**RESIDUAL STRESS INDUCED MECHANICAL PROPERTY
ENHANCEMENT IN STEEL ENCAPSULATED LIGHT METAL MATRIX
COMPOSITES**

by

Sean James Fudger

A dissertation submitted to the Faculty of the University of Delaware in partial fulfillment of the requirements for the degree of Doctor of Philosophy in Materials Science and Engineering

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Sean Fudger

Approved: _____
Darrin Pochan, Ph.D.
Chair of the Department of Materials Science and Engineering

Approved: _____
Babatunde Ogunnaike, Ph.D.
Dean of the College of Engineering

Approved: _____
Ann L. Ardis, Ph.D.
Senior Vice Provost for Graduate and Professional Education

I certify that I have read this dissertation and that in my opinion it meets the academic and professional standard required by the University as a dissertation for the degree of Doctor of Philosophy.

Signed:

Chaoying Ni, Ph.D.
Professor in charge of dissertation

I certify that I have read this dissertation and that in my opinion it meets the academic and professional standard required by the University as a dissertation for the degree of Doctor of Philosophy.

Signed:

Prashant Karandikar, Ph.D.
Member of dissertation committee

I certify that I have read this dissertation and that in my opinion it meets the academic and professional standard required by the University as a dissertation for the degree of Doctor of Philosophy.

Signed:

David Martin, Ph.D.
Member of dissertation committee

I certify that I have read this dissertation and that in my opinion it meets the academic and professional standard required by the University as a dissertation for the degree of Doctor of Philosophy.

Signed:

Ismat Shah, Ph.D.
Member of dissertation committee

I certify that I have read this dissertation and that in my opinion it meets the academic and professional standard required by the University as a dissertation for the degree of Doctor of Philosophy.

Signed:

Andrew Teplyakov, Ph.D.
Member of dissertation committee

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ABSTRACT

Macro hybridized systems consisting of steel encapsulated light metal matrix composites (MMCs) were produced with the goal of creating a low cost/light weight composite system with enhanced mechanical properties. MMCs are frequently incorporated into advanced material systems due to their tailorable material properties. However, they often have insufficient strength and ductility for many structural applications. The macro hybridized systems take advantage of the high strength, modulus, and damage tolerance of steels and high specific stiffness and low density of MMCs while mitigating the high density of steels and the poor ductility of MMCs. Furthermore, a coefficient of thermal expansion (CTE) mismatch induced residual compressive stress method is utilized as a means of improving the ductility of the MMCs and overall efficiency of the macro hybridized materials systems.

Systems consisting of an A36, 304 stainless steel, or Nitronic® 50 stainless steel shell filled with an Al-SiC, Al-Al₂O₃, or Mg-B₄C MMC are evaluated in this work. Upon cooling from processing temperatures, residual strains are generated due to a CTE mismatch between each of the phases: steel shell, metal matrix, and ceramic reinforcement. By carefully controlling the CTE mismatch between the materials and

other processing parameters, residual tensile and compressive stresses form within each phase of the composite system.

The resulting systems, when properly designed, offer higher specific properties and a more structurally efficient system can be attained. Mechanical testing was performed and improvements in yield stress, ultimate tensile stress, and ductility were observed. However, the combination of these dissimilar materials, specifically iron and aluminum, often results in the formation of intermetallic compounds. In certain loading situations, these typically brittle intermetallic layers can result in degraded performance. In this research, X-ray Diffraction (XRD), X-ray Energy Dispersive Spectroscopy (EDS), and Electron Backscatter Diffraction (EBSD) are utilized to characterize the intermetallic layer formation at the interface between the steel and MMC.

As the residual stress condition in each phase has a large impact on the improved mechanical properties of the macro composite systems, accurate quantification of these strains/stresses is paramount. X-ray Diffraction Residual Stress Analysis (XRD-RSA) or Neutron diffraction was performed on five different hybridized systems in multiple steel shell thickness variations. The analysis shows variation in the measured strain and stress results due to outer steel thickness, difference in CTE between materials, and relative position within the composite. Improvements in mechanical properties, namely ductility and yield stress, are a direct result of these measured strains.

Chapter 1

INTRODUCTION

A composite material by definition is a combination of two or more constituent materials which typically exhibit different physical properties. The resulting properties demonstrated by the composite material generally fall somewhere between those of each of the components. The starting material is typically processed by a well understood, and in this case low cost, manufacturing process. The structure of the material is a direct result of the processing method which in turn dictates the properties and performance of the material. By carefully selecting additional material(s), a system can be created which incorporates additional positive properties or removes negative properties. Common examples are composite materials which display increased strength, decreased weight, and/or lower production costs while maintaining comparable properties [1]. The combination of traditional materials into composites can often result in material properties which are otherwise unobtainable by traditional monolithic materials. A composite's increased mechanical properties are often a result of a combination of the following strengthening mechanisms: Orowan strengthening, shear lag or load transfer effect, Hall-Petch strengthening, CTE and elastic modulus mismatch, and geometrically necessary dislocation strengthening [2]–[5].

There are several categories of composites based on the constituent material type. Typically composites are defined by the matrix material, i.e. polymer matrix composite, ceramic matrix composites, and metal matrix composite. Furthermore, hybrid composites are a category of composite which include at least three different materials or phases. Each system exhibits drastically different physical and chemical properties and has unique fabrication methods and applications. Additionally, the reinforcement material type, shape, distribution, and volume fraction will all affect the resulting material properties of the composite [6].

Generally speaking, discontinuously reinforced MMC manufacturing can be grouped into three main categories; stir casting, squeeze casting, and powder metallurgy. Stir casting of aluminum and magnesium is a very well understood process, one that can easily be adjusted to accommodate the addition of a ceramic particulate reinforcement phase. During fabrication, the alloy and reinforcement are combined into an MMC then cast or infiltrated into a steel shell creating a macro hybridized materials system. By combining steel with an MMC a macro hybridized materials system can be created which exhibits increased specific stiffness, increased strength, reduced density, and greater structural efficiency. Choosing to fabricate the MMCs with cheap raw materials and the stir casting process allows for the creation of low cost/light weight macro hybridized materials systems.

A high demand exists in the aerospace, marine, and automotive industries for components which are light weight but maintain their structural integrity. Recent interest includes materials such as high strength composites, magnesium, aluminum,

steel, and combinations in the form of metal matrix composites and hybrid structures [7]–[9]. Metal matrix composites have a wide range of applicability for automotive and structural applications due to the tailorability of their mechanical and physical properties [10]–[12]. Some manufactures within the automotive industry have already begun to replace traditional steel components with low density aluminum or magnesium based MMCs with an interest in improving fuel economy and reducing CO₂ emissions [13], [14]. Some successful examples are diesel engine pistons, engine blocks, and connecting rods from Toyota, Honda, and Nissan respectively [14], [15]. Particulate reinforced aluminum or magnesium based MMCs with a high volume fraction of ceramic phase provide for very high stiffness, low CTE, and low density observed in ceramics while maintaining the damage tolerance and processing advantages of cast light metals. These mechanical and physical properties can be fine-tuned by adjusting the size, distribution, or volume fraction of the reinforcement phase, utilizing different reinforcement phases, matrix alloy selection, or adjusting the processing parameters used in fabricating the MMCs [6], [16], [17].

As cost has limited the implementation of aluminum into some automotive applications, hybrid components consisting of both aluminum and steel have drawn much attention. Joining of dissimilar metals can be performed via arc welding, spot welding, diffusion bonding, ultrasonic welding, friction stir welding, laser brazing, etc. Mechanical assembly of these materials can also be performed by using rivets [18]–[23]. Each process exhibits its own limitations such as the requirement for specific filler materials, the ability to join only plate or simple geometric configurations, high

cost, and/or the formation of intermetallic phases. The stir casting process demonstrates some of the lowest costs associated with MMC fabrication and has the ability to manufacture complex geometries as expected from traditional casting methods. Unfortunately, the combination of steel and aluminum results in the formation of several intermetallic phases at the areas of contact. These intermetallic phases are extensively evaluated to determine their effect on load transfer between the materials and overall effect on the mechanical properties of the systems.

MMC's are unique since their mechanical and physical properties can be significantly altered by simply adjusting the ratio of matrix and reinforcement. Specifically, the ability to select the MMC's CTE allows for a CTE mismatch to be created between the steel encapsulant and MMC which results in improved mechanical properties. Each material within the hybrid composite follows equation (1) for linear thermal expansion:

$$\frac{\Delta L}{L_0} = \alpha \Delta T \quad (1)$$

where L_0 is the original length, ΔL is the change in length in one dimension, α is the coefficient of thermal expansion, which is material specific, and ΔT is the change in temperature.

By selecting steel with a higher coefficient of thermal expansion than its corresponding MMC core, a residual compressive stress is introduced onto the core due to contraction of the steel upon cooling from processing temperatures. Liu and Lewandowski showed that a hydrostatic pressure of 300 MPa (43.5 ksi) increased the

reduction of area from 10 % to 80 % and quadruple failure strain in a 6061 aluminum alloy reinforced with 15 vol% Al_2O_3 particulates tested in a pressure chamber environment [24], [25]. The imposed hydrostatic pressure caused a shift in the microscopic failure mechanism. Without the induced hydrostatic pressure, typical failure observed was due to ductile failure in the matrix due to nucleation and growth of voids, void formation at the reinforcement-matrix interface, or ductile failure in the matrix in regions adjacent to the reinforcement. Following a superimposed hydrostatic pressure, the ductility significantly increases because the failure mechanism shifted to fracture of the brittle Al_2O_3 reinforcement phase which then resulted in void nucleation, growth, and coalescence in the matrix [26]–[31].

This research aims to evaluate CTE mismatch induced residual stresses of varying magnitudes to improve the mechanical properties of macro hybridized materials systems. Three steels were selected exhibiting a range of strength, ductility, and CTE: a low CTE mild steel (A36), a high CTE austenitic stainless steel (304), and a high strength/high CTE austenitic stainless steel (Nitronic® 50, cold worked 25%). The core of these steels consists of an aluminum alloy or magnesium alloy matrix discontinuously reinforced with a high volume fraction of Al_2O_3 , SiC, or B_4C ceramic particles.

