FATIGUE CRACK PROPAGATION IN FUNCTIONALLY GRADED MATERIALS

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FATIGUE CRACK PROPAGATION IN FUNCTIONALLY GRADED MATERIALS

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Abstract

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Interest in the development and application of functionally graded materials (FGM) has increased in recent years, leading to their limited use in a number of commercial and military products. More recently, interest in the use of FGMs in aircraft fuselage structures as integrated thermal protection systems has also begun to develop. However, our limited ability to predict the nucleation and fatigue propagation of cracks in these materials limits the application of FGMs to non-fracture critical structures, reducing the potential benefits of their incorporation. A first step toward addressing this technology gap is to evaluate whether linear elastic fracture mechanics methods, appropriately modified to account for material non-homogeneity, can be used to predict fatigue crack propagation in an arbitrarily graded metal/ceramic FGM.

This dissertation documents new developments for characterization and modeling of fatigue crack propagation in FGMs. Unique precracking and characterization methods, and a Paris equation dependent upon stress intensity and material phase volume
fraction and gradients are developed for FGMs. Hybrid numerical experiments are used to develop and verify a multivariable regression (MVR) method for identifying the effective crack tip coordinates and accurately recovering stress intensity from 2-D crack tip displacement field measurements in FGMs. Numerical and experimental results for Ti-TiB FGM specimens validate the MVR method for recovery of stress intensity in FGMs when allowing for the presence of manufacturing-induced residual stresses. Predicted fatigue crack propagation rates also compare favorably with experimental results, but are limited in accuracy by the effects of residual stresses. Residual stresses modify crack tip stress intensities and their effects are only accountable here by using the MVR recovered stress intensities. The results suggest that a Paris equation including material gradients as independent variables is viable. However, an accurate means of accounting for residual stresses in arbitrary FGM forms must be developed if linear elastic fracture mechanics methods are to be used effectively, otherwise excessive experimental characterization of fatigue crack propagation properties would be required.
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Chapter 1

Introduction

Functionally graded materials (FGMs) are engineered material systems that combine multiple and often significantly different materials into a single system. Compositions include but are not limited to functionally graded metals, composites, and metal/ceramic systems. The goal of producing such engineered material systems is to utilize the strengths of each material at specific locations in a component. Ideally, functionally graded materials are continuously graded, minimizing or redistributing the residual stresses generally found at the boundary between layers in bi-material systems designed for the same purpose. However, in practice FGMs are often composed of a number of layers in which the second phase increases in volume fraction with the addition of each layer. In many instances, processing produces a material system without distinct interfaces between layers [1]. As a result, interface stresses exist but are redistributed across multiple layers. This advantage, coupled with successes in manufacturing a wide range of materials and forms, have led to product introductions in areas including civil and mechanical engineering, biomechanics, and optics, to list a few. Products in these areas include fire and blast protection, thermal barrier components, medical implants, and armor.
Currently, the application of functionally graded materials and structures to aircraft and spacecraft fuselage structures is being considered. Multiple material systems focusing on graded organic composites, ceramics and metallics, and combinations thereof are being developed and evaluated for applications in advanced thermal protection systems and lightweight structurally efficient primary structures. Present designs such as the space shuttle, and proposed future vehicle designs, utilize a load carrying airframe protected from the extreme thermal environment of re-entry or high-speed flight by ceramic tiles, blankets, or titanium or superalloy metallic skins. Substitution of these traditional structures with a unitized FGM structure, where a lightweight metallic or composite load carrying airframe seamlessly transitions into a thermally resistant skin (Figure 1.1), could provide significant manufacturing, assembly, weight and life cycle cost advantages over traditional designs and structures.

However, our limited understanding of how damage progresses in these materials limits their use to non-fracture critical structures. This thesis focuses on the study of fatigue crack propagation in functionally graded materials and on determining if linear elastic fracture mechanics methods, appropriately modified to account for material non-homogeneity, can be used to predict fatigue crack propagation in an arbitrarily graded metal/ceramic FGM. In pursuit of this question: novel numerical experiments are conducted to develop and validate a multivariable regression technique with which stress intensities in a functionally graded specimen can be recovered; a new method for precracking brittle materials is developed; fatigue crack propagation rates for a Ti-TiB FGM are characterized; and a Paris equation that includes TiB volume fraction as an independent variable is developed and tested.
Fortunately, results in the literature supply many of the tools and techniques necessary to begin to address this question. Fundamental research documenting such topics as crack tip strain and displacement fields, residual stress states, and static and dynamic fracture have been and continue to be pursued. Tools such as the finite element codes provide methods for calculating stress intensity and T-stress for arbitrary material gradients in complex structures. In addition, experimental techniques for evaluating stress intensity directly from displacement measurements made on a test specimen have been partially evaluated. The following sections describe functionally graded materials that are typically discussed in the literature and are of interest here, and the research that has been accomplished that supports fatigue crack propagation research in these materials.
1.1 Functionally Graded Model Materials

There are essentially two classifications of FGMs applicable to this research. The first class is the model material systems. Two model material systems often cited include polyethylene carbon monoxide (ECO) [2, 3], a material system in which the material gradients (modulus, strength, ductility, etc.) are developed by controlled exposure to ultraviolet light, and resin or polymer material systems, which incorporate a graded distribution of a second phase particle such as ceramic spheres creating the material gradient [4]. The advantages of model materials are their availability, low cost, and the ease with which continuous material property gradients (linear, power law, exponential, etc.) can be developed and analyzed. Model materials form the basis for much of the limited validation of analytical and numerical models used to predict the fracture behavior of FGMs.

1.2 Functionally Graded Engineering Materials – Ti-TiB

In addition to model materials, functionally graded material systems of metal/metal, metal/ceramic and metal/polymer have been made and evaluated. The material system of interest in this research is a functionally graded composite of pure titanium and titanium monoboride produced by in-situ reaction of Ti and TiB₂ powders. A number of research programs have investigated the mechanical properties [5, 6] and microstructural features of this material. Related experiments and analyses have investigated the properties and microstructural features of various composites of boron and alloys of titanium such as Ti-6Al plus B and Ti-6Al-4V plus B [7,8] for very low
weight fractions of boron, typically less than 2.5 wt%. These results, while not directly applicable to the current research, do provide insight into possible behavior of the current Ti plus B FGM at low boron weight fractions. In particular, the addition of less than 0.05 wt% boron, equivalent to 11 vol% TiB, increases both the ductility and strength of Ti-6Al and Ti-6Al-4V alloys [7]. Above 0.05 wt% boron, ductility falls rapidly while strength continues to increase, with the degree of change depending strongly on the particular titanium alloy. For Ti-6Al plus 2.5 wt% B ductility decreases by 72 percent from its peak, while for Ti-6Al-4V plus 2.5 wt% B the reduction is 86 percent. In contrast, strength increases by a factor of two or more in both cases. Also, for boron addition up to 0.05 wt% the microstructure does not include the TiB whiskers observed for higher boron additions. The results provide clues that indicate that fracture properties of the pure Ti-TiB material system might initially increase, consistent with increases in ductility seen for the alloys of Ti for very low level additions of boron, before falling as the brittle TiB phase begins to dominate ductility.

However, of most interest here is research into the microstructural aspects of pure titanium with the addition of boron at concentrations that produce monolithic Ti-TiB materials with TiB volume fractions equal to and greater than 15 percent. Of particular relevance is the research on monolithic and functionally graded Ti-TiB materials produced from blended powder performs of Ti and TiB2. In this processing approach, Ti powder with average particle sizes of between 28 and 45 µm is blended with TiB2 powder with average particle sizes of approximately 2 µm. The blended powders, consisting of layers of differing volume fractions of TiB2, are hot pressed to produce a functionally graded material of Ti-TiB according to the reaction
Reported results [8-10] discuss the properties and microstructural morphology of monolithic and functionally graded Ti-TiB with TiB concentrations ranging from ~15 to over 86 vol% TiB produced using hot pressing.

From microstructural observations in the literature and in the current material it is clear that a number of microstructural changes in Ti-TiB microstructure occur over this range of TiB volume fractions. In cases where a Ti-TiB monolithic material or layer within an FGM contains less than 30 vol% TiB, the reaction of Ti and TiB₂ produces individual TiB whiskers typically 50 μm long and 5 μm wide (Figure 1.2a). Above 30 vol% TiB, a 10 μm TiB phase dominates the material due to the quantity of TiB₂ particles around each Ti particle and the associated reduction in the mean free path for whisker growth resulting from multiple TiB initiation sites. The typical microstructure above this volume fraction includes clusters of small 1 to 10 μm whiskers and relatively few large TiB whiskers (Figure 1.2b). A third transformation in the microstructure occurs when TiB volume fraction reaches approximately 70 percent. At approximately 70 vol% TiB the phase is dominated by 1 to 3 μm whiskers, few if any ~50 μm whiskers, and a small distributed population of 10 μm whiskers (Figure 1.2c).
1.3 Linear Elastic Fracture Mechanics: Analysis and Experiments in Functionally Graded Materials

A number of methods have been developed with which to calculate conditions at the tip of a crack and predict fracture behavior. Advanced methods based on cohesive zone models have been applied to monolithic materials and extended to include functionally graded materials. Ural [11] simulated fatigue crack propagation in a

Figure 1.2. TiB microstructure a) Ti-30 vol% TiB, b) Ti-45 vol% TiB and c) Ti-85 vol% TiB.
monolithic material using a damage-based cohesive zone model. Jin and Paulino [12, 13] developed cohesive zone models to predict crack propagation in FGMs. Classical methods for evaluating stress intensity analytically, numerically (finite element analysis-based) and experimentally have also been extended to include functionally graded materials.

Analytical methods based upon integral equation methods [14, 15] have been developed for a number of crack propagation problems, providing a method for verifying results from other methods. In other work, numerical methods based upon the J-integral and interaction integral have been developed. Rao [16] presents a Galerkin-based mesh free analysis method for stress intensity based on the interaction integral. Paulino and coworkers extended the J-integral and interaction integral method to evaluate mode I and mixed mode stress intensities, respectively, in functionally graded materials using conventional finite element analysis [17-24]. Coupled with development of a modified interaction integral with which to evaluate functionally graded materials, Kim developed graded finite elements for non-homogeneous materials [25]. Chi evaluated stress intensity profiles in layered and continuously graded FGMs using finite element analysis. To evaluate experimental data and calculate stress intensity and T-stress, experimental methods based upon crack tip displacement fields have been investigated for calculation of stress intensity and T-stress in functionally graded materials. *Note: T-stress is a uniform stress acting parallel to the crack plane.*
1.3.1 Crack Tip Fields

In 1987 Eischen [26] published results demonstrating that the 2-D fields for stress, strain and displacement for continuous and generally continuously differentiable FGMs were, in the limit as \( r \) collapses to zero, in part identical to those developed by Williams [27, 28] for monolithic materials. In particular, the \( r^0 \), \( r^{1/2} \) and \( r \) terms of the displacement equations remain unchanged, except that modulus and Poisson’s ratio must be evaluated at the crack tip. The higher order terms, however, are affected by the material gradient. The resulting equation for crack opening due to \( K_I \) is

\[
\frac{K_I}{2G_{tip}} \sqrt{\frac{r}{2\pi}} \sin \frac{\theta}{2} \left( \frac{3 - \nu_{tip}}{1 + \nu_{tip}} - \cos(\theta) \right) - \frac{2C_{12} \nu_{tip}}{G_{tip} (1 + \nu_{tip})} r \sin \theta
\]

(1.2)

\[
+ A_1 \cos \theta + u_{y0} + O(r^{3/2})
\]

where \( A_1 \) and \( u_{y0} \) are rigid body rotation and translation terms. Later, Erdogan [29,30] modified the crack tip stress field equations to explicitly account for an exponential material gradient at an arbitrary inclination to the crack direction

\[
\sigma_{ij} = e^{\beta r} \left( \frac{K_I}{\sqrt{2 \cdot \pi \cdot r}} f_{1y}(\theta) + \frac{K_{II}}{\sqrt{2 \cdot \pi \cdot r}} f_{2y}(\theta) \right) + T + O(r^{1/2})
\]

(1.3)

However, for the simplified material gradients studied in the literature, this form of the equation is often not used. Numerical and experimental studies into crack tip stress intensities in functionally graded materials are based upon the simpler form developed by Eischen.

Marur and Tippur [31], evaluated the applicability of the homogeneous crack tip field in graded materials and found the size of the homogeneous fields decreases as the
material gradient increases. More recently, Shukla and his students have developed crack
tip stress fields for functionally graded materials using an asymptotic analysis and
Westergaard’s stress function [32, 33]. Up to six terms of the expansion have been
derived for cracks aligned with, and inclined to, the material gradient.

1.3.2 Interaction Integral

In monolithic materials the two most common methods by which stress intensities
are computed are closed form analytical methods and J-integral methods. A similar
division of methods is possible for FGMs; however, these methods must now account for
material gradient. To account for the material gradient, closed form analytical methods
become more complex and require simplifying assumptions. Similarly the standard
interaction integral found in most finite element programs must be reformulated to
account for material gradients. Normally during formulation of the interaction integral,
terms such as the derivative of the constitutive equation are discarded. For a monolithic
material the derivative of the constitutive equation is zero and contributes nothing to the
calculation of stress intensities. However, this and other terms are non-zero for the case
of a continuously graded material and must be retained to maintain the path independence
of the integral. From a research perspective, each methodology has its advantages. The
interaction integral based methods remain general in nature and can be applied to any
combination of continuous material gradient, geometry and loading condition, allowing
actual material gradients and arbitrary shapes to be analyzed. Analytical methods are
typically considered to be exact, and as such, provide a benchmark against which other
solution methods may be evaluated. Given that the focus of the proposed research is on
numerical and experimental methods for analysis of metal/ceramic FGMs, the remainder of this review will focus on the interaction integral formulations.

1.3.2.1 Homogeneous Material Interaction Integral Formulation

The interaction integral, as implemented in most FEA codes, is derived from the J-integral assuming homogeneous material properties. Derivation of the interaction integral involves superposition of the analytical stress and displacement fields given by Eq. (1.2), with the finite element derived fields creating a J-Integral formulation

\( J^s = \int_A \left\{ \left( \sigma_{ij} + \sigma_{ij}^{aux} \right) u_{i,1} + u_{i,1}^{aux} \right\} - \frac{1}{2} \left( \sigma_{ik} + \sigma_{ik}^{aux} \right) \left( \varepsilon_{ik} + \varepsilon_{ik}^{aux} \right) \delta_{1,j} \} q_{i,j} \ dA \)  

(1.4)

\[ + \int_A \left\{ \left( \sigma_{ij} + \sigma_{ij}^{aux} \right) u_{i,1} + u_{i,1}^{aux} \right\} - \frac{1}{2} \left( \sigma_{ik} + \sigma_{ik}^{aux} \right) \left( \varepsilon_{ik} + \varepsilon_{ik}^{aux} \right) \delta_{1,j} \} \ dA \]

that depends on numerical and analytical (auxiliary) fields [18-22].

Following algebraic manipulation, this formulation can be separated into three terms: the finite element based J-Integral, the auxiliary field based J-Integral, and the interaction integral (M-Integral).

\[ J^s = J + J^{aux} + M \]  

(1.5)

where the interaction integral is given by:

\[ M = \int_A \left\{ \left( \sigma_{ij} u_{i,1}^{aux} + \sigma_{ij}^{aux} u_{i,1} \right) - \left( \sigma_{ik} \varepsilon_{ik}^{aux} \right) \delta_{1,j} \} \right\} q_{i,j} \ dA \]  

(1.6)

\[ + \int_A \left\{ \sigma_{ij}^{aux} u_{i,1} - C_{ijkl} \left( \varepsilon_{ij} \varepsilon_{kl}^{aux} \right) q \right\} dA \]
The second integrand in Eq. (1.6) includes the equilibrium equation and the derivative of the constitutive equation, terms that vanish for a homogeneous material. Thus, the standard interaction integral equation simplifies to

\[ M = \int_A \left( \sigma_{ij} u_{i,1}^{aux} + \sigma_{ij} u_{i,1} \right) - \left( \sigma_{ik} \varepsilon_{ik} \right) \delta_{ij} \{ q \} \ dA \]  

(1.7)

The strength of the interaction integral is that it includes only the cross terms between the numerical and auxiliary fields and as a result, with appropriate selection of the auxiliary fields, the stress intensity values \( K_I \) and \( K_{II} \) can be uniquely determined from the finite element numerical results using

\[ M = \frac{2 \left( K_I \cdot K_I^{aux} + K_{II} \cdot K_{II}^{aux} \right)}{E} \]  

(1.8)

### 1.3.2.2 FGM Interaction Integral Formulation

For graded materials, the equilibrium term and the derivative of the constitutive equation are non-zero. To maintain path independence these terms must be retained. Several equivalent formulations of the non-homogeneous interaction integral have been developed [18, 19, 21]: the non-equilibrium formulation, the incompatibility formulation, and the constant constitutive tensor formulation. Of the three forms, the non-equilibrium formulation

\[ M = \int_A \left( \sigma_{ij} u_{i,1}^{aux} + \sigma_{ij} u_{i,1} \right) - \left( \sigma_{ik} \varepsilon_{ik} \right) \delta_{ij} \{ q \} \ dA \]  

(1.9)

\[ + \int_A \left( \sigma_{ij} u_{i,1}^{aux} - C_{ijkl,1} \varepsilon_{ij} \varepsilon_{kl} \right) \{ q \} \ dA \]

is the simplest and has been implemented in the IFranc [34] finite element code.
Like the homogeneous formulation, this form of the interaction integral can be, with appropriate selection of the auxiliary fields, used to extract stress intensity values from Eq. (1.8). Verification of the accuracy of stress intensities calculated using the modified interaction integral has been accomplished by comparison with “exact” analytical methods and other numerical methods.

1.3.3 Experimental Methods and Results

Experimental evaluation of crack propagation in functionally graded materials has been for the most part limited to monotonic and dynamic loading fracture experiments. The predominance of this work has been in polymers modified to produce a functionally graded material. In metal/ceramic functionally graded material systems, very little work has been published, with the exception of limited monotonic loading results in Ti-TiB, and fatigue crack propagation in an Al-SiC FGM, summarized below.

1.3.3.1 Residual Stress in Ti-TiB Functionally Graded Materials

Experimental, analytical and numerical research results have been published, detailing methods for evaluating the residual stress state in a layered FGM. Hill describes a combined numerical and experimental method for determining the residual stress state of an FGM [35, 36] based upon a slotting method [37]. Experimental results in Ti-TiB specimens, similar to the specimens used in this research, reveal a residual stress state ranging from +40 MPa to -40 MPa measured through the thickness of the material from the TiB-rich surface. In other results, a closed form analytical method, verified by finite element analysis, is proposed by Panda [10].
1.3.3.2 Fatigue Crack Growth Experiments

Xu et al. [38, 39] conducted experimental research into fatigue crack propagation in an Al-SiC FGM. Single edge notch bend (SEN(B)) FGM specimens with ceramic volume fractions ranging from 0 to 30 percent were produced and tested to explore the general behavior of a fatigue crack growing parallel to the material gradient in the FGM. In these experiments cracks were propagated from the low modulus material layers into the stiffer material layers, and fatigue crack propagation progressed as would be expected, based upon the numerical results of Chi [40]. Chi reported in his work that for a layered system, load shedding into the higher stiffness layer ahead of the crack resulted in reduced stress intensity levels until the crack tip penetrated into the layer. In work by Xu, crack propagation did in fact slow as the crack entered and transitioned through the diffusion zone; however, Xu reports that in other unpublished experiments, a crack growing from the stiffer to the lower modulus material also experienced a decrease in crack propagation. This is in contrast to numerical results predicting that a crack propagating from a stiffer material into a weaker material sees a temporary increase in stress intensity as it approaches the boundary between layers. As a result, it is clear that load shedding alone cannot explain the decrease in crack propagation rate. In subsequent fractographic analysis, Xu found the crack to have a significantly different morphology in the transition zones as compared to the morphology of the crack within a layer. Significant bifurcation and kinking of the crack was found to take place in the transition zone, which would have dissipated energy with the creation of new crack surfaces. In addition, multiple crack tips would have had a shielding effect that reduced the stress intensity for any given crack tip resulting in retarded growth. Crack kinking resulting in
a rougher surface might also have contributed to crack closure. These effects along with load shedding may explain the slowed propagation rate in both directions. However, other mechanisms not identified, or not present in these experiments, may exist.

Possibly the most important result from these experiments is the report that cracks growing in both directions, metal to ceramic or ceramic to metal, experienced reduced propagation rates as they neared and traversed the diffusion zones. This may indicate path dependence relative to material gradient or local variations in material behavior unique to the transition region. If so, it may not be possible to characterize crack growth behavior in an FGM without foreknowledge of the gradient and crack path.

