent $\nu_m$ increases when the matrix plastifies and the difference in lateral contraction between fibre and matrix becomes more important. Hill [65] treated the problem as an extension of the purely elastic case under the assumption that $\nu_m = 0.5$ and that the strain-hardening rate can be taken as an instantaneous modulus for the matrix. Hill again presents bounds for the resulting Young’s modulus of the composite that are consequently larger than the bounds for the purely elastic problem.
All the approaches mentioned in Section B - 3.1 can be used to tackle plastic deformation; however, this problem is more complex than in the elastic case.

### 3.3 The Influence of Fibre Breaks on the Elastic and Elastic-Plastic Deformation

Not only nonlinear elasticity of the reinforcing fibres and plastic deformation of the metallic matrix but also fibre breaks can influence the slope of the longitudinal tensile stress-strain curve of fibre reinforced metal matrix composites. Considering a constant number of broken fibres the problem is similar to that for aligned short fibre reinforced metals. An important difference is that the distance between fibre breaks in a damaged continuous fibre reinforced aluminium matrix composite is usually far longer than the fibre length in short fibre reinforced metals. Fu et al. [66] determined the Young’s modulus of a unidirectional multi-short-fibre composite assuming a perfectly elastic matrix. They expressed the modulus by a ROM that was modified by a fibre length factor \( \lambda \).

\[
E_c = \lambda E_f V_f + E_m V_m
\]

This fibre length factor \( \lambda \) depends on the stress transfer properties of the matrix and the fibre matrix interface. This stress transfer is classically determined by a shear lag analysis, first proposed by Cox [67] for the purely elastic case. Using the elastic analysis for a moderately damaged continuous fibre reinforced aluminium matrix composite one finds that the decrease in Young’s modulus is very small.

An extensive number of refined shear-lag based stress transfer models for the analysis of single fibre fragmentation tests were developed [68-72]. An important modification for the case of metal matrix composites is the analysis of a yielding matrix. With some generally accepted approximations, a simple stress transfer rule on the basis of a constant shear yield stress can be established [73,52]. As a consequence, the axial stress in the fibre builds up linearly from zero at the broken end to the fibre remote stress after a characteristic length - often referred as to critical fibre length \( l_c \) - which is easily calculated as:

\[
l_c = \frac{r_f \sigma_m}{\tau_{y.m}}
\]

where \( r_f \) is the fibre radius, \( \sigma_m \) is the fibre remote stress and \( \tau_{y.m} \) is the matrix shear yield stress. It has to be noted that in some publications this characteristic length is defined as half the above critical fibre length [69]. It has also been called the recovery length [69] or slip length [70], sometimes with slightly different meanings.
The influence of broken fibres on the deformation behaviour was predicted on the basis of a shear lag analysis by Curtin and Zhou [46]. They also analysed the problem for an increasing number of fibre breaks during loading. It was generally found that the influence is small as long as the critical length is much smaller than the mean distance between fibre breaks – as would be expected.

3.4 Loose Fibre Bundle Fracture

The strength parallel to the fibres of a continuous fibre reinforced metal is dominated by the properties of the reinforcing fibre bundle. The strength of a brittle single fibre can be described by Weibull statistics [74].

The survival probability $S_{\text{Weibull}}$ of a single fibre of length $L$ under applied fibre stress $\sigma_f$ is given by

$$S_{\text{Weibull}} = \exp \left( \frac{-L}{L_0} \left( \frac{\sigma_f}{\sigma_0} \right)^m \right) \quad (B.7)$$

where $\sigma_0$ is the characteristic fibre strength at gauge length $L_0$ and $m$ is the shape parameter of the Weibull distribution (known as the Weibull modulus).

Under the assumption that the fibres break independently from each other and that the load carried by broken fibres is equally distributed over the intact fibres, the strength of a dry fibre bundle can readily be calculated to be [75,76]:

$$\sigma_{\text{bundle}} = \sigma_0 \left( \frac{L}{L_0} \right)^{\frac{1}{m}} \exp \left( -\frac{1}{m} \right) \quad (B.8)$$

This mean fibre bundle strength is below the mean strength of the single fibres. Equation (B.8) is an approximation for an infinite number of fibres; when the number of fibres becomes finite, the strength of the fibre bundle follows a normal distribution [77].

3.5 Tensile Failure of the Composite

Since strength is, in contrast to the longitudinal Young’s modulus, a highly structure-sensitive property, it cannot be predicted by a simple rule of mixtures [52]. Nevertheless authors have tried to use this simple rule to predict composite strength. Lee and Hwang [78] developed a modified ROM by introducing an effective fibre volume fraction $V_{\text{eff}}$. This volume fraction is related to a degradation parameter $P$ which in turn is related to the physical fibre volume fraction $V_f$. The degradation parameter $P$ can be calculated from the microstructure and should be a bilinear function of $V_f$ with a change in slope around 54 vol.%. However formulas for calculating this function are not given and the rule must thus be considered as semi-empirical.

In high volume fraction fibre reinforced metals composite failure is dominated by fibre fracture. Fracture of brittle fibres is, as mentioned, generally described by Weibull statistics [74]. As a consequence the statistical fracture behaviour of the fibres should be incorporated in the strength prediction of the composite. It is often found by in-situ SEM observations and
acoustic emission monitoring during tensile tests that a certain degree of damage in the form of fibre breaks accumulates before composite failure [79-82]. Experimental quantification of the amount of damage accumulation before failure is not found in the literature for small diameter fibre reinforced metals.

**Global Load Sharing (GLS)**

The statistical strength of a loose fibre bundle (as presented in Section B - 3.4) is often used to predict the composite strength. The assumption that the fibres in the composite show exactly the same behaviour as in the loose fibre bundle leads to the global load sharing concept (GLS), sometimes referred as to equal load sharing (ELS). It is assumed that the load carried by a fibre is distributed equally to all the remaining intact fibres when the fibre breaks. Fibre breaks are considered to be statistically independent. The fibre contribution to the strength of the composite is thus equal to the loose fibre bundle strength multiplied by the fibre volume fraction. The global tensile load carried by the matrix is, in the case of a high volume fraction of high stiffness fibres, of minor importance and sometimes difficult to quantify, because the in-situ matrix properties are not a priori known (see Section B - 4.1).

The matrix, however, plays an important role on a local scale, as soon as fibre breaks appear. Rosen [83] introduced a more realistic model that accounts for the reload of a broken fibre from its dead end by shear and that allows multiple breaks in one fibre. In this treatment the composite is “cut” in several slices each with length \( \delta \), the ineffective fibre length. These slices are treated as loose fibre bundles with a statistical strength distribution. The composite strength is then described by weakest links statistics: as soon as one of these bundles fails, the composite fails. This model, in different variations, is still widely used and referred to as the chain-of-bundles model.

On the basis of a simplified shear lag approach for yielding matrix Curtin [84] developed a statistical theory for the fragmentation of a single fibre embedded in a matrix. The maximum stress to which a fibre in a composite can be stressed \( \sigma_c \) is calculated from the fibre strength statistics, the fibre geometry and the matrix shear yield stress, as seen in Equation (B.9).

Curtin then applied this theory to a multifibre composite material and developed an explicit equation for the stress-strain curve of the composite. The maximum of this curve is the ultimate tensile strength of the composite and is explicitly given in Equation (B.10).

All the models dealing with GLS give usually an upper bound for the composite strength, which can only be approached when a weak fibre-matrix interface is present or the matrix is very weak in shear [85].
Local Load Sharing (LLS)
In most cases it is more realistic to assume that the load of a broken fibre is redistributed non-uniformly, loading neighbouring fibres more strongly, i.e. leading to stress concentrations. The stress concentration factor (SCF) is usually defined as the ratio between the maximum stress on a neighbouring fibre $\sigma_{\text{max}}$ and the applied fibre stress $\sigma_f$ carried by a fibre not affected by any fibre break.

$$\text{SCF} = \frac{\sigma_{\text{max}}}{\sigma_f}$$

The first treatment of such stress concentrations was based on the shear lag analysis [67] and was conducted by Hedgepeth [86] and Hedgepeth and Van Dyke [87]. In this Hedgepeth-Van Dyke model (HVD) the composite is approached as an infinite hexagonal or square array of fibres (approximated as one-dimensional, axial load carrying springs) embedded in a matrix that is assumed to carry no axial load but transfers only load between the fibres by shear. Perfect bonding is assumed between the fibres and the matrix. Due to the assumption that the matrix carries only shear stresses, the resulting SCFs are independent of matrix properties and interfibre distance. They are 1.146 and 1.104 for a single broken fibre in a square and hexagonal fibre array, respectively. Additionally the model only considers stress transfer to the nearest neighbours, implicitly neglecting transfer to any next-nearest neighbours.

The HVD model has formed the basis for a number of other, more general, treatments. Wagner and Eitan [88] considered the axial load carrying capacity of the matrix and calculated a SCF which varies as a function of the interfibre distance. But since only nearest neighbours are involved in stress transfer in this model, the SCF is independent of materials properties. Landis and McMeeking [89] developed a model that includes matrix axial stiffness and allows for fibre-matrix debonding with subsequent sliding along the fibre-matrix interface. They assumed a constant shear stress over the debonded region and an elastic matrix. Their SCF for one broken fibre in a square fibre array is a function of interfibre distance, the interface sliding resistance and the ratio between matrix and fibre axial stiffness. The maximum value in the elastic case (assuming no sliding) for a matrix with an axial stiffness of zero is 1.081 (independent of the interfibre distance) and therefore slightly lower in comparison to the HVD model. They compared their model to FEM calculations and found good agreement.

The SCF must be incorporated in a model predicting failure of the composite to be useful. To this end, the failure probability of the fibres seeing additional stress has to be determined. For this purpose not only the maximum extra load but the entire stress profile along the overloaded fibre axis has to be considered. Explicit stress profiles for the nearest neighbours of a broken fibre can be found in Beyerlein and Landis [90] or Wagner and Eitan [88] (in both cases based on elastic shear lag analysis), Xia et al. [91] (results from finite element modeling) and Landis and McMeeking [89] or He et al. [92] (in both cases based on shear lag analysis with interface sliding and/or matrix plasticity). In [92] the failure probability of the overloaded fibre is calculated and it is found that a lower interface shear resistance, which leads to a lower SCF, can result in a higher failure probability due to a longer overloaded length (usually called “positively affected length” PAL). Apart from the results presented by Wagner and Eitan [88] the authors predict a zone outside of the positively affected length, where the
overloaded fibre has a lower axial stress than the applied stress far away from the fibre break. This is a consequence of the requirement of overall strain compatibility within the composite. He et al. [9] measured stress concentrations in a continuous alumina fibre reinforced alumin­ium using photostimulated luminescence based piezospectroscopy at the surface of alumina fibre reinforced aluminium composites. They found very good agreement with their theoreti­cal models.

For further calculation, the stress profile in the overloaded fibre is often approximated by the two parameters SCF and PAL with a linear decrease of the axial fibre stress from the max­imum stress in the plane of the fibre break to the applied fibre stress at the distance equal to the PAL.

Even if the stress concentration factors are accurately calculated, the failure criterion is not clear in most of the models. Batdorf [93,94] presented a simplified model to determine the onset of composite failure by analysing the successive propagation of fibre breaks from a single initial fibre break. To this end Batdorf analysed the stability of a cluster of i neighbouring fibre breaks, named an “i-plet” of broken fibres. With a mathematical approximation of the Weibull-distribution (valid for low failure probabilities) the number of single fibre breaks (called “singlets”) is calculated as given by:

\[ Q_i = N \cdot \frac{L}{L_0} \left( \frac{\sigma}{\sigma_0} \right)^m \]  \hspace{1cm} (B.12)

where \( N \) is the number of fibres in the composite of length \( L \), and \( \sigma \) is the applied fibre stress. \( \sigma_0 \), \( L_0 \) and \( m \) are the parameters of the fibre Weibull distribution that can be determined by single fibre tensile tests. The number of “doublets” (two neighbouring fibre breaks) is calculated on the basis of the probability that a neighbouring fibre of a singlet breaks. Batdorf develops an explicit equation for the number of i-plets in the composite at fibre stress \( \sigma \):

\[ Q_i = N \cdot \frac{L}{L_0} \left( \frac{\sigma}{\sigma_0} \right)^m \cdot \prod_{j=1}^{i} c_j^{n_j} \cdot n_j \cdot \frac{\lambda_j}{L_0} \]  \hspace{1cm} (B.13)

where \( c_j \) is the SCF for the neighbouring fibres of a j-plet, \( n_j \) is the number of neighbours and \( \lambda_j \) is the effective positively affected length (a length overloaded by the constant SCF \( c_j \) that results in the same fibre failure probability as the real overload stress profile). A double logarithmic plot of the number of i-plets vs fibre stress results in a series of straight lines of slope \( im \). i-plets on the envelope of this lines are unstable. Batdorf then reasons that the intersection of this envelope with \( \ln Q_i = 0 \) i.e. \( Q_i = 1 \) (formation of the first unstable i-plet with near­certainty) is the failure stress of the composite.

Batdorf’s model is simple yet powerful. Still, Batdorf mentions several issues where his model fails to accurately describe the physical reality. Among those are (i) the use of the mathematical approximation for the failure probability which makes the problem easily trac­table but looses validity when the failure probability gets high, (ii) the fact that the depletion of the number of i-plets when \( i+1 \)-plets are formed is not taken into account and (iii) the fact that the calculation of the failure probability of the overloaded fibre should take into account
that it survived a lower stress without failing. Relaxing these assumptions can however be envisaged, such that Batdorf's model is a simple concept that has potential to be developed. Another key feature of this model is that it relies on the choice of realistic stress concentration factors and positively affected lengths; it therefore needs to be completed with relevant load transfer analyses.

Lienkamp and Exner [95] used the HVD model for the strength prediction of alumina fibre reinforced aluminium metal matrix composites. Monte Carlo simulations were used to calculated the number of clusters of different numbers of broken fibres as a function of stress. Their failure criterion is similar to Batdorf's [93,94] criterion. The critical cluster size is reached when a cluster of \( i \) fibres immediately transforms into a cluster of \( i+1 \) fibres; the corresponding stress is the composite failure stress. Lienkamp and Exner found good agreement with experimental results. The authors also used the method to predict the failure probability of large composite parts [96]. The Weibull modulus of the composite part was thereby – in agreement with Batdorf - predicted to be \( k \)-times that of the reinforcing fibres where \( k \) is the size of the critical cluster of broken fibres. The calculation of Lienkamp and Exner has, however, similar shortcomings as Batdorf's work.

Wisnom and Green [97] developed a model which does not need any stress concentrations for a critical cluster of broken fibres to be formed. Their key assumption is that two fibre breaks that are axially separated can be part of the same multiplet when their axial spacing is smaller than the critical length. In the following the critical length increases with the number of fibre breaks being part of the cluster. There is thus a stress at which enough – statistically independent – fibre breaks are present that this diffuse cluster of \( i \) broken fibre immediately joins up to a cluster of \( i+1 \) broken fibre and is therefore unstable.

Hu et al. [98] followed a fracture mechanics approach to the strength of continuous fibre reinforced metal matrix composites. For systems with high fibre volume fractions and high strength matrices and consequently high stress concentrations arising from broken fibres, fracture mechanics considerations become increasingly important. Assuming an initial flaw with the size of a single fibre the authors predict the ultimate tensile strength on the basis of the fracture toughness of the fibres and the stress intensity factor for a circular crack. Cao et al. [99] found reasonable agreement with experimental values only when assuming initial flaw sizes of several fibre diameters.

A number of authors use superposition techniques [91,100-103] or Monte Carlo Simulation [95,104-106] for the simulation of damage evolution and failure. The different approaches to the stress concentration factor around broken fibre outlined above are incorporated in these simulations.

Overall the literature can be summarized as follows:

(i) there is experimental evidence for a certain amount of fibre breaks before composite failure; however, quantification of this damage is only rarely found;
(ii) there is no clear experimental evidence of what causes final fracture of the composite.
4. The Matrix

4.1 *In-situ* Matrix Properties

It is widely accepted that the *in-situ* metal matrix properties can differ strongly from the properties of the same bulk material. The microstructural geometry of the composite, with a typical characteristic length scale between 1 and 50 µm (corresponding to the interfibre distance), perturbs the solidification process (see Section B - 2.5) and restricts deformation of the softer phase (the matrix). In addition to this, the mismatch of the thermal expansion coefficient between matrix and reinforcement together with high processing temperatures and subsequent cooling to room temperature results in high residual thermal stresses in the two phases.

**Indentation Techniques**

Indentations in the matrix between fibres on cross-sections perpendicular to the fibre direction could allow the measurement of *in-situ* mechanical matrix properties. Microhardness testing with Vickers or Berkovich indenters usually leaves indents with sizes in the order of 10 to 50 µm. For composites reinforced with a high volume fraction of small diameter fibres this is obviously too large. Nanoindentation techniques with indent sizes in the submicrometer range are now available and successfully used to measure phase properties in two-phase superalloys \[107\] and in thin films \[108\]. Comprehensive theoretical work to extract materials properties from instrumented sharp indentation has been done by Venkatesh et al. \[109\] and Giannakopoulos and Suresh \[110\]. Despite this recent progress, indentation can only measure the properties near the metallographically prepared surface. In addition, the influence of the neighbouring fibres must be regarded with care as it can add to the mechanical complexity of the measurements.

**Back-calculation Techniques**

*In-situ* matrix properties can also be determined from the longitudinal composite stress-strain curve \[47,111\]. The basis of this method is the assumption that the matrix and the fibre exhibit the same strain leading to the well known equistrain rule of mixtures (ROM) for the composite stress

\[
\sigma_c = \sigma_f \cdot V_f + \sigma_m \cdot (1-V_f) \tag{B.14}
\]

where \(\sigma_f\) and \(\sigma_m\) is the uniaxial tensile fibre and matrix stress, respectively, and \(V_f\) is the fibre volume fraction.

Kelly and Lilholt \[111\] carefully examined stress-strain curves of monocrystalline copper reinforced with different volume fractions of tungsten wires with a diameter of 10 and 20 µm and found the strain hardening rate of the copper matrix to be considerable higher than in unreinforced bulk material. Kelly and Lilholt stated that only a part of the difference can be accounted for by the lateral contraction mismatch between the fibres and the matrix, using Hill’s \[65\] bounds to account for this effect (see also Section B - 3.2). To solve the problem, they introduced a zone of copper surrounding each fibre that is only deforming elastically. Tanaka and Mori \[112\] explained the increasing strain-hardening rate with increasing fibre volume fraction on the basis of a self-consistent scheme based on Eshelby’s \[54\] work. This
method is only valid for low fibre volume fractions and the strain hardening predicted is independent of the fibre diameter [113], which is contrary to observations.

Neumann and Haasen [114] explained the increased strain-hardening found by Kelly and Lilholt by superimposed series of dislocation pile-ups at the fibre-matrix interface, a hypothesis originally disregarded by Kelly and Lilholt based on analysis of single pile-ups. Neumann and Haasen found that a system of dislocation pile-ups rather than an isolated pile-up can actually describe the experimental data found in [111].

Mortensen et al. [115] invoke the inevitable presence of fibre clustering and thermal expansion mismatch between the reinforcing phase and the matrix to explain the observed strain-hardening rate in Kelly and Lilholt’s data. In regions with higher local volume fraction the matrix has a high dislocation density - dislocations that where produced during cooldown from the processing temperature - and therefore a higher yield strength. With a simple rule of mixtures they were able to roughly explain the apparent strain hardening rate as a function of fibre volume fraction.