A number of macro composite material systems were mechanically evaluated by methods of shear push-out and tensile tests to determine material properties. Following mechanical testing, extensive structural characterization was performed on each system in the form of fractography via optical microscopy and scanning electron

microscopy (SEM), X-ray diffraction (XRD), X-ray energy dispersive spectroscopy (EDS), and electron backscatter diffraction (EBSD). Furthermore, X-ray diffraction residual stress analysis (XRD-RSA) and Neutron diffraction were performed on several of the macro composite systems to determine the residual stress condition of the individual phases.

Chapter 2

MATERIAL SELECTION AND PROCESSING

Aluminum and magnesium alloys are typically implemented into composite systems due to their low densities, high stiffness, relatively low cost, ease and flexibility of fabrication. When these alloys are reinforced with a ceramic phase the mechanical properties can be altered to increase stiffness and strength while maintaining low density and fabrication advantages [32]. Numerous reinforcement geometries and material types are utilized in MMCs and exhibit a wide range of properties. The reinforcement materials incorporated into the macro composite materials systems in this research are particulate Al_2O_3 , SiC , or B_4C . Typical reinforcement geometry include particulate, platelet, whisker, short fiber, long or continuous fiber, and layers of a laminate. Particulate reinforced composites exhibit isotropic material properties whereas each of the others demonstrate anisotropic properties based on the orientation of the reinforcement [33]–[35]. Platelets, whiskers, and fibers often have a preferred orientation resulting from the fabrication process or intended application where higher strength and stiffness is needed in a specific direction. Selection of reinforcement phase geometry is often limited by the composite's fabrication method, which is decided based on the starting materials, application, and other factors. In addition to the existence of numerous reinforcement

geometries, a large number of reinforcement materials also exist and are implemented based on the composites application. Hull and Clyne provide an exhaustive description on the mechanical properties and applications of carbon and glass fibers, silicon carbide, alumina and aluminosilicates and numerous other reinforcement materials [32].

2.1 Material Selection

The matrix alloys were chosen specifically for the reinforcement type in order to avoid undesirable thermodynamic instabilities. Since SiC tends to form Al_4C_3 when it reacts with aluminum, Al-10Si was chosen to avoid the formation of Al_4C_3 and subsequent degradation of the reinforcement particles which can drastically diminish mechanical properties [6], [36]–[38]. An Al-4Mg alloy was selected for use with the Al_2O_3 reinforcement as this alloy provides better ductility than Al-10Si and the inclusion of magnesium as an alloying element allows for chemical wetting via the PRIMEX process [39]. More literature exists on MMCs reinforced with SiC and Al_2O_3 as compared to B_4C due to fabrication complications, poor wetting between B_4C and aluminum alloys and higher costs [40], [41]. However, B_4C has a lower density and higher hardness along with better chemical and thermal stability compared to SiC and Al_2O_3 and thus was utilized as a reinforcement in the magnesium matrix composite [41]–[43].

2.2 Processing

A variety of processing techniques exist for the creation of metal matrix composites. Table 1 presents important features and considerations for the three main fabrication processes commonly used for discontinuously reinforced MMCs.

Table 1. Comparison between fabrication methods frequently utilized for the production of discontinuously reinforced MMCs [44], [45].

Process	Stir Casting	Squeeze Casting/ Infiltration	Powder Metallurgy
Description	Mechanically stir liquid metal and solid particles	Liquid metal injected into reinforcement preform	Consolidate blend of metallic and ceramic powders
Shape and size of composite part	Wide range of shapes and sizes	Limited by preform shape, few cm height	Wide range of shapes but size limited
Added pressure	None	Pressure assisted (low to high)	High Pressure
Distribution of reinforcement	Inhomogeneous due to settling of reinforcement	Homogeneous	Homogeneous
Interfacial bonding matrix/reinforcement	Difficulty forming interfacial bond	Strong	Strong
Wettability	Problematic	Good	Best
Grain size of matrix	Coarse	Fine	Fine-Coarse
Cost	Low	Medium	High

The focus of this thesis is not to compare processing techniques, however, it is necessary to introduce the variety of techniques which exist and reaffirm the selection of stir casting as a low cost process with a wide range of applicability. Consistent with

the low cost fabrication process, low cost raw materials are also used during the fabrication of the macro hybridized materials systems. Commercially available naturally aged aluminum and magnesium alloys were the chosen matrix alloys. Al_2O_3 , SiC , and B_4C were selected as reinforcement powders with FEPA grit sizes (corresponding particulate size in parenthesis) of P220 ($53 \pm 3 \mu\text{m}$), P240 ($44.5 \pm 2 \mu\text{m}$), and P280 ($36.5 \pm 1.5 \mu\text{m}$) respectively. These ceramic reinforcement materials are all of particulate geometry with aspect ratios of approximately 1. Alumina is produced by refining bauxite via the Bayer process (crushing and milling bauxite followed by filtering and crystallizing). The process results in a high purity Al_2O_3 product with a rhombohedral or hexagonal crystal structure. Silicon carbide is produced by heating silica sand and a carbon source (typically petroleum coke) to high temperatures in an open furnace which results in the crystallization of silicon carbide grains with a hexagonal crystal structure. Boron carbide is produced by the reaction between boron trioxide and carbon at high temperatures in an electric arc furnace resulting in a hexagonal crystal structure [46]. Although the fabrication processes vary substantially between these reinforcement powders, each results in a chemically inert, high hardness, high strength, and low density material that is exceptionally suitable for incorporation into a MMC.

Figure 1 outlines the fabrication process for each macro composite materials system, denoted 30P, 55P, and 45P process which corresponds to the volume percent of reinforcement for each case.

Figure 1. Macro composite materials system processing schematic for a) 30P Process, b) 55P Process, c) 45P Process [47]–[49].

The 30P and 55P Process follow the same first 3 steps. The ceramic powders, SiC or Al₂O₃, were pre-wet with an aluminum alloy, Al-10Si or Al-4Mg respectively, using a pressure-less infiltration technique. Wetting was achieved by mechanical (dispersion) or chemical means (PRIMEX process) based on the system [39]. The systems being fabricated with the 30P process were allowed to begin cooling in atmosphere immediately after casting whereas the 55P process has an additional hold time on the order of several hours post casting. As the viscosity of the melt increases substantially with the higher volume of the reinforcement phase, 55 volume percent vs. 30 volume percent, the additional time at processing temperature encourages sufficient flow of the MMC into the cavity. In the 45P process, the powder B₄C was

packed into the empty cavity of the sample and the molten magnesium alloy was infiltrated without any added pressure. Following infiltration there was an additional hold time at processing temperature.

The degree of wetting is an important consideration for the combination of matrix alloy and reinforcement particles. Wetting is defined as the ability of a liquid to retain contact to a solid surface. This prolonged contact is a result of intermolecular interactions as the two phases are brought together. The degree of wetting or wettability is a result of adhesive and cohesive forces and is characterized by the contact angle between a liquid droplet and a solid surface obtained from the sessile-drop technique as shown in figure 2 [50].

Figure 2. Contact angle of a liquid droplet on a solid surface.

A contact angle of $\theta = 0^\circ$ represents complete wetting, $0^\circ < \theta < 90^\circ$ represents partial wetting and $90^\circ < \theta < 180^\circ$ the nonwetting condition. Contact angles are determined from Young's equation (2). Where γ_{sv} is the interfacial tension between solid and vapor phases, γ_{sl} is the interfacial tension between solid and liquid phases and γ_{lv} the interfacial tension between liquid and vapor phases [50].

$$\cos \theta = (\gamma_{sv} - \gamma_{sl}) / \gamma_{lv} \quad (2)$$

Good wetting between the reinforcement particles and the liquid matrix is essential for the generation of good bonding and results in successful load transfer between the phases and high specific mechanical properties of the composite system [37]. Contact angle and thus wettability is affected by many factors including alloying elements in the matrix, temperature, reinforcement chemistry, reinforcement preparation method, and time (kinetic stages) [51]–[53]. Additional information is available in literature regarding the wetting and interfacial reactions of aluminum alloys and SiC, Al₂O₃ and B₄C particulates [51], [54]–[56].

The PRIMEX process, introduced by the Lanxide Corporation, utilizes magnesium as an alloying element within the matrix, along with a controlled nitrogen environment to encourage the reaction pathway outlined by equations (3), (4), and (5):

Karandikar *et al.* provide additional information about the PRIMEX process and specifics regarding the interactions between matrix alloying elements and the reinforcement phase [39].

Table 2 outlines all of the macro composite materials systems created as a part of this work. A total of eight different systems were created utilizing 3 steels, 3 matrix alloys, and 3 ceramic reinforcement materials.

Table 2. Macro composite test matrix.

Steel Encapsulant	Matrix Material	Particle Reinforcement	Reinforcement Percentage
A36	Al-10Si	SiC	30 vol%
A36	Al-10Si	SiC	55 vol%
A36	Al-4Mg	Al ₂ O ₃	46 vol%
304 SS	Al-10Si	SiC	30 vol%
304 SS	Al-10Si	SiC	55 vol%
304 SS	Al-4Mg	Al ₂ O ₃	46 vol%
Nitronic® 50	Al-10Si	SiC	55 vol%
Nitronic® 50	Mg AZ91E	B ₄ C	45 vol%

In addition to the eight materials systems, 3 conditions exist within each resulting from different steel to MMC ratios (varying steel shell thickness). The fabricated geometries of the macro hybridized bars for this research were limited to dog-bone specimens for tensile testing and cylindrical rods for shear push-out testing for a proof of concept. However, M Cubed Technologies, Inc. has the capabilities to produce macro composite systems with a variety of sizes and complex shapes.

2.3 Conclusions

1. Stir casting and pressure-less infiltration were selected as suitable fabrication processes due to their low costs and ability to create complex geometries.
2. Metal matrix composites consisting of aluminum or magnesium alloys reinforced with particulate Al₂O₃, SiC, or B₄C were successfully fabricated.
3. Three steels were chosen, A36 steel, 304 stainless steel, and Nitronic® 50 stainless steel to encapsulate the aforementioned MMCs resulting in eight different macro composite materials systems.

4. Wetting was achieved via mechanical and/or chemical means resulting in good bonding between the matrix and reinforcement particles.