1.3.3.3 Compression Precracking

Results in the literature, as well as experimental results in the present research, demonstrate that attempting to use tensile precracking methods in monolithic and FGM Ti-TiB specimens leads to mixed results. Precracking low TiB volume fraction materials can be successfully accomplished using traditional methods for metallic specimens, where a precrack is initiated in and propagated from a notch. However, when using tensile methods to precrack a high TiB volume fraction material, the lack of crack tip plasticity to absorb energy and blunt the crack tip often leads to uncontrolled pop-in of a larger than desired precrack. To produce a short precrack in these materials, a number of methods have been developed over the years. ASTM standard C1421-01b [41] describes several, including the use of the precracked beam method and surface crack in flexure method to initiate a crack. In practice the precracked beam method can produce longer than desired initial cracks and can require significant development to determine the
number of Vickers indentations, load, and bridge fixture span to produce precracks. A modification to the precracked beam method that is reported to provide more control and produce shorter initial cracks has been developed by Ray [42]. This method uses an articulated bridge fixture and is reported to be 100 percent successful in precracking small ceramic specimens. A similar method uses 3-point bending of a sandwiched specimen [43]. The approach places a grooved ceramic specimen between support beams. After crack initiation, and as the crack propagates, load transfers from the specimen to the support beams resulting in a decreasing stress intensity and crack arrest.

An improved method of precracking for brittle materials has been developed by Carpenter [44], the physics for which are described in work performed by Suresh and Brockenbrough [45-47]. In work in polycrystalline alumina and WC-Co and TiC-Ni composites, Suresh and Brockenbrough studied the impact of high compressive stresses at a notch tip and their impact on the development of microcracking. In brittle materials, microcracks begin to develop once a threshold stress $\sigma_0$ is exceeded and continue to develop until a saturation stress $\sigma_s$ is reached. If upon unloading, debris or other mechanisms wedge these microcracks open, (curves C and D in Figure 1.3) a residual tensile field develops that may, if sufficiently large, cause the microcracks to coalesce into one or more larger cracks. Continued cycling may extend the length of these cracks through development of further microcracking but only to a limited extent. Eventually, crack face closure limits the magnitude of the stress field at the tip of the developing cracks to a value below $\sigma_0$ and further microcracking halts.

Carpenter used this work as the basis for developing an approach to precracking Ti-TiB functionally graded specimens. His work utilized compression precracking in
FGM specimens including a straight-through electrical discharge machined notch. In precracking the Ti-TiB FGM the challenge was to produce a sufficiently large compressive stress to initiate cracking without introducing damage to the metal rich layers in the FGM. Initially Carpenter conducted axial compression tests as described by Suresh. However, while the axial compression method did produce the desired precrack, the axial compression stress (212 MPa) required to generate a precrack in the 60 vol% TiB layer also resulted in plastic deformation of the titanium layer, invalidating subsequent $K_R$ test results. In response, Carpenter developed a reverse four-point bend procedure similar to the method in ASTM standard C1421-01b in which a chevron notch is cycled in reverse bending. Using this approach he successfully demonstrated the capability to repeatedly generate short sharp precracks in the notch.

![Constitutive Model for microcracking of a brittle solid](image)

Figure 1.3. Constitutive Model for microcracking of a brittle solid (46).
1.3.4 Recovery of Stress Intensity from Crack Tip Displacement Fields

In 1987 McNeill [48] published research using 2-D displacement fields captured experimentally using digital image correlation (DIC) to recover crack tip stress intensity in monolithic plexiglas specimens. Using least squares regression and a 2-D field of displacements measured around the crack tip, McNeill evaluated the first 48 coefficients of the asymptotic solution for crack tip displacements in an attempt to accurately evaluate stress intensity. In general, the results overestimated K by 10 to 20 percent, but the results did demonstrate the feasibility of using experimentally measured 2-D displacement fields to recover stress intensity for comparison with analytical solutions. A possible source for the error may have been the improper determination of the crack tip coordinates. One possible method for minimizing the error uses an iterative Newton-Raphson method [49] to evaluate and correct for differences between assumed and actual crack tip coordinates; however, it does not automatically provide the number of terms required to achieve accurate stress intensity and T-stress results.

1.3.5 K Dominance in Functionally Graded Materials

Using the results of Shim [50] to evaluate the materials, geometry and loading conditions in this research program, the K dominance region is typically less than 1.0 mm in radius from the crack tip. For an ideal material, recovering stress intensity and T-stress might be possible using multivariable regression of the Williams equation, coupled with measurement of displacements within this region. However, for real materials, complex mechanisms such as plasticity, microcracking and 3-D stress fields at the crack tip limit how close to the crack tip displacement measurements can be made and exclusively
relied upon to recover stress intensity and T-stress. Within this region, the elastic Williams equations [28] for displacements do not apply. However, if the region within which complex mechanisms exist is sufficiently small, it is possible to make displacement measurements outside this region, and recover stress intensity and T-stress, if additional terms are included to account for cases where these measurements are now being made outside of the K-dominance region.

1.3.6 Recovery of Stress Intensity in Functionally Graded Materials

Multiple experiments have been conducted to develop and evaluate methods for recovering stress intensity and T-stress in functionally graded materials from optical measurements of crack tip fields. Rousseau and Tippur [51] used fringe patterns to evaluate stress intensity, and recently Abanto-Bueno and Lambros [2, 3] conducted fracture tests in functionally graded specimens manufactured from photo-sensitive poly(ethylene carbon monoxide). In the experiments by Abanto-Bueno and Lambros, digital image correlation was used to measure the 2-D displacement field at the tip of an advancing crack, and multivariable regression based upon the Williams equation was used to recover stress intensity. The quality of the recovered stress intensity was evaluated based upon agreement between the measured displacement field and the analytical displacement field, based upon recovered values of $K_I$, and rigid body translation and rotation. Relying solely on $K_I$ as the fitting parameter, their experimental and analytical results for displacement display noticeable differences. This can be attributed in part to reliance on stress intensity as a fitting parameter well outside its region of dominance [51]. To resolve this problem several additional parameters
including $K_{II}$, T-stress, and $C_{22}$ (the coefficient in the $r$ term of the mode II displacement equation) were added to the regression. The results show that significantly better agreement between measured crack tip displacement fields and analytical fields is achieved when recovered stress intensity and T-stress are both used in the analytical equations for crack tip displacements. $K_{II}$ and $C_{22}$ had little impact in this case due to the absence of mode II crack opening.

1.4 Literature Review Summary

In summary, research into crack propagation of FGMs suggests that the homogeneous crack tip field equations still apply in the limit as the radius from the crack tip approaches zero, and that for a material gradient with continuous second derivatives the square root singularity at the crack tip remains. Research has also produced a number of analytical, numerical and experimental models and methods with which to evaluate LEFM parameters and stress intensity in functionally graded materials. For very limited conditions, the models and methods have been verified and partially validated. However, nothing has been done to extend the models to include fatigue crack propagation, nor have they been validated using realistic aerospace materials.

1.5 Research Plan

Five primary tasks are pursued in this research:

1) Characterization of fatigue crack propagation in monolithic specimens representing a limited number of Ti-TiB volume fractions.
2) Development of a Paris equation spanning the range of Ti-TiB volume fractions present in the FGM.

3) Evaluation and development of a digital image correlation based method for accurately determining stress intensity directly from crack tip displacement measurements in FGMs.

4) Characterization of fatigue crack propagation properties in the FGM.

5) Evaluation, and if necessary, revision of the Paris equation and determination of whether, and under what conditions and ranges of material non-homogeneity, the model might be valid.
Chapter 2  
Titanium-Titanium Monoboride Microstructure

Normally, fatigue crack propagation models such as the Paris equation, the Walker equation and others do not explicitly account for microstructure in their formulations. Within the range of applicability of any of these models, microstructural changes are typically slight. However, the primary objective of this thesis is to develop a single Paris-style equation that spans the range of ceramic volume fraction from 0 to 85 vol% TiB in Ti-TiB FGMs. From literature results, it is known that across this range the TiB phase of the material undergoes multiple and significant microstructural transitions. As a result, the Paris equation in this case will have to account for microstructural transitions. To provide information necessary for accommodating microstructural aspects of the material, a scanning electron microscope and image analysis are used to evaluate, quantify and document microstructural features such as material phase morphology and distribution from the viewpoint of their potential impacts on fatigue crack propagation.

From this perspective two significant local microstructural variations have been identified that have the potential to impact crack nucleation and propagation behavior. These include macroscopic regions of locally elevated TiB volume fraction, and a wide
transition zone between the 30 and 45 vol% TiB layers. Experimental results (chapter 5) are inconclusive with respect to the impact of locally high TiB regions due to the limited number of times the crack has been observed intersecting these regions; however, the impact of the wide transition zone and the material morphology within it has been seen to have a significant impact on observed crack propagation.

2.1 Material Fabrication and SEM Specimen Preparation

The Ti-TiB FGM used in this investigation is a constructively produced seven layer material comprised of two primary material phases, titanium and titanium monoboride, and a minor nickel phase used as a sintering aid. The FGM is produced by Cercom Inc., now Advanced Ceramics Inc. (Vista, Ca), a BAE Systems company. Plates of Ti-TiB are produced from stacked layers of uniform blended titanium and titanium diboride powders to produce volume fractions of 0, 15, 30, 45, 60, 75 and 85 percent TiB, after hot pressing at 13.8 MPa and 1305 °C. Under these conditions the relevant reaction:

\[ \text{Ti} + \text{TiB}_2 \rightarrow 2\text{TiB} \]  

(2.1)

produces several whisker-like TiB phases, depending upon the initial volume fraction of TiB2 powder dispersed within the titanium powder. From the plates, 25 mm x 25 mm x 152 mm FGM bars were produced, from which a 25 mm x 25 mm x 2 mm specimen was cut using wire EDM for SEM analysis (Figure 2.1). The surface of the sample was prepared by polishing of the EDM surface in stages with a final polish using 1 µm diamond slurry.
2.2 General Material Microstructures

The material system exhibits four distinctly different microstructures depending upon the volume fraction of TiB. The pure titanium material is monolithic as expected, with the exception of a limited amount of nickel that appears as a lighter phase in the SEM backscatter images of Figure 2.2. With the addition of boron to levels that produce 15 and 30 vol% TiB in the FGM, a coarse phase consisting of 50 µm long whiskers develops, marking the first major change in microstructure. These whiskers tend to be evenly distributed throughout the titanium phase with relatively little clustering (Figure 2.3 a and b).
The second major transition in microstructure occurs at a TiB concentration of 45 vol%, above which the morphology is dominated by clusters of 10 μm whiskers with the occasional appearance of the larger 50 μm phase. This change in morphology, as reported by Sahay and Chandran [8, 9], is due to: particle size differences between the Ti and TiB₂ powders; powder packing arrangements; the diffusion behavior of Ti and B; and

Figure 2.2. Pure titanium layer.

Figure 2.3. a) Ti-15 vol% TiB b) Ti-30 vol% TiB layer microstructures.
changes in the mean free path (MFP) for whisker growth with increasing TiB$_2$ volume fraction in the preform. As TiB volume fraction increases to 60, 75 and 85 percent, the third change in microstructure develops as the TiB whisker phase becomes more refined, while the coarse phase disappears (Figure 2.4) and 1 to 3 $\mu$m whiskers dominate. The change to a microstructure dominated by the smaller 3 and 10 $\mu$m TiB whisker phases also marks the transformation to a much more brittle material that is very susceptible to rapid unstable crack propagation. Also observed in the microstructure are local regions of high TiB concentration. Beginning with the 30 vol% TiB layer, and persisting through the 60 vol% TiB layer, is the random distribution of local TiB rich regions (Figure 2.5).

![Figure 2.4. a) Ti-45 vol% TiB b) Ti-60 vol% TiB microstructures showing clusters of fine TiB whiskers distributed throughout the material.](image-url)
2.3 Layer Transitions

Three distinctly different transitions and transition zones between layers exist within this material depending upon TiB volume fraction. The most defined of these is the transition between the Ti and Ti-15 vol% TiB. Within this narrow transition zone the 50 μm whisker TiB microstructure is introduced (Figure 2.6 a). The second and predominant transition, occurring between all but the 30 and 45 vol% TiB layers, is dominated by changes in the volume fraction of each phase (Figure 2.6 b and c). The third transition type is found between the 30 and 45 vol% TiB layers where both 10 and 50 μm TiB phases exist simultaneously. Unique to each transition zone is a different transition width, with the most distinctive existing between the 30 and 45 vol% TiB layers (discuss later in this section).
Transition zone widths and phase distributions are evaluated using backscatter images from an electron microscope that are processed to enhance one or more of the phases in the material depending upon the desired measurement. Images of each transition zone are converted to threshold images in which gray scale values of 0 or 255 are assigned to individual pixels based upon underlying material phases. Pixels

Figure 2.6. Transition zones from a) 0 to 15 vol% TiB, b) 15 to 30 vol% TiB and c) 45 to 60 vol% TiB.
dominated by the Ti phase are assigned a value of 0, while pixels dominated by the TiB phase are assigned a value of 255. Approximately 1200 individual measurements along each contour shown in Figure 2.7 are averaged and converted from a gray scale value to approximate TiB volume fractions. Average transition zone width is estimated based upon the distance over which volume fraction increases before converging at a new level. From these measurements, two observations are made. First, transition zone width for zones between 0 and 15, 15 and 30, and 45 and 60 vol% TiB are approximately 2, 20 and 45 μm, respectively, as shown in Figure 2.8. Second, narrow continuously graded regions rather than non-continuous steps connect the layers. Above 60 vol% TiB the transition zones are difficult to detect and evaluate.

Figure 2.7. Threshold image of 15 to 30 vol% TiB transition zone including evaluation paths in and parallel to the transition zone.
Figure 2.8. Transition zone widths: a) 0 to 15, b) 15 to 30, c) 45 to 60 vol% TiB.
In contrast, the transition between the 30 and 45 vol% TiB layers is marked by a more complex transformation, a fact not previously reported. The morphology in this transition zone changes in TiB volume fraction and TiB phase over a much wider region than that observed between other FGM layers. At what appears to be the initial interface (within the box in Figure 2.9) clusters of 10 µm whiskers appear intermixed with a much larger population of 50 µm whiskers. Like other transition zones, this transition zone is continually graded. However, while an underlying gradient in TiB volume fraction certainly exists, it is masked by a gradient measured in terms of TiB microstructure. Over a width of approximately 1.0 mm, moving from the 30 vol% TiB layer into the 45 vol% TiB layer, the volume fraction of 50 µm whiskers diminishes from 100 percent of the TiB phase to nearly zero, while the 10 µm whisker population increases to 100 percent. Figure 2.10 shows the 50 µm whisker volume fraction ($v_{50\mu m}$) as a percentage of overall TiB volume fraction. Results show a linear decay of the 50 µm whisker microstructure from 100 percent at a relative position of 0.2 mm to approximately 1.0 percent at 1.0 mm, a distance that is consistent with crack propagation results presented in chapter 5. Note that within this local region, two material gradients exist, a layered gradient in which TiB volume fraction changes from 30 to 45 percent, and a superimposed local gradient in which TiB microstructure transitions continuously from 50 to 10 µm whiskers.
Figure 2.9. Transition zone between 30 and 45 vol% TiB layers.
Unfortunately, these descriptions of material microstructure and transition zone behavior do not appear to be fixed. In related experiments Quast [5] evaluated 3 mm thick Ti-TiB plates with identical reported TiB volume fractions produced by Cercom. Quast’s results for the 30 vol% TiB layer (found to be 33.8 vol% TiB based upon optical measurements) include both TiB phases. In contrast, the 30 vol% TiB layer in the 25 mm thick plate evaluated here includes only the 50 µm TiB microstructure. The difference might be explained by differences in layer thickness and material gradient. The adjacent layer in the 3 mm thick specimen is reported to be 88.5 vol% TiB, not 45 vol% TiB. This apparent dependence of the microstructure in one layer to the composition of the next may introduce microstructures that are unexpected, and may introduce a material dependency that complicates material property characterization.

Figure 2.10. 50 µm whisker TiB phase volume fraction in the 30 to 45 vol% TiB transition zone.
2.4 Conclusions

Material microstructural observations and analysis show that within the seven volume fractions investigated, several primary material phases exist: a Ti phase and 50, 10 and 3 µm whisker TiB phases. However, based upon published descriptions of Ti-TiB microstructures for other specimen geometries and manufacturing processes, the microstructure for a given volume fraction may not be fixed. In addition to the primary phases, other relevant characteristics of the material include transition zone widths ranging from 2 µm to 1000 µm. Typically within these zones, the dominant transition feature involves a narrow continuously graded change in the volume fraction of Ti and a single TiB phase. However, between the 30 and 45 vol% TiB layers a second material gradient appears. Within this zone, the TiB volume fraction gradient is superimposed with a gradient in TiB microstructure, an aspect of the material not previously reported in the literature.
Chapter 3
Material Characterization

There are at least two possible approaches to characterizing fatigue crack propagation in functionally graded materials. Should fatigue crack propagation in these material systems be found to be dependent on material gradient or crack path, it seems certain that characterization will have to be accomplished for each unique structural form. Obviously this will be a costly and time consuming process. However, if fatigue crack propagation is independent of the material gradient and crack path, it may be possible to characterize fatigue crack propagation rate in a limited number of monolithic material specimens representing discrete volume fractions. It is based upon this assumption that the Ti-TiB FGM in this research is characterized and a single Paris-style equation is developed.

Fatigue crack propagation properties in monolithic Ti-TiB specimens are characterized for six TiB volume fractions ranging from 0 to 85 vol% TiB. Based upon the results, functional relationships for the Paris equation parameters C and n and TiB volume fraction are developed, and a single model describing fatigue crack propagation rate as a function of stress intensity and TiB volume fraction is produced. A total of 18 specimens representing pure titanium and Ti-TiB at TiB volume fractions of 15, 30, 45,
60 and 85 percent are used to characterize fatigue crack propagation rate, stress intensity threshold and fracture toughness for each TiB volume fraction. Of the 18, ten are successfully used to generate fatigue crack propagation data, while several more add to results for threshold and fracture toughness behavior. The remainder failed prematurely due to difficulties initiating and propagating cracks in specimens with TiB volume fractions exceeding 30 volume percent.

All experiments were conducted in four-point bending at an R-ratio of 0.5 to limit the possibility of crack closure. Initial experiments focus on characterization of the pure titanium specimens and progress into materials with higher TiB content. For materials up to and including 30 vol% TiB, these experiments are conducted in a conventional manner typical of fatigue crack propagation testing in metals. A simple wire electric discharge machined (EDM) notch is used as a stress concentration feature from which a crack is initiated and propagated following the guidelines of ASTM 647 [52]. Above 30 vol% TiB the character of the material becomes exceptionally brittle and these specimens cannot be successfully precracked in tension from a simple notch. Tests of specimens with TiB volume fractions above 30 percent are conducted from precracks initiated in a modified notch design (see Appendix A for details). In two of three cases the specimens fail as the crack emerges from the modified notch into the full cross-section of the specimen. Only in the Ti-45 vol% TiB specimen is the crack successfully propagated out of the notch.
3.1 Monolithic an FGM Specimen Preparation and Geometry

Two sets of specimens were produced as part of this research. The first set included monolithic specimens for fatigue crack propagation rate characterization. These specimens were obtained by separating the layers of FGM bar (Figure 3.1) into monolithic plates shown in Figure 3.2a using wire EDM. This approach to producing monolithic specimens from FGM bars rather than having separate monolithic specimens produced was used to eliminate batch-to-batch variation in properties and to characterize the monolithic properties for the as-processed FGM. The resulting specimens varied in thickness depending upon the dimension of the individual layer in the FGM bar from which they were cut. Thicknesses ranged from 2.35 to 4.06 mm for the Ti-TiB layers, and from 4.0 to 5.17 mm for the pure Ti layer. To maintain configuration control, individual specimens were labeled based upon the bar of material from which they were cut (F08, F10, F11 and F12) and with a number (-1 to -7) indicating the layer from 0 to 85 vol% TiB respectively. The second set of specimens produced were FGM specimens used during experimental validation of the fatigue crack propagation models. FGM specimens 2.84 mm thick were produced from the FGM bars by cutting across the layers (Figure 3.2b).

To prevent twisting and add stability to the specimens during testing, aluminum doublers 3.2 mm thick were bonded to each end of each specimen, leaving a central test section approximately 32 mm wide. The test section was hand ground using water coolant to remove the EDM recast layer, and polished to prepare the surface for visual inspection during tests. The final configuration for each type of specimen, monolithic
and FGM, are shown in Figure 3.3a. In this four-point bend configuration, load (P) is equally divided between two points separated by 50.8 mm along the upper edge of the specimens. The load is reacted through rollers separated by 139.7 mm along the lower edge specimen. Crack growth (a) for both specimens is in the height direction (Figure 3.3b and c). Also, for the case of the FGM, crack growth is parallel to the material gradient as shown in Figure 3.3c.