Garmong and Shepard [116] produced a large set of data for iron fibre reinforced copper with different fibre diameter and a wide range of fibre volume fraction. They concluded that for high volume fraction composites triaxiality in the matrix can significantly reduce the resolved shear stress and consequently increase the axial yield stress. For lower volume fraction composites a dislocation pile-up model was used to explain the increased strain-hardening rate.

Chawla and Metzger [117] investigated the initial dislocation distribution in low volume fraction tungsten fibre reinforced copper composites by etch pitting. They found an increased dislocation density toward the fibre-matrix interface and invoked the thermal mismatch strain during cool down from the processing temperature to explain their results. They also measured the strain-hardening rate of these composites and found it to be increased compared with unreinforced copper. Similar to the explanation by Kelly and Lilholt [111] and Mortensen et al. [115] the authors explained this behaviour with the fact that the matrix does not undergo plastic deformation over the entire cross-section at once, but the zone surrounding the fibre yield later due to its higher dislocation density. Clarke [118] proposed an explanation of the enhanced strain-hardening rate based on local inhomogeneous stresses in the matrix, a mechanism called dislocation source shortening.

Bystricky et al. [47] investigated aluminium- and copper-based matrices reinforced with two different types of continuous alumina fibres. The authors calculated the complete in-situ matrix flow curves using Hill’s [65] bounds for a perfectly plastic matrix. Contrary to the work done on the copper-tungsten system they did not find an increased strain-hardening rate compared to the unreinforced matrix. For some specimens the authors even observed apparent strain-softening, which was ascribed to the significant scatter in the data.

4.2 The Influence of the Matrix Properties on the Strength

It is widely accepted that the matrix exerts a significant influence on the performance of composites. This is obvious for the strength perpendicular to the fibre direction, as this property is clearly matrix-dominated. It is obvious too for the performance of an angle-plied composite, due to the important role of shear parallel to the fibres. Johnston and Greenfield [119] found the composite shear strength parallel to the fibres to follow exactly the shear strength of the unreinforced matrix. Abe et al. [6] found the addition of 5% Cu to the pure aluminium matrix to be favorable for the strength of an angle-plied composite.
It is less obvious but there is enough experimental evidence that also the fibre-dominated longitudinal strength (in tension and compression) is strongly affected by the matrix properties. McCullough et al. [120] report tensile strength values for a number of matrices reinforced by the Nextel 610 alumina fibre. They found values between 400 MPa for a commercial 6061 Al alloy and 1550 MPa for the binary Al-2%Cu system. Abe et al. [6] found a higher tensile strength for a pure aluminium matrix reinforced with Sumitomo’s Altex fibre than for a (stronger) Al-5%Cu matrix.

Already in the early time of continuous fibre reinforced MMC it was found (mainly by Japanese researchers) that brittle second phases are detrimental for the composite longitudinal tensile strength. Fukunaga and Pan [121] recognized that fracture of a reinforced Al-4%Cu matrix is controlled by fracture of the brittle eutectic that was preferrentially found at the fibre-matrix interface. Dumant et al. [15] infiltrated two different types of SiC-fibres with pure aluminium and an aluminium-silicon alloy. Independent of the fibre type the longitudinal strength was dramatically lowered by the presence of the silicon phase. They identified two main mechanisms causing the composite strength to decrease, namely the above-mentioned embrittlement due to brittle second phases at the interface, and a decrease of the intrinsic fibre strength due to reactions between fibre and matrix. A number of authors quantified the effect of the thickness of a brittle reaction layer on the strength of the composite [122-126], usually assuming a uniform brittle layer of homogeneous thickness around the fibre. Only Ochiai et al. [127] considered a platelet-like compound adhering on the surface of the fibre. The fractured reaction compound is treated like an notch producing a stress concentration in the fibre. Experimental verification of fibre strength decrease due to brittle interface compounds is found for a number of coated fibre reinforced composites [125-128]. Ochiai and Osamura [129] performed computer simulations on the strength of continuous fibre reinforced MMC with a reaction layer at the fibre-matrix interface and found good agreement with the analytical solution developed earlier [122,126].

As a consequence it is nowadays generally accepted that most commercial wrought and cast aluminium alloys are not suitable as matrix material for continuous fibre reinforcement. There is therefore a trend towards specifically designed matrix systems. Neussl et al. [130] investigated systematically the addition of usual alloying elements (Zn, Cu, Mg and Si) to pure aluminium reinforced with different reinforcing fibres. Their goal was to identify suitable alloys for selectively (i.e. locally) reinforced MMC-structures with load-bearing unreinforced parts. They found the longitudinal tensile strength of the reinforced part to decrease for all the alloying elements at higher content (>1%) with the exception of Zn, which did not affect the strength of the composite. In the ternary age-hardenable Al-Mg-Zn system, however, Zn was found to increase the strength of the composite. The authors concluded that the system Al-Zn-Mg is the only one to have some potential for the successful production of selectively reinforced composites with load-bearing unreinforced parts. Later, Neussl et al. [131] found the addition of Ag to the system Al-Zn-Mg to be favorable in the case of T6 treatment.

Long et al. [132] investigated several binary and ternary alloys in the Al-Zn-Mg system as well as Al-Si alloys reinforced with continuous Altex fibres. They concluded that the use of a high strength matrix free of second phases increases the longitudinal tensile strength of the composite.

The strength of different matrix composites was also studied as a function of temperature. Whereas the strength of pure Al matrix composites is usually found to decrease with increas-
ing temperature [14], composites with a Al-Cu matrix often exhibit a slight initial increase in strength for moderate temperatures before their strength also decreases at higher temperature [127,133]. The strength of ceramic fibres is not found to be affected by temperatures below 1000 °C [134]. Hence the authors explain their results with the change in matrix properties with increasing temperature. The decrease in composite strength is thus related to the decrease in matrix strength with increasing temperature. Ochiai et al. [14] concluded that the increase in overloaded volume with decreasing matrix shear yield stress has a greater effect than the decrease of the SCF.

5. Fibres for the Reinforcement of Metals
Different types of fibres are used to reinforce metal matrices, and in particular aluminium. These include SiC-fibres ("Nicalon" and "Tyranno" from Nippon Carbon and Ube Industries respectively), carbon fibres of various types from several manufacturers, and alumina fibres ("Nextel" from 3M).
Small diameter multifilament SiC-fibres usually contain free carbon and silica [135] which can react with the aluminium matrix, resulting in brittle interface layers. Carbon fibres suffer the same problem when used in combination with an aluminium matrix. Plate-shaped aluminium carbides are formed, leading to a considerable embrittlement of the fibre-matrix interface. Protective coatings, such as titanium nitride, have been used, but these techniques are in general difficult and expensive for multifilaments. Pure α-alumina fibres are essentially stable in molten aluminium and most of its alloys, and can therefore be considered to be among the most suitable continuous reinforcement for aluminium matrices. Depending on the degree of porosity, these fibre can exhibit an elastic modulus close to 400 GPa. Their average strength can be more than 3 GPa, combined with a good strength retention up to several hundred degrees.
C - MATERIALS AND EXPERIMENTAL PROCEDURES

Two different forms of liquid metal infiltrated alumina fibre reinforced aluminium were investigated during this study: a continuous wire with a diameter of about 2 mm (produced by 3M, St. Paul, MN, USA) and pressure-cast parallel tensile bars with a cross-section of about 16 mm$^2$ and a length of 120 to 150 mm (produced at EPFL and EMPA Thun). Both types of composite are reinforced with Nextel 610$^{TM}$ continuous alumina fibres. Pure aluminium or aluminium-based binary and ternary alloys were used as matrix materials.

1. Reinforcing Fibre and Matrix

1.1 Nextel 610$^{TM}$ Alumina Fibre

The alumina fibre Nextel 610$^{TM}$ is a pure fine-grained $\alpha$-Al$_2$O$_3$ fibre, developed and commercialized in the early 1990s by 3M (MN, USA). A key property of this fibre is its high tensile strength, which has continuously been improved over the last decade (see Figure C.1) [136-139].

![Figure C.1: Single filament strength increase since 1989 until today for the Nextel 610 alumina fibre (from [134]).](image)

The fibre is produced by a sol-gel process (see Figure C.3). With the addition of iron oxide the nucleation rate of $\alpha$-Al$_2$O$_3$ is greatly improved and a high density, ultrafine-grained, homogeneous $\alpha$-Al$_2$O$_3$ fibre is obtained. The resulting grain size is around 0.1 µm and the fibre diameter is 10-12 µm. The fine grain size combined with a low porosity and the small fibre diameter are the reasons for the high fibre strength. The fibre also has an excellent strength retention at temperatures up to 1000 °C as long as the strain rate is high enough to prevent creep [134].
Figure C.2: Relative tensile strength retention of multi-filament strands of different Nextel™ fibres at elevated temperature (from [134]).

Figure C.3: 3M Sol-Gel-process for the production of Nextel 610 α-Al₂O₃ fibres (from [140]).

Table C.1 gives an overview of the most important properties of the Nextel 610™ alumina fibre that is currently available.
Table C.1: Main properties of the Nextel 610™ alumina fibre.

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Composition [141]</td>
<td>&gt;99% α-Al2O3</td>
</tr>
<tr>
<td></td>
<td>0.2-0.3% SiO2</td>
</tr>
<tr>
<td></td>
<td>0.4-0.7% Fe2O3</td>
</tr>
<tr>
<td>Mean UTS at L0 = 25.4 mm [134]</td>
<td>3.3 GPa</td>
</tr>
<tr>
<td>Weibull modulus [134]</td>
<td>9.7-11.2</td>
</tr>
<tr>
<td>Young's modulus [134]</td>
<td>373 GPa</td>
</tr>
<tr>
<td>Density [141,134]</td>
<td>3.75-3.9 g/cm³</td>
</tr>
<tr>
<td>Diameter [134]</td>
<td>11.98 µm</td>
</tr>
<tr>
<td>CTE (100-1100°C) [142]</td>
<td>8 x 10⁻⁶ K⁻¹</td>
</tr>
</tbody>
</table>

Comprehensive information about the fibre can be found in the Nextel Ceramic Textile Technical Notebook [142] on the 3M Webpage. This publication is regularly updated. Nextel 610™ is available with a Denier of 1500, 3000 or 10'000 corresponding to a nominal filament count of about 400, 750 or 2550 per tow respectively. In this study we used the 1500 Denier fibre.

1.2 The Matrix Materials

The continuous alumina fibre reinforced composite wire was available with two different matrices: pure Al and Al-2%Cu. Various matrices were used to infiltrate the parallel tensile bar preforms in the present study: 99.99% high purity aluminium, two binary alloys: Al-1%Mg, Al-6%Zn, and the ternary alloy Al-6%Zn-0.5%Mg. The high purity aluminium (hpAl) was purchased from VAW Aluminium AG, Grevenbroich, Germany. The binary and ternary alloys were produced at the Swiss Federal Laboratories for Materials Testing and Research (EMPA) in Thun (Switzerland) from high purity aluminium and master alloys. A detailed chemical analysis of the alloys was conducted at EMPA using the method of GDOES (glow discharge optical emission spectroscopy). The results can be found in Table C.2. The origin of the matrix materials used for the production of the composite wires by 3M is not known.

Table C.2: Chemical analysis of the different metallic matrices used in this study. All values are in mass % and represent the mean value of 3 measurements with the standard deviation in brackets. The method used was GDOES.

<table>
<thead>
<tr>
<th></th>
<th>Si</th>
<th>Mg</th>
<th>Cu</th>
<th>Zn</th>
<th>Fe</th>
<th>Mn</th>
<th>Ti</th>
<th>Cr</th>
<th>Be</th>
</tr>
</thead>
<tbody>
<tr>
<td>hp Al</td>
<td>0.004</td>
<td>&lt;0.001</td>
<td>0.001</td>
<td>&lt;0.001</td>
<td>0.002</td>
<td>&lt;0.001</td>
<td>0.001</td>
<td>0.001</td>
<td>-</td>
</tr>
<tr>
<td>AlZn6</td>
<td>0.002</td>
<td>&lt;0.001</td>
<td>0.001</td>
<td>6.12 (0.11)</td>
<td>&lt;0.001</td>
<td>0.002</td>
<td>&lt;0.001</td>
<td>&lt;0.001</td>
<td>-</td>
</tr>
<tr>
<td>AlMg1</td>
<td>0.003</td>
<td>1.03 (0.012)</td>
<td>0.002</td>
<td>0.004</td>
<td>0.004</td>
<td>0.003</td>
<td>&lt;0.001</td>
<td>&lt;0.001</td>
<td>0.005</td>
</tr>
<tr>
<td>AlZn6Mg0.5</td>
<td>0.002</td>
<td>0.43 (0.0001)</td>
<td>0.002</td>
<td>6.09 (0.033)</td>
<td>&lt;0.001</td>
<td>0.002</td>
<td>&lt;0.001</td>
<td>&lt;0.001</td>
<td>-</td>
</tr>
</tbody>
</table>

2. Preform Preparation for Parallel Tensile Bars

Net-shape fibre preforms for parallel tensile bars were produced by winding the fibre tow directly into a graphite U-profile which is part of the infiltration mold. During this winding process the fibre tow was wetted with water or an aqueous suspension of small alumina particles...
to improve the handling of the fibre tow and to achieve the desired hybridization of the composite (see Section B - 2.2). The alumina particles used for that purpose were the Sumicorundum AA-07 and AA-2 polygonal alumina particles with a mean particle size of 0.86 and 2.4 µm, respectively (purchased from Sumitomo Chemicals Ltd., Japan).

The use of net-shape graphite molds avoids machining of the cast composites. This in turn allows the accurate determination of the fibre volume fraction within the composite (see Section C - 4.3) and minimizes fibre damage at the surface of the tensile specimen (compared to machined specimens).

The rectangular tensile bars have a cross-section of 8 x 1.5-2.0 mm and a length of 120 and 150 mm for direct squeeze casting and gas pressure infiltration, respectively.

3. Liquid Metal Infiltration and Heat Treatment

3.1 Continuous Infiltration of the Composite Wire

The composite wire was produced by 3M, reportedly by means of a continuous ultrasound-driven infiltration [21,143]. The fibres are pulled through the molten matrix under agitation by an ultrasound horn, immersed in the matrix bath, which allows the infiltration.

3.2 Liquid Metal Infiltration of Parallel Tensile Bars

The preforms were infiltrated using two different liquid metal infiltration processes, namely direct squeeze casting (DSC) and gas pressure infiltration (GPI). The principles of the methods are respectively sketched in Figure C.5 and Figure C.6 and shortly outlined in the following.

For the direct squeeze casting technique the graphite molds holding the fibres are assembled and held together by massive steel plates (Figure C.4). This preform is preheated under an atmosphere of forming gas and then rapidly transferred into the die cavity. The liquid matrix is poured over the preform and the squeeze ram moves down under speed control at 5 mm/s until a threshold pressure of about 4 MPa is reached. It is assumed that infiltrations is completed at this pressure. Then pressure is increased with a rate of about 5 MPa/s to the maximum pressure of 100 MPa. The load is then held for a preset solidification time of four minutes. Solidification shrinkage is thus compensated. Directional solidification is achieved due to the temperature gradient that is introduced during preheating between the bottom of the die cavity and the squeeze ram.

For gas pressure infiltration the graphite molds containing the fibre preforms are placed at the bottom of a gas-tight alumina crucible, together with a cast matrix billet resting on top of the preform. The whole assembly is placed in a vacuum-pressure furnace and evacuated. After heating under vacuum to the infiltration temperature, pressurized argon is slowly introduced into the pressure vessel until the maximum desired pressure is reached. After a five minute hold, the pressure is reduced and the crucible is lowered and rested onto a copper chill, in order to induce directional solidification. The maximum pressure is then reapplied and held during solidification.

Processing parameters for both infiltration techniques are listed in Table C.3. Table C.4 summarizes the most important differences between the two infiltration techniques.
Figure C.4: Preform containing 8 parallel tensile bars for infiltration by direct squeeze casting.

Figure C.5: Schematic description of direct squeeze casting (DSC) as used at EMPA Thun.
C - Materials and Experimental Procedures

1. evacuation of preform and melting of the metal under vacuum
2. pressurization and infiltration
3. directional solidification under pressure

Figure C.6: Schematic description of gas pressure infiltration (GPI) as used in the present study.

Table C.3: Processing parameters for the two liquid metal infiltration techniques used for composite processing.

<table>
<thead>
<tr>
<th></th>
<th>direct squeeze casting</th>
<th>gas pressure infiltration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Melt temperature</td>
<td>750 °C</td>
<td>750 °C</td>
</tr>
<tr>
<td>Preform temperature</td>
<td>820 °C</td>
<td>750 °C</td>
</tr>
<tr>
<td>Die temperature bottom to top</td>
<td>150 - 350 °C</td>
<td>-</td>
</tr>
<tr>
<td>Preform atmosphere</td>
<td>Forming gas, Noxal3 (Ar/H₂)</td>
<td>vacuum</td>
</tr>
<tr>
<td>Max. pressure</td>
<td>100 MPa</td>
<td>9 MPa</td>
</tr>
<tr>
<td>Time under max. pressure at 750 °C</td>
<td>-</td>
<td>5 min</td>
</tr>
<tr>
<td>Time under max. pressure incl. solidification</td>
<td>4 min</td>
<td>several hours</td>
</tr>
</tbody>
</table>

Table C.4: Comparison of important parameters between gas pressure infiltration and direct squeeze casting.

<table>
<thead>
<tr>
<th>direct squeeze casting</th>
<th>gas pressure infiltration</th>
</tr>
</thead>
<tbody>
<tr>
<td>high maximum pressure applied (100 MPa)</td>
<td>moderate maximum pressure (10 MPa)</td>
</tr>
<tr>
<td>short contact time between liquid metal and reinforcing phase</td>
<td>longer contact time between liquid metal and reinforcing phase</td>
</tr>
<tr>
<td>wide variety of matrix systems (including reactive systems)</td>
<td>limited to systems that do not display excessive interfacial reactivity or matrix evaporation</td>
</tr>
<tr>
<td>partially controlled preform atmosphere</td>
<td>preform heated under vacuum</td>
</tr>
<tr>
<td>mechanically strong preform housing necessary</td>
<td>gas-tight preform housing necessary</td>
</tr>
</tbody>
</table>

26
3.3 Heat Treatment
Some specimens of the Al-2%Cu composite wire were solutionized for 16 hours at 520 °C in air and quenched in water. Some of these specimens were subsequently aged for up to 48 hours at 190 °C in air.

4. Microstructural Characterization
4.1 Metallographic Procedures
Transverse cross-sections were metallographically polished to investigate the fibre arrangement. The polishing was conducted using an automatic sample preparation system Phoenix V4000 (Wirtz-Bühler, Germany) using the procedure summarized in Table C.5.

<table>
<thead>
<tr>
<th>cloth</th>
<th>grit / grain size</th>
<th>time</th>
<th>pressure</th>
<th>RPM</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiC-paper</td>
<td>320</td>
<td>until flat by hand</td>
<td>250</td>
<td></td>
</tr>
<tr>
<td>SiC-paper</td>
<td>400</td>
<td>~ 1 min  by hand</td>
<td>250</td>
<td></td>
</tr>
<tr>
<td>SiC-paper</td>
<td>600</td>
<td>~ 1 min  by hand</td>
<td>250</td>
<td></td>
</tr>
<tr>
<td>SiC-paper</td>
<td>1200</td>
<td>~ 1 min  by hand</td>
<td>250</td>
<td></td>
</tr>
<tr>
<td>Texmet perforiert</td>
<td>9 µm</td>
<td>5 min</td>
<td>12 N</td>
<td>250</td>
</tr>
<tr>
<td>Texmet perforiert</td>
<td>6 µm</td>
<td>20 min</td>
<td>17 N</td>
<td>250</td>
</tr>
<tr>
<td>Texmet perforiert</td>
<td>1 µm</td>
<td>10 min</td>
<td>17 N</td>
<td>250</td>
</tr>
<tr>
<td>DP-NAP</td>
<td>¼ µm</td>
<td>2 min</td>
<td>10 N</td>
<td>250</td>
</tr>
</tbody>
</table>

Longitudinal cross-sections were polished by a slightly altered procedure to avoid fibre fragmentation and also to account for the usually larger specimen polish surface. This procedure is summarized in Table C.6.