Chapter 3

MECHANICAL TESTING AND FRACTOGRAPHY

Steel/MMC macro hybridized tensile bars and shear push-out specimens were produced by M-Cubed Technologies Inc. Newark DE. Subsequent tensile testing was performed at Lehigh Testing Laboratories, Inc. New Castle DE. Following mechanical testing, the fracture surfaces of the specimens were characterized utilizing optical and scanning electron microscopy, X-ray energy dispersive spectroscopy and X-ray imaging.

3.1 Optical Microscopy

Optical microscopes were used to image all of the samples in the as received condition. Moreover, they were utilized as the first step for characterization and documentation of the fracture surfaces on each of the tensile specimens. Optical microscopes combined with macro lenses allowed for documentation of the entire specimen whereas a traditional objective lens under high magnification was used for characterizing the fracture surfaces. As the maximum achievable resolution for traditional optical microscopes is limited based on the wavelength of visible light, a scanning electron microscope was utilized for high magnification imaging.

3.2 Scanning Electron Microscopy

Since many important features of a fracture surface, topography, crystal structure, chemical composition etc., occur on a scale much smaller than achievable by a traditional microscope, SEM was also performed. Typical resolutions on the order of 1 μm down to 1 nm can be achieved via SEM. This resolution is achieved by accelerating a beam of electrons from a tungsten filament, Lanthanum hexaboride (LaB_6) filament, or field emission gun at the surface of the sample. The electron beam is aligned into a fine probe via electromagnetic condenser lenses. The resulting probe is scanned across the selected area on the sample in a raster by scan coils [57]. During the electron beam interaction with the sample, secondary, backscattered, and Auger electrons along with X-ray photons can be collected with their respective detectors to give unique images of the sample. Secondary electrons are those knocked out of their orbit from the incident beam and typically give topological information about the sample. Back-scattered electrons result from the incident beam approaching the nucleus of the material and getting scattered back at a high angle [58]. This imaging method gives information about the composition of the sample where elements with a higher atomic mass appear brighter. Auger electrons are emitted from the very shallow surface of the sample giving information about its chemistry. X-ray photons are generated as the electron beam knocks out an electron from an inside shell. An electron from the outer shell will drop down from the higher to lower energy state, as this happens a characteristic X-ray is emitted [59]. By measuring and recording the

energies of these X-ray photons the chemical composition of the sample can be determined, this is the basis of EDS.

3.3 Strengthening Mechanisms

Strengthening mechanisms are the means by which composite materials exhibit increases in strength, stress, or other mechanical properties as compared with the monolithic matrix. The strengthening mechanisms result from interactions between the matrix and reinforcement from the macro down to nanoscale. The following examples represent both primary and secondary strengthening mechanisms typically observed in metal matrix composites.

3.3.1 Shear lag

The shear lag or load transfer effect deals with the transfer of tensile stresses between the matrix and reinforcement by means of interfacial shear stresses. The shear lag model was originally created by Cox [60] after observations of load transfer between matrix and fibers but has been adjusted to incorporate reinforcement geometries with small aspect ratios by Nardone and Prewo [61]. As the composite is loaded some of the matrix material adjacent to the reinforcement particles is able to transfer part of the load onto those particles. Areas of the matrix which are sufficiently separated from reinforcement particles will start to plastically deform at loads comparable to those observed in the matrix without reinforcement. Ultimately, the yield strength of the composite is much higher than the matrix alone because in

addition to the matrix deforming at its own yield stress, the reinforcement phase is bearing a higher proportion of the overall load. Following Nardone and Prewo the composite yield strength is estimated as shown in equation (6) where it is assumed the yield strength of the composite is achieved when the matrix material yields [61].

$$\sigma_{cy} = \sigma_{my} \left[\frac{1}{2} V_p (s + 2) + V_m \right] \quad (6)$$

Where σ_{cy} is the estimated composite yield strength, σ_{my} the matrix yield strength, V_p the volume of particles, V_m the volume of matrix, and s the aspect ratio. For particulate reinforcement the aspect ratio is assumed to be approximately 1. From this equation it can easily be shown that the yield strength of the composite increases with increasing reinforcement volume fraction.

3.3.2 Orowan Strengthening

Orowan strengthening is applicable for composites containing nano-sized reinforcement particles. Generally speaking for precipitation hardening, as dislocations move through the lattice and encounter a second phase particle, assuming the dislocation has sufficient energy, it will cut through or bow around the second phase particle [62]. In the case of dislocations interacting with impenetrable nanoparticles within the matrix, dislocation bowing occurs and results in an increased material strength [2]. The following Orowan-Ashby equation (7) illustrates the strengthening contribution from the Orowan mechanism [63].

$$\Delta\sigma = \frac{0.13G_m b}{\lambda} \ln \frac{r}{b} \quad (7)$$

Where G_m is the shear modulus of the matrix, b is the magnitude of Burgers vector of dislocations, r is the particle radius, d_p the diameter of the particle, and λ is the inter-particle spacing.

$$\lambda \approx d_p \left[\left(\frac{1}{2\nu_p} \right)^{\frac{1}{3}} - 1 \right] \quad (8)$$

$$\Delta\sigma = \frac{0.13G_m b}{2r \left[\left(\frac{1}{2\nu_p} \right)^{\frac{1}{3}} - 1 \right]} \ln \frac{r}{b} \quad (9)$$

When the inter-particle spacing is expanded as shown in equation (8) and plugged into equation (7), a generalized form of the equation can be obtained [64]–[66]. From equation (9) the Orowan strengthening contribution in a composite can be determined.

3.3.3 Hall-Petch Strengthening

Hall-Petch strengthening is described as the strength or hardness dependence on grain size in conventional metal alloys [67], [68]. The Hall-Petch strengthening mechanism is based on the pile-up of dislocations at grain boundaries. Due to the misorientation of the lattices between adjacent grains dislocations cannot easily transfer through a grain boundary. As the average grain size decreases within a material, the surface area of grain boundaries increases. The addition of a high

volume of reinforcement particles encourages grain refinement [2]. Eventually, the grain size can be decreased so much such that only one dislocation can be present in each grain. At this point the Hall-Petch relationship is no longer valid. The effective grain size range for the Hall-Petch mechanism to occur is a few hundred μm down to around 8-20nm based on the material chemistry and how it was prepared [69]–[71]. Below this minimal value, softening or inverse Hall-Petch behavior is observed.

The relationship is shown in equation (10) and indicates as the grain size decreases, down to and not exceeding the critical grain size, the strength of the material will increase.

$$\sigma_y = \sigma_0 + \frac{k_y}{\sqrt{d}} \quad (10)$$

Where σ_y is the yield stress, σ_0 is the starting stress for dislocation movement (material specific), k_y is the strengthening coefficient (material specific), and d is the average grain diameter. Furthermore, the smallest matrix grain size in the MMC can be estimated using the Zener equation (11) [2], [64], [72].

$$d_m = \frac{4\alpha d_p}{3v_p} \quad (11)$$

Where α is proportional constant, d_p is the particle diameter v_p is the volume fraction of reinforcement particles.

3.3.4 Thermal mismatch strengthening

Another strengthening mechanism is the plastic deformation resulting from the CTE mismatch between the matrix and reinforcement. The differential thermal contraction resulting from high CTE matrix and substantially lower CTE reinforcement results in an increased dislocation density in the matrix as shown by Vogelsang *et al.* [73]. The misfit strain is a result of the CTE mismatch and change in temperature from processing conditions to room temperature. The Taylor equation (12) describes the stress contribution resulting from CTE and elastic modulus (EM) mismatch [2], [74], [75].

$$\Delta\sigma_{CTE+EM} = \sqrt{3}\beta Gb \left[\left(\sqrt{\rho^{CTE}} \right) + \left(\sqrt{\rho^{EM}} \right) \right] \quad (12)$$

Where β is a material constant, G is the shear modulus of the matrix, b is the Burgers vector, ρ^{CTE} and ρ^{EM} are the geometrically necessary dislocation (GND) densities resulting from CTE and EM mismatches respectively.

GNDs occur to allow a system with geometrical constraints, i.e. a hard reinforcement particle inside a ductile matrix, to deform without resulting in the formation of voids surrounding the particle [73]. An increasingly dense group of dislocations accumulate in the matrix to accommodate deformation while allowing both phases to deform in a harmonious way [76]. More deformation occurs within the matrix as compared to the reinforcement particles due to the matrix having a substantially higher CTE. The generation of GNDs results in an increased strain

hardening rate of the composite compared with the unreinforced matrix [77]. Equation (13) can be used to calculate the GND density resulting from the deformation due to a CTE mismatch [2], [64], [77].

$$\rho^{CTE} = \frac{A\Delta\alpha\Delta T v_p}{b d_p (1-v_p)} \quad (13)$$

Where A is a geometric constant, $\Delta\alpha$ is the CTE mismatch between the phases, ΔT is the difference between testing and processing temperatures, d_p is the particle diameter, v_p is the reinforcement volume fraction, and b is the Burgers vector. Additionally the change in dislocation density resulting from the elastic modulus mismatch is shown in equation (14) where ε is the misfit strain due to $\Delta\alpha$:

$$\rho^{EM} = \frac{6v_p}{\pi d_p^3} \varepsilon \quad (14)$$

Typically combinations of these strengthening mechanisms are present in MMCs. Due to the size of the reinforcement particles incorporated into the macro composite materials systems in this work and the grain size in the matrix materials, the Orowan strengthening mechanism and Hall-Petch effect can be neglected [5], [17], [65]. Thus the primary strengthening mechanisms observed are the shear lag effect and CTE mismatch resulting from GND formation within the MMC. Unfortunately a direct comparison between theoretical predictions using the above equations and the results of the mechanical tests is impossible. The shear lag equation (6) predicts 15 %, 22.5 %, and 27.5 % increases in composite yield stress for 30 vol%, 45 vol%, and 55

vol% of reinforcement regardless of reinforcement type. However, it does not take into account the limited ductility of the reinforcement phase. Tensile tests were performed on the MMC systems and each fractured in a brittle fashion below the yield stress of the unreinforced matrix.