![FGM Material Gradient](image_url)

Figure 3.1. FGM bar.

![Layered Material Gradient](image_url)

a) Monolithic

Figure 3.2. Monolithic and FGM specimens.
Figure 3.2. Monolithic and FGM specimens (continued).

Figure 3.3. Typical four-point bend specimen geometry loading condition and crack propagation direction.
3.2 Calculation of Stress Intensity

Two methods for calculating stress intensity have been used depending upon the local geometry through which the crack is propagating. In cases in which the crack is propagating through the full cross-sectional area of the specimen, stress intensity is based upon the Rooke and Cartwright [53] handbook solution for a beam in four-point bending

\[
K = \frac{6M \sqrt{a}}{W^2} \left( 1.12 - 1.39 \left( \frac{a}{W} \right) + 7.32 \left( \frac{a}{W} \right)^2 - 13.1 \left( \frac{a}{W} \right)^3 + 14.0 \left( \frac{a}{W} \right)^4 \right)
\]

with \( a/W \leq 0.6 \).

In the remaining cases, initial crack propagation takes place in a modified notch geometry. Unlike the typical notch which is cut straight through the specimen, specimens with TiB volume fractions above 45 percent require a unique design to raise the stress concentration from 8 to 30 in order to be successfully precracked. The design that results (see Appendix A for details) is not cut through, but produced by saw cuts made from both sides of the specimen. This produces a thin web of material in the notch that gradually increases in thickness until it reaches the surface. For cases in which the crack is propagating within this “Y” notch detail, finite element global and local models have been constructed in ABAQUS and imported into Franc3D/NG. To calculate the stress intensity, a straight through crack profile is assumed and stress intensity is calculated along the length of the notch. To produce a continuous function of stress intensity versus crack length in the notch, near surface stress intensities are fit with a third order polynomial for each specimen. In each case, crack length is taken as the length
along the surface of the notch to be consistent with the locations at which stress intensity results are obtained.

Typically two finite element analyses for each specimen incorporating the “Y” notch detail have been conducted. A pretest analysis, based upon the assumed as-manufactured notch geometry (Figure 3.4) is used to estimate stress intensity. Results are used to determine the appropriate loads to be applied during the experiment in order to maintain crack propagation and avoid overloading the specimen. For experiments in the 60 and 85 vol% TiB specimens, in which cracks were never successfully propagated beyond the notch, a post test analysis is conducted. In these cases, the post-test analysis, based upon the actual notch geometry measured after failure, is used to calculate ΔK and characterize da/dN. In each case, stress intensity for the notch detail is combined with handbook solutions [53] for a four-point bend specimen to produce a complete stress intensity profile as a function of crack length covering the entire range of possible crack lengths, both internal and external to the notch (Figure 3.5).
Figure 3.4. Typical assumed and actual notch profiles.
3.3 Experimental Fatigue Crack Growth Techniques

Two techniques for precracking and propagating cracks in these materials were required during characterization. For titanium, and Ti-TiB specimens with TiB volume fractions equal to or less than 30 percent, the standard ASTM method for metals was used successfully. However, due to the exceptionally brittle behavior of the Ti-TiB material when TiB volume fraction exceeds 30 percent, modifications to the precracking notch design and loading method were required. In general, stable initiation and propagation of fatigue cracks in brittle materials is a challenge. In ductile materials, plasticity at the crack tip is both the source of initiation and crack arrest, resulting in a stable precracking process. However, in brittle materials no such mechanism exists. Without a plastic zone

Figure 3.5. Stress intensity profiles for "Y" notch geometry in Ti-60 vol% TiB specimen.
at the crack tip to dissipate energy and blunt the crack, or other toughing mechanisms such as phase transformation or crack bridging, unstable extension during precracking is a constant threat. In addition, once a precrack is initiated, fast fracture remains a problem due to the same lack of toughing mechanisms that plague precracking.

Both of these issues have been experienced in the Ti-TiB monolithic specimen experiments where TiB volume fractions exceeded 30 percent. Attempts to use tension-tension precracking led to sudden initiation and fast fracture. Also, constant amplitude load control testing inevitably led to fast fracture and premature failure at some point in a test. The cause for this type of sudden failure appears to be due to the tendency for cracks in these TiB-rich materials to suddenly advance a short distance in a single cycle, after hundreds or thousands of cycles in which no observable growth takes place. In load control, as the crack advances, loads are reduced and the control system continues to displace the crosshead in an effort to reach the desired load. This "following of the crack" appears in several cases to have caused the crack to run through the part instead of arresting.

To resolve the precracking issue, a novel notch design, discussed in detail in Appendix A, was developed, which increases the typical notch stress concentration factor from 8 to 30. To resolve the propagation problem in materials with TiB volume fractions above 30 percent, tests were conducted in displacement control, and all four specimens that employed the method were conducted successfully. This approach has the advantage that excessive propagation during a single cycle results in unloading of the specimen. It also has the disadvantage of foregoing the convenience of constant-amplitude loading for
monotonically varying $\Delta K$ conditions, and therefore requires constant monitoring of loads to ensure minimum and maximum load values are achieved throughout the test.

A final complication in successful characterization of specimens with TiB volume fractions of 60 and 85 percent dealt with fast fracture outside the notch. While in the notch, crack propagation was stable due to the decreasing stress intensity gradient versus crack length produced by the “Y” notch design. However, upon transition to the full cross-section of the specimen, the cracks propagated to failure. Despite several attempts to lower the loads at this transition point, cracks were never successfully propagated out of the notch into the full cross section of the 60 and 85 vol% TiB specimens. As a result, a crack propagation procedure designed to characterize the material with the crack in the notch has been devised, without which data for the 60 and 85 vol% TiB specimens could not have been generated.

### 3.3.1 Low TiB Volume Fraction Crack Propagation Procedures

The crack propagation method used for specimens with TiB volume fractions less than or equal to 30 vol% TiB followed ASTM standard 647 for metals. Precracking was accomplished at a maximum load equal to the planned load for characterization and at an R-ratio of 0.1. Following precracking, R-ratio was incrementally increased from 0.1 to 0.3 and finally to the planned test value of 0.5 while propagating the crack at least 1.0 mm. In general, experiments were conducted at a constant maximum load resulting in a rising $\Delta K$ test until failure, or until the test was terminated. In some cases, after propagating the crack to near failure, load was dropped to collect additional data in region II of the crack propagation curve.
3.3.2 High TiB Volume Fraction Crack Propagation Procedures

Crack propagation testing of specimens employing the "Y" notch configuration was significantly more complex due to a combination of circumstances. First, the stress intensity range between $\Delta K_{th}$ and $\Delta K_c$ in materials above Ti-45 vol% TiB is very narrow, ranging from 0.1 to 0.3 MPa$\sqrt{m}$. Second, stress intensity falls as the crack advances over most of the length of the notch (Figure 3.5), requiring periodic incremental increases in load as the crack propagates to prevent $\Delta K$ from falling below $\Delta K_{th}$. Third, uncertainties in $\Delta K_{th}$ and $\Delta K_c$, coupled with likely errors in pretest predictions of stress intensity due to differences between the assumed and actual notch profile, prevent accurate prediction of the load at which the crack will arrest, or the maximum incremental load increase at which fast fracture will occur. The challenge is to follow a load path between threshold and toughness limits, achieving stable propagation and populating the entire region II portion of the $da/dN$ versus $\Delta K$ curves, without allowing the crack to arrest or propagate unstably through the specimen.

The procedure to start and to maintain crack propagation from an initial precrack in the notch begins with cyclic loads at an artificially low level. The intent is that the initial cycles be at an alternating stress intensity level below threshold. At this starting load, an initial 5000 cycles are applied while visually monitoring crack propagation. If after 5000 cycles no crack propagation is measured, load is increased slightly, generally no more than 2 to 5 percent, 10 to 25 N for the current materials and geometries, and an additional 5000 cycles are applied with this step repeated until crack propagation is observed. Once a load is achieved at which the crack propagates, several intervals of
between 1000 and 5000 cycles based upon observed crack propagation are run and crack extension is measured. Because $\Delta K$ decreases with increasing crack length over most of the length of the notch, crack propagation rates decrease with each interval. Once the crack propagation rate falls below $1.0 \times 10^{-8}$ m/cycle, load is increased with the new load picked to attempt to fill gaps in data recorded during previous intervals. If the new load results in rapid propagation, cycling is stopped and the load in the next interval is reduced before being incrementally increased again. In the final stage of crack propagation in the notch, as the crack tip approaches the transition to the surface of the specimen, load is reduced to minimize $\Delta K$ and minimize the likelihood of fast fracture as the crack emerges onto the surface of the specimen.

The procedure as implemented for the Ti-60 vol% TiB specimen is shown in Figure 3.6, and includes the load history from start to failure, incremental crack growth, and load limit curves, based upon fracture toughness and threshold values and post-test measurements of notch geometry. Initial loads of 400 and 425 MPa, applied in 5000 cycle increments and at an R-ratio of 0.1, failed to propagate the crack despite appearing to be well above threshold for this length of crack. Increasing the load to 455 MPa resulted in 0.97 mm of growth in 4185 cycles. At this point the R-ratio was increased to 0.5 and 11,673 cycles were run with the crack propagating an additional 0.25 mm, while the propagation rate fell to $4.86 \times 10^{-9}$. To populate region II, load was increased to 480 MPa causing rapid crack advance, 1.0 mm in 1049 cycles, indicating that the toughness value had been approached or exceeded. In response, load in the next interval was decreased to 460 MPa to avoid further rapid crack advance. In retrospect, the load should have remained at 480 MPa. With the decreasing stress intensity profile in this
portion of the notch, crack propagation would have slowed as the crack advanced, populating region II more completely. Over the next 52,643 cycles, load was increased to 470 and 480 MPa each time crack propagation dropped below $1.0 \times 10^{-8}$ m/cycle. As the crack approached the end of the notch, load was dropped to 470 MPa, reducing stress intensity range to approximately 2.6 MPaVm, the estimated threshold value for the material. Despite the reduction in $\Delta K$, the specimen failed as it transitioned out of the notch. In fact, the only specimen in which the crack was successfully propagated into the full cross-section was the Ti-45 vol% TiB specimen. As a result, data for the Ti-45 vol% TiB material is based upon crack propagation outside of the notch, while data for the Ti-60 vol% TIB and Ti-85 vol% TiB materials is based upon results within the notch.
Characterization of crack propagation rate versus $\Delta K$ and TiB volume fraction was attempted on a total of eighteen specimens with ten successfully yielding fatigue crack propagation data. Three successful experiments were conducted in pure titanium, and two each in Ti-15 vol% TiB and Ti-30 vol% TiB specimens. Due to the exceptionally poor toughness of the materials with TiB volume fractions above 30 percent only a single successful test was achieved in each of these materials. The remaining eight specimens, all containing TiB volume fractions of 45 percent and higher, failed prematurely.

**Figure 3.6.** Load and Crack Path History relative to threshold and fracture toughness load limits for the Ti-60 vol% TiB specimens.

### 3.3.3 Fatigue Crack Propagation Results

Characterization of crack propagation rate versus $\Delta K$ and TiB volume fraction was attempted on a total of eighteen specimens with ten successfully yielding fatigue crack propagation data. Three successful experiments were conducted in pure titanium, and two each in Ti-15 vol% TiB and Ti-30 vol% TiB specimens. Due to the exceptionally poor toughness of the materials with TiB volume fractions above 30 percent only a single successful test was achieved in each of these materials. The remaining eight specimens, all containing TiB volume fractions of 45 percent and higher, failed prematurely.
Beyond the expected reduction of $\Delta K_{th}$ and $\Delta K_{\text{max}}$ with increasing TiB volume fraction, three distinctly different fatigue crack propagation behaviors have been observed in the six materials evaluated. These, like other fracture characteristics, appear to be strong functions of TiB microstructure. Experimental results in the pure titanium specimens display a significant level of scatter not seen in the Ti-TiB materials. Scatter in the Ti material and lack thereof in the Ti-TiB materials can be attributed to a number of factors, all stemming from differences in crack propagation behavior. Unique to crack propagation in Ti, and significant contributors to scatter in all three experiments (specimens F08-1, F10-1, and F12-1), were the strong propensity for crack kinking and crack tip bifurcation (Figure 3.7). Both behaviors had a detrimental impact on the accuracy of the measured crack length and on the actual magnitude of stress intensity experienced by the crack tip(s). However, despite the scatter, the pure Ti layer produced results that on average match published data for Ti-70 [54]. In contrast, all of the TiB bearing materials produced straight cracks without significant kinking and no indication of bifurcation. Results for each of the Ti-TiB materials are shown in Figure 3.8.
Figure 3.7. An example of crack kinking and bifurcation in the pure Ti material.

Figure 3.8. Fatigue crack propagation results for the Ti-TiB monolithic specimens.
Figure 3.8. Fatigue crack propagation results for the Ti-TiB monolithic specimens (Concluded).
3.3.3.1 Threshold and Toughness Stress Intensities

As with other characteristics of these materials, changes in threshold behavior appear to be closely related to changes in material microstructure. Common to each material from 0 to 60 vol% TiB is an obvious threshold region, though scatter within each region appears to be a strong function of TiB content and morphology. Devoid of the TiB phase, pure titanium results exhibit significant scatter in the threshold region from a low of 7.38 MPa√m to a high of 9.15 MPa√m, consistent with the crack kinking and bifurcation seen throughout experimental results in this material. With the addition of 15 and 30 vol% TiB and associated introduction of the 50 μm TiB whisker phase, results present clearly defined threshold regions, typically with scatter of no more than 0.54 MPa√m. Above 30 vol% TiB, microstructural changes dominated by the 10 μm TiB whiskers are accompanied by a reduction in scatter to 0.15 MPa√m. At 85 vol% TiB, microstructural changes to a 3 μm TiB whisker appear to result in the loss of the threshold region. Even at the minimum growth rate measured in the experiment, 3.88 x 10⁻⁹ m/cycle, results do not show a tendency for a lower limit. By comparison, threshold behavior for the 45 and 60 vol% TiB materials is observed at approximately 1.0 x 10⁻⁷ m/cycle.

In light of the observed existence or absence of threshold in the materials, ΔKth is estimated based upon either the average of the data within region I or upon the lowest value observed during testing. For materials ranging from Ti to Ti-60 vol% TiB, threshold is based upon an average of the data points that fall within a definable region I. For the Ti-85 vol% TiB material, threshold is based upon the minimum value recorded. Results for all six materials are given in Table 3.1.
3.3.3.2 Fracture Toughness

Due to the tendency for any of the Ti-TiB materials to fracture unexpectedly, most experiments were terminated once crack propagation rates approached or exceeded $1.0 \times 10^{-6}$ m/cycle to preserve the specimens should additional testing be desired. Other experiments ended when an inadvertent overload caused failure. In some cases, experiments that ended unexpectedly in fast fracture still yielded enough data to estimate fracture toughness. As a result, the change in slope that accompanies the transition from region II to region III, providing definition of fracture toughness, is typically not well defined, with the exception of the pure Ti material. Without a detectable transition to region III, one of three criteria is used to evaluate fracture toughness in these materials.

For pure titanium, fracture toughness is based upon the maximum calculated alternating threshold ($\Delta K_{th}$) and fracture toughness ($K_c$) stress intensity and values.

<table>
<thead>
<tr>
<th>Material</th>
<th>$\Delta K_{th}$</th>
<th>$K_c$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti</td>
<td>8.6</td>
<td>39</td>
</tr>
<tr>
<td>Ti-15 vol% TiB</td>
<td>7.4</td>
<td>23</td>
</tr>
<tr>
<td>Ti-30 vol% TiB</td>
<td>5.7</td>
<td>17</td>
</tr>
<tr>
<td>Ti-45 vol% TiB</td>
<td>3.9</td>
<td>9</td>
</tr>
<tr>
<td>Ti-60 vol% TiB</td>
<td>2.6</td>
<td>5.8</td>
</tr>
<tr>
<td>Ti-85 vol% TiB</td>
<td>2.7</td>
<td>5.6</td>
</tr>
</tbody>
</table>

Note: Fracture toughness ($K_c$) not $\Delta K_{max}$ is presented in this table. Given that experiments were run at an R-ratio of 0.5, $K_c$ is twice the maximum $\Delta K$ achieved in the experiments.
stress intensity of 19.5 MPa√m. For materials from 15 to 60 vol% TiB, fracture toughness is based upon fatigue crack propagation results and overload events, which suggest an upper limit of approximately $1.0 \times 10^{-6}$ m/cycle for stable crack growth. Fracture toughness in materials with TiB volume fractions from 15 to 60 percent is estimated to be either the maximum stress intensity achieved and associated with stable crack growth, or the stress intensity at which the Paris equation for a material crosses $1.0 \times 10^{-6}$ m/cycle. Based upon these criteria, estimates for fracture toughness for each of the Ti-TiB materials are shown in Table 3.1.

### 3.3.3.3 Coefficients and Exponents of the Paris Equation

For each of the six TiB volume fractions, the Paris equation

$$\frac{da}{dN} = C \cdot \Delta K^n$$

(3.2)

has been fit to the region II data to estimate values of $C$ and $n$. Where multiple specimen results are available, results from all specimens are combined into one data set and linear regression of the data is used on the complete set of data. Experimental results for $C$ and $n$ for each of the material systems are shown Table 3.2.
3.3.4 Single-Phase Paris Equation Model

The primary objective of research into the fatigue crack propagation behavior of the Ti-TiB FGM material system is to determine if a continuous model describing crack propagation rate as a function of both $\Delta K$ and TiB volume fraction can be developed. The model used here is a modified Paris equation assuming a single TiB phase in the form of randomly oriented high aspect ratio whiskers. To include the new degree of freedom associated with changes in TiB volume fraction, a single TiB phase Paris equation (referred to as the single-phase Paris equation from this point forward), which incorporates functional descriptions for $C$ and $n$ with $V_f$ (TiB volume fraction) as a new independent variable Eq.(3.3), is investigated. Initially the model is assumed to be bounded over some range, but based upon literature review results, not necessarily over the entire range of TiB volume fractions. The model is also bounded by threshold and toughness limits, which are functions of $V_f$ as well.

$$\frac{da}{dN}(\Delta K, V_f) = C(V_f) \cdot \Delta K^{n(V_f)}$$

Table 3.2. Paris equation parameter values.

<table>
<thead>
<tr>
<th>Material</th>
<th>$C$</th>
<th>$n$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti</td>
<td>$6.39 \times 10^{-12}$</td>
<td>3.6</td>
</tr>
<tr>
<td>Ti-15 vol% TiB</td>
<td>$1.16 \times 10^{-16}$</td>
<td>9.5</td>
</tr>
<tr>
<td>Ti-30 vol% TiB</td>
<td>$1.19 \times 10^{-15}$</td>
<td>9.71</td>
</tr>
<tr>
<td>Ti-45 vol% TiB</td>
<td>$5.67 \times 10^{-16}$</td>
<td>14.14</td>
</tr>
<tr>
<td>Ti-60 vol% TiB</td>
<td>$1.59 \times 10^{-15}$</td>
<td>18.93</td>
</tr>
<tr>
<td>Ti-85 vol% TiB</td>
<td>$3.76 \times 10^{-36}$</td>
<td>63.54</td>
</tr>
</tbody>
</table>
3.3.4.1 Determination of C(Vf) and n(Vf)

Development of equations describing changes in C and n as functions of Vf is accomplished in three steps: 1) determination of the range of TiB volume fraction over which crack propagation behavior (C and n) change smoothly and continuously; 2) identification of appropriate functional forms for C(Vf) and n(Vf); and, 3) evaluation of the coefficients of C(Vf) and n(Vf).

Determination of the range of TiB volume fractions, over which C and n can be described by simple continuous functions, is based upon evaluation of C and n for individual Ti-TiB monolithic specimen results and the literature review. Plots of C and n versus TiB volume fraction are shown in Figure 3.9 and Figure 3.10, respectively. Significant departure of values for C and n at 0 and 85 vol% TiB from trends between 15 and 60 vol% TiB suggest that significant changes in crack propagation behavior exist for TiB volume fractions below 15 percent and above 60 percent. Similar departures are seen in results for $\Delta K_{th}$ and $\Delta K_{max}$. The results also suggest that between limits of 15 and 60 vol% TiB, C and n, and therefore crack propagation rate, change smoothly and continuously with changes in TiB volume fraction. Further supporting the observed TiB volume fraction limits are literature review results for mechanical properties for Ti alloys with small additions of boron, and material microstructural changes observed for Ti-TiB. Results in the literature for titanium alloys plus boron suggest that differences in material behavior would be expected below 15 vol% TiB as ductility begins to increase from levels at or near zero for materials containing greater than 15 vol% TiB. The literature also suggests changes in behavior would be expected above approximately 73 vol% TiB, when a change in TiB microstructure to one composed primarily of 1 to 3 \( \mu \)m whiskers.
dominates the TiB phase. Without characterization in these TiB volume fraction ranges, the current models are limited to the intermediate range of 15 to 60 volume percent TiB.