<table>
<thead>
<tr>
<th>cloth</th>
<th>grit / grain size</th>
<th>time</th>
<th>pressure</th>
<th>RPM</th>
</tr>
</thead>
<tbody>
<tr>
<td>diamond disc</td>
<td>125 µm</td>
<td>until flat by hand</td>
<td>250</td>
<td></td>
</tr>
<tr>
<td>diamond disc</td>
<td>20 µm</td>
<td>~ 1 min  by hand</td>
<td>250</td>
<td></td>
</tr>
<tr>
<td>SiC-paper</td>
<td>400</td>
<td>~ 1 min  by hand</td>
<td>250</td>
<td></td>
</tr>
<tr>
<td>SiC-paper</td>
<td>600</td>
<td>~ 1 min  by hand</td>
<td>250</td>
<td></td>
</tr>
<tr>
<td>Texmet perforiert</td>
<td>9 µm</td>
<td>30 min</td>
<td>30 N</td>
<td>250</td>
</tr>
<tr>
<td>Texmet perforiert</td>
<td>6 µm</td>
<td>10 min</td>
<td>30 N</td>
<td>250</td>
</tr>
<tr>
<td>Texmet perforiert</td>
<td>1 µm</td>
<td>10 min</td>
<td>30 N</td>
<td>250</td>
</tr>
<tr>
<td>DP-NAP</td>
<td>¼ µm</td>
<td>10 min</td>
<td>20 N</td>
<td>250</td>
</tr>
</tbody>
</table>

The grain structure of the composites was revealed by dipping in Keller’s etch. A short activation of the surface was done before. The procedure is summarized in Table C.7.
Materials and Experimental Procedures

Table C.7: Etching procedure for revealing the grain structure.

<table>
<thead>
<tr>
<th>agent</th>
<th>temperature</th>
<th>time</th>
</tr>
</thead>
<tbody>
<tr>
<td>NaOH (1 M)</td>
<td>50-60 °C</td>
<td>10 s</td>
</tr>
<tr>
<td>water</td>
<td>30 °C</td>
<td>5 s</td>
</tr>
<tr>
<td>Keller's etchant (HCl/HNO₃/HF/H₂O)</td>
<td>50-60 °C</td>
<td>30 s</td>
</tr>
<tr>
<td>water</td>
<td>30 °C</td>
<td>20 s</td>
</tr>
</tbody>
</table>

4.2 Transmission Optical Microscopy (TOM)

Since the alumina fibres are translucent for visible light, these composites offer one of the rare applications of transmission optical microscopy in metallurgy. For this purpose composite slices perpendicular to the fibre direction were cut to different thicknesses and polished on both sides as described above. An Axioplan 2 optical microscope (Zeiss, Germany) with a 100 W halogen transmission light source was used. A slight amount of reflected light was used at the same time to illuminate the matrix. A maximum slice thickness of about 2 mm could be investigated with this light intensity. Figure C.7 shows a transmission optical micrograph from a composite wire cross-section. The slice thickness is about 1.8 mm. The fibres are clearly visible as bright spots, whereas the matrix is somewhat darker (the matrix would actually appear completely black if no reflected light were used). The brightness of the fibres depends, among other parameters, on the light intensity and slice thickness. One can easily see that some of the fibres are not bright but black. These were identified not as being broken continuous fibres but rather as dead-ending fibres; i.e. fibres which do not traverse the slice. Figure C.8 shows the principle of the transmission optical microscopy technique used.

Figure C.7: Cross-section of a composite wire in transmission optical microscopy. The black spots are dead ending fibres whereas the bright spots are continuous fibres. The slice thickness is about 1.8 mm.
The brightness of the continuous fibres was found to be influenced by at least two additional factors, namely their orientation relative to the microscope axis, and possible fibre breaks where the fracture surface reflects a portion of the incoming light. However it was found that fibre breaks cannot be reliably detected with the setup at hand. Fibre misalignment on the other hand can readily be seen during live observation in the microscope: a misoriented fibre moves relative to its well-aligned neighbours when the focus of the microscope is slightly changed (see Figure C.9).

![Principle of the transmission optical microscopy (TOM) of alumina fibre reinforced composites.](image)

**Figure C.8:** Principle of the transmission optical microscopy (TOM) of alumina fibre reinforced composites.

![A misaligned fibre is moving relative to their neighbours when the focus on the microscope is changed in transmission optical microscopy. The slice thickness is about 1.8 mm.](image)

**Figure C.9:** A misaligned fibre is moving relative to their neighbours when the focus on the microscope is changed in transmission optical microscopy. The slice thickness is about 1.8 mm.

The method is used in this study to investigate initial damage that is present in the form of dead fibre ends in the composite material. Automated image analysis was performed using the

---

1 This slight reflection of light from the fracture surface can actually be detected in reflected light microscopy when the fibre break is very close to the specimen surface (< 15 µm). Such fibres are then clearly brighter than others. Unfortunately the probed volume is so small that the method cannot be used to make a quantitative measure of fibre breaks within the composite.
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UTHS CSA Image Tool program (developed at the University of Texas Health Science Center at San Antonio, Texas, and available from the Internet at http://ddsdx.uthscsa.edu/dig/) to count the number of black and white fibres in the image.

4.3 Fibre and Particle Volume Fraction Determination

The fibre volume fraction for the pressure-cast parallel tensile bars was determined from the specimen geometry (mainly imposed by the graphite mold) and the number of fibre tows, known by counting the number of fibre tows during the fibre winding process. The nominal number of fibres per tow is 400 to 420. Since some fibres might have been lost during all handling processes from fibre production through the fibre winding process, the fibre mass per meter was measured after the winding process. Knowing the fibre density, the tow volume per meter, and from the number of tows, the fibre volume fraction could be accurately calculated (to an experimental error of roughly ±0.5 vol.%).

The volume fraction of particles – added to some of the specimens as fibre spacers – is measured by comparing the mass per meter of a fibre tow with and without particles.

The fibre volume fraction for the composite wire was measured by densitometry; the estimated accuracy is ±0.6 vol.%.

5. Mechanical Characterization

5.1 Tensile Testing

Tensile tests were performed on the cast parallel tensile bars and on the composite wire in the fibre parallel direction. The tests were conducted on a computer-controlled screw-driven universal testing frame (Alliance RT/50 from MTS, MN, USA) with a 50 kN load cell (for parallel tensile bars) or a 5 kN load cell (for the composite wire). The machine was equipped with servohydraulic wedge grips attached to an alignment fixture that allowed minimizing the bending moments.

To avoid damage and fracture in the gripping section, aluminium tabs were glued on both ends of the cast parallel tensile bars (see Figure C.10). The composite wire was gripped directly in the hydraulic wedge grips using wooden inserts to protect the composite from damage in the gripping section.

![Figure C.10: Parallel tensile test specimen with glued aluminium end tabs.](image)

Tests were performed at constant crosshead speeds. The speed was chosen according to the specimen gauge length to have a comparable deformation rate in all tests of around $10^{-4}$ s$^{-1}$. 
Deformation was measured with a double-sided biaxial extensometer (gauge length: 25 mm) and occasionally with two strain-gauges glued on opposite sides of the specimen (see Figure C.11). Deformation readings from up to four channels were compared to evaluate bending and averaged to get the valid strain value.

Figure C.11: Strain measurement with double sided extensometre and strain gauges for the parallel tensile bars (left) and for the composite wire (right).

5.2 Measurement of Young’s Modulus

The evolution of Young’s modulus was measured on the parallel tensile bars by the following procedure. The composite was cycled twice between 1000 and -600 MPa. During these cycles, each 50 MPa, three unload-reload-cycles over a range of 150 MPa were performed. Finally the composite was loaded in tension to failure, still continuing the unload-reload cycles each 50 MPa.
5.3 Determination of the Stress-Strain Curves

For the determination of the stress-strain curve the composite was loaded up to 1000 MPa, unloaded to 10 MPa and -600 MPa for the wire material and the parallel tensile bars, respectively, and reloaded to 1000 MPa. This loop was usually repeated twice. The amount of compression applied depends on the thickness of the specimen because high compressive stresses on thin specimens induce buckling, making in turn the strain measurement unreliable.

5.4 Tensile Testing of Composite Wire at Elevated Temperatures

For measurement of their fracture strength, the composites were monotonically loaded to failure. The alumina fibre reinforced composite wire was additionally tested in tension at different temperatures ranging from room temperature up to 775 °C. From room temperature up to 175 °C an MTS environmental chamber was used, which exposed the grips to the same temperature as the specimen. Strain was measured with a standard clip-on extensometer. For higher temperatures the gauge length of the wire was heated by a home-built resistance tube furnace and the gripping section was kept cold. For the tests at 775 °C (which is above the melting temperature of the matrix) the length over which the matrix was melted was varied using different numbers of furnace zones and different furnace sizes. The liquid length varied between 15 and ~500 mm and was determined after testing by measuring the length of the unmolten parts and comparing with the initial specimen length (the molten and the unmolten regions were easily distinguished visually). Only crosshead displacement and load were measured at higher temperatures (i.e. there was no direct strain measurement).
D - RESULTS

1. Microstructure

1.1 Fibre Volume Fraction
The fibre volume fraction of the composite wire was determined by densitometry to be 49.7 ±0.6 vol.%. For the parallel tensile bars, the fibre volume fraction varied between about 40 and 70 vol.%. By counting the fibre tows during preform preparation, the volume fraction for the specific specimen was determined with a precision better than ±0.4 vol.%.

1.2 Particle Volume Fraction
Particles were added to some of the fibre preforms for parallel tensile bars. Their volume fraction was determined in the same way as the fibre volume fraction and found to vary between 1 and 2 vol.% of the composite (equivalent to 2 to 3.5 vol.% of the matrix).

1.3 Fibre Spatial Distribution
The global fibre spatial distribution in higher volume fraction composites \((V_f > 50 \text{ vol.\%})\) is quite homogeneous, although locally the fibre volume fraction varies to some extent, Figure D.1. The lower volume fraction specimens \((V_f < 50 \text{ vol.\%})\) always exhibit reinforcement free matrix regions and higher fibre volume fraction regions, where the fibres were pushed during pressure infiltration, Figure D.2.

![Figure D.1: Representative optical micrograph of a composite cross-section with \(V_f = 70 \text{ vol.\%} \) without particles. Variations in local fibre volume fraction are small yet visible.](image)

The effect of the alumina particles added to some of the parallel tensile bar preforms is shown in Figure D.2. They successfully reduced fibre-fibre contacts, although these did not completely disappear.
There was no difference found in fibre distribution between gas pressure infiltrated and direct squeeze cast specimens.

![Figure D.2: Cross-section of a low volume fraction specimen (Vf = 38 vol.%) (left hand side). The matrix flows through channels at the extremities of the specimen and pushes the reinforcement together resulting in large unreinforced matrix regions and higher Vf regions. Alumina particles reduced successfully fibre-fibre contact (right hand side).](image)

The composite wire shows a characteristic fibre distribution in the form of an “S” with some lower volume fraction regions. This feature is apparently related to the processing where a flat fibre tow bundle is folded to form the round wire: the low volume fraction regions are former surface regions. An equivalent diameter of 1.99 mm was determined by volume measurement of specimens with a length of about 70 mm.
1.4 Matrix Grain Size
The matrix grain size was determined by macrography using Keller’s etch to be in the range of 1-4 mm. The grain size is therefore about two orders of magnitudes greater than the other matrix characteristic, microstructural length, namely the interfibre distance.

1.5 Al-2%Cu Matrix Microstructure
The composite wire with the binary Al-2%Cu matrix in the as-cast condition contains a considerable amount of second phases – Al₂Cu according to the phase diagram – preferentially located at the fibre-matrix interface and in regions where two fibres are in close contact, Figure D.4. Solutionizing at 520 °C for 16 hrs followed by water quenching successfully dissolved most of these intermetallics; however there were still some intermetallics found at contact points between two fibres, Figure D.5.
Figure D.4: Backscattered electron micrograph (BSE) of a metallographically polished cross-section of the Al-2%Cu/Nextel 610 composite wire. The Al$_2$Cu intermetallics are clearly visible as white regions around some of the fibres.

Figure D.5: Backscattered electron micrograph (BSE) of a metallographically polished cross-section of the Al-2%Cu/Nextel 610 composite wire after solutionizing at 520 °C for 16 hrs and quenching in water.

1.6 Transmission Optical Microscopy

The pressure-cast parallel tensile bars showed only a negligible number of dead fibre ends. However a difference in brightness between distinct fibre regions with about 400 fibres each was clearly visible (Figure D.6).
The composite wire on the other hand contains a considerable amount of dead fibre ends, visible in TOM as black fibres in Figure D.7. The ratio of dead fibre ends to the total number of fibres in the cross-section is shown in Figure D.8 for three different slice thicknesses (three different volumes). The measurement indicates a linear increase in the number of dead fibre ends with thickness, as expected for a constant distribution of these in the wire volume. There is an offset at a slice thickness of zero mm. There is no change in this behaviour detected as the composite is prestressed to various stress levels up to values close to the composite fracture stress (it will be shown that fibre fragmentation occurs when the composites are strained; however, it is not detected by the TOM-method applied in this study).
Figure D.7: Transmission optical micrograph of the entire cross-section of a virgin composite wire (the equivalent diameter of the wire is 1.99 mm). The black spots are dead fibre ends, the bright spots are continuous fibres, the rest is matrix.

Figure D.8: Percentage of dead fibre ends as a function of slice thickness in a virgin composite wire.
A damage parameter $D_0$, denoting the number of fibre ends per mm of fibre, is defined as the slope of the curve in Figure D.8. This parameter takes a value of 0.0068 mm$^{-1}$ for the pure aluminium matrix composite wire. The parameter was not measured for the Al-2%Cu matrix composite wire.

2. Mechanical Characterization

2.1 Room Temperature Tensile Behaviour

A typical stress-strain curve (load-unload loop) of the composite systems tested in this study is shown in Figure D.9. All the systems showed similar curves exhibiting the same features. A slight knee point at a strain of about 0.05% (varying with fibre volume fraction) combined with a decrease of the slope was found in the first loading. Upon unloading a marked Bauschinger effect was observed. As shown in Figure D.10, upon cycling up to more than 600 times between fixed stress values the difference between the maximum and minimum strain values was constant. This indicates the absence of cyclic hardening or softening of the composite.

![Figure D.9: Stress-strain curve of a hpAl/Nextel 610 (V_r = 59.9 vol.%, hybridized) parallel tensile bar produced by direct squeeze casting. This curve is typical for all the composite systems tested in this study. All values are engineering.](image)
2.2 Ultimate Tensile Strength
The parallel tensile bars broke often just at, or behind, the beginning of the gripping section, seemingly due to stress concentrations. Tensile strength values can therefore only be viewed as a lower bound for the intrinsic specimen strength. Valid strength values also show considerable scatter, preventing identification of a clear trend for the composite strength as a function of the different matrix alloy composites (see Appendix 1). Generally the strength varied between 1200 and 1400 MPa for specimens with a fibre volume fraction of about 60 vol.%. The pure aluminium matrix composite wire has a mean strength of about 1380 MPa at room temperature. No effect of specimen size on strength was found within the scatter of the strength values.

2.3 Evolution of Young’s Modulus
The Young’s modulus evolution as measured by short unloadings on parallel tensile bars is illustrated in Figure D.11. It is found that the modulus decreases linearly with increasing strain regardless of the matrix composition. This behaviour is completely reversible as the strain is decreased and increased again. The same behaviour was also found for the composite wire.
Figure D.11: The modulus development as measured by short unloadings on parallel tensile bars a) with different matrix compositions and b) upon increasing and decreasing strain increments ($V_f = 59.9$ vol.%, hybridized matrix).

2.4 Tensile Strength at Elevated Temperature

The ultimate tensile strength of the pure aluminium matrix composite wire decreased monotonically with increasing temperature from room temperature up to $600 \, ^\circ\text{C}$. The length of the hot zone was in most of the tests about $100 \, \text{mm}$. As with the tests at room temperature any size effect in wire strength was hidden by experimental scatter.
2.5 Tensile Strength at $T > T_{m, \text{matrix}}$

The tensile strength of the pure Al / Nextel 610 composite wire at 775 °C is shown in Figure D.13. A clear strength decrease with increasing molten wire length is visible. It can additionally be seen in Figure D.13 that prestressing the wire before melting leads to a further decrease in strength.

Figure D.12: Ultimate tensile strength of the pure aluminium matrix composite wire as a function of test temperature (each data point represents the average of three to seven measurements).

Figure D.13: Influence of molten length and prestressing on the tensile strength of the pure aluminium matrix composite wire at 775 °C.
Figure D.14 shows the strength of the Al-2%Cu / Nextel 610™ composite wire at 775 °C. The strength decrease with increasing molten length $l$ in the as-cast condition (left) is roughly the same as for the pure Al matrix composite. The strength at shorter lengths is somewhat lower. The effect of prestressing, however, is much greater with the alloyed matrix. After solutionizing (Figure D.14 right) the strength at shorter molten length is greater again and reaches almost that of the pure aluminium matrix wire. The effect of prestressing is similar to that found in the pure aluminium matrix composite.

![Figure D.14: Fracture strength of the Al-2%Cu matrix composite wire in the a) as-cast and b) solutionized condition as a function of the molten length for different values of prestress.](image-url)
The composite fracture behaviour also changes gradually from abrupt fracture at shorter lengths to a smooth load decrease after maximum load at longer molten zone lengths, see Figure D.15. For specimens with molten length $l$ smaller than 50 mm, macroscopic failure of the specimen was usually observed at the solid liquid transition (Figure D.16). For $l \leq 50$ mm no macroscopic specimen separation was observed and a smoothly decreasing remanent wire flow stress after failure of up to 40 MPa was measured. This value varied roughly in proportion with molten length.

Figure D.15: Difference in fracture behaviour depending on the molten length of the specimens (pure Al matrix composite wire).

Figure D.16: Macroscopic failure at the solid-liquid transition for specimens with a molten length $l < 50$ mm (pure Al matrix composite wire). The upper specimen on the right picture was pulled apart after failure.
**E - DISCUSSION PART I:**
**COMPOSITE DEFORMATION AND *IN-SITU* MATRIX FLOW CURVE**

The importance of the matrix flow properties was outlined in the literature review together with some evidence that the *in-situ* flow behaviour of the matrix in the composite may greatly differ from that of the same unreinforced matrix material. In this section we draw from measurements of the composite flow stress to assess the *in-situ* deformation behaviour of the matrix in the present composites.

1. **General Deformation Behaviour**
   
The composites tested in this study invariably exhibit an essentially brittle tensile behaviour; however, there is some plastic deformation prior to rupture. Generally the stress-strain curves of fibre reinforced metals exhibit a Stage I where matrix and fibres behave in purely elastic fashion, and where the slope $E_{c,1}$ is roughly given by the rule of mixtures (ROM)

$$E_{c,1} = E_f \cdot V_f + E_m \cdot V_m$$  \hspace{1cm} (E.1)

where $E$ denotes the phase Young’s modulus, $V$ the phase volume fraction and the subscripts $f$ and $m$ refer to fibre and matrix, respectively. It was found that measured values for the composite Young’s modulus agree very well with this rule (see Figure B.1).