Similarly, predictions of the effect of dislocation densities on stress for the composite are also highly dependent on the reinforcement percentage but do not closely match the measured results. Thermal mismatch strengthening can be calculated following equations (12-14) for Al-SiC: $\beta = 0.5$, $G = 26.4$ GPa, $b = 2.84 \times 10^{-10}$ m, $A = 12$, $\Delta\alpha = 20 \times 10^{-6}$, $\Delta T \approx 650$, v_p varies from 0.3 - 0.55 based on reinforcement percentage, and d_p varies between 36.5 and 53 μm according to reinforcement type [61], [64], [78], [79]. For the Al-SiC 55P case, an increase of approximately 28 MPa should be observed in composite strength as compared to the unreinforced alloy. Dislocation generation resulting from CTE mismatch is approximately 100 times greater than dislocation generation due to EM mismatch which indicates the CTE mismatch should have a substantially larger effect on the improvement in mechanical properties. However, these results are not observed in the composite system since the material fractures prematurely due to the high volume percentage and brittle nature of the reinforcement particles.

3.4 Tensile Testing

For the macro hybridized tensile bars, steel (A36, 304, and Nitronic ® 50) bar with a diameter greater than 0.5” dia. was bored to produce a tube with an inside

diameter of 0.188". The steel tubes were then processed to fill the internal void with various compositions of particulate MMCs: Al-SiC, Al-Al₂O₃, and Mg-B₄C using a cast or infiltration approach [47]–[49]. Following fabrication of the macro hybridized tensile bars; they are machined such that the gauge section of each bar was turned to a diameter of 0.300", 0.400" or 0.500" creating cylindrical dog-bone tensile samples as per ASTM B557-10, see figure 3 for a schematic. The various MMC cores remained constant at 0.188" but with 3 different thicknesses of steel to provide for 3 different residual stress conditions.

Figure 3. Schematic of tensile test specimen [47].

Each of the macro hybridized systems was tested and results were obtained for yield strength (0.2% offset), tensile strength, elongation, and reduction of area. Figure 4 presents specific stress vs. strain data for Al/SiC 55 vol%, 0.500" diameter gauge section, macro hybridized systems with three different encapsulating steels. To

realistically compare the various steel/MMC macro hybridized systems, the specific properties were calculated. For stress, the specific stress is the stress divided by the density of the material system in question. Specific properties account for density differences between the steels and MMC systems, different reinforcement volume fractions, and the volume of MMC in each given hybridized system which allows for a normalized comparison between systems.

Figure 4. Specific stress vs. strain for Al-SiC 55P MMC within each steel [80].

As shown in Figure 4, the Nitronic® 50/Al-SiC hybridized system has a drastically higher specific stress as compared to the 304 stainless and A36 steels yet retains in excess of 20% strain to failure despite the strain to failure of the Al-SiC 55P

MMC of less than 0.2%. The 304 stainless steel has about a 40% higher specific stress than the A36 steel and nearly double the strain to failure.

The specific stress versus strain for Al-SiC 30P, Al-SiC 55P, and Al-Al₂O₃ 46P encapsulated with 304 stainless steel (0.500" dia. gauge section) is shown in Figure 5. Table 3 shows 304 stainless steel has a much higher CTE than Al-SiC 55P and Al-Al₂O₃ 46P, and slightly higher than the Al-SiC 30P. Therefore, one would anticipate that following the thermal excursion required to fabricate the macro hybridized tensile bars, there would be a residual compressive stress in the MMC. The confirmation was made by both FEA simulation and neutron diffraction measurements and will be discussed in later chapters.

Figure 5. Plot of Specific Stress vs. Strain for 3 different 304 stainless steel/MMC macro hybridized tensile bars [80].

The 304SS 55P curve represents a monolithic 304 stainless steel tensile bar (no MMC core) subjected to the same thermal cycle as the macro hybridized system during fabrication. In this case, all the specific stress-strain curves in Figure 5 show the macro hybridized systems have a higher specific stress, particularly yield stress, than the 304 stainless steel alone. The strain to failure of the hybridized systems is similar to the 304 SS. In this case, the macro hybridized systems offer a specific yield strength performance advantage over the monolithic steel while maintaining a high level of ductility.

Table 3. Material properties.

Material	Matrix	Vp	ρ (g/cc)	E (GPa)	CTE (ppm/K)
A36	N/A	N/A	7.8	200	11.7
304 SS (annealed)	N/A	N/A	8.03	200	16.6
Nitronic ® 50	N/A	N/A	7.88	200	16.1
Al ₂ O ₃	N/A	N/A	3.9	370	5.1
SiC	N/A	N/A	3.22	476	3
B ₄ C	N/A	N/A	2.51	445	5.5
Al/Al ₂ O ₃	Al-4Mg	46	3.3	160	11.2
Al/SiC	Al-10Si	30	2.78	120	15.6
Al/SiC	Al-10Si	55	2.96	202	11.8
Mg/B ₄ C	AZ91E	45	2.22	165	16.5

Vp – particle volume fraction, ρ - density, E – Young’s modulus; ν - Poisson’s ratio, CTE – coefficient of thermal expansion (20-100°C). Sources: M Cubed Technologies (MCT) [81] or directly from the manufacturer’s websites or datasheets [46], [82].

CTE measurements were performed on 5 x 5 x 25 mm samples of the MMCs using a Netzsch TMA 402 F1 with a heating rate of 5°C/minute from 20°C to 100°C

with a helium purge gas. The expansion of the sample holder was corrected by a calibration measurement of a fused silica standard. The calibration run was carried out under identical measurement conditions as were used for the test samples. Two measurements were made on each material type and an average value was reported. In each case the results of both samples showed very similar and reproducible results [47].

The residual stresses resulting from the CTE mismatch between the steel and MMCs in these systems result in an increase in dislocation density and subsequent increase in yield stress. Equations (12) and (13) indicate how the yield stress of the composite varies as a result of the change in dislocation density resulting from a CTE mismatch. Vogelsang *et al.* performed an in situ High Voltage Electron Microscope (HVEM) investigation on 6061 Al reinforced with 5 vol% SiC whisker and 20 vol% SiC whiskers and platelets [73]. The research shows drastically higher measured UTS values compared with their continuum mechanics formulations for the Al/SiC 20 vol% case. CTE mismatch in MMCs is accepted in literature as a means of increasing dislocation density and thus increasing yield stress and ultimate tensile stress in composites [73], [83]–[85]. Furthermore, an increased flow stress resulting from additional dislocations in the matrix is a direct result of superposed pressure [30]. Increased dislocation density is however expected to be localized and observed most in areas of high reinforcement volume fraction resulting in high strain. [61], [73], [86].

Confirmed by SEM micrographs, the increase in ductility of the systems is a result of a change in failure mechanism within the MMC. Figure 6 shows the multimode failure mechanism observed in the 304SS/Al-SiC 55P system.

Figure 6. SEM micrograph of MMC fracture surface within 304SS/Al-SiC 55P system.

The high magnitude residual compressive stress in the MMC resulting from the high CTE mismatch leads to cracking of the hard SiC particles during mechanical testing. This indicates good bonding between the matrix and reinforcement particles exists as the load successfully transfers from the continuous matrix to the discontinuous reinforcement particles. Also consistent with the high strain observed for each system in figure 5, ductility is observed in the aluminum in the form of microvoid coalescence in the areas adjacent to the reinforcement particles.

Figure 7 presents specific stress vs. strain for Al-SiC 30P, Al-SiC 55P, and Al-Al₂O₃ 46P encapsulated with A36 steel (0.500" diameter gauge section). Contrary to the high CTE mismatch systems, each of the systems containing A36 steel (low CTE mismatch systems) regardless of the reinforcement type, exhibits a substantial drop in both specific yield stress and specific ultimate tensile stress.

Figure 7. Plot of Specific Stress vs. Strain for 3 different A36 steel/MMC macro hybridized tensile bars [80].

Evident from figure 8, failure occurs via brittle intergranular fracture within the matrix. Brittle fracture occurs when the local strain energy resulting from external load(s) becomes greater than or equal to the energy necessary to separate the atomic layers [87]. The grain boundaries are the weakest region of the material in this case resulting in failure between the grains and exhibiting the sharp/straight edges of the

grains seen in the micrograph [88]. In addition, none of the reinforcement particles appear to have cracked which indicates there is a limited strength contribution from the SiC particles. In this case, there appears to be no advantage to the hybridized system.

Figure 8. SEM micrograph of MMC fracture surface within A36/Al-SiC 30P system.

Figure 9 presents specific stress vs. strain for A36/Al-Al₂O₃ 46P of varying gauge section diameter. These systems exhibit decreased specific yield stress, specific ultimate tensile stress, and strain as compared to monolithic A36 steel that has undergone the same thermal cycle.

Figure 9. Plot of Specific Stress vs. Strain for A36 steel/Al-Al₂O₃ 46P macro hybridized tensile bars.

Figure 10 shows optical and scanning electron micrographs of the fracture surfaces of A36 steel/Al-Al₂O₃ 46P macro hybridized tensile bars. Several of the tensile bars exhibit microshrinkage or interdendritic porosity within the MMCs. This casting defect is a result of a steep thermal gradient between the matrix and high volume fraction of thermally insulating reinforcement particles. This result was not entirely unexpected as the Al-4Mg alloy is inherently difficult to cast. These casting defects ultimately result in decreased mechanical properties for the macro composite systems as illustrated in figure 9.

Figure 10. (a,b) optical micrographs (c,d) SEM micrographs of fracture surface of A36 steel/Al-Al₂O₃ macro hybridized tensile bars.

To evaluate the relative effectiveness of the ratio of steel to MMC, the specific yield stress was calculated for each of the macro hybridized materials systems and plotted vs. the %MMC. The %MMC refers to the ratio between the area of MMC core to the total area of the hybrid system (MMC + steel). The MMC core diameter remains constant and the outer diameter of the steel varies from 0.300" to 0.400" to 0.500" resulting in 14.1, 22.1, and 39.3% MMC respectively. The measured specific yield stress of the steel alone is indicated at 0% MMC while the specific yield stress of the MMC is indicated at 100%. These values were obtained by performing tensile tests on a steel dog-bone sample and MMC dog-bone sample as shown in figure 11.

Figure 11. Image of monolithic steel dog-bone (top) and Al-SiC dog-bone (bottom).

The dashed line between these points represents the rule of mixtures (ROM) prediction for specific yield stress at a given %MMC. Two identical samples for each macro composite system configuration were tested and the average value reported with standard error reported as error bars. Figure 12 presents specific yield stress vs. %MMC for each of the systems and reinforces the trends observed in figures 5, 7, and 9. For the low CTE A36 steel, and all %MMC combinations, the specific yield stress is below the ROM. However, for the high CTE 304 and Nitronic® 50 stainless steels, with all %MMC combinations, the specific yield stress is above the ROM prediction.