Between 15 and 60 vol% TiB, identification of the appropriate low order functional forms for $C(V_f)$ and $n(V_f)$ is based on observed trends in plots of $C$ and $n$ versus TiB volume fraction. For $C(V_f)$ the best fit is obtained with an exponential equation of the form:

$$ C(V_f) = e^{\alpha_0 \cdot V_f + \alpha_1} \quad (3.4) $$

For $n(V_f)$ the best fit is obtained with a second order polynomial given by

$$ n(V_f) = \alpha_2 \cdot V_f^2 + \alpha_3 \cdot V_f + \alpha_4 \quad (3.5) $$

Figure 3.9. Paris equation "C" coefficients for each TiB volume fraction.
With TiB volume fraction limits and functional forms established for $C(V_f)$ and $n(V_f)$, two options are available to evaluate the coefficients $\alpha_i$ ($i = 0, 1, 2, 3, 4$). The coefficients can be evaluated by fitting Eqs. (3.4) and (3.5) directly to values of $C$ and $n$ evaluated for the individual monolithic materials. However, using the original values produces functions $C(V_f)$ and $n(V_f)$ that are based upon fits of the original Paris equation to individual monolithic material data sets. The most significant disadvantages of using the data sets individually to evaluate the coefficients of $C(V_f)$ and $n(V_f)$ are: 1) the coefficients for $C(V_f)$, ($\alpha_0$, $\alpha_1$), and $n(V_f)$, ($\alpha_2$, $\alpha_3$, $\alpha_4$), are determined independently, minimizing or eliminating potential coupling between $C(V_f)$ and $n(V_f)$ that may exist across the TiB volume fraction range of interest; and 2) the coefficients for $C(V_f)$ and $n(V_f)$ are based upon a single independent variable, TiB volume fraction. A better approach to determining $C(V_f)$ and $n(V_f)$ is to evaluate the coefficients of the equations

![Figure 3.10. Paris equation exponent "n" values for each TiB volume fraction.](image)

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for C(Vf) and n(Vf) jointly. Joint evaluation of the coefficients maintains any correlation that may exist over the TiB volume fraction range of interest, and bases the coefficients of C(Vf) and n(Vf) directly on both independent variables, TiB volume fraction and ΔK, along with the response variable da/dN.

To evaluate the coefficients αi using the complete set of experimental data, the single-phase Paris equation

\[
\frac{da}{dN} = e^{\alpha_0 \cdot V_f + \alpha_1 \cdot \Delta K + \alpha_2 \cdot V_f^2 + \alpha_3 \cdot V_f + \alpha_4}
\]  

(3.6) is written in the standard form

\[
\ln \left( \frac{da}{dN} \right) = \alpha_0 \cdot V_f + \alpha_1 + \alpha_2 \cdot V_f^2 \cdot \ln(\Delta K) + \alpha_3 \cdot V_f \cdot \ln(\Delta K) + \alpha_4 \cdot \ln(\Delta K)
\]

(3.7) for solution of the coefficients using multi-variable regression. In Eq. (3.7) Vf, ΔK and da/dN are known experimental values, leaving the coefficients αi as the only unknown quantities. The coefficients are found by solving the matrix equation

\[
\tilde{\alpha} = \left( X^T \cdot X \right)^{-1} \cdot X^T \cdot Y
\]

(3.8) where X

\[
X = \begin{bmatrix} V_{f1}, 1, V_{f1}^2 \cdot \ln(\Delta K), V_{f1} \cdot \ln(\Delta K), \ln(\Delta K) \end{bmatrix}
\]

(3.9) is a matrix based upon measured experimental values, and Y

\[
Y = \ln \left( \frac{da}{dN} \right)
\]

(3.10) is the natural log of measured fatigue crack propagation rate.

The resulting equations for C(Vf) and n(Vf) are:
\[ C(V_f) = e^{4.742V_f - 36.84} \quad (3.11) \]

\[ n(V_f) = 50.88 \cdot V_f^2 - 16.35 \cdot V_f + 10.61 \quad (3.12) \]

### 3.3.4.2 Evaluation of \( \Delta K_{th} \) and \( \Delta K_{max} \) Limits

Evaluation of plots of \( \Delta K_{th} \) and \( \Delta K_{max} \) versus TiB volume fraction shown in Figure 3.11 and Figure 3.12 respectively yield two observations. First, with the exception of \( \Delta K_{th} \), at 0 vol\% TiB, \( \Delta K_{th} \) and \( \Delta K_{max} \) support establishment of the 15 to 60 vol\% TiB range over which the properties can be modeled as continuous functions of TiB volume fraction and \( \Delta K \). Similar to results for C and n, trends of \( \Delta K_{th} \) and \( \Delta K_{max} \) change smoothly and continuously between TiB volume fraction limits of 15 and 60 percent, while values deviate significantly from the trends at 0 and 85 vol\% TiB. Second, between 15 and 60 vol\% TiB, \( \Delta K_{th} \) and \( \Delta K_{max} \) can be modeled by simple linear equations, with TiB volume fraction as the independent variable. Unfortunately, due to uncertainty in calculated stress intensity values for the experimental conditions, the very narrow range between \( \Delta K_{th} \) and \( \Delta K_{max} \) (0.3 MPa \( \sqrt{m} \)) for the 60 vol\% TiB material, and inherent scatter in experimental results for \( da/dN \), unrestricted linear models for \( \Delta K_{th}(V_f) \) and \( \Delta K_{max}(V_f) \) cross and predict a lower toughness value than threshold value for TiB volume fractions above 58 percent. To prevent this inversion, linear equations representing \( \Delta K_{th} \) and \( \Delta K_{max} \) are forced through measured values of \( \Delta K_{th} \) and \( \Delta K_{max} \) at 60 vol\% TiB. The results yield limiting equations

\[ \Delta K_{th} = -10.28 \cdot V_f + 8.768 \text{ (MPa} \sqrt{m}) \quad (3.13) \]
that are valid between TiB limits of 15 and 60 percent.

\[ \Delta K_{\text{max}} = -18.72 \cdot V_f + 14.132 \text{ (MPa}\sqrt{m}) \]  \hspace{1cm} (3.14)

With limiting equations for threshold and toughness established, the complete model for crack propagation is given by:

\[ \frac{da}{dN} (K, V_f) = e^{4.742 \cdot V_f - 36.84 \cdot \Delta K - 50.88 \cdot V_f^2 - 16.35 \cdot V_f + 10.61} \]  \hspace{1cm} (3.15)
3.4 Conclusions

The forgoing discussion documents fatigue crack growth experiments in six monolithic Ti-TiB volume fractions ranging from 0 to 85 percent, and the methods necessary to characterize these materials. For materials with TiB volume fractions of 30 percent or less, traditional metal precracking and propagation techniques have been successfully used to characterize the materials. However, the exceptionally brittle behavior of materials containing 45 volume percent TiB or more required the development of a notch design with two key attributes to enable stable precracking and fatigue crack propagation. First, the notch produces a stress concentration factor of approximately 30 to ensure critical compression precracking stress levels are reached in
bending, while limiting the tensile stresses that developed along the opposite edge of the specimen to below ultimate. Second, the notch provides a slightly decreasing stress intensity gradient along its length, ensuring stable crack extension during fatigue crack growth experiments. This aspect became particularly important in characterization efforts in monolithic specimens in which TiB volume fraction equaled or exceeded 60 percent. In these materials, stable crack propagation in the increasing stress intensity gradient outside the notch inevitably led to uncontrollable and abrupt failure. Combined, the two experimental characterization methods enabled successful characterization of fatigue crack propagation properties in all six material volume fractions.

Evaluation of $C$, $n$, $\Delta K_{th}$ and $K_c$ for each volume fraction, and observed trends in these parameters as a function of TiB volume fraction, indicate that, despite the significant change in TiB microstructure from a 50 µm to a 10 µm whisker with the transition from 30 to 45 vol% TiB, fatigue crack propagation rates appear to vary smoothly between TiB volume fraction limits of 15 and 60 percent. Based upon these observed trends, functional forms for $C(V_f)$ and $n(V_f)$ have been developed and incorporated into a Paris equation to develop a single predictive model spanning a TiB volume fraction range from 15 to 60 percent.
Chapter 4

DIC Based Stress Intensity Recovery

Digital image correlation is a computational method which uses multiple photographic images to track displacements and distortions of the surface of a body in up to three dimensions. In post test analyses, thousands of user defined points, where values of strain and displacement are required, can be specified. Centered on each point in the baseline image, pixel subsets are defined and a characteristic gray scale value for each subset is calculated. In the deformed images, the material element surface need not, and normally would not, maintain a one to one correspondence with the pixel matrix. In most cases, the material element surface will strain, translate and rotate such that it occupies a non-integer number of pixels (Figure 4.1). A computer algorithm searches the vicinity of the original points in the deformed image, evaluating subpixel regions until a best match of the original characteristic gray scale value is found at each point. Over the surface of the body the results provide a two or three dimensional map of displacements and strains similar to those obtained from finite element analysis. From an experimental standpoint, this technique and its results allow experimental measurements acquired directly from a test article to be manipulated much like finite element analysis results. Contour maps of
strain and displacement can be plotted, and results can be post-processed to evaluate such quantities as stress intensity.

Figure 4.1. Baseline undeformed and deformed images showing relationship between the material element and the pixel matrix before and after the body displaces and strains.
Recently, Abanto-Bueno and Lambros [2, 3] used digital image correlation and the first two terms of the Williams equation to evaluate stress intensity and T-stress in functionally graded polyethylene carbon monoxide specimens. However, attempts to use their approach in monolithic titanium produce poor results. When compared to values of $K_I$ based upon handbook [53] and finite element analysis solutions from IFranc [34] and Franc2DL [55], differences exceeding 10 percent are common (Figure 4.2).

To address the problem, a hybrid analysis using numerical experiments to produce crack tip displacements and simulate DIC data is coupled with experimental data analysis algorithms to evaluate potential sources of error in recovered stress intensity and T-stress. After a series of numerical experiments, a reliable method for accounting for and minimizing the errors is developed. The method is validated by reanalysis of the initial data from monolithic titanium experiments. Additional numerical experiments are conducted to evaluate the applicability of the method for recovery of stress intensity and T-stress in exponentially and constructively graded (layered) materials.
In these experiments, digital image correlation is used to measure the 2-D displacement fields \((u, v)\) in the vicinity of a crack tip. Images approximately 6 mm wide and 8 mm high, centered on the crack tip, are recorded at 50 and 100 percent of the peak constant amplitude load for a given fatigue propagation experiment, with the objective of later recovering \(\Delta K_I\) at \(R = 0.5\). To acquire these images, the test is temporarily paused at the required load levels. Using a digital image correlation research code, written by Dr. Jorge Abanto-Bueno and provided by Dr. John Lambros, a rectangular array of measurements \((u, v, du/dx, dv/dy, du/dy\) and \(dv/dx)\) at approximately 3000 points (Figure 4.3) around the crack tip is produced.
To minimize regression errors, displacement measurements made in the region around the crack tip are filtered and averaged locally. Displacements for which correlation values are low due to a locally poor speckle pattern, and invalid displacements due to a crossing of the crack by a DIC subset, are removed. Displacements in a 1.5 mm square region ahead of the crack tip, which might be subject to nonlinear processes such as plasticity and microcracking, and complex 3-D stress fields that violate the assumption of plane stress upon which the Williams equations are based are also removed.

Figure 4.3. Typical DIC image measurement locations.
Figure 4.4. Unfiltered and filtered displacement measurements.
Typical unfiltered, along with filtered and averaged crack opening displacement results are shown in Figure 4.4. Note that the displacements plotted in the vertical direction in the plots are actually in-plane displacements in the direction of crack opening. Apparent in the unfiltered data in Figure 4.4a are invalid measurements along the crack flanks and typical measurement variability seen throughout the region. Filtered and averaged results are shown in Figure 4.4b. Figure 4.5 shows filtered measurement locations relative to the crack tip, which is located at the origin.


4.2 Recovery of Stress Intensity and T-Stress

Stress intensity and T-stress are recovered from DIC displacement measurements using MVR (multivariable regression) to solve for rigid body translation and rotation and for the unknown coefficients \( \beta_n \) in the asymptotic equation for crack opening displacements at the crack tip given by:

\[
v_i = \beta_0 + \sum_{n=1}^{m} \left( \frac{n}{2} \right) \beta_n \left[ \kappa - \frac{n}{2} - (-1)^n \sin \left( \frac{n}{2} \theta_i \right) + \sin \left( \frac{n}{2} \left( -2 \theta_i \right) \right) \right] + \beta_{m+1} r_i \cos \theta_i
\]

where

\[
\kappa = \frac{3 - \nu}{1 + \nu}
\]

\( i \) is the data point number

\( m \) is the maximum number of terms of the asymptotic equation used in the regression

\( \beta_0 \) is rigid body translation in \( y \)

\( \beta_{m+1} \) is equal to \( \sin(\varphi) \)

\( \varphi \) is the angle of rigid body rotation

\( \kappa \) is the plane stress factor

The equation is in the form necessary to perform multiple linear regression:

\[
V = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \ldots
\]
where:

- $V$ is of the filtered 2-D displacement field
- $\beta$ is a vector of the unknown coefficients of the asymptotic equation for crack tip displacements
- $X$ is a matrix of known values of the asymptotic equation for crack tip displacements and rigid body translation and rotation terms given by:

$$X_{i,n} = 1 + \left[ \frac{r_i}{2G} \left( \kappa - \frac{n}{2} - (-1)^{\alpha} \right) \sin \left( \frac{n}{2} \pi i \right) + \frac{n}{2} \sin \left( \frac{n}{2} \pi i \right) \right] + r_i \cos \theta_i. \quad (4.4)$$

Solutions for $\beta$ are obtained from the solution of the matrix equation:

$$\beta = \left( X^T \cdot X \right)^{-1} \cdot X^T \cdot V \quad (4.5)$$

From the solution for $\beta_1$ and $\beta_2$, $K_I$ and T-stress are found from the equations:

$$K_I = \beta_1 \sqrt{2\pi} \quad (4.6)$$

$$T = 4 \left[ \beta_2 - \frac{2G}{\kappa - 3} (\cos(\phi) - 1) \right] \quad (4.7)$$

In this case, the value recovered for T-stress is corrected to remove the influence of rigid body rotation, to which it is extremely sensitive. However, this can only be done if mode II stress intensity is zero; otherwise the contributions of mode II displacements, rigid body rotation and T-stress cannot be uniquely determined, and equation (9) is invalid.
4.3 Numerical Experiments

Three series of numerical experiments and analyses have been conducted for four-point bend specimens. The first is conducted assuming monolithic material properties. Subsequent numerical experiments assume exponential and layered FGM forms. The objectives of the experiment include: development, implementation and verification of an algorithm for recovery of model I stress intensity and T-stress from experimentally measured 2-D displacement fields; development of a better understanding of the impact of identified sources of error on recovered stress intensity and T-stress; and development of a procedure for mitigating the impact of these sources of error. Subsequent analyses based on experimental data are used to validate the methods and algorithm.

In the first series of experiments, monolithic material properties are used to verify the multivariable regression algorithm for recovery stress intensity and T-stress, and to evaluate suspected sources of error. Starting with a monolithic material has the advantage of using a well-understood problem for which the analytical displacement equations for an ideal elastic material are known. It also allows direct comparison between interaction integral solutions and multivariable regression results, without the uncertainties that would develop if initial experiments were to assume functionally graded properties. Numerical experiments and analyses in FGMs extend the research to evaluate the methods and algorithm for use with exponentially graded and constructively graded (layered) materials.
4.3.1 Sources of Error in Regressed Stress Intensity

Several variables can be sources of error when using the experimentally measured crack tip displacement field to recover stress intensity and T-stress. These include aspects of the geometry of the problem, including the region over which displacement measurements are made and the proximity of the crack tip to a boundary. Both impact the number of terms of the Williams equation that must be retained in the regression to obtain accurate values of stress intensity and T-stress. The objective of numerical experiments in this case is to evaluate the number of terms of the Williams equation that must be retained in the regression, based upon the proximity of the crack tip to a boundary and the size of the region over which displacement measurements are made.

A second and more significant source of error in recovered stress intensity is the accuracy with which the crack tip is located. Here the problem is different in that if the crack tip is not accurately located, stress intensity and T-stress results will not converge regardless of the number of terms retained in the regression. In this case, the Williams equation, unmodified to account for the fact that the origin of the problem is not at the crack tip, is inappropriate for describing the crack tip displacement field. The objectives of numerical experiments here are to evaluate trends in recovered stress intensity and T-stress as coefficients are added to the regression algorithm, and to develop a method by which the effective crack tip can be identified. Once identified, the assumed multivariable regression origin can be moved to the effective crack tip coordinates, allowing accurate recovery of stress intensity and T-stress.
To achieve the objectives outlined above, crack tip displacement fields from finite element analyses are used in place of experimental measurements to test the data analysis algorithms. Using numerical experimental results in this case has multiple advantages. Displacements are essentially noise free, and the precise location of the crack tip is known. These attributes eliminate two primary and uncontrollable variables associated with experimental data, allowing for accurate assessments of the impacts of measurement window size and boundary proximity. They also permit the effect of errors in the assumed crack tip location, to be evaluated independently of measurement noise.

4.3.2 Numerical Models and Procedures

Numerical experiments and subsequent multivariable regression are intended to support specific experimental studies of fatigue crack propagation in four-point bend monolithic and functionally graded specimens. As a result, the finite element models used to produce crack tip region displacement fields closely match the geometry, material properties (monolithic and functionally graded), loading and boundary conditions of the four-point bend specimens used in actual experiments (Figure 4.6).
The objectives of numerical experiments (an experiment being defined as an analysis for a single crack length and material system) are to generate two dimensional plane stress displacement fields over the surface of the model, and to evaluate stress intensity and T-stress values based upon the interaction integral. In general, numerical experiments are conducted in 1.0 mm increments for cracks lengths ranging from 3.0 to 13.5 mm (a/W from 0.12 to 0.54). However, for the seven layer FGM, increments as small as 0.125 mm are made in order to generate results as the crack approaches, transits and propagates away from a boundary between layers. The smaller increments are necessary to adequately capture the trends that develop in these regions. In a typical case, displacement data at each crack increment are filtered to produce a rectangular array of displacement measurements centered on the crack tip, which approximately matches DIC measurements in terms of the number of data points and their spatial distribution. In addition, in some analyses, larger and smaller arrays representing larger and smaller measurement windows around the crack tip are produced, to study the impact of the size
of the measured displacement field on the convergence rate of the multivariable
regression algorithm.

4.3.3 Monolithic Titanium

Numerical experiments and analyses have been conducted for three primary
conditions that are likely to exist in an actual experiment, and that may have an impact on
recovered values of stress intensity and T-stress. Assuming the crack tip has been
accurately located, the first set of numerical experiments evaluates the combined impact
of measurement window size and crack tip proximity to a specimen boundary on the
number of terms required to achieve converged results for stress intensity and T-stress.
Separately, the impact of erroneously locating the crack tip in the x and y coordinate
directions is evaluated, and a method for identifying the effective crack tip location is
developed.

4.3.3.1 Measurement Window Size and Boundary Proximity Results

The analysis of measurement window size and crack tip proximity to a boundary
addresses the issue of defining the number of terms required to achieve converged results
for stress intensity and T-stress. Three questions are answered by the analysis. 1) Typically how many terms must be included when using multivariable regression to
achieve converged results for stress intensity? 2) How do changes in the size of the
measurement window impact the number of required terms? 3) How does proximity of
the crack tip to a boundary modify the above results? Based upon analyses of
measurement window sizes ranging from 4.0 mm x 5.0 mm to 7.2 mm x 9.0 mm, in
combination with crack lengths from 3.0 mm to 13.5 mm, it is clear that the window of observation size within this range has a limited impact on the number of terms required to achieve a converged solution for stress intensity and T-stress. For short cracks (3.0 mm), where the crack tip is relatively close to the boundary, the size of the measurement window does have an effect on the number of terms required for convergence. However, stress intensity variability before convergence is achieved is small, 7 percent in the worst case for the 7.2 mm x 9.0 mm window, and less than 4 percent for a typical experimental window size of 5.1 mm x 6.4 mm (Figure 4.7).