Depending on the fibre volume fraction there is a more or less explicit knee point in the composite stress-strain curve marking the transition to the Stage II. The slope of the curve in Stage II is not constant but rather determined both by the (usually decreasing) strain-hardening rate of the matrix and by the Young’s modulus of the fibre, Figure E.1.

The composite stress-strain curve can thus give access to the elasto-plastic matrix flow curve in the presence of the fibres. Deducing this curve from that of the composite is, however, a difficult task because it is inevitably computed by subtraction of two large numbers (the composite flow stress and the fibre contribution to this stress). For this reason, prior attempts to derive matrix *in-situ* curves in composites with ceramic fibres have yielded results which feature a large degree of relative uncertainty [51,47]. We attempt in what follows to reduce this uncertainty by close examination of several second-order effects which, if taken into account, improve significantly the final result. They concern the fibre contribution to the composite flow stress, and the mechanical relation between the composite and the matrix flow stress.
2. The Fibre Stress

2.1 Strain Dependence of Young’s Modulus

Figure D.11 presents the change of Young’s modulus of the composite as a function of strain. The measurement was done via short unloadings (compare Figure C.12). Both phases can be considered as being fully elastic in these unloadings. One observes a linear variation of the modulus as a function of strain, both in compression and in tension. This observation is consistent for all composite systems investigated in this study (apart from the Al-2%Cu composite wire, for which this measurement was not done). The behaviour was found to be completely reversible within the performed stress-strain loop (i.e. at a given composite strain, the same modulus is found regardless of strain history). To explain the observed behaviour two different hypotheses can be proposed:

Fibre Fragmentation

A gradually increasing number of broken fibres with strain would lead to a gradual decrease of the slope of the stress-strain curve of the composite. As the Young’s modulus is measured during the short unloading cycles, the matrix can be considered as being purely elastic. For an elastic matrix the effect of fibre breaks on the composite modulus is very small as long as fragmentation is not extensive. The effect can be estimated by a shear lag calculation [67]. For a fibre fragment length (mean axial distance between two breaks) of 10 mm the modulus decrease compared to an intact composite is only in the order of 1 to 2 GPa. Most importantly, this modulus decrease would persist when the strain is subsequently decreased, which is in contradiction with experimental data.
Elastic Non-linearity of Al$_2$O$_3$

The observed evolution of Young’s modulus can alternatively be attributed to the intrinsic elastic nonlinearity of the ceramic fibres. For uniaxial stress, second order elasticity for the tensile modulus of an isotropic material can be described using the following equation:

\[ \sigma_{f,\text{applied}} = E_f^0 \cdot \varepsilon + \frac{k}{2} \cdot \varepsilon^2 \]  

or in a differential form:

\[ \frac{d\sigma_{f,\text{applied}}}{d\varepsilon} = E_f^0 + k \cdot \varepsilon \]  

The experimental data can be well described with the producer’s data for the fibre Young’s modulus \( E_f^0 = 373 \) GPa, and a fitted nonlinearity parameter \( k = -2540 \) GPa. This value for \( k \) can be compared to literature values. Taking elastic constants from References [61,62] leads to \( E_f^0 = 403 \) GPa and \( k = -3506 \) GPa at \( \rho = 3.98 \) g/cm$^3$. This is slightly higher, but of the same order of magnitude, as the value of \( k \) measured here for the Nextel™ 610 alumina fibres. The discrepancy can be attributed to the lower density of the Nextel™ 610 fibres due to the presence of porosity in the fibres (their first order nominal modulus of 373 GPa is lower than 403 GPa for this same reason [64]), or perhaps to the fine-grained fibre structure (amorphous ceramic materials have a positive nonlinearity parameter \( k \) [144,145]).

In conclusion, the observed decrease of Young’s modulus with increasing strain is therefore attributed to the non-linear elastic behaviour of the alumina fibres.

2.2 The Effect of Fibre Fragmentation on the Effective Fibre Young’s Modulus

As mentioned above, the effect of fibre fragmentation on the elastic composite modulus is very small. As soon as the matrix undergoes plastic deformation, however, the shear transfer length increases significantly. The fibres then carry over a non-negligible length a stress that is lower than the applied fibre stress. This effect can be described as equivalent to an ineffective fibre volume fraction, which is easily calculated from a simplified shear lag approach for a plastic matrix.

Assuming a constant shear stress in the matrix equal to the shear yield strength of the matrix, \( \tau_{y,m} \), the axial stress in the fibre builds up linearly from zero at the fibre break to the applied fibre stress \( \sigma_{f,\text{applied}} \) at a distance \( \delta \) (Figure E.2, solid gray line). This distance is easily calculated from a simple force equilibrium and is

\[ \delta = \frac{\sigma_{f,\text{applied}} \cdot R_f}{2 \cdot \tau_{y,m}} \]  

where \( R_f \) is the fibre radius. For the axial integral of the stress borne by the fibre the stress profile can be approximated as a rectangular profile (Figure E.2 dashed line). The effective stress-bearing fibre length is then deduced. From this, an effective fibre stress (Figure E.2, solid black line) can be defined by the following condition:
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\[ \sigma_{f,\text{eff}} = \frac{(l_b - \delta)}{l_b} \cdot \sigma_{f,\text{applied}} \]  \hspace{1cm} (E.5)

where \( l_b \) is the mean axial distance between two fibre breaks. Differentiating this equation and using Equation (E.2) for the fibre stress leads to a formulation for an effective fibre Young's modulus due to fibre fragmentation:

\[ E_{f,\text{eff}} = E_f \left(1 - \frac{\delta}{l_b}\right) = E_f \cdot \left(1 - \frac{\sigma_{f,\text{applied}} \cdot r_f}{2 \cdot \tau_{y,m} \cdot l_b}\right) = \left(\frac{E_f^0 + k \cdot \varepsilon}{2 \cdot \tau_{y,m} \cdot l_b}\right)^2 \]  \hspace{1cm} (E.6)

For an initially damage-free composite the fragmentation length can be calculated using Weibull statistics (as will be shown in the next chapter, this is a valid approach for the present composites). The fragmentation length is then simply the inverse failure probability of a fibre of unit length \( l_0 = 1 \text{ mm} \).

\[ l_b = \left\{1 - \exp \left[-\frac{l_b}{l_0} \cdot \left(\frac{\sigma_{f,\text{applied}}}{\sigma_0}\right)^n\right]\right\}^{-1} \]  \hspace{1cm} (E.7)

It can be seen that for an initially non-damaged composite, at a strain of \( \varepsilon = 0.6\% \), corresponding to \( \sigma_{\text{applied}} = 2200 \text{ MPa} \), the mean distance between fibre breaks is on the order of magnitude of meters. Assuming a shear yield strength in the matrix of about 20 MPa, the effective fibre modulus is very close to the fibre modulus \( E_f^0 \): the difference is at most about 0.03 GPa. In this case the correction can safely be neglected because the uncertainty in matrix work hardening rate is well above this value.

For the case of the composite wire, where initial damage was detected by transmission optical microscopy (TOM), \( l_b \) is already initially much lower. From TOM data (Section D - 1.6) the mean distance between two fibre ends \( l_b \) can be calculated as the inverse of the number of fibre ends per mm \( D_0 \): this is on the order of 150 mm in the wire. The reduction of \( l_b \) due to damage accumulation during straining is, as shown above, very small within the region where the in-situ matrix flow curves are evaluated. The fibre fragment length can therefore be taken as constant in the wire. In this case the effective fibre Young’s modulus is lowered by 0.22% (at a strain of 0.6%) compared to the uncorrected fibre Young’s modulus. This effect thus introduces a negative (underestimation) error of about 0.5 GPa in the wire matrix work hardening rate at 0.6% strain upon initial loading.

The difficulty with this effect is that, if the stress state sketched in Figure E.2 is quite valid for monotonic loading of the composite, upon unloading the stress state on a broken fibre near the fibre break becomes far more complex. Taking this effect into account for cyclic loading is, thus, far more difficult. Since an error of 0.5 GPa is below the total uncertainty in the matrix
in-situ work hardening rate, the effect of fibre breaks will hereafter be neglected in computing the matrix in-situ flow curve from that of the composite.

Figure E.2: Stress profile on a broken fibre according to a simplified shear-lag approach for a perfect plastic matrix.

3. The Composite Flow Stress

3.1 Deviation of the Composite Flow Stress from the ROM

At the simplest level of analysis, the composite stress is approximately given by the rule of mixture:

\[ \sigma_c = \sigma_f V_f + \sigma_m (1-V_f) \]  

(E.8)

As the fibres are in all cases deforming in purely elastic fashion, the fibre stress is calculated from the composite strain using Equation (E.2). The matrix stress could then easily be calculated if Equation (E.8) were valid; however, the phase Poisson’s ratios of matrix and reinforcement differ by a non-negligible amount \( (\nu_f = 0.235, \nu_m = 0.345 \) and more when the matrix undergoes plastic deformation). Hence the lateral contraction mismatch in the composite cannot be neglected and the ROM is not an exact solution for back-calculation of the matrix flow stress knowing that of the composite, as was pointed out earlier by Kelly and Lilholt [111] and Bystricky et al. [47]. Hill’s bounds [58,65] for the effective longitudinal modulus of a continuous fibre reinforced composite are used to account for this effect in the elastic regime of deformation. These bounds are the most precise calculation of the composite modulus as long as an arbitrary transverse fibre distribution is considered. They represent two extreme cases of fibre configuration, namely a single fibre surrounded by matrix (lower composite modulus) and a tube of fibre material filled with matrix (upper composite modulus; this upper bound can be viewed as relevant to the case of a close-packed array of touching fibres). These bounds are always positive; consequently, the real composite modulus always deviates above the ROM.

Hill gives analytical solutions for the cases where both phases are elastic (Equation (E.9)) or where one of the phases is fully plastic with zero strain-hardening rate (Equation (E.10)). For
the latter case the slope of the matrix stress-strain curve at the composite strain is considered as an instantaneous modulus and the Poisson’s ratio of the plastic phase is set to 0.5. The bounds are then wider as compared to the elastic case because the difference in the (apparent) Poisson’s ratios between the phases is larger. All other constants retain their elastic values. Numerical results for the bounds for several fibre volume fractions are summarized in Table E.1.

\[
\frac{4V_mV_f}{V_m + V_f + \frac{1}{k_f}} \left( v_f - v_m \right)^2 \leq E_c - E_fV_f - E_mV_m \leq \frac{4V_mV_f}{V_m + V_f + \frac{1}{k_f}} \left( v_f - v_m \right)^2
\]  
(E.9)

\[
\frac{V_fV_m \left( 1 - 2v_f \right)^2}{\left( \frac{1}{k_m} \right) \left( \frac{1}{k_f} + \frac{1}{G_m} \right) \left( \frac{1}{k_f} + \frac{1}{G_f} \right)} \leq \frac{d\sigma_f}{d\varepsilon} - \frac{d\sigma_m}{d\varepsilon} = \frac{d\sigma_m}{d\varepsilon} - \frac{d\sigma_f}{d\varepsilon} \leq \frac{V_fV_m \left( 1 - 2v_f \right)^2}{\left( \frac{1}{k_m} \right) \left( \frac{1}{k_f} + \frac{1}{G_m} \right) \left( \frac{1}{k_f} + \frac{1}{G_f} \right)}
\]  
(E.10)

Table E.1: Bounds for the deviation from the ROM of the composite longitudinal response, calculated for elastic and purely plastic matrix according to Equations (E.9) and (E.10) for different \( V_f \).

<table>
<thead>
<tr>
<th>( V_f )</th>
<th>Equation (E.9)</th>
<th>Equation (E.10)</th>
</tr>
</thead>
<tbody>
<tr>
<td>lower</td>
<td>upper</td>
<td>lower</td>
</tr>
<tr>
<td>50 vol.%</td>
<td>0.26 GPa</td>
<td>0.81 GPa</td>
</tr>
<tr>
<td>60 vol.%</td>
<td>0.24 GPa</td>
<td>0.73 GPa</td>
</tr>
<tr>
<td>70 vol.%</td>
<td>0.20 GPa</td>
<td>0.57 GPa</td>
</tr>
</tbody>
</table>

It is seen in Table E.1 that Hill’s bounds are quite close compared to the Young’s modulus or the slope of the stress-strain curve of the composite (which is between 200 and 300 GPa). On the other hand – and more important for the purposes of the present analysis – the bounds are quite far apart when back-calculation of the matrix flow curve is intended, since the matrix has a strain-hardening rate of only a few GPa. A more precise relation between composite and matrix flow stress is therefore developed in the following by means of finite element analysis (FEA).

### 3.2 Finite Element Analysis of Composite Flow Stress

**Bounds in Analogy to Hill’s Method**

By analogy to Hill’s calculation a hexagonal fibre array is investigated by finite element analysis (FEA). The unit cell of the model is shown in Figure E.3. The stiffness of this array is evaluated under generalized plane strain conditions, for fibre and matrix both elastic and for an elastic fibre and an elastic-plastic matrix. This configuration is the analogue to Hill’s lower bound. To create an analogue with Hill’s upper bound, the phases are simply inverted. Table E.2 summarizes the input parameters together with the analytical results calculated by Equation (E.9) or (E.10) and the numerical results from FEA. The calculation was performed for two purely elastic cases, namely for a stiff matrix (\( E_m = 70 \) GPa), for a very soft, but still elastic matrix (\( E_m = 0.007 \) GPa), and for an elastic-plastic matrix with a constant strain-
hardening rate $\theta_m$. In this case the tangential composite strain-hardening rate of the FEA output between 0.45 and 0.50% strain was measured. For the analytical computation, the constant strain-hardening input was used in the ROM and a Poisson’s ratio of 0.5 was used to calculate the bounds that are added to the ROM-result.

Perfect agreement was found between results of these two finite-element simulations and Hill’s bounds for the purely elastic case.

In the elastic-plastic case the upper bounds are still in good agreement; however, it is found that the analytical lower bound is considerably above the curve predicted by finite element analysis. This implies that the extension of the elastic bounds to plastic deformation proposed as an approximation by Hill [65] is in fact not reliable.

![Figure E.3: Unit cell for a hexagonal fibre array for FEM stiffness evaluation in analogy to Hill’s analytical calculation.](image)

| $V_f$ (%)| $E_f$ (GPa)| $E_m$ (GPa)| $\sigma_m$ (MPa)| $\theta_m$ (GPa)| $\nu_m$| $E_o$ or $\theta$ from Equation (E.9) or (E.10) (GPa)| bounds from FEA lower (GPa)| bounds from FEA upper (GPa) |
|----------|------------|------------|-----------------|----------------|------|-----------------|----------------|----------------|------|
| fibre and matrix elastic | | | | | | | | |
| 50.3204 | 373 | 70 | - | - | 0.235 | 0.345 | 222.47 | 0.26 | 0.80 | 0.26 | 0.84 |
| 68.5172 | - | - | - | - | - | - | 277.61 | 0.21 | 0.62 | 0.22 | 0.65 |
| 50.3204 | 70,10$^6$ | - | - | - | 0.4999999 | 187.70 | 0.00 | 5.54 | -0.01 | 5.51 |
| 68.5172 | - | - | - | - | - | 255.57 | 0.00 | 4.46 | 0.00 | 4.45 |
| fibre elastic, matrix elastic-plastic with constant strain hardening | | | | | | | | |
| 50.3204 | 373 | 70 | 20 | 0.007 | 0.235 | 0.345 | 187.70 | 1.50 | 4.67 | 0.46 | 4.88 |
| 68.5172 | - | - | - | - | - | - | 255.57 | 1.25 | 3.60 | 0.46 | 3.67 |

Hill’s proposed analytical lower elastic-plastic bound is based on an approximation for the Von Mises yield criterion where any shear stresses in the plane perpendicular to the fibre axis are neglected. This approximation – certainly acceptable when evaluating the flow stress of the composite from the phase properties – slightly overestimates the flow stress of the matrix. This provides an explanation for why Hill’s lower elastic-plastic “bound” is in fact not a rigorous lower bound. In the case of the upper bound, the shear stresses in the matrix are negligible and the agreement with FEA is acceptable.
A Modified Upper Bound

To determine the upper bound of the axial Young’s modulus Hill considers a simple but not very realistic scenario of a cylindrical tube made of the fibre material filled with matrix. Although this bound is physically possible, in a fibre composite the hard phase geometry is fixed since it must take the shape of a circular cylinder. One could, therefore, aim to narrow somewhat the bounds on the composite flow stress by searching for the most rigid configuration knowing that the fibres must retain their shape. This configuration is the one that introduces the maximum stress triaxiality in matrix: in turn, this is to be expected when fibres contact one another to the greatest possible extent.

Regular close-packed fibre arrangements, in which the entire matrix is found in small isolated regions surrounded by touching fibres, are therefore expected to provide the highest composite flow stress. Since touching fibres are laterally less rigid than a cylindrical tube of fibre material (the contact-area between two fibres consists essentially of a line where compressive stresses and associated strains can become very important), we expect these configurations to be less rigid than a tube of reinforcement surrounding a cylinder of matrix.

To quantify this difference between Hill’s upper bound and the proposed modified upper bound, the hexagonal unit cell from Figure E.3 was modified by increasing the fibre volume fraction to the limiting case of $V_f = 90.7\ \text{vol.\%}$; this results in the hexagonal closed fibre packing (Figure E.4). A new square unit cell with a fibre volume fraction of 78.5 vol.% was also drawn to simulate square fibre packing (Figure E.5). The resulting Young’s modulus or strain-hardening rate $\theta_e$ for the purely elastic and the elastic-plastic calculation were compared to the result from Equations (E.9) and (E.10). The positive deviation of the FEA results from Hill’s lower bound can be given as a simple percentage from the difference between Hill’s upper and lower bound. The numerical results are given in Table E.3 (hexagonal close-packed) and Table E.4 (square packing).

Figure E.4: Unit cell for a hexagonal closed packed fibre array.
Discussion Part 1: Composite Deformation and In-situ Matrix Flow Curve

Table E.3: Modified upper bound from the hexagonal closed fibre packing compared to Hill's lower and upper bound.

<table>
<thead>
<tr>
<th>$V_f$ (%)</th>
<th>$E_f$ (GPa)</th>
<th>$E_m$ (GPa)</th>
<th>$\sigma_{\text{m}}$ (MPa)</th>
<th>$\theta$ (GPa)</th>
<th>$\nu_f$</th>
<th>$\nu_m$</th>
<th>$E_i$ or $\theta_i$ from Equation (E.9) or (E.10) (GPa)</th>
<th>ROM+ lower</th>
<th>ROM+ upper</th>
<th>$E_i$ or $\theta_i$ from FEA (GPa)</th>
<th>deviation from Hill's lower bound</th>
</tr>
</thead>
<tbody>
<tr>
<td>fibre and matrix elastic</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.906903</td>
<td>373</td>
<td>70</td>
<td>70.10 $10^{-6}$</td>
<td>-</td>
<td>0.235</td>
<td>0.345</td>
<td>344.79</td>
<td>344.87</td>
<td>345.01</td>
<td>344.93</td>
<td>43%</td>
</tr>
<tr>
<td>fibre elastic, matrix elastic-plastic with constant strain hardening</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.906903</td>
<td>373</td>
<td>70</td>
<td>20</td>
<td>0.007</td>
<td>0.235</td>
<td>0.345</td>
<td>338.27</td>
<td>338.27</td>
<td>339.89</td>
<td>338.44</td>
<td>10%</td>
</tr>
</tbody>
</table>

Figure E.5: Unit cell for the FEA of the square fibre packing.