Figure 12. Specific yield stress vs. %MMC for each macro hybridized materials system.

As evident from figure 13, each macro composite materials system incorporating A36 or 304SS exhibits improvement in % elongation (ductility) above the rule of mixture prediction at some steel shell thickness. The condition which appears to be most consistent in improving the ductility for all systems is the 14.1% MMC (0.500" steel shell) case. As expected, a larger volume of steel shell applies a higher confining stress onto its core compared with a thinner shell, which is consistent with the neutron diffraction results. The residual stress caused by confinement in these cases suppresses void generation in the matrix resulting in an overall increase in the ductility of the system.

Figure 13. % Elongation vs. % MMC for each macro composite materials system.

Table 4 presents mechanical properties for two macro composite materials systems, namely the Nitronic/Al-SiC 55P and Nitronic/MgB₄C 45P systems. Measured yield stress (YS), ultimate tensile stress (UTS), and % elongation values along with their corresponding relation to ROM predictions are included. Apparent from the measured results, yield stress values and ultimate tensile stress values increase with an increasing outer steel thickness. The values in the table which exhibit green font indicate an improvement over the ROM properties and red font indicates a decrease in the measured properties. Larger increases in YS and UTS over ROM tend to be associated with the thinner steel conditions for two major reasons. First, the MMC core is significantly contributing to the overall strength of the macro composite system. Second, comparable YS and UTS values are obtained between the three

thicknesses but the weight reduction is substantial when decreasing the thickness of the high density steel. In both composite systems, the thickest steel condition (0.500" OD) results in an approximate 10% decrease in weight compared to the monolithic steel material. In addition the thinnest steel condition (0.300" OD) results in a weight reduction of approximately 25%. The weight reduction is a result of lower densities of the MMC materials compared to the surrounding steels, see table 3. The higher weight reduction of the systems containing Mg and B₄C are due to their lower densities compared to Al and SiC. Increases in yield stress and ultimate tensile stress are observed for each system at almost every steel thickness. Conversely the percent elongation is lower for each system as compared to the monolithic Nitronic® 50 steel.

Table 4. Mechanical properties of Nitronic® 50 encapsulated macro composite materials systems.

Composite system	Steel diameter	YS (ksi)	YS (MPa)	UTS (ksi)	UTS (MPa)	%e	ΔROM YS	ΔROM UTS	ΔROM %e	Weight Reduction
Nitronic/ Al-SiC-55P	0.3	86.15 ± .025	594.0 ± 1.7	92.65 ± 0.5	638.8 ± 3.5	13.25 ± 1.25	13.30%	7.47%	-24.92%	24.53%
	0.4	106.35 ± 0.45	733.2 ± 3.1	118.65 ± 0.05	818 ± 0.3	17.75 ± 0.25	7.04%	3.84%	-21.51%	13.80%
	0.5	116.2 ± 0.1	801.2 ± 0.7	131.85 ± 0.05	909.1 ± 0.3	22	4.50%	2.55%	-11.69%	8.83%
Nitronic/ Mg-B₄C-45P	0.3	90.5 ± 2.3	623.9 ± 15.9	95.8 ± 0.6	660.5 ± 4.1	8.5 ± 1.5	29.13%	-3.37%	-51.81%	28.21%
	0.4	111.5 ± 0.3	768.8 ± 2.1	123.45 ± 0.05	851.1 ± 0.3	15.5 ± 2.5	15.04%	-0.23%	-31.44%	15.87%
	0.5	122.4 ± 1.1	843.9 ± 7.6	135.9 ± 0.8	936.9 ± 5.5	20	10.46%	0.03%	-19.71%	10.16%

These systems show decreased elongation due to the fact that they were cold worked. Cold working strengthens the material by generating dislocations during plastic deformation [89]. These dislocations begin to accumulate as cold working progresses until they eventually act as pinning points to impede the movement of other dislocations. The result is an increased force required to overcome the barrier which means increased strength for the material. However, the movement of dislocations is what allows a material to plastically deform, thus any obstacles to this dislocation movement will decrease the ductility of the material [62]. In addition, the decreased ductility observed in the systems containing magnesium as a matrix alloy is due to the HCP structure of magnesium which has limited slip systems compared to the FCC aluminum [75], [90]–[92].

Table 5 presents the same results as table 4, this time for the macro composite systems encapsulated with 304SS. For each of these systems, substantially lower values for yield and ultimate tensile stresses are observed but the %elongation is much larger compared to the Nitronic® 50 systems. Measured YS, UTS, and %elongation tend to increase with increasing steel diameter. This indicates that the composite material properties are somewhat dominated by the steel material. Improvements over ROM for YS and UTS for the Al-Al₂O₃ 46P and Al-SiC 55P systems tend to increase with increased steel thickness. This can be attributed to the higher residual stress resulting from the thicker steel condition.

Table 5. Mechanical properties of 304SS encapsulated macro composite materials systems.

Composite system	steel diameter	YS (ksi)	YS (MPa)	UTS (ksi)	UTS (MPa)	%e	Δ ROM YS	Δ ROM UTS	Δ ROM %e	Weight Reduction
304SS/Al-Al₂O₃-46P	0.3	38.7	266.8	57.2 ± 0.5	394.4 ± 3.4	37.5 ± 0.5	1.08%	-6.02%	27.77%	23.13%
	0.4	51.15 ± 0.4	352.6 ± 2.8	73.95 ± 0.35	509.8 ± 2.4	41	20.50%	0.99%	9.31%	13.01%
	0.5	52.1 ± 0.05	359.2 ± 0.3	81.2	559.8	44	17.59%	2.42%	6.57%	8.33%
304SS/Al-SiC-55P	0.3	38.55 ± 4.55	265.7 ± 31.4	56.95 ± 0.05	392.6 ± 0.3	35 ± 5	5.39%	1.22%	19.92%	24.79%
	0.4	48.85 ± 0.65	336.8 ± 4.5	73.75 ± 0.75	508.4 ± 5.2	36.5 ± 1.5	17.92%	4.87%	-2.45%	13.95%
	0.5	53.15 ± 0.25	366.4 ± 1.7	81.6 ± 0.2	562.6 ± 1.4	44	21.81%	5.51%	6.73%	8.93%
304SS/Al-SiC-30P	0.3	40.1 ± 1.4	276.4 ± 9.7	55.45 ± 3.35	382 ± 23.1	22	-21.60%	-17.45%	-29.13%	25.68%
	0.4	47.15 ± 1.95	325.1 ± 13.4	73.05 ± 0.75	503.6 ± 5.2	42 ± 4	-13.60%	-6.62%	5.60%	14.44%
	0.5	50.55 ± 0.05	348.5 ± 0.3	80.95 ± 0.25	558.1 ± 1.7	46.5 ± 0.5	-9.36%	-2.97%	6.13%	9.24%

Additionally the Al-Al₂O₃ 46P and Al-SiC 55P systems exhibit high magnitude residual compressive stress in the MMC cores resulting from a high CTE mismatch of 5.4 and 4.8 ppm/K respectively, which results in improved YS, UTS, and %e in almost every steel thickness case. Whereas for the Al-SiC 30P system, which has a CTE mismatch of 1 ppm/K between outer steel and MMC core, substantially lower magnitude residual stresses are present and thus only minor improvements in %elongation were observed. Quantitative results of the residual stress measurements will be presented in chapter 4. The observed weight reductions follow those presented in table 4, in that the thinner steel condition results in the highest weight reduction. For these materials, it is also apparent that as the reinforcement percentage increases

from 30P to 55P, the weight reduction decreases slightly since SiC has a higher density than the aluminum matrix.

Another important parameter for many structural systems is the damage tolerance, or the specific energy, which is the area under the stress strain curve. The specific energy was calculated for each of the macro hybridized materials systems and plotted vs. the %MMC. The specific energy of the monolithic steel is indicated at 0% MMC while the specific energy of the MMC is indicated at 100%. The dashed line between these points represents the ROM prediction for specific energy at a given %MMC. The results are shown in figure 14 for the macro hybridized tensile bars of each surrounding steel thickness.

Figure 14. Plot of specific energy vs. % MMC for each hybridized system [80].

Due to the extreme strain to failure of the annealed 304 SS, the specific energy is highest for these systems. More importantly, and unlike the specific YS ROM results, both the A36 and 304 SS macro hybridized systems exhibit specific energy greater than the ROM would predict at all %MMCs evaluated. These systems exhibit the synergistic effect in that the properties of the overall system perform better than the sum of the components. As previously mentioned, the improvements in strength and ductility which result in increased specific energy are due to increased dislocation density and a shift in failure mechanism which result from a coefficient of thermal expansion mismatch. As the Nitronic samples exhibit decreased strain as shown in figure 13 due to the cold working, the specific energy is also below the rule of mixtures results for each steel thickness condition.

Figure 15 presents specific energy for the macro hybridized materials (red) evaluated in this work compared to several common structural materials (blue). The A36 mild steel has a specific energy similar to 5083 aluminum while maintaining a lower raw material cost. The 304 SS has substantially higher specific energy, similar to 4340 steel and much greater than Ti-6Al-4V (annealed). However, this material has a relatively low yield stress.

Figure 15. Specific energy for macro composite systems and structural materials [80].

Finally, the hybridized systems composed of Nitronic® 50 (cold worked) with either an Al-SiC or Mg-B₄C MMC, which has greatly increased yield strength, exhibits specific energy similar to Ti-6Al-4V (annealed). This is significant as such a system utilizes lower cost raw materials and lower cost manufacturing processes than titanium.

Further characterization via X-ray scans was performed on each of the tensile specimens post fracture. The X-ray images in figure 16 show the failure of the MMC cores characterized by a series of transverse cracks and relatively uniform segments of MMC. Additional X-ray images for the remaining macro composite systems can be found in appendix A.

Figure 16. 304SS/Al-Al₂O₃ 46P macro composite materials systems.