Figure 4.7. Effect of window size on a short crack ($K_I = 5.18$ MPa $\sqrt{m}$).

\[ a/W = 0.12 \]
As the crack length increases, the impact of measurement window size quickly diminishes. The impact of measurement window size for 7.0 mm and 11.0 mm crack lengths ($a/W = 0.28, 0.44$) is shown in Figure 4.8 and Figure 4.9. Note that at these distances from the boundary, the number of terms required to converge does not change with crack length. The primary impact as window size increases, regardless of the proximity of the crack tip to the boundary, is to increase the error in recovered stress intensity when relatively few terms are included in the equation. This result is consistent with published results for the K-dominance region [50].

![Figure 4.8. Effect of window size on an intermediate length crack ($K_I = 8.36 \text{ MPa } \sqrt{m}$).](image)
Results for T-stress are a stronger function of measurement window size only when the crack tip is near a boundary. Here the magnitude of the error is large if insufficient terms are used, particularly for the larger window sizes. For short cracks the magnitude of error that develops for T-stress is up to 450 percent for the largest window (7.2 mm x 9.0 mm), as shown in Figure 4.10. For the smallest measurement window evaluated, the difference between interaction integral results and the regression based T-stress is still 200 percent. In contrast, for longer cracks, a/W > 0.28, convergence is quickly achieved in five to six terms regardless of window of observation size (Figure 4.11).

Figure 4.9. Effect of window size on a long crack ($K_I = 12.69 \text{ MPa} \sqrt{\text{m}}$).
Figure 4.10. T-stress results for a short crack (T = -18.07).

Figure 4.11. T-stress results for an intermediate length crack (T = -5.63).
These results demonstrate that for an ideal material: 1) except for the case of a short crack, the measurement window size does not affect the number of terms required to achieve a converged solution for stress intensity or T-stress; 2) the number of terms needed to achieve an accurate solution is typically on the order of four or five for stress intensity and six for T-stress; and 3) for the short crack, adequate accuracy can be achieved for stress intensity in relatively few terms before the results are fully converged. However, accurate T-stress results for a short crack require that enough terms be retained to achieve convergence, typically more than 10.

4.3.3.2 Identification of Effective Crack Tip Coordinate Location

The most significant errors in recovered stress intensity and T-stress stem from inaccuracies in identifying the crack tip location. Sources of inaccuracies include experimental equipment limitations such as camera resolution and magnification, which limit the ability to observe the physical limits of the crack on the surface of the specimen. Crack tip attributes such as plasticity, microcracking, surface and subsurface crack bifurcation, and crack front curvature also limit the accuracy with which the effective crack tip can be located. Unfortunately, most of these aspects of the cracking process cannot normally be detected or accounted for directly at any magnification or camera resolution. As a result, an indirect method for determining the coordinates of the effective crack tip is required. To develop such a method, numerical experiments have been conducted to evaluate the impact on recovered stress intensity for cases where the assumed crack tip location differs by a small amount from the actual crack tip location. Analytically, errors are introduced by shifting the origin of the regression analysis away
from the known crack tip in increments up to ±0.2 mm in each coordinate direction (Δx, Δy) (Figure 4.12). At each increment, stress intensity and T-stress are evaluated to assess the trends that develop as terms are added to the regression. The results reveal that consistent trends in recovered stress intensity and T-stress develop, and that they are a function of the magnitude of error in the effective crack tip coordinates. As a result, the trends can be used to adjust the assumed crack tip coordinates to match the effective crack tip coordinates, allowing accurate results for stress intensity and T-stress to be recovered.

4.3.3.2.1 Mode I Stress Intensity Results

The interaction integral in Franc2DL [55] has been used to evaluate stress intensity and T-stress for a/W from 0.12 to 0.54. Results at a/W = 0.12, 0.28 and 0.44
(3.0, 7.0 and 11.0 mm), shown in Table 4.1, form the basis against which regression-based stress intensity results are evaluated. For each a/W, multivariable regression is used to evaluate stress intensity and T-stress as a function of the number of terms in the expansion as the errors in identifying the effective crack tip coordinates, Δx and Δy, take on values of 0.0, ±0.10 and ±0.20 mm.

Table 4.1. Franc2DL stress intensity results and IFranc T-stress results.

<table>
<thead>
<tr>
<th>Finite Element Results</th>
</tr>
</thead>
<tbody>
<tr>
<td>a/W</td>
</tr>
<tr>
<td>ΔK_I (MPa v/m)</td>
</tr>
<tr>
<td>T-stress (MPa)</td>
</tr>
</tbody>
</table>

It has already been established that for Δx and Δy both equal to zero (i.e., the effective crack tip has been accurately located), the trends that develop with the addition of higher order terms of the Williams equation in the regression converge to accurate stress intensity and T-stress results after approximately five and seven terms, respectively. The current objectives are to evaluate, for values of Δx and Δy other than zero: the magnitude of errors in recovered stress intensity and T-stress; the trends that develop for stress intensity and T-stress versus number of terms used in the regression; and, whether these trends can be used to locate the effective crack tip and accurately recover stress intensity and T-stress.

For values of Δx other than zero, the error in stress intensity that develops as a percentage of the actual stress intensity appears to be independent of crack length.
Virtually identical trends in percent error, defined as \((K_{reg} - K_{int})/K_{int}\), develop independently of crack length as seen in Figure 4.13 for \(\Delta x = \pm 0.2\) mm at \(a/W = 0.12, 0.28\) and 0.44. In absolute terms the degree of non-convergence in stress intensity increases as \(\Delta x\) assumes larger absolute values. Shown in Figure 4.14 are results at \(a/W = 0.44\) and \(\Delta x = 0, \pm 0.1\) and \(\pm 0.2\) mm.

![Figure 4.13. Error in stress intensity trends with error in crack tip location.](image)

Evident in Figure 4.14 are several important trends in stress intensity versus crack tip location error \(\Delta x\). First, for non-zero values of \(\Delta x\), stress intensity fails to converge as the number of terms used in the multivariable regression increases. Second, as \(\Delta x\) assumes larger positive values, the slope of stress intensity versus number of higher order terms (terms above 4) becomes more negative, and as \(\Delta x\) assumes larger negative values the slope becomes more positive. Third, in both cases the change in slope appears to be
linearly proportional to the magnitude of \( \Delta x \) (Figure 4.15). Given the consistency of the trends, the results can be used to identify whether the x coordinate of the effective crack tip has been located accurately and, if not, to determine the relative magnitude of the error \( \Delta x \). Using the magnitude of the slope of \( \Delta K \) versus number of terms as a guide, the origin can be shifted iteratively until the slope is zero, at which point stress intensity will have been accurately recovered.

Figure 4.14. Stress intensity trends with error in crack tip location (\( a/W = 0.44 \)).
In contrast, the impact of error locating the crack tip in the y coordinate direction on mode I stress intensity is significantly less than the effect of $\Delta x$. For all values of $a/W$ from 0.12 to 0.44, and for all window sizes, relatively large values of $\Delta y$ have only a slight impact on the accuracy of recovered stress intensity. Figure 4.16 shows the change in stress intensity as a function of the number of terms using the regression algorithm for a 7.0 mm crack ($a/W = 0.28$). Errors in recovered stress intensity for values of $\Delta y$ up to 0.2 mm are no more than -1.3 percent based upon a five term regression, which is well within acceptable experimental limits. Also, in contrast to trends for $\Delta x$, positive and negative values for $\Delta y$ yield similar results in sign and magnitude for the trends that develop. These characteristics for $\Delta y$ have two important implications. First, precise identification of the y coordinate of the effective crack tip does not appear to be necessary for mode I stress intensity to be accurately recovered. Second, the

![Figure 4.15. Error in recovered stress intensity based upon 5 terms as a function of $\Delta x$.](image)
insensitivity of stress intensity to Δy limits the usefulness of this parameter for determining the effective crack tip coordinates.

4.3.3.2.2 T-stress Results

Results for T-stress follow similar trends to those observed for stress intensity. Positive slopes develop if the crack tip coordinate is underestimated (Δx < 0) and negative slopes develop if it is overestimated. However, while the general trends are similar for Δx, T-stress is much more sensitive to crack tip location inaccuracies. T-stress based upon the interaction integral solver in IFranc [34] for a/W = 0.28 is 5.63 MPa, while the regression results for a seven term regression are -102 MPa for Δx = 0.2 mm and 168 MPa for Δx = -0.2 mm, or 1700 and 3100 percent (Figure 4.17) respectively.

![Figure 4.16. Impact of crack tip location error Δy on stress intensity.](image)
T-stress results also exhibit significant dependence on crack tip coordinate inaccuracies in the y direction. Figure 4.18 shows T-stress results for Δy ranging from 0 to 0.2 mm. Here the six term solution for T-stress for non-zero values of ±Δy produces significant inaccuracies, up to ~600 percent for a value of Δy = ±0.2 mm.

4.3.3.3 Procedure for Recovery of Mode I Stress Intensity and T-stress

Based upon the preceding results, it is clear that accurate stress intensity results can be obtained if the trends that develop with changing window size, crack length, Δx and Δy are understood. It is also clear that for mode I stress intensity, Δx is the dominant variable that must be accounted for in the analysis, and that if Δy is less than a few tenths of a millimeter, its influence on recovered stress intensity is minimal.
Based on the results, an iterative procedure for identifying the coordinates of the effective crack tip and accurately recovering mode I stress intensity has been developed.

1) Identification of the surface-connected crack tip, and measurement of crack opening displacements in a two-dimensional region centered on this point.

2) Approximation of stress intensity using a multivariable regression algorithm based upon the Williams equation for plane stress mode I displacements, for orders of the equation from $r^{\frac{1}{2}}$ to $r^{10}$.

Figure 4.18. Impact of $\Delta y$ on T-stress.

Refinement of the method for T-stress is not accomplished for reasons that will become clear later. The procedure is a four-step process including:
3) Evaluation of the trend in stress intensity that develops as the order of the equation increases, noting that:

a) A positive slope indicates that the observed crack length underestimates the effective crack length, requiring a shift in the origin of the measurement region in the positive x direction by an amount $\Delta x$. *(Note that new measurements are not required, just an adjustment of the x coordinates by $\Delta x$ such that the measurements now are with respect to a new origin.)*

b) A negative slope indicates that the observed crack length overestimates the effective crack length, requiring a shift in the x direction by $-\Delta x$.

c) The magnitude of the slope is proportional to the error in locating the crack tip and thus larger slopes require greater correction of the assumed effective crack tip location.

4) Iteration and re-evaluation of stress intensity and stress intensity trends, and correction of the assumed crack tip location until the addition of higher order terms (typically above four or five) have little significant impact on recovered stress intensity (i.e., the slope of stress intensity as a function of equation order is approximately zero).

This procedure assumes that the crack tip y coordinate has been found to within 0.2 mm and that mode II and III displacements are insignificant.

**4.3.3.4 Validation of Numerical Experiments**

To validate the developed algorithm and methods, the data that motivated this research has been re-evaluated. Stress intensity results for each crack length in the
original data set are recalculated and plotted along with the original values and analytical solutions in Figure 4.19. Using the method for locating the effective crack tip, results for stress intensity are typically within ±5 percent. In contrast, recovery of meaningful T-stress values is not possible due to the magnitude of noise in measured displacements, which, while small, are of the same order of magnitude as the v displacements due to T-stress.

Figure 4.19. Experimental results for a wrought titanium four-point bend specimen showing ±5 percent bounds.
4.3.4 Functionally Graded Material Results

Theoretical and limited experimental results suggest that with a minor modification, the Williams equation used for monolithic materials can be used to recover stress intensity and T-stress in functionally graded materials. The following work extends the multivariable regression and crack tip identification methods, developed and validated for the monolithic case, to include exponentially and constructively graded materials. The objectives here, as suggested by the work of Abanto-Bueno and Lambros [2, 3], are to determine if the regression algorithm based upon the Williams equation can be used to recover stress intensity and T-stress in two FGM forms, exponentially and constructively graded, and if so, to determine how quickly the results converge. Two specific questions are answered by the numerical experiments: under ideal conditions, with what accuracy can stress intensity and T-stress be recovered; and are there conditions for which the use of the Williams equation does not produce accurate results?

4.3.4.1 Exponentially Graded FGMs

The applicability of multivariable regression based upon the Williams equations for crack tip displacements, with material properties evaluated at the crack tip as suggested by Eishen [26], is tested by comparison with stress intensity and T-stress results generated using the interaction integral in IFranc [34]. Using IFranc, a plane stress four-point bend model 152.4 mm x 25.0 mm x 1.0 mm is analyzed at crack lengths from 3.0 to 13.0 mm at a total applied load of 200 N to generate crack tip displacements. Load point locations are identical to previous dimensions for the monolithic model. The analyses assume two exponential material gradients based upon the modulus and
Poisson’s ratio for Ti (110 GPa, 0.32) and Ti-85 vol% TiB (321 GPa, 0.18). In the first analysis the crack propagates from the pure titanium edge of the specimen into an increasing modulus and decreasing Poisson’s ratio gradient. Here, modulus increases according to the equation $E(x) = E_0 \cdot e^{\beta x}$ GPa (where $E_0=110$ GPa, $\beta = 0.0428$ and $x$ is the distance from the edge of the specimen, in a direction parallel to the material gradient). Poisson’s ratio decreases linearly (a limitation in IFranc) between 0.32 and 0.18. In the second case the crack propagates from the Ti-85 vol% TiB edge into a decreasing modulus and increasing Poisson’s ratio gradient. The modulus profile in this case is given by $E(x) = e^{-0.0428x}$ GPa and Poisson’s ratio again varies linearly, this time between 0.18 and 0.32. Crack propagation and modulus gradient orientations are shown in Figure 4.20.

Figure 4.20. Exponential material gradients.
The numerical experiment approach is identical to the approach and methods used for the monolithic material investigation, though this investigation is focused exclusively on material property gradient effects on convergence rate and convergence accuracy. Finite element displacement results within a measurement window 5.1 mm wide and 6.4 mm high are filtered to remove data along the crack and within a rectangular region around the crack tip. The data are then used with the multivariable regression algorithm to recover stress intensity and T-stress for comparison with interaction integral results.

Table 4.2 and Figure 4.21 show the convergence behavior of recovered stress intensity for cracks ranging in length from 3.0 to 13.0 mm for the exponentially increasing material properties case. In general, convergence is achieved with the inclusion of the fifth term of the Williams equation in the regression. Table 4.3 and Figure 4.22 show absolute results for finite element and regression based stress intensities (based upon five terms) for increasing and decreasing material property gradients. Typically, the multivariable regression solutions, based upon five terms, agree with interaction integral results to within less than one percent.
Table 4.2. Normalized \((K_{I-DIC}/K_{I-M-int})\) convergence behavior.

<table>
<thead>
<tr>
<th>Terms</th>
<th>Crack Length (mm)</th>
<th>3 (mm)</th>
<th>5 (mm)</th>
<th>7 (mm)</th>
<th>9 (mm)</th>
<th>11 (mm)</th>
<th>13 (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td></td>
<td>1.02</td>
<td>1.05</td>
<td>1.05</td>
<td>1.06</td>
<td>1.06</td>
<td>1.06</td>
</tr>
<tr>
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<tr>
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<td>1.00</td>
<td>1.00</td>
<td>1.00</td>
<td>1.00</td>
<td>0.99</td>
</tr>
</tbody>
</table>

Figure 4.21. Typical convergence behavior.
Table 4.3. Comparison of IFranc (34) and regression based solutions for $K_I$ (MPa√m).

<table>
<thead>
<tr>
<th>a (mm)</th>
<th>Material Gradient</th>
<th>$\beta = 0.0428$</th>
<th>$\beta = -0.0429$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>IFranc</td>
<td>Regression</td>
<td>IFranc</td>
</tr>
<tr>
<td>3</td>
<td>3.2</td>
<td>3.2</td>
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<tr>
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<tr>
<td>13</td>
<td>11.4</td>
<td>11.4</td>
<td>15.8</td>
</tr>
</tbody>
</table>

Figure 4.22. Comparison of IFranc and DIC based solutions for stress intensity.
Similar evaluation of T-stress produces less satisfactory results. Figure 4.23 shows the convergence behavior of T-stress for an exponentially graded (β = 0.0428) material specimen. Results for 3.0 and 9.0 mm long cracks are poor for both material gradients, for each crack length studied. Convergence requires from 15 to 20 terms, far more than the six required for the monolithic case, and the converged values of -13.6 and -8.6 MPa do not agree with interaction integral results of -17 and 3.4 MPa respectively. Figure 4.24 shows 15-term regression and modified interaction integral results for monolithic and exponentially graded materials, for crack lengths ranging from 3.0 to 13.0 mm. The results clearly show a general failure of the regression results to converge to the interaction integral results across the entire range of gradients and crack lengths. Together, poor convergence behavior and consistent failure to converge to values which match the interaction integral results lead to the conclusion that T-stress cannot be recovered for an exponentially graded material using a measurement window size of 5.1 mm x 6.4 mm, even under ideal conditions.
However, the possibility remains that T-stress might be accurately recovered if displacements are confined to a small enough region around the crack tip. To evaluate this possibility, a limited evaluation of recovered T-stress versus measurement window size has been conducted. To maximize the likelihood that results converge to accurate T-stress values, the 1.5 mm region typically removed from displacement data measurements is retained and the exclusion zone along the length of the crack is reduced to 0.1 mm. Normalized T-stress results (T_{reg}/T_{M-int}) shown in Figure 4.25 for the 3.0 and 9.0 mm cracks demonstrate that while convergence may occur in fewer terms as measurement window size diminishes, the converged solution still does not correlate with interaction integral based results.

Figure 4.23. T-stress convergence behavior for 3.0 and 9.0 mm long cracks.
Figure 4.24. T-stress results for monolithic and exponentially graded materials.
Figure 4.25. Normalized T-stress ($T_{reg}/T_{M-int}$) convergence for a) 3.0 and b) 9.0 mm cracks.
4.3.4.2 Constructively Graded FGMs

The final step in the analysis is to test the applicability of the developed methodology for crack tip location identification and recovery of stress intensity for a crack propagating through a constructively graded FGM, the focus of this research. However, given the validation results in monolithic titanium and the foregoing analysis for exponentially graded materials, only stress intensity is considered in this analysis.

For the analysis, material properties and general layer thicknesses and dimensions of actual FGM specimens (Table 4.4) are used in Franc2DL [55] to generate the crack tip displacement fields. Results for cracks ranging in length from 3.0 to 13.75 mm, growing into increasing and decreasing material property gradients, are shown in Figure 4.26. Despite the large region over which measurements are made, and the fact that in most cases the measurement window overlaps two or more layers of the specimen, the regressed values of stress intensity typically converge with the addition of the 4th or 5th term (Figure 4.27). Furthermore the results match the interaction integral results produced by Franc2DL within two percent in most cases. The worst correlation, six percent or less, is always seen after the crack tip has passed into the next layer. However, the magnitude of the error quickly diminishes as the crack propagates away from the interface. Also, trends in stress intensity versus the number of terms in the regression are similar to those that develop for the monolithic and exponential FGM cases (Figure 4.27).
Table 4.4. As measured FGM material properties and FEA model layer thicknesses.

<table>
<thead>
<tr>
<th>Ti (%)</th>
<th>TiB (%)</th>
<th>E (GPa)</th>
<th>ν</th>
<th>Thickness (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>0</td>
<td>110</td>
<td>0.32</td>
<td>6.25</td>
</tr>
<tr>
<td>85</td>
<td>15</td>
<td>144</td>
<td>0.28</td>
<td>3.125</td>
</tr>
<tr>
<td>70</td>
<td>30</td>
<td>171</td>
<td>0.28</td>
<td>3.125</td>
</tr>
<tr>
<td>55</td>
<td>45</td>
<td>213</td>
<td>0.24</td>
<td>3.125</td>
</tr>
<tr>
<td>40</td>
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</tr>
<tr>
<td>15</td>
<td>85</td>
<td>321</td>
<td>0.18</td>
<td>3.125</td>
</tr>
</tbody>
</table>

Figure 4.26. M-integral and regression based stress intensity results for constructive FGMs.
Conclusions

Using a hybrid numerical experimental approach, the foregoing research results make several important contributions to our understanding of the accuracy and applicability of a multivariable regression method used to recover stress intensity and T-stress from displacement measurements made in a two dimensional field centered on the crack tip. Numerical experiments in monolithic materials demonstrate, in contrast to published results, that a minimum number of terms of the Williams equation must be retained if accurate stress intensity and T-stress solutions are to be achieved. Typical results indicate that three and preferably five terms should be retained to achieve accurate stress intensity results, while six is normally required for T-stress. These numerical
experiments also demonstrate the sensitivity of mode I stress intensity to error (Δx) locating the effective crack tip location along the crack plane, while also demonstrating its relative insensitivity to errors in the opening direction. Fortunately, the trends for stress intensity as the number of terms in the regression increase can be used to evaluate Δx. Using the insight provided by these trends and an iterative procedure, Δx can be minimized, the effective crack tip can be located, and stress intensity and T-stress can recovered.