Table E.4: Modified upper bound from the square fibre packing compared to Hill's lower and upper bound.

<table>
<thead>
<tr>
<th>$V_f$ (%)</th>
<th>$E_f$ (GPa)</th>
<th>$E_m$ (GPa)</th>
<th>$\sigma_{\text{m}}$ (MPa)</th>
<th>$\theta$ (GPa)</th>
<th>$\nu_f$</th>
<th>$\nu_m$</th>
<th>$E_i$ or $\theta_i$ from Equation (E.9) or (E.10) (GPa)</th>
<th>ROM+ lower</th>
<th>ROM+ upper</th>
<th>$E_i$ or $\theta_i$ from FEA (GPa)</th>
<th>deviation from Hill's lower bound</th>
</tr>
</thead>
<tbody>
<tr>
<td>fibre and matrix elastic</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.785398</td>
<td>373</td>
<td>70</td>
<td>70.10 $10^{-6}$</td>
<td>-</td>
<td>0.235</td>
<td>0.345</td>
<td>307.98</td>
<td>308.14</td>
<td>308.43</td>
<td>308.31</td>
<td>59%</td>
</tr>
<tr>
<td>fibre elastic, matrix elastic-plastic with constant strain hardening</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.785398</td>
<td>373</td>
<td>70</td>
<td>20</td>
<td>0.007</td>
<td>0.235</td>
<td>0.345</td>
<td>292.96</td>
<td>293.91</td>
<td>295.61</td>
<td>294.21</td>
<td>18%</td>
</tr>
</tbody>
</table>

It is clearly seen from the results for the elastic case that Hill’s upper bound yields an overestimate of the highest expected axial stiffness of a continuous fibre reinforced composite. The Young’s modulus evaluated by the contacting-fibre hexagonal and square packings is roughly...
near the average between Hill’s lower and upper bound. Since it is not expected that a real composite will exhibit a configuration stiffer than the ones presented in Figure E.4 and Figure E.5, it is concluded for the elastic case that Hill’s upper bound can be replaced by the upper bound found by FEA without losing much universality for cylindrical fibre reinforced composites.

For the elastic-plastic case the same observation is made as for the hexagonal packing with lower fibre volume fraction: even though all matrix is found in isolated regions surrounded by touching fibres, the FEA results in an output that is quite close or even below the analytical lower bound. The reason for this is, as mentioned, the overestimation of the matrix flow stress due to a simplified yield criterion in Hill’s analytical bounds.

*FEA of a Complex Unit Cell*

To determine which of the two bounds is closer to the real fibre spatial distribution present in the composites, various complex cells containing 52 fibres and one cell containing 71 fibres were evaluated by FEA. The cells are represented in Figure E.6. Their fibre volume fraction is ~50 vol.% and ~69 vol.%, respectively. The cells differ in the number of fibres touching each other and in the area of matrix surrounded by fibres touching each other (isolated matrix regions). Touching fibres have one common node. Hence, they cannot slide relative to each other but the configuration can be bent to some extent (the node has no rotative stiffness). The stiffness of the cell is evaluated under generalized plane strain conditions assuming both phases are purely elastic, and also for elastic fibres and an elastic-plastic matrix. Results from these simulations are compared to the bounds determined by the hexagonal fibre array (Table E.2) and Hill’s analytical bounds.
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Figure E.6: Complex cells used for FEM. Their fibre spatial distributions are representative for the composites investigated during this study. a) a cell with extensive fibre-fibre contact and some almost surrounded by touching fibres ($V_f = 50$ vol.%); b) a cell with some completely isolated matrix regions ($V_f = 50$ vol.%); c) a cell with no fibre-fibre contact ($V_f = 50$ vol.%); d) a cell with extensive fibre-fibre contact and a higher fibre volume fraction ($V_f = 68$ vol.%).

The results of the analysis of the complex unit cells are summarized in Table E.5. It is clearly seen that the behaviour of all four complex cells is closer to the lower bound than to the upper bound. This means that the cell is quite "soft" in the lateral direction and that deformation of the matrix is not as extensively hindered by the presence of touching fibres as it is in a perfectly hexagonal array.

The expected trend discussed above is moreover clearly visible when comparing the results from cells a) to c) in Figure E.6. The complex cell with no touching fibres (Figure E.6 c) is the softest. Its behaviour is very close to the behaviour of a perfect hexagonal array, although the fibre distribution is not very regular.

The configuration where some matrix regions are surrounded by touching fibres (Figure E.6 b) exhibits the highest Young's modulus, because lateral deformation of the isolated matrix regions is hindered by the fibre rings surrounding them. These additional stresses perpendicular to the loading direction reduce the equivalent von Mises stress and raise the material stiffness or flow stress locally. Looking at the stress triaxiality level (defined as the hydro-
static stress divided by the equivalent von Mises stress) in the matrix, Figure E.7, it is clearly seen that isolated matrix regions that are almost circular exhibit a higher triaxiality than the rest of the matrix. On the other hand, if the surrounding fibres are not in a nearly circular arrangement but rather in a more ellipse-shaped configuration, triaxiality is hardly greater than in the rest of the matrix. This configuration is relatively soft in the transverse direction and can deform and follow to some extent the lateral contraction of the matrix. This effect is probably reinforced in the real composite, where fibres can also slide relative to each other (this was, as mentioned, not allowed in the present FEA). Figure E.8 shows the triaxiality in the fibres for the same stress state and the same unit cell as in Figure E.7. A lower triaxiality—lowered by contact pressure— is found at the contact lines between touching fibres, and along the interface with “circular” isolated matrix regions.

The cell with some fibre-fibre contact but only limited isolated matrix regions, Figure E.6 a) is considered to be the most realistic cell for composites with a fibre volume fraction of about 50 vol.% (its design was inspired by micrographs from the continuous wire composite). It can be seen that its modulus is only slightly higher than that of cell c) where no fibre to fibre contact is present.

Table E.5: Summary of input parameters and results for the evaluation of the complex unit cells.

<table>
<thead>
<tr>
<th>$\nu_f$ (%)</th>
<th>$E_f$ (GPa)</th>
<th>$E_m$ (GPa)</th>
<th>$\sigma_m$ (MPa)</th>
<th>$\theta$ (GPa)</th>
<th>$\nu_r$</th>
<th>unit cell (Figure E.6)</th>
<th>$E_c$ or $\theta_c$ from ROM (GPa)</th>
<th>$E_c$ or $\theta_c$ from FEA of the hexagonal array (GPa)</th>
<th>$E_c$ or $\theta_c$ from FEA complex unit cell (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>50.3204</td>
<td>373</td>
<td>70</td>
<td>-</td>
<td>-</td>
<td>0.345</td>
<td>a)</td>
<td>222.47</td>
<td>222.73</td>
<td>222.82 (16%)</td>
</tr>
<tr>
<td>68.5172</td>
<td>70</td>
<td>-</td>
<td>-</td>
<td>0.4999999</td>
<td>0.235</td>
<td>b)</td>
<td>187.70</td>
<td>187.69</td>
<td>187.84 (3%)</td>
</tr>
<tr>
<td>50.3204</td>
<td>70-10$^{-4}$</td>
<td>70-10$^{-4}$</td>
<td>-</td>
<td>-</td>
<td>0.4999999</td>
<td>c)</td>
<td>255.57</td>
<td>255.57</td>
<td>255.89 (7%)</td>
</tr>
<tr>
<td>68.5172</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>d)</td>
<td>187.70</td>
<td>187.69</td>
<td>187.84 (3%)</td>
</tr>
</tbody>
</table>

fibre elastic, matrix elastic-plastic with constant strain hardening

<table>
<thead>
<tr>
<th>$\nu_f$ (%)</th>
<th>$E_f$ (GPa)</th>
<th>$E_m$ (GPa)</th>
<th>$\sigma_m$ (MPa)</th>
<th>$\theta$ (GPa)</th>
<th>$\nu_r$</th>
<th>unit cell (Figure E.6)</th>
<th>$E_c$ or $\theta_c$ from ROM (GPa)</th>
<th>$E_c$ or $\theta_c$ from FEA of the hexagonal array (GPa)</th>
<th>$E_c$ or $\theta_c$ from FEA complex unit cell (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>50.3204</td>
<td>373</td>
<td>70</td>
<td>0.007</td>
<td>0.235</td>
<td>0.345</td>
<td>a)</td>
<td>187.70</td>
<td>188.16</td>
<td>188.22 (1%)</td>
</tr>
<tr>
<td>68.5172</td>
<td>20</td>
<td>0.007</td>
<td>0.235</td>
<td>0.345</td>
<td>-</td>
<td>b)</td>
<td>188.70</td>
<td>188.36</td>
<td>188.36 (5%)</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>c)</td>
<td>188.70</td>
<td>188.36</td>
<td>188.36 (5%)</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>d)</td>
<td>255.57</td>
<td>256.03</td>
<td>256.23 (6%)</td>
</tr>
</tbody>
</table>
**Figure E.7:** Matrix stress triaxiality in the complex unit cell of Figure E.6 b) from FEA for a matrix with a yield stress of 20 MPa and a constant strain hardening rate of 3 GPa. The output corresponds to an axial strain of 0.6%.

**Figure E.8:** Stress triaxiality in the fibres in the complex unit cell of Figure E.6 b) from FEA for a matrix with a yield stress of 20 MPa and a constant strain hardening rate of 3 GPa. The output corresponds to an axial strain of 0.6%.
Comparison of the Complex Cells with Micrographs

Examples of the spatial distribution of the fibres in the continuous composite wire are shown in Figure E.9. By carefully investigating these microstructures one can draw the following conclusions:

- There is a number of fibres touching each other or situated very close to one another.
- The total area of isolated matrix regions is small and only few of them are surrounded by touching fibres in a nearly circular configuration.
- The complex cell in Figure E.6 a) is thus a representative configuration for the wire material.

![Micrographs](image)

*Figure E.9: Micrographs showing representative spatial fibre distributions in the continuous composite wire. The fibre diameter is ~11.5 µm.*

The complex cell shown in Figure E.6 c) can be compared with the micrograph shown in Figure D.1 in Section D - 1.3. The area fraction of isolated matrix regions in actual composite micrographs is seen to be small, and hence similar to what is simulated using the complex cell given in Figure E.6 c). This cell thus provides a realistic model for modelling the behaviour of higher volume fraction composites (with a fibre volume fraction greater than 60 vol.%).
3.3 Back-calculation the Matrix in-situ Flow Curve

Conclusions from FEA

To back-calculate the matrix flow curve from that of the composite, we make the simplifying assumption that, in each of the elastic and elastic-plastic matrix deformation regimes, the deviation, \( \alpha \), of the composite rate of work hardening from the rule of mixtures:

\[
\frac{d\sigma_c}{d\varepsilon} = \frac{d\sigma_f}{d\varepsilon} V_f + \frac{d\sigma_m}{d\varepsilon} (1-V_f) + \alpha
\]

is constant. This is an assumption; however, it provides a method for back-calculating the matrix in-situ average stress-strain curve without having to perform for each sample a series of iterative finite element computations.

By finite element computation of the stiffness of complex unit cells it was found that the deviation from the ROM is significantly closer to Hill’s lower bound than to Hill’s upper bound in the elastic case. Results from the calculations suggest that it is appropriate for fibre volume fractions around 50 vol.% to use the lower bound plus 16% of the difference between Hill’s upper and lower elastic bound to back-calculate the in-situ matrix flow curve during elastic deformation.

For the elastic-plastic regime, the analytical bounds developed by Hill are not rigorous: the strain-hardening of the complex cells is found to be well below the lower Hill bound. This error is small when evaluating the flow curve of the composite from the phase properties but cannot be accepted for the inverse problem, where the flow stress of the matrix is back-calculated from the composite stress-strain curve.

To estimate realistic constant values for \( \alpha \), the deviation from the ROM for the complex cells in Figure E.6 a) and d) was calculated using \( E_f = 373 \) GPa, \( E_m = 70 \) GPa, \( \sigma_{f,m} = 20 \) MPa and \( \theta_m = 3 \) GPa as input parameters. The values for \( \alpha \) found by this procedure were 0.45 GPa and 0.70 GPa, for the complex unit cells in Figure E.6 a) and d) with \( V_f \approx 50 \) vol.% and \( V_f \approx 68 \) vol.% respectively.

The in-situ matrix flow curves were then computed from the composite stress-strain curve using Equation (E.12). This equation accounts for the non-constant Young’s modulus of the fibre (Equation (E.3)) and for the deviation from the ROM, quantified using the estimated values for parameter \( \alpha \); the value of the elastic lower Hill bound plus 16% for the elastic regime of matrix deformation, and the values determined by FEA outlined above for the elastic-plastic regime:

\[
\frac{d\sigma_m}{d\varepsilon} = \frac{\frac{d\sigma_c}{d\varepsilon} - \left( \frac{E_f^0 + k \cdot \varepsilon}{1-V_f} \right) \cdot V_f - \alpha}{(1-V_f)}
\]

Influence of the Input Parameters

Being the difference between two big numbers, the result of the back-calculation is very sensitive to the input parameters fibre Young’s modulus and fibre volume fraction. The fibre vol-
Volume fraction was carefully measured to an uncertainty of ±0.4 vol.%, as detailed in Section C-4.3. Figure E.10 illustrates the effect of such an error on the back-calculated *in-situ* matrix curve. Adding to this uncertainty an – not easily quantifiable – uncertainty in the fibre Young’s modulus makes a precise calculation of the *in-situ* flow curve quite challenging.

This uncertainty can be lowered by checking the matrix Young’s modulus –measured in the elastic regime of the back-calculated matrix flow curve – for consistency. Elastic properties are not very structure-sensitive and it is therefore legitimate to assume that the elastic properties of the phases are not affected by the presence of one another. It is hence concluded that the matrix Young’s modulus measured from the elastic part of the *in-situ* matrix flow curve has to correspond to literature values (which are quite nearly isotropic for aluminium, and will not vary much with strain since, unlike the fibres, the matrix stresses are relatively small). Values for the stiffness of pure aluminium were taken from [62] and converted to engineering constants for polycrystals using the Hashin-Shtrikman bounds. These bounds are 69.80 GPa and 69.84 GPa, respectively. To account for the influence of fibre preform hybridization, which results in the presence of small ceramic particles in the matrix of some composites, the modulus increase as a function of particle volume fraction was calculated with the Hashin-Shtrikman bounds and a 3-phase self-consistent scheme. The following table shows upper and lower bound for three different particle volume fractions used in the production of hybrid composites, for a nominal matrix modulus of 70 GPa.
Table E.6: The matrix Young's modulus as a function of the particle volume fraction according to the Hashin-Shtrikman bounds and a 3D-self-consistent scheme.

<table>
<thead>
<tr>
<th>particle volume fraction</th>
<th>Hashin-Shtrikman lower bound</th>
<th>Hashin-Shtrikman upper bound</th>
<th>3-phase-self-consistent</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.0 %</td>
<td>72.08 GPa</td>
<td>74.09 GPa</td>
<td>72.08</td>
</tr>
<tr>
<td>2.4 %</td>
<td>72.50 GPa</td>
<td>74.91 GPa</td>
<td>72.50</td>
</tr>
<tr>
<td>2.8 %</td>
<td>72.92 GPa</td>
<td>75.74 GPa</td>
<td>72.93</td>
</tr>
</tbody>
</table>

To measure the composite modulus, unload – reload loops are particularly suitable as they exhibit extended elastic deformation of the matrix when going from tension to compression or vice-versa. A typical loop of an in-situ matrix stress-strain curve is shown in Figure E.11 (qualitative drawing). Using Equation (E.12) it is verified that the matrix Young's modulus at different composite strains remains constant when – and only when – the elastic nonlinearity of the alumina fibres is considered.

The fibre volume fraction can now be calculated so as to yield a correct matrix Young's modulus and – as a consequence – an in-situ matrix flow curve with a minimum of $V_f$-related experimental error.

![Figure E.11: Loop of in-situ matrix stress-strain curve used for comparing the matrix Young's modulus measured from the back-calculated curve with the literature value.](image)

### 3.4 Pure Aluminium Matrix

The in-situ matrix curves for the different specimens produced with pure aluminium matrices are shown in Figure E.12 and Figure E.13. The fibre volume fraction and the corresponding matrix Young's moduli measured upon unloading and reloading are summarized in Table E.7. It is seen that the fibre volume fraction used for the back-calculation is not always within experimental error of the measured fibre volume fraction. The deviation, however, is small and the estimation of experimental error for the fibre volume fraction measurements might have been optimistic.
Table E.7: Fibre volume fractions measured and used for the back-calculation of the in-situ matrix flow curves together with the matrix Young's moduli measured from the elastic part of the matrix stress-strain curves (for Figure E.12 and Figure E.13).

<table>
<thead>
<tr>
<th></th>
<th>( V_f ) measured</th>
<th>( V_f ) used for back-calculation</th>
<th>( E_m ) in-situ unload</th>
<th>( E_m ) in-situ reload</th>
<th>( E_m ) theoretical</th>
</tr>
</thead>
<tbody>
<tr>
<td>Figure E.12 DSC</td>
<td>59.9±0.4 vol.%</td>
<td>59.65 vol.%</td>
<td>72.9 GPa</td>
<td>68.3 GPa</td>
<td>72.75 GPa</td>
</tr>
<tr>
<td>Figure E.12 GPI</td>
<td>59.9±0.4 vol.%</td>
<td>58.80 vol.%</td>
<td>72.5 GPa</td>
<td>71.3 GPa</td>
<td>72.75 GPa</td>
</tr>
<tr>
<td>Figure E.13 a)</td>
<td>71.3±0.5 vol.%</td>
<td>72.35 vol.%</td>
<td>70.3 GPa</td>
<td>69.1 GPa</td>
<td>69.8 GPa</td>
</tr>
<tr>
<td>Figure E.13 b)</td>
<td>49.7 vol.%</td>
<td>49.9 vol.%</td>
<td>70.1 GPa</td>
<td>69.9 GPa</td>
<td>69.9 GPa</td>
</tr>
</tbody>
</table>

Figure E.12: In-situ matrix flow curves for a direct squeeze cast (DSC) and a gas pressure infiltrated (GPI) parallel tensile bar with a fibre volume fraction of about 60 vol.%. The matrix is hybridized with about 2.3 vol.% alumina particles.

The yield stress of the matrix can be estimated as half the vertical distance between the two parallel lines for the linear strain-hardening in tension and compression. It is, for both specimens, on the order of 45 MPa. Overall it can be seen that the in-situ matrix behaviour for a pure aluminium matrix is the same for both infiltration methods used. The high rate of strain-
hardening compared with unreinforced aluminium can partly be attributed to the hybridization of the matrix, which itself is in fact a low volume fraction particle reinforced composite.

Figure E.13 shows in-situ matrix flow curves for a) pure aluminium matrix reinforced with ~70 vol.% Nextel 610™ and for b) the continuous composite wire with a pure aluminium matrix and a fibre volume fraction of about 50 vol.%. Both matrices do not contain particles. The same features as in Figure E.12 can be observed. The yield stress is about 50 MPa, and work-hardening is kinematic, with no cyclic hardening within experimental error. The rate of work-hardening is about 10 GPa in the case of the 70 vol.% reinforced matrix and about 7 GPa for the composite wire, which contains about 50% fibres by volume. This is only about half of the rate measured for the hybrid matrices but still considerably higher than would be expected for pure unreinforced bulk aluminium. It is thus found that the higher fibre volume fraction results in a higher rate of matrix work-hardening. For the composite wire material the rate of work-hardening may, as mentioned above, be influenced by the observed initial fibre damage. As shown in Section E.2.2 the effect can be quantified by means of an effective fibre Young’s modulus during monotonic tensile loading. The positive correction for the measured rate of work-hardening is in the order of 0.5 GPa: this is far lower than the difference in work-hardening between the two different fibre volume fractions. The trend towards a higher rate of work-hardening for higher fibre volume fractions is consistent with what is found in the literature [111, 116].