These cracks are due to a transfer of tensile stresses between surrounding steel and MMC core by interfacial shear stresses explained by the shear lag effect [60]. During tensile testing as the composite system is loaded sufficient axial stresses will develop in the matrix which causes rupture. This situation is the inception of the first transverse crack. Since the strain to failure is much higher in the surrounding steel compared to the matrix, the composite system continues stretching. As bonding has been achieved between the matrix and surrounding steel, axial stresses will continue to accumulate within the resulting MMC segments until the next transverse crack forms. Generally speaking this process continues until failure within the steel shell occurs resulting in catastrophic failure of the composite system [93]. Table 6 presents the average number of transverse cracks in each macro composite materials system.

Table 6. Transverse crack count within macro composite system tensile bars [80].

Average Number of Cracks by Diameter			
	A36	304	Nitronic
0.500"	17.7	31.4	26.5
0.400"	13.8	35	32.5
0.300"	11.7	29	30
Average Number of Cracks by Reinforcement Type			
Al/Al ₂ O ₃	10.8	35.6	-
Al/SiC	16.2	30.3	21.7
Mg/B ₄ C	-	-	37.7
Average Number of Cracks by Reinforcement Percent			
30P	11.0	24.2	-
45P	-	-	37.7
55P	16.1	35.7	21.7
Average Number of Cracks by Surrounding Metal			
	14.4	31.8	29.7

Analysis of the number of cracks and the resultant length of each MMC segment indicates substantial variability between the macro composite systems. It is apparent that outer steel thickness, steel type, reinforcement percent, and reinforcement type all affect the load transferring and resulting number of cracks. There are roughly double the number of cracks in the 304 SS and Nitronic® 50 systems as compared to the A36 steel. This is likely due to the higher CTE mismatch and resulting higher stresses offered by the stainless steels compared to the A36 steel. Similarly, the 55 vol% reinforcement MMCs resulted in 50% more cracks than its 30 vol% counterpart. For this case, a higher percentage of reinforcement means a higher CTE mismatch between steel and MMC, as the ceramics particles have very low CTE values. Furthermore, as the reinforcement particles are brittle in nature, increasing the ratio of a brittle material within the MMC will work to decrease the overall ductility of

the MMC likely resulting in additional transverse cracks. Figure 17 shows the number of transverse cracks for each system plotted vs. the residual stress measured via neutron diffraction. Sign convention is negative for residual compressive stresses and positive for tensile stresses.

Figure 17. Number of transverse cracks vs. the residual stress (measured via neutron diffraction).

There is an obvious linear trend observed where the systems which provide more residual compressive stress in the MMC core display more cracks and shorter segments. Thus, each element of the MMC can carry more of the load due to the residual compressive stress. The one case which exhibits a residual tensile stress in the matrix (A36/Al-Al₂O₃) also has the lowest number of cracks in the MMC. The shear lag model explains the transfer of tensile stresses between the shell and core via interfacial shear stresses [32]. As interfacial shear stresses are affected by contact pressure it makes sense that load transfer occurs more efficiently in the presence of an additional contact pressure which the surrounding steel impacts on the MMC core.

3.5 Theoretical Investigation

A 3D finite element modeling framework was implemented in the commercial software (ABAQUS/Explicit) to predict the residual stresses due to thermal expansion mismatch and predict the effect of hydrostatic confining pressure on the tensile damage behavior in steel encapsulated MMC tensile bar specimens. The framework involves a two-step process in which a thermo-mechanical simulation is first conducted to determine the residual stresses present in the hybrid structure after cooling down from MMC processing conditions.

The stress state from this simulation is imported into a simulation of a tension test as the initial stress state. The density, elastic modulus, and coefficient of thermal expansion of the MMC were assumed to be 2.96 g/cc, 200 GPa, and $11.8 \times 10^{-6}/^{\circ}\text{C}$ respectively. The density, elastic modulus, and coefficient of thermal expansion of the steel were assumed to be 7.85 g/cc, 200 GPa, and $17.3 \times 10^{-6}/^{\circ}\text{C}$ respectively. In the tensile loading simulations the MMC was modeled as elastic to failure with a hydrostatic pressure dependent failure criterion using a user subroutine. In tension, the MMC was assumed to have an ultimate strength (σ'_u) of 300 MPa. In compression, the MMC was assumed to have an ultimate strength (σ^c_u) which was dependent upon the confining stress state. In the absence of experimental data for the pressure dependent strength of the current MMC system, it was necessary to assume this behavior for this preliminary work which was designed to test the implementation of the pressure dependence in the finite element framework. The compressive strength

was assumed to follow the following hydrostatic pressure (P) dependent relation illustrated in equation (15):

$$\sigma_u^c(P) = 300 + 3P \text{ (MPa)} \quad (15)$$

The steel was modeled as elastic-plastic using Mises plasticity with an assumed initial yield strength of 1027 MPa with isotropic hardening to an ultimate strength of 1138 MPa at a true equivalent plastic failure strain of 0.19. The thermo-mechanical simulation did not consider heat transfer and assumed a uniform temperature profile which decreased linearly from an assumed stress free temperature of 400°C to room temperature (25°C). The interface between the two materials was assumed to be perfectly bonded. Figure 18 shows the FEA model and corresponding X-ray image of a dog-bone tensile specimen. The predicted damage patterns from the tensile simulations considering hydrostatic pressure dependent strength are shown in figure 18 and qualitatively predict the experimentally observed shear lag fracture patterns. Relatively uniform residual stresses are observed within the gauge section of each phase of the composite. Transverse cracking within the MMC core is a result of successful load transfer between steel shell and MMC core during a tensile test. The residual stress results from simulations are presented with the experimental results from neutron diffraction in section 4.3.

Figure 18. Transverse cracking within 304SS/Al-Al₂O₃ 46P dog-bone specimen (a) X-ray scan post fracture (b) FEA model [80] (c) Predicted confining pressure within the composite system; sign convention is negative for tension.

3.6 Shear Push-out Testing

A similar fabrication process was used for the production of the shear push-out specimens. However, in this case, three different diameter macro hybridized rods were produced, 0.626", 0.874", and 1.0". In all cases, the MMC core remained constant at 0.364" diameter. The rods were then sliced to a thickness of 0.236" as shown in figure 19 below. Shear push-out testing was utilized to measure the bond strength between the steel and MMC. A cylindrical punch was placed on the center of the MMC and incrementally loaded until the core pushed out of the surrounding steel shell. Three distinct stages exist during a push-out test; elastic loading up to the point of debonding, propagation of the debonding front, and consequent interfacial motion

by frictional sliding [32]. The maximum load value reached (during debonding event) was recorded for each of the macro hybridized systems for comparison.

Figure 19. Schematic of shear push-out test specimen [47].

Figure 20 shows images of the shear-push out specimens prior to and after loading. The sample on the left shows the core is flush with the steel shell whereas the sample in the middle shows the MMC core protruding out of the shell following the push-out test. The thinnest steel condition (0.626" diameter) is illustrated here and the corresponding results are presented in figure 21 below, but each of the steel thicknesses was tested and similar trends observed.

Figure 20. Shear push out specimens: untested (left), core pushed out bottom view (middle), and core pushed out top view (right).

Figure 21. Shear strength results for the 0.626" steel diameter composite [80].

Interfacial shear strengths between the steel and MMC core are expected to be affected by interfacial roughness, sliding distance, and contact pressure [32]. Since the inner diameter of the steel was fabricated in the same manner for all materials, the roughness contribution from the steel is expected to be the same for all samples. Furthermore, the sliding distance has a large influence on shear strength in the third stage (interfacial motion) but can be neglected for the debonding event. Thus the contact pressure is likely to have the largest effect on the experimental interfacial shear strengths. The nature of the materials systems investigated herein means that this shear push-out test is measuring not only the bond strength between the steel and the MMC, but also the contact pressure (residual stress) due to the CTE mismatch.

The 304 stainless steel shows much higher shear strength partly due to the much higher residual compressive stress on the MMC as compared to the A36 steel. It

is also apparent that MMCs with a higher reinforcement content result in greater shear strength as these MMCs have a lower CTE which leads to a larger CTE mismatch with the steel and subsequently a greater residual compressive stress in the MMC. Figure 22 presents shear strength vs. CTE mismatch for all three steel thickness conditions.

Figure 22. Shear Strength vs. CTE mismatch for each macro composite system grouped by outer steel diameter.

It is apparent from figure 22 that the highest shear strengths coincide with a substantially higher CTE in the steel than the MMC core (positive sign convention). Conversely, comparable CTE values between steel and MMC result in lower levels of shear strength and in the cases where the CTE of the MMC is higher than the steel (negative sign convention) shear strength values are the lowest. In addition to the residual stress resulting from the CTE mismatch, the confinement also ensures the steel and MMC maintain intimate contact during cool down. In the case of the

A36/Al-SiC 30P, the MMC actually has a higher CTE than the steel, 15.6 vs. 11.7 ppm/K. For several of these samples, the MMC contracted away from the steel so much during cooling that the cores actually fell out of the steel shell without any external load. For the A36/Al-SiC 30P samples which were loaded, the applied stress required for push-out was only ~600 psi (4 MPa), a fraction of the stress required compared to the 304/Al-SiC 30P system which has a CTE mismatch of 1.

Comparing the shear strength results from figure 21 and the transverse cracks documented in table 6, several trends can be observed as shown in figures 23 and 24.

Figure 23. Shear Strength vs. # Transverse Cracks.

A nearly linear trend is observed in figure 23 for shear strength vs. # transverse cracks. As the transverse cracking occurs as a result of successful load transfer between the materials, higher shear strengths between the steel and MMC materials develop indicating a more efficient macro composite system. The ability to transfer

load between the phases results in increased mechanical properties, namely yield stress and ultimate tensile stress, as each phase is contributing to the overall strength of the system. To reaffirm what was mentioned previously with the A36/Al-SiC 30P push-out samples, with shear strengths of ~600 psi (4 MPa), since contact between the steel and MMC is not maintained during processing, poor load transfer occurs during testing indicated by the very few transverse cracks (between 8 and 13) that form in the MMC. Conversely, over 40 transverse cracks are observed in the gauge length of the systems with a high CTE mismatch as a result of successful load transfer between phases.

Figure 24 presents shear strength vs. segment length resulting from the transverse cracking. As the gauge length of the tensile specimens is a constant value between all samples, the average segment length is measured as a consequence of the number of transverse cracks present, representing the spacing between cracks.

Figure 24. Shear Strength vs. Segment length.