Numerical experiments for two FGM forms, exponentially graded and layered, build upon results for monolithic material specimens. For this limited set of forms, numerical experiment results demonstrate that using the methods developed for monolithic materials, stress intensity can be very accurately recovered. The results show that the stress intensity convergence rate, and trends for stress intensity versus number of terms in the regression algorithm, remain unchanged. However, T-stress results for exponentially graded materials exhibit significant differences when compared to interaction integral results. As a result, it is recommended that T-stress in exponentially graded specimens not be reported if the standard Williams equation is used. Instead, if this quantity is desired, it is recommended that a more appropriate displacement equation, perhaps the equation being developed by Shukla, be investigated.

Finally, experimental results in monolithic titanium and layered FGM specimens validate the proposed procedures. Evaluation of stress intensity from DIC data for a crack growing through a monolithic titanium four-point bend specimen produces results that are typically within ±1 percent of analytical solutions. Experimental results in the layered FGM are normally within ±2 if residual stresses are accounted for. However, the
results also demonstrate that without significantly better displacement resolution, T-stress, even in a monolithic metal specimen, cannot be accurately recovered. Similarities in the magnitude and distribution of displacement measurement variability to displacements due to T-stress prevent an accurate evaluation.
Chapter 5
Fatigue Crack Propagation in the Ti-TiB FGM

Two avenues of research have been conducted to enable testing of a hypothesis that states: “linear elastic fracture mechanics methods, appropriately modified to account for material non-homogeneity, can be used to predict fatigue crack propagation in an arbitrarily graded metal/ceramic FGM”. Along the first avenue of research, several related topics have been evaluated. Microstructural aspects of the FGM, that may have an impact on fatigue crack propagation behavior, have been evaluated, quantified and documented. Fatigue crack growth properties have been characterized for six monolithic Ti-TiB materials ranging from 0 to 85 volume percent TiB. In addition, a predictive model, based upon the Paris equation, has been developed. This single-phase Paris equation given by:

\[
\frac{da}{dN}(\Delta K, V_f) = C(V_f) \Delta K^{n(V_f)}
\]  \hspace{1cm} (5.1)

assumes the dominant independent variables controlling fatigue crack propagation are stress intensity and TiB volume fraction.

Along the second avenue of research, multivariable regression methods using digital image correlation measurements of a two dimensional array of displacements, centered on the crack tip, have been developed and partially validated. These methods
provide tools with which the effective crack tip can be located, after which accurate values of stress intensity can be calculated. Through numerical experimentation, the applicability of these methods for monolithic, exponentially graded and layered FGMs has been verified.

With the single-phase Paris equation defined and experimental tools in place with which to evaluate stress intensity, experiments on FGM specimens have been conducted to test the hypothesis. Three successful experiments have been conducted in layered FGM specimens for cracks propagating from the TiB rich side of the specimen. Three additional experiments have also been conducted with the crack propagating from the pure titanium side of the specimen. However, none of these cracks were successfully propagated into the Ti-15 vol% TiB layer. In each case, though minimum loads sufficient to propagate the crack to the interface were maintained, the test articles immediately failed when the crack front reached the 15 percent layer. As a result, only test results with the crack propagating from the TiB-rich edge of the specimens have been used to evaluate the single-phase Paris equation and pretest predictions of stress intensity in the as-manufactured FGM.

Stress intensity and fatigue crack propagation results from the experiments span TiB volume fractions from 30 to 75 percent, with the bulk of the results in the 30 and 45 vol% TiB layers. No data was collected in the 15 vol% TiB layer due to overload and fast fracture which occurred as the crack approached this layer. Crack propagation results in the 60 and 75 vol% TiB layers have been evaluated, but the sensitivity to slight differences in stress intensity of this FGM at high concentrations of TiB make comparing results between the monolithic and FGM experiments difficult. Therefore, results in the
60 and 75 vol% TiB layers are limited to comparison of finite element analysis and DIC based stress intensity, and evaluation of the single-phase Paris equation is limited to the 30 and 45 vol% TiB layers and the transition zone between them.

5.1 Functionally Graded Material Specimen Preparation

Seven FGM four-point bend specimens were cut from a 25 mm x 25 mm x 152 mm FGM bar and notched with a simple through notch using wire EDM. In two instances, the EDM processes for producing the notch resulted in uncontrolled pop-in of an initial crack on the order of 10 mm long. In both cases, the specimens that cracked during manufacturing were the outer layers (i.e., specimens 1 and 7) of the FGM bar. Each of the five inner layers were separated from the FGM bar without initiating cracks. Three of the seven specimens, including one of the cracked specimens (FGM-1-1), were prepared for experiments using the same grinding and polishing methods used for the monolithic specimens. During final polishing, cracks initiated in FGM-1-2 and FGM-1-3 eliminating the need to precrack these specimens. Final pretest specimen dimensions, precrack lengths (given for both sides of the specimen), layer coordinates and material properties are given in Table 5.1 and Table 5.2.
5.2 Stress Intensity Calculations

Two methods for estimating stress intensity for cracks growing in the FGM specimens have been used: a finite element method based upon the interaction integral in Franc2DL; and a multivariable regression method based upon DIC measurements of the crack tip displacement fields. Digital image correlation measurements were made using the Trilion QualitySystems ARAMIS software. Both the layered FGM finite element and
multivariable regressions analyses use material properties that are consistent with those used to evaluate stress intensity and characterize fatigue crack propagation rate in the monolithic materials. However, multivariable regression results for stress intensity also include the effects of residual stress due to the use of digital image correlation to measure crack tip displacement fields.

5.2.1 Finite Element Calculation of Stress Intensity

A plane stress finite element model (Figure 5.1) based upon layer thicknesses measured in the FGMs has been constructed in Franc2DL and used to solve for stress intensity as a function of crack length and local TiB volume fraction. The model assumes the specimen is free of residual stresses, an unrealistic but necessary assumption without reliable experimental or numerical results for residual stress. Model results for $\Delta K$, normalized by the alternating load $\Delta P$, are shown in Figure 5.2. Observed stress intensity discontinuities are due to step changes in modulus between layers.
Figure 5.1. Finite element model of as produced FGM Specimens.

Figure 5.2. Finite element results for $\Delta K/\Delta P$ in FGM-1, 2, and 3.
5.2.2 Digital Image Correlation Based Stress Intensity

Improved techniques for lighting, polishing and speckling (see Appendix D) have been developed to achieve accuracy and precision from digital image correlation measurements needed to resolve the very small displacements and low stress intensity values in the stiffer layers of the FGM material (see Appendix C). As a result, multivariable regression solutions for the FGM specimens are relatively free of noise, and results for stress intensity are clear. Figure 5.3 shows typical examples of trends in stress intensity versus the number of terms used in the multivariable regression for the layered FGMs. Convergence is comparable to the numerical experiment results, reducing uncertainty in identification of the effective crack tip location, and improving confidence in the stress intensity results.

Figure 5.3. Typical convergence behavior for $\Delta K_I$ versus number of terms in the layered FGM.
For each increment of crack growth in the three FGM specimens tested, experimental stress intensities are calculated based upon DIC-based measurements of the crack tip displacement field and compared to interaction integral solutions. Assuming the DIC based results are an accurate reflection of crack tip stress intensity, including the influences of residual stress, it appears that the interaction integral results tend to overestimate stress intensity (Figure 5.4). Furthermore, the magnitude of overestimation increases as the crack propagates into each successive layer. Results for $\Delta K_{DIC}/\Delta K_{FEA}$ (Figure 5.5) show the magnitude of disagreement between interaction integral and DIC-based results for $\Delta K$ as the crack extends through the FGM. Differences range from as little as -1.0 percent in the 75 vol% TiB layer to 18 percent in the 30 vol% TiB layer, due to the influence of residual stress.

Figure 5.4. FEA and DIC based $\Delta K$ results.
The exact origins of residual stresses influencing stress intensity in the FGM specimens are not clear. They may arise from combinations of coefficient of thermal expansion, or modulus differences between layers as processing temperatures (1305 °C) and pressures (13.8 MPa) are relieved. They may also result from volumetric changes due to boron diffusion and transformation of Ti and TiB₂ into TiB. Unfortunately, accurately accounting for these mechanisms is not trivial, as conflicting results between Ti-TiB FGM experiments and numerical modeling show. Experimental results from the
work of Hill [35] imply that an approximate debit of 2 MPa \( \sqrt{m} \) in stress intensity should be realized regardless of crack length. In contrast, published finite element results suggest that residual stress based upon coefficient of thermal expansion and modulus mismatches would add to bending loads and accelerate crack growth throughout the specimen, rather than retarding the growth rate as observed in these experiments. Further complicating the problem is that the residual stress state influencing stress intensity in an FGM would be a strong function of material gradient, geometry and material processing parameters. As a result, it is clear that significantly more work is required to understand the residual stress state in the material and account for its influence on stress intensity in finite element analysis solutions. However, allowing for residual stresses, the results validate the use of multivariable regression and the standard Williams equation for recovery of stress intensity in the layered FGM.

5.3 FGM Layer Results

To evaluate the single-phase Paris equation, measured and predicted crack propagation rates in the FGM are compared. Two predictions are made with the single-phase equation, the first using FEA-based stress intensities for the FGM, and the second using DIC displacement measurement-derived stress intensities. As shown in Figure 5.6, the single-phase Paris equation, using FEA-based stress intensities, is in agreement with experimental results in the 45 vol% TiB layer but fails to produce accurate results in the transition region or in the 30 vol% TiB layer. In contrast, using DIC based stress intensities, the single-phase Paris equation results are in good agreement with measured results in both layers, supporting the previous conclusion that significant
residual stresses are influencing stress intensity, and therefore fatigue crack propagation rate, in the material. However, DIC based fatigue crack propagation rate results in the transition region remain poor due to microstructural aspects not accounted for in the single-phase Paris equation.

As described in chapter 2, the morphology of this wide transition region includes a continuous gradient of two TiB phases, the 50 $\mu$m whiskers associated with 30 vol% TiB layer and the 10 $\mu$m whiskers found in the 45 vol% TiB layers (Figure 5.7 and Figure 5.8). The single-phase Paris equation does not account for the presence of the graded TiB microstructure, and is therefore incapable of accounting for

![Graph showing comparison of measured and predicted fatigue crack propagation rate based upon the initial modification of the Paris equation.](image.png)

Figure 5.6. Comparison of measured and predicted fatigue crack propagation rate based upon the initial modification of the Paris equation.

As described in chapter 2, the morphology of this wide transition region includes a continuous gradient of two TiB phases, the 50 $\mu$m whiskers associated with 30 vol% TiB layer and the 10 $\mu$m whiskers found in the 45 vol% TiB layers (Figure 5.7 and Figure 5.8). The single-phase Paris equation does not account for the presence of the graded TiB microstructure, and is therefore incapable of accounting for
increased toughening provided by the 50 µm TiB whiskers. As a result, the model significantly overpredicts crack propagation rate in the transition region.

5.4 Dual-Phase Paris Equation

To account for multiple TiB microstructures within the 15 to 60 vol% TiB range, and the continuous gradient of these phases in the transition region between the 30 and 45 vol% TiB layers, a dual-phase Paris equation, incorporating an additional parameter, \( v_{10\mu m} \), to account for the volume fractions of the 10µm and 50µm TiB microstructures as a function of overall TiB content, is developed. The equation applies the modeling approach used for the single-phase Paris equation to develop two submodels, separately predicting the fatigue crack propagation rate over the ranges of 15 to 30 vol% TiB and 45 to 60 vol% TiB. Over both TiB ranges, the models have the form:

\[
\frac{da}{dN} = e^{\alpha_0 V_f + \alpha_1} \Delta K^{\alpha_2 V_f + \alpha_3}
\]  

(5.2)
where

\[ C = e^{\alpha_0 V_f + \alpha_1} \]  

and

\[ n = \alpha_2 V_f + \alpha_3 \]  

To evaluate the coefficients \( \alpha_i \) (i = 0, 1, 2, 3) in each submodel, the equations for \( \frac{da}{dN} \) are written in the standard form for multivariable regression by taking the natural log of each side of the equation:

\[
\ln \left( \frac{da}{dN} \right) = \alpha_0 V_f + \alpha_1 + \alpha_2 V_f \ln(\Delta K) + \alpha_3 \ln(\Delta K)
\]

Using multivariable regression, the data sets of crack propagation rates and stress intensities for the ranges from 15 and 30, and 45 and 60 percent TiB volume fraction are used separately to define the coefficients for each submodel. The resulting models for the 15 to 30 vol% TiB layers is

\[
\frac{da}{dN_{\text{coarse}}} = e^{3.576 V_f - 36.879 \cdot \Delta K^{6.355 V_f + 8.538}}
\]

and for the 45 to 60 vol% TiB layers is

\[
\frac{da}{dN_{\text{fine}}} = e^{6.884 V_f - 38.202 \cdot \Delta K^{31.935 V_f - 234}}
\]

To account for the local TiB microstructure gradient in the transition region, the submodels are combined using a simple rule of mixtures to evaluate intermediate values of \( C \) and \( n \)

\[
C = C_{50\mu m}(1 - \nu_{10\mu m}) + C_{10\mu m}\nu_{10\mu m}
\]

120
where \( v_{10\mu m} \) is the volume fraction of the 10\( \mu \)m TiB microstructure relative to overall TiB content (\( v_{10\mu m} = 100 \) percent in the bulk of the material with TiB volume fraction equal to or greater than 45 percent. Within the transition zone \( v_{10\mu m} \) decreases linearly from 100 to 0 percent as the point of interest extends into the 30 vol\% TiB layer.)

The combined model for crack propagation in the transition zone is given by:

\[
\frac{da}{dN} = \left[ e^{C_{\text{coarse}}(1-v_{10\mu m})} + e^{C_{\text{fine}} v_{10\mu m}} \right] \cdot \Delta K \cdot \left[ n_{\text{coarse}}(1-v_{10\mu m}) + n_{\text{fine}} v_{10\mu m} \right] \tag{5.9}
\]

Using the dual-phase Paris equation to predict crack propagation rate in the FGM, including the transition region, assumes the following:

1. The transition region is on average 1.0 mm wide.

2. The proportions of the 10 \( \mu \)m and 50 \( \mu \)m TiB phases relative to total TiB volume fraction change linearly across the transition region, each ranging from 0 to 100\% (Figure 5.9).

3. The 50 \( \mu \)m TiB phase in the transition region ranges from 0 to 30 vol\% as a proportion of the overall material volume.

4. The 10 \( \mu \)m TiB phase in the transition region ranges from 0 to 45 volume percent as a proportion of the overall material volume.
Results for the single- and dual-phase Paris equations are shown in Figure 5.10 and Figure 5.11, along with actual crack propagation rate results from the FGM. Several observations can be made from these results. Within each layer, dual-phase Paris equation results typically match measured results. Absent is any indication that fatigue crack propagation rate or stress intensity is dependent on material gradient, supporting the pretest hypothesis of independence. The dual-phase Paris equation is clearly better in the transition region between layers, typically producing results that are within an order of magnitude of the observed crack propagation rates, though further improvement appears possible. It may be that additional toughening mechanisms in addition to the 50 μm TiB phase exist, or that the influence of the coarse microstructure is not linear as assumed by the rule of mixtures model. However, to test these possibilities, additional data, under very tightly controlled test conditions is needed.
Figure 5.10. Predicted versus measured FGM crack propagation rate results (specimen FGM-1-2).

Figure 5.11. Predicted versus measured FGM crack propagation rate results (specimen FGM-1-3).
5.5 Conclusions

The performance of linear elastic fracture mechanics tools for prediction of stress intensity and fatigue crack propagation rates in a layered FGM has been evaluated. Stress intensities based upon digital image correlation displacement measurements and multivariable regression of the Williams equation have been compared with interaction integral solutions. Results show the accuracy of finite element based interaction integral solution accuracies to be limited. The comparison shows a clear trend toward increasing under-prediction of stress intensity by the interaction integral method, due to the inability to account for residual stresses in the FGM. This conclusion is supported by fatigue crack propagation rate results. When fatigue crack propagation rates are predicted based upon finite element analysis stress intensity calculations, results consistently exceed measured propagation rates. In contrast, propagation rates based upon multivariable regression results for stress intensity, which include the effect of residual stress, more consistently match measured rates. Fundamentally, the finite element analysis method is sound. However, a means for accounting for residual stresses in the numerical analysis is required if this tool is to be used to predict stress intensity in FGMs when material processing results in a significant residual stress state.

In parallel, two equations modeling fatigue crack propagation rate have been evaluated. A single-phase Paris equation, assuming the dominant independent variables controlling fatigue crack propagation are stress intensity and TiB volume fraction, has been evaluated. Using stress intensity solutions based upon MVR of measured displacements, results in monolithic TiB layers match measured fatigue crack
propagation results; however, performance in the graded transition region between the 30 and 45 vol% TiB layers is poor. To address the deficiency, a dual-phase Paris equation, accounting for the superposition of the locally graded TiB microstructure of 10 and 50 µm TiB whiskers onto the global layered gradient, has been developed. Comparisons with measured propagation results show significant improvement. Overall, these results support the hypothesis upon which this research is based. Neither stress intensity nor fatigue crack propagation rate appears to be a function of global or local material gradients. As a result, both can be evaluated considering only material properties, phase volume fractions and phase microstructures at the crack tip.

However, effective use of linear elastic fracture mechanics to predict fatigue crack propagation in functionally graded materials is limited by, and dependent upon, at least two conditions. First, an accurate method to account for residual stresses in arbitrary FGM gradients and structural forms is required if accurate finite element stress intensity predictions are to be made. The alternative is to use an experimental method for evaluating stress intensity in every case, a costly proposition. Second, development of a relatively simple predictive model is possible if microstructural changes within the FGM are limited in number and impact on fatigue crack propagation rate. However, results here show that multiple material microstructure transitions, and superimposed local and global material gradients, can develop, and can have a significant effect on the complexity of fatigue crack propagation properties throughout the material. In cases, where these complexities become extreme, modeling fatigue crack propagation with a single relatively simple model may not be possible.
Chapter 6

Conclusions and Extensions

The focus of this investigation has been to evaluate the hypothesis that linear elastic fracture mechanics methods, appropriately modified to account for material non-homogeneity, can be used to predict fatigue crack propagation in an arbitrarily graded metal/ceramic FGM. To accomplish this task, contributions have been made in multiple technical areas.

1. The Ti-TiB FGM microstructure has been investigated and documented including superimposed material gradients.

2. MVR techniques for accurate recovery of stress intensity in monolithic and FGM specimens have been developed.

3. A notch geometry enabling compression precracking of, and stable fatigue crack propagation in, brittle materials has been devised.

4. Material characterization methods have been developed and a predictive equation for fatigue crack propagation in a FGM formulated.

5. Experiments have been conducted to evaluate stress intensity and fatigue crack propagation in a layered Ti-TiB FGM, and test the predictive model and hypothesis upon which this work is based.
Evaluation of the microstructure of the Ti-TiB FGM has identified and documented the existence of a more complex material gradient than previously reported. The Ti-TiB FGM includes two significant material gradients, a global layered gradient, and a local gradient. Within the global gradient two material transitions exist. Stepwise changes in material phase volume fractions and material microstructure are connected by rapid and continuous transitions. Superimposed upon the global gradient, is a local gradient, unique to the transition between the 30 and 45 vol% TiB layer in this FGM. This gradient appears over an approximately 1 mm transition width and involves the linear variation of two TiB microstructures.

A hybrid analysis combining numerical and experimental tools has been used to develop and verify multivariable regression methods, expectations and criteria for recovery of stress intensity in monolithic materials. Trends in stress intensity versus the number of terms used in the MVR have been shown to be indicative of accuracy estimates of the effective crack tip location and recovered stress intensity. Criteria have been established with which these trends may be evaluated, effective crack tip location corrected, and stress intensity and T-stress recovered. Additional numerical experiments have been used to extend the method to include a limited set of exponential and layered FGM gradients. Results demonstrate that accurate stress intensities can be recovered, and the trends and criteria established for monolithic materials also apply to this limited set of functional gradients. In contrast, T-stress cannot be accurately recovered using the Williams equation. Recovery of this term for FGMs requires significant improvements in displacement precision along with a more appropriate displacement equation, perhaps one being developed by Shukla.
A novel compression precracking notch configuration has been developed for testing of the brittle monolithic and FGM Ti-TiB specimens. The notch offers several advantages over conventional straight through notches in brittle materials. In reverse four-point bending, short sharp stable precracks initiate in compression at relatively low loads. The lower loading requirement limits the tensile stresses that develop along the opposite edge of the specimen, avoiding potential tensile failure in materials that may be very strong in compression but weak in tension. During characterization tests the notch provides a slightly decreasing stress intensity profile along its length, enabling stable fatigue crack propagation and affording the opportunity to characterize a material/specimen that does not otherwise exhibit stable propagation.