The yield stress estimated from the stress-strain curves is 46 MPa for the high volume fraction tensile bar and 44 MPa for the wire composite. Contrary to the rate of work hardening, the matrix yield stress (i.e., one-half the matrix flow stress amplitude between tensile and compressive deformation) seems not to be affected by the fibre volume fraction nor by the presence of the few percent ceramic particles introduced by the hybridization process.

The yield stress of 50 MPa is in broad agreement with the data of Bystricky et al. [47] for the same class of composites. The corresponding matrix dislocation density \( \rho \) is on the order of \( 1.5 \times 10^{14} \, \text{m}^{-2} \), corresponding to what is found in high-purity aluminium deformed to a tensile true strain of 22% [146]. This dislocation density is very high for an essentially annealed matrix, also similar to what was found in Refs. [47,51]. The present work thus confirms the ob-
observation made in earlier investigations that the matrix dislocation density induced by thermal mismatch strains between matrix and fibre during composite cooldown from processing temperatures is very high, being far above the density expected from simple geometrically necessary dislocation punching models.

The matrix rates of work hardening fall within the range of values found by Bystricky [47] for these composites; however, present data are far more precise than those in this earlier investigation. More importantly, whereas the precision of earlier investigations was too low to conclude to a positive matrix work hardening rate [47, 51], the present data yield values which are positive and near one-tenth of the matrix tensile modulus. Although far lower than values reported by Kelly and Lilholt [111] for copper/tungsten composites (values were as high as one-half the matrix Young’s modulus), a relatively constant work hardening rate of 10 GPa for pure aluminium is relatively high compared with what is found in the unreinforced metal (this is on the order of 0.1 GPa in polycrystalline pure aluminium after work hardening to a flow stress of 50 MPa) [146]. Aluminium as a composite matrix thus work hardens much faster than when it is unreinforced. The present data also show that this high rate of work hardening is, within experimental precision, entirely kinematic in nature, since there is no cyclic hardening of the matrix. Two reasons can be invoked to explain the observed matrix kinematic work hardening rate:

(i) triaxiality in the matrix stress; we note, however, that this has, at least to a significant degree, already been taken into account in the estimated value of parameter $\alpha$ used in the matrix work-hardening rate back-calculation;

(ii) back-stresses in the matrix resulting from dislocation pile-ups within the matrix, these pile-ups being caused by the fibres.

Precise quantification of these effects would require a full iterative back-calculation of the matrix flow curve from that of the composite using more detailed and extensive finite element simulations than were performed here.

### 3.5 Binary and Ternary Aluminium Alloy Matrices

The back-calculated *in-situ* matrix flow curves of the Al-2%Cu matrix composite wire with varying heat treatments are given in Figure E.14. The calculation parameters are shown in Table E.8.
Table E.8: Fibre volume fractions measured and used for the back-calculation of the in-situ matrix flow curves together with the matrix Young’s moduli measured from the elastic part of the matrix stress-strain curves (for Figure E.14 and Figure E.15).

<table>
<thead>
<tr>
<th></th>
<th>V_f measured</th>
<th>V_f used for back-calculation</th>
<th>E_m in-situ unload</th>
<th>E_m in-situ reload</th>
<th>E_m theoretical</th>
</tr>
</thead>
<tbody>
<tr>
<td>Figure E.14 a)</td>
<td>49.7±0.6 vol.%</td>
<td>49.60 vol.%</td>
<td>70.3 GPa</td>
<td>69.5 GPa</td>
<td>69.8 GPa</td>
</tr>
<tr>
<td>Figure E.14 b)</td>
<td>49.7±0.6 vol.%</td>
<td>50.85 vol.%</td>
<td>70.2 GPa</td>
<td>70.3 GPa</td>
<td>68.8 GPa</td>
</tr>
<tr>
<td>Figure E.14 a)</td>
<td>49.7±0.6 vol.%</td>
<td>50.40 vol.%</td>
<td>70.3 GPa</td>
<td>70.0 GPa</td>
<td>68.9 GPa</td>
</tr>
<tr>
<td>Figure E.14 b)</td>
<td>49.7±0.6 vol.%</td>
<td>49.50 vol.%</td>
<td>69.9 GPa</td>
<td>70.7 GPa</td>
<td>69.8 GPa</td>
</tr>
<tr>
<td>Figure E.15</td>
<td>59.9±0.4 vol.%</td>
<td>60.57 vol.%</td>
<td>74.2 GPa</td>
<td>73.8 GPa</td>
<td>74.0 GPa</td>
</tr>
</tbody>
</table>

Figure E.14: In-situ matrix flow curves for Al-2%Cu matrix continuous composite wires in different heat treatment conditions: (a) as-cast (b) solutionized (520 °C, 16 hrs, water quenched) (c) solutionized (520 °C, 16 hrs, water quenched) and cooled to liquid nitrogen temperature and heated up to room temperature (d) solutionized and age-hardened (190 °C, 48 hrs).
Comparing Figure E.13 b) with Figure E.14 it is obvious that the Al-2%Cu matrix is – as expected – considerably harder than the pure aluminium matrix. The matrix yield stress is not as clearly measurable because the plastic regime is not as pronounced upon cycling as it is the case for the pure aluminium matrix. Nevertheless, the effect of heat treatment on the behaviour of the binary Al-2%Cu matrix is clearly visible in the \textit{in-situ} matrix flow curves.

The solutionized matrix (Figure E.14 b) has a better defined yield stress and a lower rate of work-hardening than the as-cast matrix (Figure E.14 a). The initial rate of work hardening in the as-cast matrix is 4.5 GPa whereas in the solutionized matrix it is only about 0.2 GPa. Also, higher residual tensile stresses in the matrix are introduced when the wire is quenched in water from the solutionizing temperature. This explains the very early occurrence of plastic deformation of the matrix upon first loading. Additionally the low rate of work-hardening is associated with the absence of a hard second phase in the matrix after solutionizing. The yield stress is in both cases clearly higher than for the pure aluminium matrix.

The residual stresses can be reversed by quenching the composite in liquid nitrogen and heating up to room temperature again before testing. The resulting \textit{in-situ} matrix flow curve is shown in Figure E.14 c). It is clearly seen that yielding upon first loading now occurs at a higher strain.

Aging increases the rate of work-hardening upon initial tensile loading, from almost 0 GPa for the solutionized matrix to a value of about 8 GPa after 48 hrs at 190 °C. Although the yield stress cannot easily be quantified it can be seen that aging increases also the yield stress: for a comparable strain difference in the loop, the stress difference is about 25% higher in the aged matrix.

Contrary to the pure aluminium matrix there are signs of cyclic hardening in the binary alloy; however, more data would be needed for a clear conclusion in this regard.

\begin{figure}[h]
\centering
\includegraphics[width=0.5\textwidth]{figureE15}
\caption{\textit{In-situ} matrix flow curve for the Al-Zn6-Mg0.5 matrix reinforced with ~60 vol.\% Nextel 610™ continuous fibres.}
\end{figure}

The \textit{in-situ} matrix flow curve of the ternary Al-6%Zn-0.5%Mg alloy is shown in Figure E.15. A bilinear behaviour is seen upon initial loading with a strain-hardening rate of about 15 GPa. No important cyclic hardening was observed within a few cycles. This matrix and as a consequence the composite can deform purely elastically over a significantly increased strain interval compared to the pure aluminium matrix composites.
3.6 Uncertainty in \textit{in-situ} Matrix Flow Curves

The accuracy of the back-calculated \textit{in-situ} metal matrix flow curves presented in this study is greatly improved compared to earlier work [47]: this is because several features, not captured before, were introduced in the analysis. These include

(i) the non-constant Young’s modulus of the reinforcing alumina fibre;
(ii) adjustment of the fibre volume fraction on the basis of the measurement of the matrix Young’s modulus on the back-calculated curve and
(iii) finite element analysis of complex unit cells to reduce the uncertainty due to lateral contraction mismatch between matrix and reinforcement.

Taking these factors into account, together with the higher measurement accuracy (particularly in strain measurement), results in a significantly more reliable derivation of the \textit{in-situ} matrix flow curves. In particular, the adjustment of the fibre volume fraction from the unload and reload modulus of the matrix, made possible by taking into account second order elasticity in the fibres, actually results in a measurement of the slope difference between the plastic and elastic regime, where the elastic slope is fixed. This procedure eliminates to a certain extent errors that could result from a slightly unprecise extensometer, strain-gauge or load cell calibration, as long as these devices exhibit no nonlinearity.

However, it was seen in a few (rare) cases that the matrix Young’s modulus upon unloading and reloading differed by up to 5 GPa. The reason is probably bending when the specimen is in compression. Bending is found to strongly affect the strain measurement [147,148]. In most of the cases the unloading and reloading modulus differ not by more than 1.5 GPa, the reload modulus usually being slightly lower. This could be a sign of a slightly unprecise correction for the non-constant fibre Young’s modulus (factor $k$).

Considering that the difference between unload and reload Young’s modulus of the matrix is usually small it is concluded that the precision for the rates of work-hardening given above can be estimated as better than $\pm 1$ GPa.

3.7 Conclusions

The measured flow stress amplitude and strain-hardening rates for the different composite matrices are summarized in Table E.9. From this comparison it is seen, that

- in the pure Al matrix system a rate of work hardening of 7 to 10 GPa was measured. This value is high but lower than the values found for copper matrix reinforced with tungsten fibres [111]. This work hardening is found to be entirely kinematic in nature.
- the rate of work hardening increases with increasing fibre volume fraction for the pure Al matrix composites. This would be expected as the amount of geometrically necessary dislocations punched during cool-down from the processing increases with increasing fibre volume fraction. These measurements need further confirmation.
- the addition of 2-3 vol.% of alumina particle lead to an additional increase in the rate of work hardening of about 10 GPa. Assuming an equistrain rule of mixture for the particle reinforcement (which is clearly an overestimation) such an increase can be
expected. The increase due to the particle addition must thus be considered high, since this is an overestimation of the particle load-bearing capacity.

- the strain-hardening rate of the solutionized Al-2%Cu matrix is almost zero and thus lower than the that of pure aluminium. The binary matrix is – in contrary to the pure Al matrix – water quenched. There could thus be an effect of a different state of recovery in the two matrices.
- the aged Al-2%Cu matrix has again a higher rate of work hardening. This is the hardening effect of the $\theta$-phase similar to the effect of the alumina-particles in the pure aluminium matrix.

Table E.9: Summary of the strain-hardening rates and stress amplitudes of different composite specimens. GPI, DSC and USI stand for gas pressure infiltration, direct squeeze casting and ultrasound infiltration, respectively.

<table>
<thead>
<tr>
<th>matrix</th>
<th>hp Al non-hybridized</th>
<th>Al wire</th>
<th>hp Al hybridized</th>
<th>hp Al hybridized</th>
<th>Al-Zn6-Mg0.5 hybridized</th>
<th>Al-2%Cu wire as cast</th>
<th>Al-2%Cu wire solutionized</th>
<th>Al-2%Cu wire aged</th>
</tr>
</thead>
<tbody>
<tr>
<td>$V_r$</td>
<td>72 vol.%</td>
<td>50 vol.%</td>
<td>60 vol.%</td>
<td>60 vol.%</td>
<td>50 vol.%</td>
<td>50 vol.%</td>
<td>50 vol.%</td>
<td>50 vol.%</td>
</tr>
<tr>
<td>processing</td>
<td>GPI</td>
<td>USI</td>
<td>GPI</td>
<td>DSC</td>
<td>DSC</td>
<td>USI</td>
<td>USI</td>
<td>USI</td>
</tr>
<tr>
<td>$\Delta\epsilon$</td>
<td>0.45 %</td>
<td>0.51 %</td>
<td>0.65 %</td>
<td>0.65 %</td>
<td>initial rate</td>
<td>initial rate</td>
<td>initial rate</td>
<td>initial rate</td>
</tr>
<tr>
<td>$\theta_m$</td>
<td>10 GPa</td>
<td>7 GPa</td>
<td>20 GPa</td>
<td>18 GPa</td>
<td>15 GPa</td>
<td>4.5 GPa</td>
<td>0.2 GPa</td>
<td>8 GPa</td>
</tr>
<tr>
<td>$\Delta\sigma$</td>
<td>90 MPa</td>
<td>90 MPa</td>
<td>90 MPa</td>
<td>90 MPa</td>
<td>160 MPa</td>
<td>195 MPa</td>
<td>210 MPa</td>
<td></td>
</tr>
</tbody>
</table>
1. In-situ Fibre Bundle Strength

1.1 Pure Al Matrix Composite Wire: Molten Wire Experiments

It was observed in tensile testing of molten wires with shorter lengths that:

(i) fracture of the wire (i.e. of the fibres in molten aluminium) occurred invariably at the solid/liquid transition, Figure D.16, and

(ii) that the measured wire strength is significantly lower than the expected bundle strength for the respective length (by about a factor of about 1.7).

The most logical explanation for these observations is that, at the solid/liquid matrix interface, stress concentrations are created in the fibres: Most likely, fibres that emerge from the aluminium liquid/solid interface (which is expected to be smooth given the high purity of the matrix) experience stress concentrations due to bending whenever their axis is not strictly parallel to the stress axis. These, in turn, should lower the apparent average fibre fracture stress.

As the molten wire segment length is increased, the macroscopically observed fibre fracture becomes increasingly less concentrated at the liquid/solid matrix interface. Fibres still fracture at the solid/liquid matrix interface; however, an increasing fraction of fibres pull out of the molten zone, consistent with the observation of a finite friction stress after failure, which increases as the wire length increases, Figure D.15. Additionally, the wire fracture strength decreases as the molten zone length increases: there is thus a size effect.

This observed size effect does not match the expected size effect for the virgin fibre bundle strength, as calculated according to the virgin fibre Weibull statistics (Equation (B.8)): the molten wire strength undergoes a significantly greater size effect. Since the fibres are known to be inert in molten aluminium, it is highly unlikely that their strength would be degraded by the presence of the molten matrix. Hence, it must be concluded that the fibres in the composite wire were damaged beforehand, most likely during processing of the wire.

The presence and nature of such damage are confirmed by transmission optical microscopy (Section D - 1.6) which identified the presence of “dead” fibre ends within the wire, i.e., fibres which terminate within the matrix. The presence of such fibre ends can be explained either by the presence of ending fibres in the initial fibre tows, or by fibre fracture followed by relative displacement of the fractured fibre ends during wire processing prior to matrix solidification. Since any fractured or terminating fibre can clearly not carry load within the molten zone (since it does not bridge the two solid matrix zones), and since the number of fractured or terminating fibres increases as the molten zone length increases, the strength of the wire should decrease and the proportion of fibres broken at the liquid/solid interface should decrease with increasing molten segment length. All observations are thus qualitatively explained.
It was observed that, after the peak load was passed, the remanent wire strength is small: friction loads exerted by broken fibres within the molten composite are thus negligible. The fracture load of the molten wires can thus be calculated with the following two assumptions: (i) all fibres that end within the molten part do not carry any load and (ii) fibres that are continuous throughout the molten part break at the solid-liquid transition, at an average stress value that is independent of the molten length.

Under these assumptions, the composite fracture strength is proportional to the number of intact continuous fibres bridging the entire molten region (in the following called “surviving fibres”), this number being governed by initial damage in the wire. If the damage parameter $D_0$ denotes the probability that a given fibre of unit length $l_0$ ends or is fractured within this length, the survival probability $S$ (i.e., the probability that a fibre is intact) over length $l$ can readily be calculated as the inverse probability over length $l$ (equal to 1-$D_0$ over length $l_0$) equal to

$$S = (1 - D_0 \cdot l_0)^{\frac{l}{l_0}} \quad (F.1)$$

The fracture strength $\sigma$ of a composite wire containing a molten region of length $l$ can therefore be written as

$$\sigma = C \cdot (1 - D_0 \cdot l_0)^{\frac{l}{l_0}} \quad (F.2)$$

where $C$ is the wire fracture stress for the limiting case where all fibres are intact (molten length $l = 0$ mm), equal to the bundle strength of the fibres at the liquid/solid matrix interface. According to Equation (F.2) a plot of $\ln \sigma$ vs $l$ should yield a straight line. As can be seen in Figure F.1, this is not the case: the relation between the logarithmic strength of the composite and $l$ is not linear. Rather, the plot obtained suggests a bilinear correlation.

Figure F.1: Logarithmic strength vs length of the molten part in the pure aluminium matrix composite wire. It is clearly visible that the relation is not linear.
The reason for this can be found from TOM observations of fibre damage in the wire: as discussed in Section D - 1.6, initial damage in this composite is better described by a bimodal damage distribution. This is apparent on close examination of Figure D.7, which shows the distribution of defective fibres within the wire: the majority of these are situated either at the periphery of the wire, or at the border of essentially fibre-free matrix matrix regions, which correspond to regions between individual fibre tows which were bundled and infiltrated when making the composite. These observations indicate that ending fibres in the wire originate from surface damage of the fibre tows and the molten matrix composite wire during spooling and handling operations in the composite wire production process.

If we then separate the fibres into two populations with different fracture statistics and corresponding respectively to the more damaged peripheral fibres and to less damaged fibres within the tows, a good description of the strength of the molten wires as a function of the molten zone length \( l \) is obtained, Figure F.2. The corresponding equation for the strength of the molten wire is a simple modification of Equation (F.2):

\[
\sigma = C \left( c_1 \cdot \left(1 - D_{\text{virgin}} \cdot l_0 \right)^{l_0} + c_2 \cdot \left(1 - D_{2,\text{virgin}} \cdot l_0 \right)^{l_0} \right)
\]

where the subscripts 1 and 2 denote the two different damage regions and \( c \) their respective fraction (\( c_1 + c_2 = 1 \)). The parameters used to describe the experimental values are:

\[
C = 870 \text{ MPa} \quad c_1 = 0.25 \quad D_{\text{virgin}} = 0.02 \text{ mm}\text{ }^{-1} \quad D_{2,\text{virgin}} = 9.3 \cdot 10^{-4} \text{ mm}\text{ }^{-1}
\]

These parameters correspond to an average damage parameter of \( 5.7 \cdot 10^{-3} \text{ mm}\text{ }^{-1} \). This value is close, but not quite equal, to the average damage parameter measured by TOM and reported above (\( D_0 = 6.8 \cdot 10^{-3} \text{ mm}\text{ }^{-1} \)).