It is evident from figure 24 that higher values of shear strength coincide with smaller average segment lengths. Although there is slightly more scatter in the data, these results are consistent with the results of figure 23, the systems with higher shear strengths tend to have more transverse cracks in the gauge length are thus result in smaller segment lengths. Since a large number of variables exist between the systems, namely reinforcement percent, material elastic constants, CTE mismatch, processing conditions, etc. scatter in the data is not unexpected.

3.7 Conclusions

1. Of the numerous primary and secondary strengthening mechanisms which can be present in MMCs, shear lag strengthening and thermal mismatch strengthening dominate in the macro composite materials systems investigated in this work. For the case of the Al-SiC 55P MMC shear lag is expected to increase composite yield stress by 27% or nearly 70 MPa compared with the unreinforced matrix. Similarly, the strength increase resulting from thermal mismatch strengthening is projected to be over 10% or 28 MPa.
2. Significant improvements in strength are observed in the systems which exhibit a high CTE mismatch between steel and MMC. The strengthening is a result of high magnitude residual stresses and increased dislocation density in the core due to the high CTE mismatch. For the systems which have comparable CTE values between the steel and MMC no improvements in strength are observed.

3. The 0.400" steel configuration in the macro composites results in a nearly 15% decrease in weight whereas the 0.300" configuration results in approximately 25 % weight savings as the MMC materials (matrix and reinforcement) both exhibit substantially lower densities than the surrounding steel.
4. Each system containing A36 and 304SS demonstrate better than rule of mixtures specific energy. The A36/Al-SiC 30P system shows improvements of nearly 75 %, the A36/Al-SiC 55P system ~30 % and the 304SS/Al-SiC 55P of 15 % for the 0.500" steel condition. Conversely, the systems surrounded by Nitronic® 50 stainless steel show reduced specific energy of approximately 25 % for the 0.500" steel condition due to cold working of the steel.
5. Macro hybridized systems can offer specific energy greater than or similar to more expensive high performance conventional monolithic metals, specifically Ti-6Al-4V. The systems containing Nitronic steel exhibit specific energies between 155 and 170 ksi*in³/lb. The 304SS/Al-SiC 55P system exhibits specific energy in excess of 260 ksi*in³/lb.
6. The FEA model predicts residual stresses due to thermal expansion mismatch and agrees with the experimentally observed shear lag fracture patterns resulting from a hydrostatic confining pressure.
7. The results of the shear push-out tests indicate that higher shear strengths, on the order of 13,000 psi (90 MPa), are a direct result of a high CTE mismatch and thus a high residual stress condition. Conversely substantially lower

values are necessary for debonding the MMC from the steel when CTE values are comparable: around 3,000 psi (21 MPa) or when the MMC's CTE is higher than the steel: approximately 600 psi (4 MPa).

8. Transverse cracking was observed in each macro composite tensile specimen. The number of observed transverse cracks increased nearly linearly with the shear strength of the systems up to a maximum of 43 cracks at a shear strength of ~16,500 psi (114 MPa). Similarly, the average segment length or distance between cracks decreases with increasing shear strength, resulting in a segment length of ~0.095" for the sample with 43 transverse cracks.

Chapter 4

RESIDUAL STRESS ANALYSIS

Residual stresses are the stresses that remain in a material after the original source, whether an external load or force, has been removed. Residual stresses result from excessive external loads which cause yielding of the material [94]. Residual stresses can also result from eigenstrain, a term introduced by Mura to characterize nonelastic strains resulting from thermal expansion, phase transformation, misfit strains, plastic strains, or initial strains [95]. The resulting eigenstress is defined as a self-equilibrated internal stress resulting from eigenstrain in a body which is free from any external load or force [95]. Residual stresses can have either a beneficial or detrimental effect on the material properties depending on the application and the residual stress [96]. For example, shot peening the surface of metals can significantly improve fatigue life by imparting a residual compressive stress at the surface. These residual compressive stresses must be overcome by the applied load in order for cracks to initiate [97]. Conversely, the presence of a residual tensile stress in an MMC prior to loading is typically considered as a flaw or defect. The tensile stress can cause microcracking during the initial stages of testing and result in decreased strength [98]. A simplified example of a residual stress field can be considered in the case of a spherical cavity within a homogeneous material in which an oversized sphere is placed

[96]. The solution was originally solved by Lamé and later generalized by Eshelby to describe the residual stresses resulting from the placement of a misfitting inclusion having identical elastic constants as the matrix [99].

Numerous residual stress measurement methods exist and can be categorized into destructive, semi-destructive, and non-destructive to the measurement sample. The non-destructive residual strain/stress measurement techniques include laboratory X-ray diffraction, Magnetic Barkhausen Noise Analysis, Synchrotron X-ray, Neutron Diffraction, Ultrasonic Testing, and Electron Backscatter Diffraction [97], [98], [100]. Each technique has its own advantages and disadvantages i.e. equipment accessibility, resolution, penetration depth, collection time etc.

Laboratory X-ray and neutron diffraction techniques are utilized for this research. In X-ray diffraction, X-rays are scattered by the electrons which surround the nucleus of an atom. As a result, heavier atoms with more electrons scatter X-rays more than light atoms which X-rays tend to penetrate through. The interaction between X-rays and electrons is a strong electromagnetic interaction and the beam cannot penetrate deep into the material. In the case of neutron diffraction, neutrons have no charge and interact with atomic nuclei via the strong nuclear force. Since the spacing between adjacent nuclei is orders of magnitude larger than the nuclei themselves neutrons can penetrate much deeper into materials [101].

All residual stress measurements were performed on cylindrical samples with 0.625", 0.875", or 1.0" outer steel diameter and a constant MMC core diameter of 0.375". The lengths of the macro composite materials systems varied slightly, with

lengths between 3-4 inches long. Measurements with both X-ray and neutron diffraction techniques were performed in the axial center of the sample to avoid any edge effects resulting from the cutting method or stress relaxation.

The source of the observed strains varies greatly with the composite material constituents. Generally speaking, they can be divided into 3 categories based on the length scale over which the strain acts. Type I stresses, also called macro-stress and engineering stress, typically fluctuate on a length scale proportional to many grains (several millimeters in size). These stresses are often the result of fabrication including processing parameters, phase constituents and structural geometry. Type II stresses typically fluctuate on a length scale proportional to individual grains. These stresses result from inhomogeneity within a material or between phases in a composite, specifically a coefficient of thermal expansion mismatch, elastic modulus mismatch, or discontinuity in plastic flow resulting from elastically deforming particles. Type III stresses fluctuate over the smallest distances down to atomic length scales. These stresses can result from variations in local stress fields around dislocations [96], [102], [103]. Type-I and Type-II stresses contribute to the shifts in the positions of diffraction lines and Type-II and Type-III stresses contribute to diffraction line-widths [104], [105].

4.1 X-ray Diffraction

X-Ray diffraction residual stress analysis was performed with a Technology for Energy Corporation (TEC) Model 1610 X-Ray Stress Analysis System employing

the $\sin^2\psi$ technique. Following Bragg's law (16) atomic spacing can be measured within a given sample.

$$n\lambda = 2d \sin \theta \quad (16)$$

The incident X-ray beam penetrates the sample, interacting with some of the crystallites in its path. The beam diffracts to a characteristic 2θ angle based on the atomic spacing of the material. Here d is the atomic spacing between diffracting planes, θ is the incident angle, n is any integer, and λ is the wavelength of the beam.

Figure 25 shows the setup for the TEC Model 1610 with a 304/Al-SiC shear push-out specimen being measured in the -45 to $+45$ ψ angle arrangement. The resulting intensity vs. 2θ plot is presented in figure 26. Utilizing a large -45 to $+45$ ψ angle arrangement for collection increases the effective gauge volume of measurement and measures more grains within that volume due to the alignment of the incident diffracting beam. In addition, measuring both positive and negative values of ψ allows the user to check for texturing within the sample which may be present as a result of the fabrication method.

Figure 25. XRD-RSA with TEC Model 1610 experimental setup.

Figure 26. Intensity vs. 2θ plot of Al_2O_3 contribution within 304/Al- Al_2O_3 sample.

Numerous macro composite systems with different constituents, orientations, and sizes were measured. The stress values obtained during measurements were too inconsistent and the error values too high for this technique to be considered a reliable method for surface measurement of the materials in their current condition. It was realized that additional variables, such as thermal and mechanical stresses resulting

from the cutting method and polishing technique, might adversely affect the results of surface measurements. Furthermore, it was decided more meaningful data can be obtained from measuring a larger gauge volume away from the surface thus alternative measurement techniques were sought after.

4.2 Neutron Diffraction

Similar to XRD-RSA, neutron diffraction also follows Bragg's law; however the determination of strain within a material requires measurement of a stress-free "standard" for quantification. In equation (17), ε is strain, d is the measured atomic spacing of the sample, and d_0 is the measured atomic spacing of the stress-free "standard".

$$\varepsilon = \frac{d-d_0}{d_0} \quad (17)$$

Neutron diffraction was utilized as it provides a method capable of measuring through thickness strain profiles in each phase of the material, substantially larger gauge volumes, and can separate stress contributions from each phase within the hybrid composites. Neutron diffraction does however have limitations in the type of materials that it can measure, specifically the B_4C in some of the systems. Boron has an extremely high neutron absorption cross section of 767 ± 8 barns for 2200 m/s neutrons [106]. This makes neutron diffraction measurements nearly impossible in the MMCs reinforced with B_4C .

All neutron diffraction measurements were performed at the Canadian Neutron Beam Centre, (CNBC) Chalk River site utilizing the L3 diffractometer. (115) reflection of a single crystal Ge monochromator was used to filter the spectrum resulting in a beam of neutrons that possess a narrow energy band exhibiting a wavelength of 1.5509 Å.

4.2.1 Stress-free standard fabrication and measurement

Stress-free “standards” were fabricated in the form of rectangular prisms, approximately 2 x 2 x 20 mm in size, similar in appearance to match-sticks. In an effort to limit the amount of residual stress introduced during fabrication of these specimens, electrical discharge machining was utilized. Since there is no direct contact between the tool and sample, machining is performed without introducing additional mechanical stresses which results from traditional cutting methods. Samples were fabricated for each of the steels, the Al-10Si matrix, and the Al-SiC MMC. In addition, equivalent match-sticks were made from the MMC raw materials: unreinforced Al-10Si and Al-4Mg alloys and powder SiC and Al₂O₃. The powders were poured into a vanadium crucible so a similar gauge volume could be measured. Vanadium is typically used as a container since its nuclei hardly scatter neutrons, having a neutron absorption cross section of 5.08 ± 4 for 2200 m/s neutrons [106]. This feature guarantees there will be no influence on the SiC and Al₂O₃ diffraction peaks. The average stress-free d_0 was measured from each of these samples with a gauge volume of 1 x 1 x 10 mm to be used for the corresponding strain calculations.