A method for characterizing fatigue crack propagation properties using a limited set of monolithic specimens representing the range of material constituent volume fractions present in the FGM has been developed and used to characterize the Ti-TiB FGM. To utilize the data effectively, a dual-phase Paris equation, including additional independent variables, has been formulated. Accounting for TiB volume fraction and local changes in TiB microstructure in addition to stress intensity, the equation predicts fatigue crack propagation rate for the Ti-TiB FGM.

Experiments in layered Ti-TiB FGM four-point bend specimens have been used to evaluate the MVR methods and the dual-phase Paris equation. Combined MVR stress intensity and dual-phase Paris equation results validate the experimental methods used to recover stress intensity for FGM specimens. These results also document the need to account for residual stresses when using finite element based methods to evaluate stress intensity in a FGM. Comparison of dual-phase Paris equation and measured fatigue
crack propagation in the FGM specimens also support the hypothesis upon which this research has been based. For the limited range of TiB volume fraction over which reliable data could be measured, predicted and measured fatigue crack propagation correlation compare within an acceptable margin of error. Absent from the data is any obvious indication that stress intensity or fatigue crack propagation rate is dependent upon the material gradient. These results lead to the conclusion that these stress intensity and fatigue crack propagation rates are only a function of the material properties at the crack tip in this layered FGM. However, residual stresses and material gradients do have an impact that must be understood.

In practical engineering terms the results of this research have the following impact. Linear elastic fracture mechanics methods appear to apply to this class of materials for the limited set of material gradients studied. It is possible to characterize the fatigue crack propagation properties of an arbitrarily graded FGM using a limited number of monolithic volume fraction specimens and to formulate a predictive model for propagation within the FGM. However, the microstructural investigation of Ti-TiB clearly shows that changes beyond simple volume fraction gradients can develop within a FGM. These changes can include variation in microstructure, and distributions of multiple microstructures, as they do here, that must be accounted for in the predictive equation. Further complicating the problem is that these changes may be a function of the material gradient and the size of the structure produced. At this point the predictive equation is no longer a material model but must now incorporate aspects of variations in gradients that develop with geometry. As a result, modeling these gradients and accounting for their effect on fatigue crack propagation can become prohibitively
complex. In these cases, an alternate method of characterizing and modeling fatigue crack growth will become necessary.

An additional complication is the residual stress state that may often exist in these materials. Experimental studies clearly show that stress intensity can be accurately solved for in this material if residual stresses are accounted for, as demonstrated by MVR results. Unfortunately, accounting for them will not be a simple matter. Residual stresses will certainly be a function of material gradient, processing parameters, and may in many cases be a function of structural size and shape. To model residual stresses reliably, the complex interactions between the properties of the material constituents, in-situ transformations, and material processing methods will have to be understood and modeled. Our understanding will have to be such that these calculations can be reliably made for complex structural geometries. Currently the only reliable method to account for residual stress during material characterization or component testing is to use a data intensive DIC approach. To address these technical challenges, research in a number of areas must be pursued.

Numerical analyses and experiments should be conducted to demonstrate the extent of applicability of the criteria and MVR methods developed here for recovery of stress intensity in a broad range of functionally graded materials. Considerations in such research should include a wide range of material properties and complex material gradients, specimen geometries, and loading conditions. If necessary, other more appropriate equations for FGMs could be explored. Part of such work could be to automate the iterative method for locating the effective crack tip and accurately recovering stress intensity. Successful methods must be robust enough to accommodate
displacement measurement variability and its impact on convergence behavior of stress intensity versus the number of terms used in the MVR.

Future material characterization of FGMs could take multiple paths. As demonstrated here, the use of multiple monolithic specimens to provide the data base with which to develop a predictive equation appears to be an efficient method for characterizing a layered FGM. However, this may not be true if monolithic material specifically produced for characterization is used to characterize continuously graded FGMs. Observed differences in microstructure (reported here and in the literature) due to changes in thickness, and layer-to-layer TiB volume fraction variations, suggest that microstructures could develop in the monolithic materials that are different than the microstructure in a continuously graded materials at a given volume fraction. Also, there remains the possibility that fatigue crack propagation rate could be a function of material gradient, and dependent upon crack path in continuously graded materials. If this is the case, an alternate approach would need to be explored. A possible candidate direction would evaluate non-traditional methods, such as cohesive zone models, for predicting fatigue crack propagation.

Finally, a critical area of research that must be addressed before reliable fatigue crack propagation predictions can be made is the study of residual stress. Methods that account for the complex interactions between material properties, in-situ chemical reactions, microstructural transformations and processing methods (sintering, plasma spray, diffusion, etc.) should be investigated. The objective should be to develop reliable methods for modeling the residual stress state of arbitrary FGMs and structural configurations.
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Appendix A

Fatigue Precracking of Monolithic Ti-TiB Specimens

Consistent with results in the literature, precracking of monolithic specimens with TiB volume fractions equal to or greater than 45 percent proved to be impossible using tension-tension precracking methods. Owing to the brittle nature of the materials, using the conventional tension-tension precracking method resulted in uncontrolled pop-in of a crack that generally failed the specimen. In response, a limited investigation of compression precracking methods has been explored, and a reverse four-point bend method developed by Carpenter [44] specifically for Ti-TiB FGMs has been modified to support fatigue crack propagation experiments in the current monolithic material. The primary objectives of the research have been the development of a notch configuration: 1) that can easily be precracked in compression after machining; 2) that can be precracked at a sufficiently low load in reverse four-point bending such that no damage will be done in tension on the opposite edge of the specimen; and 3) from which a stable fatigue crack can be grown in a controlled fashion. A secondary objective, identified during development, was the need for a geometry that can easily be inspected to verify the existence of a precrack, in-situ on the test frame.
A.1 Progression of Notch Geometry

Several notch geometries have been evaluated numerically and experimentally, progressing from a simple through notch (Figure A.1a), to a 90° chevron notch (Figure A.1b) based on ASTM standard C1421-01b [41], concluding with the more complex "Y" notch shown in Figure A.1c. Both the through notch and the chevron notch are produced by straight cuts using wire electric discharge machining. The “Y” notch is cut from both sides of the specimens using a diamond wheel, producing intersecting curved profiles in a compact notch geometry. The intent of the “Y” notch design is to produce a notch with an interconnecting web tapering to a sharp edge, in which a precrack can easily be initiated. However, in practice, spalling of material at the knife edge of the web results in a finite width edge, typically 0.03 mm thick if properly refined.
Figure A.1. Typical monolithic specimen geometry, Chevron and "Y" notch configurations.
A.2 Numerical Analysis Results for Compression Pre-cracking

Based upon the work of Suresh and others, the key characteristic upon which to evaluate the potential effectiveness of a notch design is the level of compressive stress that can be developed within it. To establish the compressive pre-cracking threshold stress requirement for the Ti-TiB materials, a finite element analysis of Carpenter’s [44] specimens and notch geometries has been conducted. Additional finite element analyses of proposed notch configurations for the materials and specimen geometries of this research program are conducted to: assess the stresses that develop at the notch, and compare them to the requirement; estimate the loads necessary to successfully achieve the compression threshold stress levels required to precrack each of the 45 vol% TiB and higher specimens; and to document the overall stress distribution in the specimens, and in particular, the peak tension stresses that develop along the edge of the specimen opposite the notch.

A.2.1 Analysis of Carpenter's Experiments in FGMs

Carpenter conducted precracking experiments in seven-layer Ti-TiB FGM specimens in which the notch was introduced through the 85 and 75 vol% TiB layers into the 60 vol% TiB layer. Experiments investigating both axial and reverse four-point bend loading conditions produced precracks in the 60 vol% TiB layer. To apply the reverse four-point bend method to the current monolithic materials and specimen geometry, an elastic finite element analysis of Carpenter's reverse four-point bend and axial compression experiments has been conducted using the Franc2DL [55] finite element analysis code (Figure A.2).
Finite element results for the four-point bend experiment reveal that a notch tip stress of approximately 4500 MPa is generated at the reported loading condition of $P = 6900 \text{ N}$ (Figure A.3). A similar result is found for Carpenter’s axial compression experiment where a 212 MPa applied axial compression stress generates a notch tip stress of 4300 MPa. Based upon analysis results and successful precracking of both specimens, it appears that a reasonable value for the compressive precracking threshold stress, required to precrack the Ti-60 vol% TiB materials, is approximately 4500 MPa. Evaluation of finite element analysis results also reveals a tensile stress generated on the opposite edge of the specimen of 395 MPa in the four-point bend experiments. This exceeds the reported ultimate strength of pure titanium (179 MPa) and indicates that the straight through notch did not provide a sufficiently high stress concentration to preclude damage to the Ti layer of the monolithic FGM. As a result, some plastic flow and redistribution of stresses would be expected to have occurred. The result also suggests that tensile stresses that develop in the monolithic Ti-TiB specimens will exceed the tensile strength of the materials before compressive stresses sufficient to precrack the specimens are achieved.
Figure A.2. Franc2DL model of Carpenter's reverse four-point bend experiments.

Figure A.3. Longitudinal stress profile for Carpenter's FGM specimen at 6900 N.
A.2.2 Analysis and Experiments in Monolithic Specimen Configurations

Three notch geometries in the monolithic specimens have been evaluated numerically and experimentally. Numerical analysis has been used to evaluate notch tip stress concentration, and determine which geometries generate the required compressive stress, while limiting tensile stress levels on the edge opposite the notch to levels below the tensile strength of the material. Geometries that demonstrated sufficient stress concentrations have been evaluated experimentally to demonstrate precracking and detectability requirements.

A.2.2.1 Simple Notch Geometry

Finite element analysis results for the typical specimen geometry and loading conditions (Figure A.4), estimate a notch tip stress concentration of $K_t \approx 8$ for the simple through notch geometry. Using this $K_t$ value and limiting the applied load, such that tensile stresses in the specimen do not exceed 80 percent of the ultimate strength of the material, yields compressive stresses below 4500 MPa in all cases (Table A.1). Comparing the peak compressive stress value of 1485 MPa for the Ti-60 vol% TiB specimen, with the 4500 MPa target established from analysis of Carpenter’s experiments, clearly shows that the simple through notch is insufficient to precrack the monolithic specimens. In fact, the finite element results show that when sufficient load has been applied to generate the required notch tip stress, the opposite edge experiences a tensile stress of 533 MPa (Figure A.5), well above the strength (359 MPa) of even the strongest of the high TiB volume fraction materials.
Table A.1. Maximum notch tip stresses for a through notch based upon allowable tensile stress limits.

<table>
<thead>
<tr>
<th>TiB $V_f$ (%)</th>
<th>Tensile Strength (MPa)</th>
<th>Notch Stress $\sigma_{\text{notch}}$ (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>$K_t = 8$</td>
</tr>
<tr>
<td>45</td>
<td>258</td>
<td>1651</td>
</tr>
<tr>
<td>60</td>
<td>232</td>
<td>1485</td>
</tr>
<tr>
<td>75</td>
<td>306</td>
<td>1958</td>
</tr>
<tr>
<td>85</td>
<td>359</td>
<td>2298</td>
</tr>
</tbody>
</table>

$\sigma_{\text{notch}} = U_{\text{ts}} \times 80\% \times K_t$
A.2.2.2 Chevron Notch

Based upon guidance in ASTM standard C1421-01b, a 90° chevron notch (Figure A.6) has been evaluated. The configuration has been analyzed using a 3-D model and quadratic tetragonal elements in ABAQUS, and results show the notch develops a stress concentration of 20 to 22 in this specimen geometry. For a $K_t = 20$ the compressive stress that develops at the tip of the notch is marginal, falling short of the target value if tensile stress is limited to 80 percent of the tensile strength in the 60% layer (Table A.2). Assuming that the compressive precracking threshold stress increases with increasing tensile strength, it can be assumed that, even though the peak notch stresses in the 75 and 85 volume percent TiB layers exceed 4500 MPa, they may not exceed the threshold stresses for those layers. Further limiting the potential of the chevron notch is the rate at which these stresses decay. Within 0.10 mm of the tip, $\sigma_y$ has decayed to less than half...
the peak value, well below what might be reasonably expected to produce or extend a precrack. As a result, any microcracking that might develop would be expected to be very short and hard to detect inside the notch.

Figure A.6. Chevron $\sigma_y$ stresses.

Table A.2. Maximum chevron notch tip stress based upon tensile stress limit of 80 percent of material tensile strength.

<table>
<thead>
<tr>
<th>TiB $V_f$ (%)</th>
<th>Tensile Strength (MPa)</th>
<th>Notch Stress $\sigma_{notch}$ (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>45</td>
<td>258</td>
<td>4128</td>
</tr>
<tr>
<td>60</td>
<td>232</td>
<td>3712</td>
</tr>
<tr>
<td>75</td>
<td>306</td>
<td>4896</td>
</tr>
<tr>
<td>85</td>
<td>359</td>
<td>5744</td>
</tr>
</tbody>
</table>
In fact, experimental results for the chevron notch have produced less than satisfactory results. While several of the experiments produced precracks, they were only detectable using a scanning electron microscope. Further limiting the utility of the chevron notch were unsuccessful crack propagation results. In each case, attempts to propagate from precracks in the chevron notch resulted in abrupt failure.

A.2.2.3 Y Notch

To increase the likelihood for generating a precrack in these materials, a "Y" notch geometry has been devised. The primary intent in choosing this geometry is to address the deficiencies of the chevron notch, marginal stress concentration, short precrack length and poor in-situ precrack detectability. A 3-D ABAQUS finite element model of the “Y” notch, composed of quadratic tetrahedral elements, has been built and analyzed. Results demonstrate that a notch tip stress concentration of 30 or higher is generated, depending upon the exact dimensions of the notch and thickness of the web produced. The results also indicate that stresses exceeding the 4500 MPa target extended as far as 0.5 mm from edge of the notch (Figure A.7), potentially producing a longer initial precrack than can be achieved with the chevron notch, improving detectability.
Figure A.7. Cross section stress profiles for a monolithic specimen loaded to 80 percent of tensile strength.

A.2.2.4 Experimental Precracking Results for the Y Notch

The biggest challenge to precracking the specimens using the "Y" notch configuration has been the ability to produce a refined edge from which to initiate a precrack. Spalling of material from the edge in the 60 and particularly the 85 vol% TiB materials limited the thickness of the edge that could be achieved with the available machine tools. The impact to the Ti-85 vol% TiB specimen was a thicker than desired edge and difficulty detecting microcracking in the notch. In spite of the thick edge produced on the single Ti-85 vol% TiB specimen successfully tested, 20,000 cycles at a compression load of 5000 N were applied to precrack the specimen. Post compression
inspection in a scanning electron microscope produced inconclusive results with no firm evidence of a precrack. However, subsequent tension tests did result in a crack, propagated to a length of 5.0 mm, from which fatigue crack propagation data was successfully generated.

The second specimen successfully precracked from a "Y" notch configuration was the Ti-60 vol% TiB specimen. Initial precracking attempts with a 50 μm notch edge thickness proved ineffective after 30,000 cycles. Post cycling finite element results estimate that a peak notch compression stress of only 2200 MPa was generated due to the thick web. Subsequently the notch edge was refined to a 30 μm width, producing a compression stress comparable to the requirement established by analysis of Carpenters [44] experimental results. After 20,288 cycles at a peak compression stress of 4900 MPa, a 60 μm precrack was evident in-situ on the test frame (Figure A.8). The specimen was immediately reversed in the four-point bend fixture and successfully tested for 93,000 cycles with the crack growing an additional 4.94 mm.
Based upon results in the Ti-60 vol% TiB specimen, particular attention was given to producing a refined edge in the Ti-45 vol% TiB specimen. Post machining inspection using an optical microscope revealed a refined notch edge of 23 µm and scanning electron microscope inspection revealed the presence of an 80 µm crack on side "a" shown in Figure A.9. The inspection also revealed the crack had not penetrated through the thickness of the web to the opposite ("b" side) of the notch. Attempts to propagate from the initial 80 µm crack failed despite 50,000 cycles, and the specimen was reoriented to apply a compression load. 415 compression cycles at peak load of 1200 N and an R-ratio of 0.1 were applied, producing a notch tip compressive stress based upon post test analysis of approximately 5000 MPa. The cycles had no detectable impact on the initial 80 µm crack, a result that is consistent with the constitutive model for compression precracking. However, the compression cycles did propagate the crack

Figure A.8. In-situ observation of a compression precrack in the Ti-60 vol% TiB specimen.
through to the “b” side of the notch (Figure A.10). In subsequent tensile cycles the crack was successfully propagated the length of the notch and into the full cross-section of the specimen.

Figure A.9. Pre-compression "a" side precrack in Ti-45 vol% TiB specimen.
A.3 Conclusions

Precracking thin monolithic Ti-TiB specimens, where the volume fraction of TiB exceeds 30 percent proved to be impossible in tension. To overcome this problem, compression precracking methods, first developed by Suresh [45, 47, 59] and extended to Ti-TiB functionally graded specimens by Carpenter [44], have been modified for use with monolithic Ti-TiB specimens. Several notch geometry variations have been analyzed and evaluated experimentally before settling on a "Y" notch configuration. The “Y” notch configuration substantially increases the notch stress concentration, from eight for a simple straight through notch to greater than 30, minimizing the required bending load and limiting the magnitude of the tensile stresses that develop during compression precracking. The geometry of the notch also maximizes the distance over which elevated
compression stresses exist and a precrack develops, making in-situ observation on the hydraulic test machine possible. Post test analysis of the experiments also support results from analysis of Carpenter’s [44] specimens, establishing a compressive threshold stress of approximately 4000 to 5000 MPa, to achieve precracking in materials with TiB volume fractions equal to or greater than 45 percent.
Appendix B
Mechanical Properties of Ti-TiB

Published material properties for Ti-TiB FGMs vary widely, and may do so for a variety of reasons, not the least of which is variability in material processing. As a result, the use of published data in place of characterization of needed properties on a lot-by-lot basis may produce spurious results when evaluating stress intensity. To avoid the problem, limited characterization of mechanical properties, E, ν and ultimate strength, has been conducted to support development of compression precracking procedures and analysis of stress intensity and T-stress. The following summarizes the results and compares them when possible to published data.

B.1 Modulus and Poisson’s Ratio

Several test methods and measurement procedures have been used to characterize E and ν for each of the seven Ti-TiB material volume fractions that exist in the FGM used here. Test methods include simple tension tests using flat “dog bone” specimens and an ultrasonic method used on thin, typically 3.25 mm thick, 152 mm long and 25 mm wide specimens. Modulus results obtained from tension tests are based directly upon strain gage data and P/A calculations. In some cases, strain gages were installed on both front and rear surfaces of the tension specimens to detect the presence of bending during
tests. In each case, slight differences between strain on the front and rear surface is observed. To account for the differences, front and rear surface strain values are averaged. As an added check on strain, digital image correlation has been used on the rear surface of the specimen. In most cases, good results have been obtained for modulus using the DIC data as a virtual extensometer to calculate strain. In a few cases, poor correlation prevented evaluation of strain using the DIC method and results have been dismissed. However, Poisson’s ratio results are generally not valid, particularly for materials with moderate to high TiB volume fractions. In the lateral direction, displacements due to Poisson’s effect appear to have been too small for the DIC code being used at the time to resolve. As a result, Poisson’s ratio is not reported for the DIC measurements. Modulus and Poisson’s ratio derived from ultrasonic tests\(^1\) are based upon measurement of shear and dilatational wave speeds and are calculated using the equations:

\[ C_s = \sqrt{\frac{\mu}{\rho}} \quad \text{(B.1)} \]

\[ C_d = \frac{2(1-v)\mu}{\sqrt{(1-2v)\rho}} \quad \text{(B.2)} \]

\[ v = \frac{1}{2} \left( \frac{C_d}{C_s} \right)^2 - 1 \quad \text{(B.3)} \]

\(^1\) Thanks to Jay Carrol at the University of Illinois for conducting these experiments.
\[
\mu = C_s^2 \cdot \rho \quad \text{(B.4)}
\]

\[
E = 2\mu(1 + \nu) \quad \text{(B.5)}
\]

Figure B.1 and Table B.1 show results from each of the experiments and methods used to evaluate modulus and Poisson’s ratio.

![Figure B.1. Combined strain gage, DIC and ultrasonic results for modulus.](image-url)
Figure B.3 and Figure B.4 present current results for modulus and Poisson’s ratio compared to typical published results from a range of sources, and characterized using a

Table B.1. Average Properties.