It was found that the reason for this discrepancy of about 20% has its origin in the fact that the mechanical test data and the TOM quantitative metallography were not made on the same wire spool. The first wire spool supplied by 3M ran off during the mechanical tests. As mechanical tests were continued with a new spool, it was found that the molten wire strength increased, indicating that the new spool contained somewhat less initial damage. As can be seen in Figure F.3, for a molten length of \( l = 230 \text{ mm} \) the strength for the old spool was roughly about 440 MPa, whereas for the new spool (Figure F.2) it was around 530 MPa. Taking damage parameters corresponding to the strength of the wire from the old reel, an average damage parameter of \( 6.6 \cdot 10^{-3} \text{ mm}\text{ }^{-1} \) is found. This value is in excellent agreement with the TOM measurement from this same material. Given the extensive work required for TOM measurements of initial damage and the already good agreement found between the two measures of damage, TOM measurements were not repeated on the new spool.
1.2 Al-2%Cu Matrix Composite Wire: Molten Wire Experiments

The Al-2%Cu matrix composite wire exhibits similar behaviour to that of the pure Al matrix composite. The wire strength at short molten length is lower in the as-cast Al-2%Cu wire than with pure Al as the matrix. After the wire is solutionized the short molten zone values almost approach those measured for the pure Al matrix composites. Again the wire strength depend-
ence on the length of the molten zone, \( l \), can be described by a bimodal damage distribution with only slightly changed parameters as compared to the pure aluminium matrix composite. The resulting average damage parameter is \( 4.56 \cdot 10^{-3} \) mm\(^{-1} \). It is likely that the different value of \( C \) reflects a difference in the liquid/solid interface morphology (and hence in the resulting stress concentration factor on the fibres), while differences in damage distribution parameters reflect spool to spool variations noted previously, rather than an influence of the matrix.

\[
C = 730 \text{ MPa} \quad c_1 = 0.28 \quad D_1^{\text{virgin}} = 0.015 \text{ mm}^{-1} \quad D_2^{\text{virgin}} = 5.0 \cdot 10^{-4} \text{ mm}^{-1}
\]

![Figure F.4: Fracture strength of the Al-2%Cu matrix composite wire at 775°C as a function of the molten length \( l \).](image)

**2. Damage Accumulation**

**2.1 Pure Al Matrix Composite Wire**

As demonstrated in what precedes, testing partially molten composite wires provides a method of assessing the level of fibre damage in the composite wire. Whether damage in the form of isolated fibre ends or broken fibres (two opposite fibre ends close together) is present does obviously not change the effect. Hence the method is used in the following to measure the amount of additional damage introduced in the composite after it is subjected in the solid state to a given tensile stress \( \sigma_{\text{pre}} \).

Testing of partially molten prestressed wires shows indeed that composites prestressed to a stress \( \sigma_{\text{pre}} \) have a lower ultimate tensile strength when tested at 775 °C. This effect is illustrated for the pure aluminium matrix wire in Figure D.13. As can be expected, the strength difference between prestressed and virgin composite also decreases as the length \( l \) decreases. As scatter in the wire strength was found to increase as the molten zone length decreases, molten wire strength measurements for the prestressed wires were only done for \( l > 100 \) mm.
The model developed in Section F - 1 to describe the in-situ fibre bundle strength as a function of the damage parameter can now be used to describe the behaviour of the prestressed and hence damaged composite. Only one parameter (damage parameter $D_2$) was changed independently to describe the experimental values of the prestressed composites, while the ratio between $(1-D_1)$ and $(1-D_2)$, and as also the respective concentrations $c_1$ and $c_2$, were assumed to be constant, on the reasoning that damage accumulates identically in intact fibres regardless of which region of the initial wire they belong to. The result of the fitting process can be seen in Figure F.5: as seen it agrees reasonably well with the data.

![Figure F.5: Damage accumulation in the pure aluminium matrix composite described via bimodal damage distribution (according to Equation (F.3)).](image)

This evolution of damage with prestress in the wire can be confronted with that expected from the intrinsic fibre strength Weibull distribution. If fibre fracture during composite straining is uncorrelated and not influenced by pre-existing fibre damage in the composite, it would be expected that the fraction of fracturing intact fibres during prestressing is the same as the probability of fibre fracture for a fibre of length equal to the molten zone length $l$ subjected to the same prestress. From the change in the damage parameter $D$ used to describe the virgin and the prestressed specimens, the increase in damage for a certain prestress and hence the Weibull distribution for the fibres can be calculated. Assuming that fibre fracture is governed by Weibull statistics with the same Weibull modulus $m$ as the initial virgin fibre, known to be $m = 12$ [134], the survival probability $S_{\text{Weibull}}$ for a fibre of length $L$ subjected to a stress $\sigma$ is

$$S_{\text{Weibull}} = \exp \left( \frac{L}{L_0} \left( \frac{\sigma_f}{\sigma_0} \right)^m \right)$$  \hspace{1cm} (F.4)

where $\sigma_0$ is the characteristic fibre strength at gauge length $L_0$ and $m$ is the shape parameter of the Weibull distribution (Weibull modulus, equal to 12 in the present case). The fibre survival probability according to Equation (F.4) is equivalent to the survival probability $1 - D_{\text{vir}}^m$ of
fibres in the prestressed composite normalized with the survival probability $1 - D^x_{\text{virgin}}$ in the virgin composite (where for the subscript $x$ either 1 or 2 can be used, as the ratio of the two parameters is constant for uniform fibre fracture statistics in the wire). As the damage parameters $D_1$ and $D_2$ are normalized on the basis of unit length, this probability is for a fibre of unit length $l_0$.

$$S_{\text{Weibull}} = \frac{1 - D^x_{\text{virgin}} \cdot l_0}{1 - D^x_{\text{virgin}} \cdot l_0}$$  \hspace{1cm} (F.5)

The applied fibre stress in the solid matrix composite during prestressing can roughly be calculated from the fibre volume fraction, assuming that the matrix carries negligible load (see chapter E):

$$\sigma_f = \frac{\sigma_{\text{pre}}}{V_f}$$  \hspace{1cm} (F.6)

where $V_f$ is the fibre volume fraction. By combining Equations (F.4), (F.5) and (F.6) the characteristic fibre strength $\sigma_0$ can be calculated if the fibre damage evolution is known as a function of applied wire prestress:

$$\sigma_0 = \frac{\sigma_{\text{pre}}}{V_f} \left[ \frac{l_0}{l_0} \cdot \ln \left( \frac{1 - D^x_{\text{virgin}} \cdot l_0}{1 - D^x_{\text{virgin}} \cdot l_0} \right) \right]^{\frac{1}{m}}$$  \hspace{1cm} (F.7)

Table F.1 provides the measured evolutions of parameters $D_1$ and $D_2$, together with the calculated characteristic fibre strength from Equation (F.7) for a fibre gauge length of $L_0 = 25.4$ mm.

**Table F.1: Damage accumulation in pure aluminium matrix composite wires during prestressing and calculated characteristic fibre strength.**

<table>
<thead>
<tr>
<th>$\sigma_{\text{pre}}$</th>
<th>$C$</th>
<th>$c_1$</th>
<th>$D_1$</th>
<th>$D_2$</th>
<th>$\sigma_0 (L_0 = 25.4 \text{ mm})$</th>
</tr>
</thead>
<tbody>
<tr>
<td>virgin</td>
<td>870 MPa</td>
<td>0.25</td>
<td>0.0200 mm$^{-1}$</td>
<td>0.00093 mm$^{-1}$</td>
<td></td>
</tr>
<tr>
<td>1200 MPa</td>
<td>870 MPa</td>
<td>0.25</td>
<td>0.0205 mm$^{-1}$</td>
<td>0.00145 mm$^{-1}$</td>
<td>3593 MPa</td>
</tr>
<tr>
<td>1300 MPa</td>
<td>870 MPa</td>
<td>0.25</td>
<td>0.0209 mm$^{-1}$</td>
<td>0.00180 mm$^{-1}$</td>
<td>3462 MPa</td>
</tr>
</tbody>
</table>

The values found for the characteristic fibre strength for $L_0 = 25.4$ mm are in good agreement with single fibre test data from Wilson and Visser [139] ($\sigma_0 = 3.47$ GPa) and Wilson and Visser [134] ($\sigma_0 = 3.3$ GPa), independently of the value of $\sigma_{\text{pre}}$. It can thus be concluded that intact fibres fracture in the solid composite as if they were in the virgin state. In turn, this implies that (i) intact fibres are not degraded in the composite production process, and (ii) fibre fractures are uncorrelated, from one another and also from pre-existing fibre damage in the composite at room temperature for applied composite stresses of up to 1300 MPa.
2.2 Al-2%Cu Matrix Composite Wire

In the as-cast condition the A-2%Cu matrix wires could only be prestressed to 1100 MPa because in this condition the average tensile stress is below 1200 MPa. The same procedure was applied as for the pure aluminium matrix wire to describe the composite behaviour after prestressing. The result can be seen in Figure F.6 and Table F.2.

![Figure F.6](image)

**Figure F.6:** In-situ bundle strength of the Al-2%Cu matrix composite (as-cast) after prestressing.

**Table F.2:** Damage parameters and calculated characteristic fibre strength for the Al-2%Cu matrix composite in the as-cast condition.

<table>
<thead>
<tr>
<th>$\sigma_{pre}$</th>
<th>$C$</th>
<th>$c_1$</th>
<th>$D_1$</th>
<th>$D_2$</th>
<th>$\sigma_0$ ($L_0 = 25.4 \text{ mm}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>virgin</td>
<td>730 MPa</td>
<td>0.28</td>
<td>0.0150 mm$^{-1}$</td>
<td>0.0005 mm$^{-1}$</td>
<td></td>
</tr>
<tr>
<td>1000 MPa</td>
<td>730 MPa</td>
<td>0.28</td>
<td>0.0161 mm$^{-1}$</td>
<td>0.0016 mm$^{-1}$</td>
<td>2722 MPa</td>
</tr>
<tr>
<td>1100 MPa</td>
<td>730 MPa</td>
<td>0.28</td>
<td>0.0171 mm$^{-1}$</td>
<td>0.0026 mm$^{-1}$</td>
<td>2837 MPa</td>
</tr>
</tbody>
</table>
Discussion Part II: Damage Accumulation and Fracture

Figure F.7: Strength of virgin and prestrained composite wires with Al-2%Cu-matrix in the solutionized condition. The grey lines represent the model calculation from the parameters for the virgin wire in the as-cast condition and with adapted parameters for a prestress level of 1200 and 1300 MPa from the effect found in the pure aluminium matrix composite.

Table F.2 shows clearly that in this case the characteristic fibre strength, calculated for two different prestress levels, is consistent, but well below the literature value. Figure F.7 shows clearly that after solutionizing, damage accumulation during prestraining is again much lower and comparable with what is observed in the pure aluminium matrix wire. It can therefore be concluded that the intrinsic in-situ fibre properties in the composite are not changed by the addition of copper to the aluminium matrix, but rather that the matrix second phases at the fibre-matrix interface probably initiate premature cracking of the fibres. As shown in the literature review (Section B - 4.2), this is a well-known phenomenon: brittle second phases or a brittle coating adhering to fibres in a metal matrix composite cause a significant lowering of the composite longitudinal strength.

Quantification of that phenomenon is not trivial. In order to successfully predict the strength of alumina fibres with patches of second phases adhering to the fibre surface, the strength of this intermetallic (possibly featuring a strength distribution), its geometrical distribution on the fibres, its Young’s modulus, the interface strength and the fracture toughness of the fibres have to be known.

While some simplified approaches are proposed in the literature (see Section B - 4.2), these are too simple to be used predictively, as they are based on the assumption of a homogeneous brittle layer around the fibre and do not consider the statistical fracture behaviour of the fibres [123,124,126]. Rather, we attempt to compare the two observed fibre strengths to assess the physical significance of the influence of the intermetallics on the strength of the fibres in the composite by assimilating these to a change in the pre-existing distribution of intrinsic flaws in the fibres. Neglecting the smallest defects, the probability of finding a defect with a size...
between $a_0$ and $a_0 + da_0$ in a volume element $V_0$ can be assumed, by analogy with what has been assumed for other brittle solids [149], to be governed by:

$$P(a_0) \cdot da_0 = \frac{\alpha}{a_0^\beta} \cdot da_0$$

(F.8)

where $\alpha$ and $\beta$ are material constants if $V_0$ is fixed. Integrating this probability over the values of $a_0$ from the critical flaw size $a_0^c$ to infinity results in the failure probability of the volume element $V_0$. The critical flaw size is linked to the applied stress $\sigma$ and the fracture toughness $K_{lc}$ of the material by

$$a_0^c = \frac{K_{lc}^2}{\pi \cdot Y^2 \cdot \sigma^2}$$

(F.9)

where $Y$ is a geometrical constant. The failure probability for the volume element $V_0$ is therefore:

$$P_{V_0}(\sigma) = \int_{a_0}^{\infty} P(a_0) \cdot da_0 = \left( \frac{\sigma}{\sigma_{0,\nu}} \right)^{2\beta - 2} = \left( \frac{\sigma}{\sigma_{0,\nu}} \right)^m$$

(F.10)

where $\sigma$ is the applied far field stress to the volume element, the Weibull modulus $m$ is equal to $2\beta - 2$ and $\sigma_0$ is a material constant that can be linked to the material constant $\alpha$ by:

$$\sigma_{0,\nu} = \left( \frac{Y \cdot \sqrt{\pi} \cdot (1 - \beta)}{\alpha \cdot K_{lc}^2 \cdot (1 - \beta)} \right)^{-\frac{1}{2\beta - 2}}$$

(F.11)

It is now possible – with certain assumptions – to determine a parameter $\alpha$ of the flaw distribution of the fibres when the Weibull parameters $\sigma_0$, $L_0$ and $m$ are known. The Weibull modulus $m$ is taken from the literature to be roughly 12 [134]. The volume element $V_0$ is, somewhat arbitrarily, chosen to be $V_0 = 0.1 \mu m \cdot A$, where $A$ is the fibre cross-section. The corresponding characteristic strengths for this volume elements are calculated from the Weibull parameters $\sigma_0$ and $L_0$ that are measured by the molten wire technique (see Table F.1 and Table F.2) and are:

$\sigma_{0,\nu} = 5.7$ GPa $\quad$ for pure aluminium matrix composite

$\sigma_{0,\nu} = 4.3$ GPa $\quad$ for Al-2%Cu matrix composite in the as-cast condition

The fracture toughness of the alumina fibres is assumed to be around 4 MPa $\sqrt{m}$. From a plot of the resulting flaw density distributions for the two cases, Figure F.8, it is visible that the brittle second phases in the Al-2%Cu matrix have the same effect on the fibre strength as an increase in the flaw density in the fibre by nearly an order of magnitude compared to the fi-
bres in virgin condition, or equivalently within the pure aluminium or homogenized Al-Cu matrices. This effect is considerable.

![Figure F.8: Flaw density as a function of flaw size for the two cases of an alumina fibre in a pure Al matrix (black line) and an alumina fibre in Al-2%Cu matrix in the as-cast condition (grey line).](image)

3. Composite Fracture

3.1 Room Temperature Fracture Strength

It was shown in what precedes that damage development in these composites can at least in the early stage roughly be described by the Weibull statistics of the virgin reinforcing fibres. The definition of which amount of damage, or of which internal event, triggers final composite fracture has not been treated up to here. In what follows two models that have previously been proposed to predict the longitudinal strength of unidirectional continuous fibre reinforced composites are invoked and compared with the experimental values found for the pure aluminium matrix composite wire.

**GLS Predictions of Ultimate Tensile Strength**

Curtin’s [150] GLS-model for prediction of the ultimate tensile strength of a continuous fibre reinforced composite can readily be used (see Equations (B.9) and (B.10)). The necessary input parameters and their respective values are

- characteristic fibre strength $\sigma_0 = 3.3$ GPa
- characteristic fibre length $L_0 = 25.4$ mm
- Weibull modulus $m = 12$
- matrix shear yield strength $\tau_{y,m} = 20$ MPa
The resulting composite UTS is roughly 1700 MPa and therefore much higher than the measured composite strength. This model is clearly not appropriate for the material under investigation.

**Batdorf’s Model using LLS**

The following model proposed to predict the strength of the alumina fibre reinforced composite is based on Batdorf's work [93,94]. Batdorf’s methodology consists in predicting the stress necessary for the formation of the first unstable i-plet, an i-plet being a cluster of i correlated fibre breaks (see Section B - 3.5). The mathematical approximation used by Batdorf to simplify the Weibull expression will be relaxed in the following.

The failure probability of an alumina fibre of length L subject to stress $\sigma_f$ is described by the Weibull statistics and is simply the inverse of the survival probability (Equation 0)

$$P_{\text{Weibull}} = 1 - \exp \left( \frac{-L \cdot \left( \frac{\sigma_f}{\sigma_0} \right)^m}{L_0} \right)$$

We can now calculate the number of single fibre breaks (“singlets”) in the composite. Let $N$ be the number of fibres in the composite, all of length $B$. All fibres are intact over the composite length, i.e. no initial damage is considered. The matrix has a shear yield strength of $\tau_{\text{sm}}$.

To account for the possibility of multiple fracture of one given fibre the fracture probability of one fibre segment of length $2 \cdot \delta$ is calculated and then multiplied with the number of such segments in the composites. The number of singlets $Q_1$ is therefore

$$Q_1 = N \cdot \frac{B}{2 \cdot \delta} \left[ 1 - \exp \left( -\frac{2 \cdot \delta \cdot \left( \frac{\sigma_{f,\text{applied}}}{\sigma_0} \right)^m}{L_0} \right) \right]$$

where $\delta$ is the shear length (Equation (E.4)) and therefore itself a linear function of $\sigma_{f,\text{applied}}$.

Figure F.9 shows the logarithmic number of singlets as a function of the logarithmic fibre stress in a qualitative plot. It is seen that the mathematical approximation used by Batdorf results in a linear function (solid line) whereas Equation (F.13) “bends over” at higher stresses. Even at very high stresses the number of fibre breaks can physically not go to infinity and will always be lower or equal to the number of segments in the composite (given the mechanics of stress transfer from the matrix). This condition is not fulfilled with the mathematical approximation used by Batdorf.
When a singlet is formed, a stress increase $s_1$ is locally introduced in neighbouring fibres. In the following this stress concentration is calculated under the assumption that all the load shed by a broken fibre is distributed over its nearest neighbours only (fully local load sharing). This will probably underestimate the strength of the composite. For a hexagonal fibre array a singlet has six nearest neighbours and the stress concentration is therefore

$$s_1 = 1 + \frac{1}{6} \approx 1.17$$

(F.14)

In general the stress concentration for an $i$-plet is calculated from $i$ and the number of neighbours $n_i$

$$s_i = 1 + \frac{i}{n_i}$$

(F.15)

It is a rough approximation to assume that all the nearest neighbours see the same stress concentration. Depending on the configuration of the $i$-plet it is easily possible that some fibres are more overloaded than others and are therefore more likely to break. Taking an average stress concentration factor is thus not rigorous; however, this facilitates the calculation enormously.

The number of nearest neighbours for the singlet and doublet in a hexagonal fibre array is six and eight respectively. For a triplet and higher order multiplets this number depends on the spatial arrangement of the broken fibres and can therefore only be estimated. For more or less round clusters of broken fibres the following values for the number of neighbours were graphically determined and are given in Table F.3.
Table F.3: Number of neighbours and the corresponding stress concentrations as a function of the cluster size for approximately round clusters of broken fibres.