<table>
<thead>
<tr>
<th>TiB Vf</th>
<th>E (GPa)</th>
<th>ν</th>
<th>Tensile Strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>110</td>
<td>0.32</td>
<td>389</td>
</tr>
<tr>
<td>15</td>
<td>145</td>
<td>0.29</td>
<td>596</td>
</tr>
<tr>
<td>30</td>
<td>171</td>
<td>0.26</td>
<td>476</td>
</tr>
<tr>
<td>45</td>
<td>214</td>
<td>0.23</td>
<td>258</td>
</tr>
<tr>
<td>60</td>
<td>268</td>
<td>0.20</td>
<td>232</td>
</tr>
<tr>
<td>75</td>
<td>324</td>
<td>0.19</td>
<td>306</td>
</tr>
<tr>
<td>85</td>
<td>323</td>
<td>0.18</td>
<td>359</td>
</tr>
</tbody>
</table>

Figure B.2. Combined strain gage, DIC and ultrasonic results for Poisson’s ratio.

Figure B.3 and Figure B.4 present current results for modulus and Poisson’s ratio compared to typical published results from a range of sources, and characterized using a
number of methods. Tuegel [6] presents results based upon shaker table tests of 3.15 mm beams using composite theory to evaluate $E$ and $v$. The similarity between Tuegel’s results and those of this program can be attributed to the fact that the material used in both cases was recently purchased from Cercom. The results from Hill [35, 36] are also for Cercom material, but for specimens manufactured before 2002. The results from Atri [57] and Grosse [58] are for materials of unknown pedigree.

![Figure B.3. Comparison of present modulus results and typical published data.](image)

Figure B.3. Comparison of present modulus results and typical published data.
B.1.1 Tensile Properties

Tensile strength properties for Ti-TiB composites are typically not found in the literature. Cercom did publish results, but inconsistencies in strength as well as modulus and Poisson’s ratio in the data raise questions about its accuracy, so none of the results are presented here. Strength data in this program is from the same tensile tests used to derive modulus and Poisson’s ratio. Two tests were conducted for each Ti-TiB volume fraction with a primary goal of establishing a general expectation for tensile strength. Results were only used to limit loads in reverse four-point bend precracking to minimize the possibility of tensile failure. Tensile strength results are shown in Figure B.5, with average values of 389, 596, 476, 258, 232, 306 and 359 MPa. Note that the pure titanium material is the only material that exhibits a yield point, approximately 300 MPa.

Figure B.4. Comparison of present Poisson’s ratio results and typical published data.
B.2 TiB Physical Properties

Most available data for TiB, TiB₂ and Ti-TiB FGM properties focus on mechanical properties. There are however, a few sources for physical properties [8, 10, 36] (Table B.2).

Table B.2. Physical properties.

<table>
<thead>
<tr>
<th>Property</th>
<th>Ti</th>
<th>TiB</th>
<th>85vol%TiB</th>
<th>TiB₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density (g/cc) [8]</td>
<td>4.57</td>
<td>4.56</td>
<td>-</td>
<td>4.52</td>
</tr>
<tr>
<td>CTE. at RT (10⁻⁶/°C) [8]</td>
<td>8.6</td>
<td>7.15</td>
<td>-</td>
<td>6.2</td>
</tr>
<tr>
<td>CTE. at RT (10⁻⁶/°C) [10]</td>
<td>10</td>
<td>6.2</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>CTE. at RT (10⁻⁶/°C) [36]</td>
<td>10.9</td>
<td>-</td>
<td>8.69</td>
<td>-</td>
</tr>
<tr>
<td>Vickers hardness (kg/mm²) [8]</td>
<td>150</td>
<td>1,800</td>
<td>-</td>
<td>2,200</td>
</tr>
</tbody>
</table>

Figure B.5. Tensile test results.
Appendix C
DIC Measurement Uncertainty

While not exhaustive, the following is an attempt to evaluate the likely statistical distributions of recovered stress intensity and T-stress due to inherent variability in measured crack opening displacements, and material modulus, and determine if these might be the source of differences seen between finite element and multivariable regression results for stress intensity in the FGM specimens. The approach taken to accomplish this objective includes characterizing displacement variability from rigid body and crack propagation tests in terms of distribution, mean and standard deviation. The results are used to modify finite element analysis based displacements, which are in turn used in the multivariable regression algorithm to evaluate stress intensity and T-stress. Stress intensity and T-stress, recovered from the modified displacements for a range of crack lengths and material systems, are compared with interaction integral and noise free multivariable regression solutions to evaluate the level of error that may be expected in experimental results for these quantities.

C.1 Displacement Measurement Uncertainty

Two experimental series have been conducted to evaluate the statistical distribution of displacement measurement variability. Rigid body displacement
experiments have been used to isolate and evaluate displacement measurement variability. The results are used to characterize the statistical distribution of noise within displacement measurements, evaluate accuracy and identify any bias that may exist in the measurement. The results also establish a baseline against which results obtained from crack opening experiments, which include strain, can be compared to determine if strains present in the vicinity of a crack tip significantly influence the statistics of the noise present in displacement measurements.

C.1.1 Rigid Body Translation

Rigid body translation experiments have been conducted using an extensometer calibrator with defined increments of displacement of 0.0001 in (2.54 μm) to move a target by rigid body displacements from 0 to 50.8 μm (0.0001 to 0.0020 in). Images taken at each increment are used in digital image correlation to evaluate rigid body displacement. Within each image, approximately 2900 independent measurements of displacement in each coordinate direction are made. The results are analyzed to evaluate two aspects of the digital image based displacements, measurement accuracy in terms of average measured displacement throughout the image, and measurement precision defined by the statistical distribution of displacement results. Note that pretest evaluation of the extensometer calibrator determined that the precision with which it can be set is ±0.46 μm, based upon one standard deviation. As a result, differences between extensometer calibrator and average DIC results of less than ±0.46 μm are essentially indistinguishable.
Typical rigid body displacement results, shown in Figure C.1, do not have a
spatial bias, indicating that measurement results are not a strong function of spatial
distributions of the speckle pattern, light intensity, or lens distortion. Table C.1 shows
accuracy and precision results for intended rigid body displacements (IRBD)
displacements from 2.54 to 50.8 µm. Digital image correlation results, average
displacement, accuracy and precision are shown in the remaining columns. Accuracy
across the range of measurements ranges from -0.16 to 0.44 µm with an average value of
0.09 µm. Results for precision are shown in Table C.1 and Figure C.2 and are observed
to be normally distributed with an average standard deviation of 0.12 µm. Based on this
limited data set, and the precision limit with which the extensometer calibrator can be
adjusted, several conclusions can be made. Digital image correlation results, for the
combination of speckle size and coverage, lighting, and measurement window size in the
experiments, appear to be: accurate to within at least ±0.46 µm, do not exhibit a
significant bias, and are normally distributed with a standard deviation of 0.12 µm.
Qualitatively, overall experimental results suggest that measurement accuracy is much
to better than ±0.46 µm.
Figure C.1. Typical digital image correlation displacement results for rigid body displacements.

Table C.1. Rigid body displacement results.

<table>
<thead>
<tr>
<th>Calibrator</th>
<th>DIC</th>
<th>Average</th>
<th>Accuracy (µm)</th>
<th>StDev (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>IRBM</td>
<td>v (µm)</td>
<td>v (µm)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2.54</td>
<td>2.47</td>
<td>0.07</td>
<td>0.12</td>
<td></td>
</tr>
<tr>
<td>5.08</td>
<td>4.64</td>
<td>0.44</td>
<td>0.11</td>
<td></td>
</tr>
<tr>
<td>7.62</td>
<td>7.66</td>
<td>-0.04</td>
<td>0.11</td>
<td></td>
</tr>
<tr>
<td>10.16</td>
<td>10.32</td>
<td>-0.16</td>
<td>0.12</td>
<td></td>
</tr>
<tr>
<td>12.70</td>
<td>12.60</td>
<td>0.10</td>
<td>0.11</td>
<td></td>
</tr>
<tr>
<td>25.40</td>
<td>25.36</td>
<td>0.04</td>
<td>0.12</td>
<td></td>
</tr>
<tr>
<td>38.10</td>
<td>38.21</td>
<td>-0.11</td>
<td>0.15</td>
<td></td>
</tr>
<tr>
<td>50.80</td>
<td>50.47</td>
<td>0.33</td>
<td>0.14</td>
<td></td>
</tr>
<tr>
<td>Average</td>
<td></td>
<td>0.09</td>
<td>0.12</td>
<td></td>
</tr>
</tbody>
</table>

Figure C.2. Typical measurement distribution.
C.1.2 Crack Opening Displacement Measurement Uncertainty Results

As with rigid body displacements, experimental displacement measurements can be viewed as two superimposed components of displacement, a mean component and a variable component. The mean component is ideally composed of actual (accurate) values of displacement throughout the window of observation. The variable component is the difference between the actual and measured values of displacement at each measurement location. However, unlike rigid body displacement data, actual displacement values vary from point to point. Each point in the data set includes a unique actual displacement value upon which measurement variability is superimposed. These components must be separated if precision is to be evaluated.

Here, evaluation of displacement measurement precision in the near crack tip region uses previously measured results for a crack propagating through a layered FGM from the TiB rich surface toward the Ti layer. Measurement variability data is extracted from DIC results by subtracting the analytical solution for crack tip displacements from measured displacement results. Figure C.3 shows the process for estimating measurement variability.

Multivariable regression of measured displacements (Figure C.3a) is used to evaluate the first five terms of the asymptotic equation for displacement at the crack tip. Based on the coefficients, a surface assumed to represent actual crack tip displacements is generated (Figure C.3b) and subtracted from measured displacements, leaving a data set dominated by measurement variability (Figure C.3c). Statistical evaluation of the data set for a range of crack lengths produces results for precision that range from 0.09 to 0.12 µm, with an average standard deviation of 0.10 µm. The results are essentially
identical to the rigid body displacement results, and reinforce the conclusion that displacement results typically vary by a standard deviation of ±0.1µm.

Figure C.3. a) DIC displacement measurements, b) Analytical displacements, c) Measurement variability (a-b).

C.2 Impact of Measurement Uncertainty on K and T

A series of statistical analyses have been conducted to evaluate the impact of displacement measurement variability on stress intensity and T-stress recovered using
multivariable regression. Each analysis is conducted by superimposing displacement “noise” onto finite element analysis displacements around a crack tip, essentially reversing the procedure used earlier to extract measurement variability from displacement measurements. Artificial displacement noise is generated point-by-point using a random number generator, assuming the noise is normally distributed with a mean of zero and a standard deviation of 0.1 µm. The resulting displacements are used as input to the multivariable regression algorithm used to recover stress intensity. In each analysis, multivariable regression solutions for stress intensity and T-stress are repeated 100 times, with a new “noise” component generated for each point in each repeat.

Several variables and combinations thereof are considered in this series of analyses including: stress intensity, material modulus and gradient, and crack tip proximity to a boundary between layers, both approaching and extending away from the boundary (Figure C.4). Results for a crack growing in three material systems (monolithic and layered TiB to Ti and Ti to TiB) are shown in Table C.2, Table C.3 and Table C.4. In each case, stress intensity based upon the interaction integral, and multivariable regression assuming noise free displacements ($v_{num}$), are shown for reference. Multivariable regression-based stress intensity results with “noise” ($v_{num} + “noise”) superimposed are also shown. Franc2DL does not provide T-stress and as a result, only regression-based T-stress results are shown. Based upon earlier work, T-stress in the monolithic material should closely match expected interaction integral results. However, the accuracy of T-stress based upon multivariable regression in the FGMs is suspect and is only shown here for completeness.
In analyses assuming monolithic Ti material properties, crack lengths range from 5 to 13 mm, with interaction integral stress intensity results ranging from 6.83 to 16.25 MPa√m respectively. Table C.2 shows mean and standard deviation results for stress intensity based upon 100 independent analyses at each crack length. In each case, differences between mean stress intensity and baseline values for stress intensity are insignificant. Mean stress intensity results match the noise-free and interaction integral results, and the standard deviation ranges from 0.049 to 0.037 MPa√m. The lack of sensitivity of stress intensity to this level of noise can be attributed to the difference in displacement magnitudes due to stress intensity and measurement variability (Figure C.5a and c). Typically displacements due to stress intensity over much of the region around a crack tip are one to two orders of magnitude larger than the variability seen in digital image correlation measurements.

Figure C.4. Crack tip position relative to layer boundaries in analyses with the crack propagating from the Ti side of the specimen.
In contrast, T-stress results show significant sensitivity to measurement variability. T-stress, based upon regression of noise-free displacements, ranges from -12.10 to 24.06 MPa. While average T-stress matches noise-free results after 100 simulations, any single evaluation can include significant error. The standard deviation for T-stress in the results averages 6.5 MPa. This sensitivity can also be attributed to the magnitudes of displacement due to T-stress and noise, both of which are typically of the same order of magnitude (Figure C.5b and c).

Similar results are observed for a crack propagating in the layered FGMs, even for cases where the crack is approaching or has just crossed a boundary. Mean stress intensity results closely match the reference values established for noise-free displacements, and standard deviation results remain low. Even for the most extreme case, of a short crack propagating in the Ti-85 vol% TiB layer of the FGM at a stress intensity of 2.7 MPa√m, where displacements due to stress intensity are small, the
standard deviation in stress intensity remains low, $0.13 \text{ MPa} \sqrt{\text{m}}$ or 5 percent of the mean (Table C.3 and Table C.4). In addition, consistent with monolithic results, T-stress is very sensitive to measurement variability, particularly for the short crack referenced above.

Figure C.5. Displacements due to a) stress intensity equal to $3.1 \text{ MPa} \sqrt{\text{m}}$, b) accompanying T-stress equal to 29 MPa and c) measurement variability.
From these results it can be concluded that the measurement variability observed for digital image correlation does not, for the conditions in these experiments, have a direct and significant impact on stress intensity results. In the worst case, one standard deviation amounts to a five percent error. The same, however, is not true for T-stress, which exhibits extreme sensitivity to measurement variability.

Table C.3. Stress intensity and T-stress for a crack propagating from the TiB side of the specimen.

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<td></td>
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<table>
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C.3 Conclusions

Measurement variability has limited impact on recovered stress intensity, with a standard deviation of typically less than 0.1 MPa√m. It also imparts little if any bias in the results to explain the difference seen between finite element and DIC based stress intensity results. The primary impact of displacement measurement variability is its effect on recovered T-stress values. The typical standard deviation in T-stress observed in the analysis is on the order of 6 MPa, with some cases exceeding 21 MPa, representing a significant potential error compared with the maximum T-stress value of 38 MPa.
Appendix D

Digital Image Correlation Experimental Techniques

When digital image correlation is only employed to measure strains and displacements on the surface of a body, many options exist regarding preparation, lighting and speckling of the surface. The ideal surface has a matte finish to minimize specular returns, and is therefore easily illuminated. However, in this case, competing requirements exist. The surface of the specimen must be prepared to enhance visual observation of the crack tip and provide an appropriately speckled surface to enable accurate digital image correlation measurements. The following discussion documents techniques for lighting and speckling specimens to simultaneously satisfy requirements for digital image correlation and crack visualization.

D.1 Specimen Lighting

Crack visualization, particularly in the stiffer layers of the FGM where low toughness limits crack opening displacements, requires a finely polished surface. However, a polished surface is hard to properly illuminate. Normally any off-axis lighting reflects in a direction other than down the axis of the lens system, the only exception being light scattering from edges of surface defects or the flanks of a crack.
The result is a dark-field image with little detail. Figure D.1 shows a dark field image resulting from direct lighting by a ring of LEDs arranged around the objective lens.

To eliminate the dark-field produced by a polished surface, the specimen can be coated with a very thin (less than 0.001 mm) white semi-transparent layer to create a matte finish. In early experiments, India ink diluted in alcohol and atomized by briefly agitating the mixture in an ultrasonic bath was used to produce a matte surface. In practice, the ink produces a surface that scatters light, producing a bright-field image against which cracks and other defects appear dark. Figure D.2 shows such a bright-field including a crack, and black India ink speckles applied for digital image correlation measurements. The primary disadvantage to using a sprayed-on matte surface is hiding of the crack tip, particularly for small crack opening displacements such as those occurring in the TiB layers of the specimen. Even a thin layer of ink tends to stretch before breaking, masking the crack tip.

Figure D.1. Dark-field image using direct LED lighting (note crack propagating from the left).
A more effective approach is to light the specimen with a coaxial light source. Coaxial lighting of the surface assures that most of the light reflecting from the surface returns down the axis of the lens system and strikes the camera CCD. This minimizes the amount of light required to illuminate the surface, reducing specular returns from edges and defects and producing images with good contrast and clarity. Coaxial lenses and light sources can be purchased; however, for these experiments none was available, and an alternative approach was developed and adopted.

Figure D.3 shows a pseudo-coaxial lighting system developed for this research. Two rings of white LEDs, each with an intensity of 55,000 millicandles, are positioned facing the lens. The design allows for adjustment of the distance between the light ring and the objective lens to achieve the best illumination of the surface of the test article. In
this design, light reflecting from the surface of the lens strikes the polished surface of the test article at a small enough angle that most of the light is reflected back along the axis of the lens system producing a bright-field image.

Figure D.3. Pseudo-coaxial lighting arrangement.
At the micro-scale the design, if properly set up, produces images that rival those taken by high quality light microscopes. Figure D.4 shows the same region presented in Figure D.1, but this time the image was taken with the pseudo-coaxial lighting arrangement on a hydraulic test frame. A crack propagating from the left hand side of the image can be seen (arrow). Digitally zoomed in, the crack, unloaded in this image, is visible to within less than 5 µm of the tip identified in the high magnification light microscope image inset on the right. Note that in-situ and microscope images are shown with relative sizes maintained. However, the in-situ image contains more contrast and shows more surface detail than the microscope image, despite having been taken at a much lower magnification than is possible with the microscope. At the macro level, the pseudo-coaxial system produces high quality images with limited specular returns that can be used in a digital image correlation analysis (Figure D.6).

The primary disadvantage of this lighting design is that it is not appropriate for all lens systems. It works in this case because the lens system, a 12X navitar lens and 1X adapter tube, is long enough to prevent light from the LEDs from propagating the length of the lens system and striking the camera CCD. In shorter lens systems, this lighting design creates a cloudy image, which is much less satisfactory for crack visualization and digital image correlation.
Several options may be available and have been used to produce a speckle pattern on specimens when images are taken at relatively high magnifications. Natural variations in surface coloration can be used when appropriate. In a limited number of cases, specular returns from a dark-field image can also be used effectively. However, the recommended approach is to use sprayed on patterns of black and/or white paints or inks. Each of these methods has been used to speckle specimens for DIC in this research. Initially, black India ink was successfully used to speckle specimens; however, particular care was needed to ensure that speckle sizes remained within size limits of 5 to 15 µm.
appropriate for the image sizes being used. Natural surface coloration has also been used in limited cases (Figure D.5). However, the majority of images in this research have relied on application of laser toner to produce a speckle pattern appropriate to the length scale in the image.

Figure D.5. Digital image correlation image based upon natural material coloration variation in the Ti-45 vol% TiB layer.
D.3 Toner Speckling

The typical image sizes used for DIC in this research range from 5 to 6 mm wide and 6 to 8 mm high, depending upon the camera resolution. Images taken with a 1 mega pixel Sony XCD910 camera are typically 5.1 mm x 6.4 mm in size, while images taken with a 2 megapixel Sony XCD-U100 camera are typically 5.5 mm x 7.1 mm in size. Pixel sizes in both cases vary slightly, but are typically 4 to 5 µm/pixel. In comparison, typical laser toner particle sizes range from 3 to 15 µm, and conform to the general rule requiring speckle size range from approximately 1x to 3x the pixel size.

Two methods for applying laser toner have been used to speckle specimens for DIC. Initially, dry laser toner, dusted onto a dry surface directly or through a screen to aid in dispersing the toner, was used to speckle the specimen surface. Digital image correlation results using the dry toner application produced acceptable results; however, this approach tended to produce an uneven speckle pattern. Locally, variations in apparent speckle size due to clumping and inconsistency in coverage had an impact on the quality of digital image correlation results, increasing measurement variability and at times resulting in dropped measurements due to poor correlation.

An improved application method, developed over the course of the research effort, applies wet toner using an airbrush. The wet toner approach suspends a small amount of toner in alcohol. To ensure the toner does not cluster together in the suspension before or during application, the mixture is agitated for five minutes in an ultrasonic bath. Speckling is accomplished by applying very light coats of the alcohol/toner mixture until the desired speckle coverage is attained. Applied properly, an even distribution of toner can be easily achieved over the surface of a specimen.
(Figure D.6). Using this method produces a much more consistent speckle pattern, with less measurement variability and fewer dropped data points.

Figure D.6. Macro-scale image used for digital image correlation.