<table>
<thead>
<tr>
<th>cluster size $i$</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
<th>9</th>
<th>10</th>
<th>11</th>
<th>12</th>
</tr>
</thead>
<tbody>
<tr>
<td>number of nearest neighbours $n_i$</td>
<td>6</td>
<td>8</td>
<td>9</td>
<td>10</td>
<td>11</td>
<td>12</td>
<td>13</td>
<td>14</td>
<td>15</td>
<td>15</td>
<td>16</td>
<td></td>
</tr>
<tr>
<td>stress concentration $s_i$</td>
<td>1.17</td>
<td>1.25</td>
<td>1.33</td>
<td>1.40</td>
<td>1.45</td>
<td>1.50</td>
<td>1.54</td>
<td>1.62</td>
<td>1.64</td>
<td>1.67</td>
<td>1.73</td>
<td>1.75</td>
</tr>
</tbody>
</table>

For a cluster size $i > 10$ the number of neighbours can be approximated by a root function of the cluster size $i$:

$$n_i = 5 + \sqrt{8 \cdot i} \quad \text{(F.16)}$$

The overload on the neighbouring fibres can be approximated by a triangular stress profile where the peak value is in the fracture plane and the remote fibre stress is again reached at a distance $\delta$ away from the fracture plane in either direction (Figure F.10). For the sake of simplicity this stress profile is approximated by a constant stress concentration $s_i$ that is acting over a length $\lambda_i$ which is defined such that the resulting failure probability is the same as for the triangular profile [93].

$$\lambda_i = \beta \cdot \frac{\delta_i^{m+1} - 1}{s_i^m \cdot (s_i - 1) \cdot (m+1)} \quad \text{(F.17)}$$

The failure probability for the overloaded fibre is now

$$P_{\text{overloaded}} = 1 - \exp \left( \frac{\lambda_i \cdot \left( s_i \cdot \sigma_f / \sigma_0 \right)^m}{I_0} \right) \quad \text{(F.18)}$$

Figure F.10: Simplified axial load profile on a broken fibre and its nearest neighbour.
The probability that a singlet transforms into a doublet (by breaking one of its neighbours) is then calculated by assuming that the total of the overloaded length is $\lambda_1$ times the number of nearest neighbours $n_1$.

$$P_{1\to 2} = 1 - \exp\left(\frac{n_1 \cdot \lambda_1}{L_0} \left(\frac{s_1 \cdot \sigma_f}{\sigma_0}\right)^n\right) \quad (F.19)$$

And the number of doublets formed by this process is

$$Q_2 = Q_1 \cdot P_{1\to 2} = Q_1 \left[1 - \exp\left(\frac{n_1 \cdot \lambda_1}{L_0} \left(\frac{s_1 \cdot \sigma_f}{\sigma_0}\right)^n\right)\right] \quad (F.20)$$

or in general

$$Q_{i+1} = Q_i \left[1 - \exp\left(\frac{n_i \cdot \lambda_i}{L_0} \left(\frac{s_i \cdot \sigma_f}{\sigma_0}\right)^n\right)\right] \quad (F.21)$$

An explicit formula for the number of $i$-plets can finally be found from Equation (F.21).

$$Q_i = Q_1 \prod_{j=1}^{i-1} \left[1 - \exp\left(-\frac{n_j \cdot \lambda_j}{L_0} \left(\frac{s_j \cdot \sigma_{f,\text{applied}}}{\sigma_0}\right)^n\right)\right] \quad (F.22)$$

This equation represents a family of curves. The first five curves are schematically represented in Figure F.11.
When the curve for the number of \( i+1 \)-plets joins the curve for the number of \( i \)-plets — the respective numbers are though only almost equal — the \( i \)-plet can be considered as being unstable; it immediately transforms into an \( i+1 \)-plet. The family of curves for all possible number of \( i \)-plets thus defines an envelope, which physically describes an asymptotic envelope of \( i \)-plet fracture instability.

The intersection of this envelope with the x-axis where \( \ln(Q_i) = 0 \) i.e. \( Q_i = 1 \) has a particular significance: it denotes the stress where the first unstable \( i \)-plet is formed with near-certainty, and is therefore considered as being the composite failure stress. The failure stress of the composite is therefore an asymptotic function of \( i \).

Neglecting the axial load-bearing capacity of the matrix, the composite failure stress is thus a function of the fibre properties (described by \( \sigma_p, L_0 \) and \( m \)), of the shear flow stress of the matrix (via the shear length \( \delta \) and of the composite volume (via the number and length of fibres). The parameters used to predict the strength of the pure aluminium composite wire are summarized in Table F.4. The strength statistics for the alumina fibre Nextel 610™ is taken from the literature [134]. The number of fibres in the cross-section was determined by counting during the investigation of TOM-micrographs. The specimen length \( B \) is the average length between the grips, used for most of the tensile testing. The fibre volume fraction was determined by a density measurement and was confirmed by back-calculation of the \textit{in-situ} matrix flow curves (see Chaper E). The matrix shear yield stress is taken from the \textit{in-situ} matrix flow curves from the preceeding chapter as equal to one fourth of the stress amplitude in the cyclic stress-strain curves.

The stresses \( \sigma_i \) for the appearance of the first \( i \)-plets as a function of \( i \) are plotted in Figure F.12. It is clearly seen from this plot, that from a cluster size of about 10 broken fibres the stress increment required to break an additional fibre is negligible. A 10-plet can thus be considered unstable and the stress required to form the first 10-plet should therefore provide a good prediction of the composite strength. The ultimate tensile stress for the composite predicted in this way is in very good agreement with the experimentally measured composite strength of 1380 MPa for the pure aluminium matrix composite wire (Section D - 2.2).
Table F.4: The input parameters and the resulting tensile strength of the pure aluminium matrix composite wire.

<table>
<thead>
<tr>
<th>Parameter</th>
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<tr>
<td>$\sigma_0$</td>
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<tr>
<td>$L_0$</td>
<td>25.4 mm</td>
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<tr>
<td>$m$</td>
<td>12</td>
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<tr>
<td>$N$</td>
<td>14600</td>
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<tr>
<td>$B$</td>
<td>200 mm</td>
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<tr>
<td>$V_f$</td>
<td>50%</td>
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<tr>
<td>$\tau_{v,m}$</td>
<td>20 MPa</td>
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<tr>
<td>$\sigma_{\text{comp}}$</td>
<td>1373 MPa</td>
</tr>
</tbody>
</table>

Figure F.12: Stress for the appearance of the first cluster of size $i$.

The failure stress is not the only information that can be drawn from the model. The slope of the envelope at $\ln(Q_i) = 0$ i.e. $Q_i = 1$ in Figure F.11 is in Batdorf's original model the Weibull modulus of the composite strength. In the present, somewhat less simplified, version where the composite failure stress is an asymptotic function of the cluster size, the first unstable cluster is not clearly defined. As a consequence, the apparent composite Weibull modulus is also an asymptotic function of the cluster size $i$, which can be defined by the slope of the envelope at $\ln(Q_i) = 0$ i.e. $Q_i = 1$ in the plot of $\ln(Q_i)$ vs $\ln(\sigma)$. Determining the Weibull modulus experimentally can however give some information about the size of the unstable $i$-plet, if it is not too large. Several lengths of the composite wire were tested. Although the number of tested samples and lengths was not sufficient to lead to a reliable estimate of $m$ for the wire (to this end more than about 50 samples would be needed, particularly for a high value of $m$), it was found that the modulus of the apparent Weibull distribution of composite wire is – in accordance with the presented model – significantly above 12.
For comparison with the present model, the unmodified Batdorf model predicts, with the same parameters, a composite wire strength of 1348 MPa, governed by instability of the first 7-plet. The discrepancy with the present calculation is slight, despite the fact that Batdorf assumes, in the stress range of fibre fracture, an approximate form of the Weibull strength that is not valid here. One can, indeed, argue that the error due to the approximations used by Batdorf is small compared to the uncertainty in realistic load-sharing rules and stress concentrations.

3.2 Elevated Temperature Composite Fracture Strength

It is found, see Section D - 2.4, that the strength of the pure aluminium composite wire decreases with increasing temperature. This is, at first sight, not surprising, as both matrix and fibres see a decrease of their strength as temperature increases. Knowing that the fibres do not lose more than about 2-3% of their room temperature strength at temperatures up to 600 °C [134], this strength decrease must be attributed to the matrix. The matrix in turn carries at room temperature a tensile stress on the order of 50 MPa. Assuming that it carries no stress at all at 600 °C, a composite strength decrease of 25 MPa ($V_f = 0.5$) could be explained. The measured composite strength decrease of almost 400 MPa between room temperature and 600 °C requires thus a more complete explanation: to this end Batdorf's model is used again. The flow stress of pure aluminium decreases with increasing temperatures. Experimental data can be found in [151] for temperatures up to 545 °C: it is found that the strength decreases between room temperature and 545 °C by almost a factor of 20. This decrease in strength of the matrix has two major effects apart from the loss of axial load bearing capacity in the matrix:

(i) the ability of the matrix to transfer stress to fibres by shear decreases, causing in turn the ineffective length $\delta$ of the broken fibres and the overloaded length of the neighbouring fibres to increase;

(ii) the stress concentration factor in the vicinity of broken fibres decreases.

These two effects are competitive and are discussed in what follows.

Increasing Shear Length $\delta$

The shear length $\delta$ is, to a first order approximation, inversely proportional to the shear yield strength of the matrix (Equation (E.4)). It is intuitively expected that the overloaded length of intact fibres neighbouring a broken fibre is also proportional to the matrix shear yield strength. The calculation of the failure probability of an overloaded fibre has to account for this effect. Additionally the shear length may no longer be small compared to the mean axial distance between two fibre breaks when the matrix shear yield strength becomes very small. In this case, the far field stress on a fibre is higher than the applied fibre stress calculated using Equation (F.6), for a given load over the composite cross-section has to be constant. This effect can be taken into account by introducing an effective fibre volume fraction.

Decreasing Stress Concentration Factor

The evolution of the stress concentration factor SCF with decreasing matrix shear yield stress is less evident. Keeping the assumption made above that all the stress of the broken fibres is distributed to the nearest neighbours only, the SCF does not change with matrix shear yield
strength; however, it is intuitively expected that it changes and vanishes for the limiting case of a vanishing matrix shear yield strength, in which case global load sharing is expected. A decrease in the stress concentration, in turn, implies inevitably that the stress is redistributed not only over the nearest but also over the next-nearest neighbours and perhaps beyond (again because the load that has to be transferred over the cross-section has to be constant).

Stress concentrations as a function of the normalized matrix shear yield strength can be found in [89]; these calculations show that the SCF in fact decreases relatively slowly for higher normalized shear yield strength. Only for matrix shear yield strengths close to zero does it decrease rapidly towards zero.

Resulting Effect on Composite Strength

The important quantity for the strength of the composite is neither the stress concentration factor nor the overloaded volume but the failure probability of the fibre affected by these two effects. Landis and McMeeking [89] quantified the failure probability of a fibre neighbouring a broken fibre on the basis of a shear-lag approach. Their model neglects the axial matrix load-bearing capacity but accounts for sliding at the fibre-matrix interface. In metal matrix composites such as aluminium/alumina with a strong interface bonding, relative matrix/fibre sliding is governed by shear yielding of the matrix. Landis and McMeeking found that for decreasing sliding stress the failure probability of the overloaded fibres first increases, passes by a maximum, and only for very low sliding resistance decreases and reaches zero for the limiting case where the sliding resistance is zero. These findings can be transferred to the present composite system in a way that a decreasing matrix shear yield stress (in analogy to the sliding fibre-matrix interface) first results in an increase in the fracture probability of an overloaded fibre neighbouring a broken one. Only for a matrix shear yield stress close to zero (molten matrix), does the fracture probability of the neighbouring fibre approach zero. At this point the global load sharing regime would be reached; however, this regime is not reached in the present experiments when the matrix melts, due to the stress concentrations at the solid-liquid interface.

Interpretation with Batdorf's Model

In the model presented so far the stress concentration factors are only a function of the cluster size of broken fibres but independent of matrix materials properties. The overloaded length on the other hand is inversely proportional to the matrix shear yield strength. Composite strength according to this model therefore decreases with decreasing matrix shear yield strength. The strength decrease predicted from the present model using the temperature-dependent pure aluminium matrix shear yield strength given in [151] together with the measured strength decrease from tensile tests on the continuous pure aluminium matrix composite wire is shown in Figure F.13. Reasonable agreement between the model and the experiment is found, even if some differences between prediction and experiment can be seen at higher temperature. At these temperatures, however, yield strength values for the matrix are probably less reliable than at lower temperatures, and stress transfer may perhaps be influenced by diffusion-governed sliding along the fibre/matrix interface.
Comparison of Model Predictions with Damage Measurements

Damage evolution measurements in Section F - 2 can also be confronted with the present model. From the fact that the calculated characteristic fibre strength is independent of the applied fibre stress during prestressing of the composites it was concluded that fibre fracture occurs mainly uncorrelated. Comparing this with the numbers of \( i \)-plets predicted from the present model the conclusion is confirmed: for a wire length of 500 mm and a composite stress of 1300 MPa (equal to a fibre stress of 2600 MPa), the number of singlets is roughly 13'500, whereas the number of doublets and triplets is only about 320 and 20, respectively. This low number of doublets and triplets has a negligible influence on the total amount of damage measured.

Conclusion

What emerges from the present data and analysis is that a strong matrix is beneficial for composite strength. Matrix alloying should, thus, provide a method for strengthening metal matrix composites both in the transverse and longitudinal direction; however, there are several limitations to this approach:

(i) any brittle second phase adjacent to the fibres must be avoided;
(ii) alloying elements in the matrix must preserve the virgin fibre strength; and
(iii) the matrix must remain significantly tougher than the fibre (otherwise cracking will begin in the matrix, as in ceramic matrix composites for example).

These are strong limitations, which explain why matrix alloying has most often caused a lowering in composite longitudinal strength compared with a pure matrix. Yet, there are instances where matrix strengthening, by alloying and/or heat treatment, has been shown in the literature to provide for some level of strengthening of fibre reinforced metals, see Section B - 4.2.
With Batdorf's model, the strengthening effect of a stronger matrix can also be explored. The matrix shear yield strength is varied, all other parameters keeping their values as summarized in Table F.4. The matrix shear yield stresses explored together with the predicted composite strengths are summarized in Table F.5. The predicted composite strengths corresponding to a high-strength matrix are considerably higher than for low-strength matrices.

Table F.5: Matrix shear yield stresses used as input parameter together with the composite strength predicted by Batdorf's model.

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<th>$\tau_{vm}$ (MPa)</th>
<th>UTS (MPa)</th>
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<tr>
<td>100</td>
<td>1529</td>
</tr>
<tr>
<td>200</td>
<td>1602</td>
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Continuous alumina fibre reinforced aluminium composites are produced by pressure assisted liquid metal infiltration techniques, namely gas pressure infiltration and direct squeeze casting. Net shape preforms are suitably produced by means of a fibre winding technique directly into graphite molds which comprises wetting the fibre tows with an alumina particles suspension during winding. When tested in tension, these composites display the well known two-stage behaviour of fibre reinforced metals.

It is shown that the longitudinal Young's modulus of the composite is a univoqual function of strain. This is explained as resulting from the intrinsic elastic nonlinearity of the alumina fibres. A simple second order approach, assuming a linear modulus decrease as a function of composite strain, provides a good agreement with measured data.

*In-situ* matrix flow curves are back-calculated from composite stress-strain curves by means of a refined relation between composite and matrix flow stress. This relation is based on the simple rule of mixtures, modified to incorporate several phenomena not captured in earlier studies. The calculation of the fibre stress accounts for the nonlinear elasticity of the alumina fibres; it is also shown that fibre breaks present in the composite can be neglected in the derivation. The deviation from the rule of mixtures due to lateral contraction mismatch between the fibre and matrix is investigated by finite element analysis of complex unit cells. These calculations are used to propose a back-calculation method yielding reduced uncertainty compared to the usually used Hill’s bounds. The remaining uncertainty in the knowledge of the fibre volume fraction is further reduced by calibration of composite unloading and reloading moduli with the constant matrix Young’s modulus.

A transmission optical microscopy technique is developed to detect initial damage present in the form of dead fibre ends in the composites. No such damage is found in samples pressure infiltrated at EPFL or EMPA; however, in the wire produced at 3M a significant number of such fibre ends exists. A non-homogeneous distribution of this damage is suggested by the fact that damage is more frequently observed near the wire surface and in low fibre volume fraction regions that are situated between individual fibre tows which were thus near the tow surface during wire production.

A technique for the measurement of damage in composite wires is presented. The method consists in measuring the variation of the wire strength on the length of a zone within which the matrix is molten. Data from this measurement show good agreement with the amount of damage measured for the as-processed wires by transmission optical microscopy.

The evolution of internal damage as a function of the applied stress on the composite wire is measured using the developed technique of tensile testing wires with sections of molten matrix. It is found for pure aluminium matrix composite wire that fibre breaks are mainly uncorrelated for stresses up to 1300 MPa and that the *in-situ* fibre fracture statistics follow the
Weibull statistics known for the virgin fibres. It is thus concluded that the fibre strength is not altered by the pure aluminium matrix.

Brittle second phases at the fibre-matrix interface, as are found in the Al-2%Cu matrix composite wire in the as-cast condition, induce premature fibre failure during composite straining, increasing greatly the amount of damage and reducing composite strength. After solutionizing, the in-situ fibre fracture statistics follows essentially those found for pure aluminium matrix or the virgin fibre. The addition of copper is thus not an intrinsic factor degrading the fibre strength in the composite.

The fracture strength of the continuous composite wire can be reasonably well predicted by a model based on the stability of clusters of neighbouring broken fibres. Particularly the observed composite strength decrease with increasing temperature is in excellent agreement with the proposed model and is attributed to the decrease in shear yield strength of the matrix, which in turn increases the shear transfer length.

It can be concluded from the data and their analysis that the matrix of fibre reinforced metals can be used to strengthen the composite: a strong matrix is beneficial for the composite strength if (i) brittle second phases are avoided, (ii) the matrix does not affect the intrinsic fibre strength and (iii) the matrix remains significantly tougher than the composite.

The usually high performance of continuous fibre reinforced composites with a (essentially weak) pure aluminium matrix is probably based on the increased in-situ matrix flow strength found in the composite; however, it is not clear to what extent the in-situ tensile flow strength of the matrix is representative for the in-situ matrix shear strength, which is an important parameter for the composite strength.
H - Future Work

From the work presented in this study four main areas for further research can be identified:

Transmission optical microscopy was only rarely used to date in metallurgy. In the present study TOM was shown to reliably detect dead-ending fibres in the composites. Broken fibres, however, were not detected with the setup at hand. It might, however, be possible with other light sources to detect the presence of a fracture surface in a given fibre. The method would then probably not be limited to alumina fibre reinforced aluminium but could be transferred to other composite systems where the fibre material is translucent (e.g. glass fibre reinforced polymer matrix composites).

Tensile tests at temperatures higher than the matrix melting temperature were shown to be a useful tool to quantitatively assess damage in continuous alumina fibre reinforced aluminium matrix composites. This tool could be used in a number of studies to monitor the damage evolution. Damage introduced by mechanical or thermal fatigue can, for example, be measured by this method. Quality control in the production of composite wires could be another application. A limiting factor, however, is the high specimen length needed to get reliable results.

A number of improvements in the back-calculation of the in-situ matrix properties from the composite flow curve are presented; however, full and accurate solution of this inverse problem remains challenging. Finite element analysis was shown to be a tool to improve the accuracy of the back-calculation by “calibrating” the analytical equations. Solving the problem by an iterative finite element analysis is time consuming but feasible and could increase the reliability of the results, even though it will be a challenge to model representative complex unit cells that capture well the real composite behaviour.

The reliable prediction of fracture strength of continuous fibre reinforced metal matrix composites is still very demanding. There are several powerful tools available from simple models (like the one presented here) to sophisticated 3D fracture simulations; however, success of the strength prediction depends on numerous input parameters such as the fibre spatial distribution, static and dynamic stress transfer properties of the matrix, statistical strength distribution of the fibres and possibly other parameters, all known with varying reliability. Considerable work would thus be needed in measuring micromechanical features such as stress transfer from a broken fibre to its neighbours to check the validity of current micromechanical models.
I - Appendix

Longitudinal fracture strength of parallel tensile bars with varying fibre volume fraction and matrix alloy.

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