For each sample, limited by geometry, there are three measurement areas within the steel shell and four measurement areas within the MMC core. Since the steel thickness varied between the samples, step sizes were chosen such that they represent even spacing between measurements and span the thickness of the steel wall. The first measurement is $\frac{1}{2}$ step size inside the outer diameter of the steel and subsequent measurements penetrate deeper into the material. This process is repeated for the MMC core except the final measurement is in the center of the MMC, see figure 27. Since the core is a composite consisting of Al-10Si and SiC or Al-4Mg and Al_2O_3 , separate measurements were made at each MMC location since each of the constituent materials diffracts at different angles and will be subjected to different strains.

Figure 27. Diagram of the measurement locations on a hybrid-composite; measurements 1-3 are within the steel, measurements 4-7 are within MMC core [107].

4.2.2 Peak selection

Careful selection of 2θ diffraction peaks was also important for the accurate measurement of strain in the composite systems. As neutron diffraction is a non-destructive technique, the neutron beam must pass through the outer steel material and penetrate into the MMC in order to measure the strain on the core. The ability to distinguish between diffraction peaks created by each phases was accomplished with the help of the Pauling file database. Prior to measurement, files from each phase are compared and one peak is selected for each phases with considerations of peak intensity, peak overlap or shouldering, and peak angle relative to 90° . Figure 28 gives an example of the Pauling file for SiC at a wavelength of $1.55 \text{ \AA}$.

Figure 28. Pauling file: Intensity vs. 2θ for SiC at 1.55 Å wavelength.

High intensity peaks are observed at 34, 36, 38, 60, and 72 degrees 2θ , representing the (101), (102), (103), (110), and (116) directions respectively. Although the intensity of the (102) peak is highest for this material, the (110) peak was ultimately selected due to the lack of peak overlap from the steel or aluminum. Furthermore, the closer the peak is to $90^\circ 2\theta$, the more symmetric the gauge volume will be within the sample based on the geometry of the incident and diffracted beams. It is necessary to measure three separate orientations, axial, radial, and hoop for the cylindrical geometry, see figure 29, to properly map out stresses within the sample.

Figure 29. Cylindrical coordinate system illustrating axial, radial, and hoop directions.

For example, equation (18), Hooke's law, solves for stress, σ . Where E is Young's Modulus, ν is Poisson's ratio, and ϵ is strain as measured from equation (17). Subscripts A, R, and H represent axial, radial, and hoop directions respectively.

$$\sigma_A = \frac{E}{1+\nu} \left[\epsilon_A + \frac{\nu}{1-2\nu} (\epsilon_A + \epsilon_R + \epsilon_H) \right] \quad (18)$$

The measurement gauge volume was maximized to decrease the required collection time and associated error. Radial and hoop measurements were performed with a 1 x 10 mm beam slit whereas the axial measurement was limited to a 1 x 1 mm beam slit due to geometry. Although measuring a larger area may be more representative of the bulk material condition, the smaller beam geometry is still capable of obtaining narrow peaks with a high signal to noise ratio, see figure 30 below, when the collection time is increased. A 32-wire position sensitive detector was used to capture the diffracted beam resulting in the 3.2 degree collection width observed in figure 30.

Figure 30. Example of a typical Bragg peak (with Gaussian distribution) measured in 304 SS [107].

Throughout the set of experiments, each sample must be examined with exactly the same calibrated set-up, wavelength, collimators, and slits as the reference standards. Furthermore, the alignment of the beam and position of the samples is also

a crucial step in guaranteeing accurate results. First the sample is lined up optically to establish a datum, typically the edge of the sample. This step is then followed by a second alignment, this time utilizing the count rate of the neutrons to more accurately determine the samples position relative to the path of the beam. Figure 31 shows the steady-state neutron diffraction setup utilized at the Chalk River site for strain measurement. The current configuration illustrated in figure 31b is set up to measure the hoop direction on 3 macro-composite specimens.

Figure 31. a) Schematic [108] b) photo of steady-state neutron diffraction setup for strain measurement [107].

1. Neutron beam source
2. Single crystal Germanium monochromator resulting in a 1.5509 Å wavelength.
3. Beam defining optics: Collimator and Cadmium slit to shape incident beam
4. Specimen(s)
5. Interaction volume
6. Detector
7. Beam defining optics: Collimator and Cadmium slit to shape diffracted beam
8. Beam stop: Lead shielding

4.2.3 Origin of Residual Stresses

During fabrication, the hollow steel rods are heated to a processing temperature between 600 and 750 °C. At these temperatures, the molten MMCs are cast or infiltrated inside the hollow cavity created in the steel. Upon cooling the materials attempt to shrink proportional to their coefficient of thermal expansion. Evident from literature for 2009Al-25 vol% SiC_p [109], 2124Al-17 vol% SiC_p [110], and 6061Al-15 vol% SiC_w [111] thermal residual stresses due to a CTE mismatch between the phases results in tension in the matrix and compression in the reinforcement when MMC's are cooled from processing temperatures.

The ceramic particles have a substantially lower CTE compared to the aluminum matrix, 3 and 6 for SiC and Al₂O₃ respectively vs. 23 ppm/K for both aluminum matrix alloys, refer back to table 3. The aluminum attempts to shrink around the reinforcement particle, upon doing this, the aluminum transfers a load onto the particle. Due to the high stiffness of the ceramic particle, it does not necessarily yield or break but imparts a residual compressive stress on the particle. At the same time, the forces balance on the aluminum which can result in yielding and plastic deformation within the matrix, leading to residual tensile stress. The yield stress of the unreinforced aluminum matrix alloys, A36 steel, and 304SS are approximately 250 MPa (36.3 ksi), 415 MPa (60 ksi), and 325 MPa (47.5 ksi) respectively. When a material's internal stresses exceed their yield stress, plastic deformation occurs. For many of the macro composite materials systems which exhibit a high CTE mismatch, plastic deformation occurs both within the steel and the matrix material. For the case

of the A36/Al-Al₂O₃ samples, plastic deformation occurs in the matrix material as a result of the CTE mismatch between the matrix and reinforcement. However, since the CTE is comparable between the steel and MMC, low magnitude residual stresses are observed in the steel and no plastic deformation is expected.

Neutron diffraction measurements were performed on numerous macro composite materials systems. It is clear that the type II stresses do not normally average to zero in each phase and thus, like the macro stress, cause a shift in the Bragg scattering angles which allows the average strain in each phase over the sample gauge volume to be measured. Uncertainty in the strain measurement and the resulting stress calculation is a result of many factors relating to the measurement and reference values and include: uncertainty in the measurement of d and d_0 , the incident beam wavelength, and the measurement position within the specimen.

4.2.4 Residual Stress Results

Figure 32 presents the results of residual stress calculations using equation (18) for the A36 and 304 stainless steels, each with the Al-SiC 55P MMC and varying outer steel thicknesses. Since the core is a composite of two materials: matrix and reinforcement, the strains were measured separately.

Figure 32. Neutron diffraction residual stress results a) A36/Al-SiC 55P 0.626" steel outer diameter b) A36/Al-SiC 55P 0.875" c) 304/Al-SiC 55P 0.626" d) 304/Al-SiC 55P 1.0" [107].

It can be seen that within the respective material systems, a thicker outer steel diameter results in a higher magnitude residual compressive stress in the SiC reinforcement particles. The MMC core diameter is the same for all materials however the outer steel thickness varies according to the figure description. Furthermore, an additional strain is applied to the MMC core via the steel encapsulation. The surrounding steel contracts onto the MMC core affecting the residual stresses based on the CTE mismatch. Considering the MMC as having one averaged value of CTE based on the volume percentage of its constituents the following trends can be observed. Since the CTE is comparable between the A36 steel

and the Al-SiC 55P MMC, the residual stresses observed in the MMC are dominated by the CTE mismatch between matrix and reinforcement. The weak confinement offered by the A36 steel has little effect on the observed stresses on the core.

Conversely, the 304 SS with the Al-SiC 55P samples have a much higher CTE difference. The surrounding 304 SS contracts around the MMC compressing both the matrix and reinforcement. Lower tensile values are observed in the aluminum matrix and higher compressive values are observed in the SiC reinforcement. The effect of the steel can be isolated since the MMC core is identical between the samples and the elastic constants between the steels are comparable. This indicates the type II thermal misfit stresses are the dominant stress type present in these materials.

Figures 31-33 present the neutron diffraction residual stress results for several additional systems: A36/Al-Al₂O₃ 46P 1.0", A36/Al-Al₂O₃ 46P 0.626", 304/Al-Al₂O₃ 46P 1.0", 304/Al-Al₂O₃ 46P 0.626", 304/Al-SiC 30P 1.0", and 304/Al-SiC 30P 0.626" respectively.

Figure 33. Neutron diffraction residual stress results in A36/Al-Al₂O₃ 46P a) 1.0" outer steel diameter, b) 0.626" outer steel diameter.

Consistent with the previously presented neutron diffraction data, thicker surrounding steel results in lower tensile stress in the aluminum matrix and higher compression in the reinforcement when compared to thinner steel for each system. The low CTE mismatch between the A36 and Al-Al₂O₃ (0.5 ppm/K) results in low magnitude residual stresses in the steel. The residual stresses within the MMC are a result of the CTE mismatch between matrix and reinforcement. Comparing figure 33 to figure 34, the higher CTE mismatch offered in the 304/Al-Al₂O₃ 46P compared to the A36/Al-Al₂O₃ 46P systems results in higher magnitude compression in the reinforcements and also compressive stress in the matrix. The additional confinement from the steel brings the entire core into compression. In addition, the 304SS exhibits higher residual tensile stresses than the A36 due to load balancing from the core.

Figure 34. Neutron diffraction residual stress results in 304/Al-Al₂O₃ 46P a) 1.0" outer steel diameter, b) 0.626" outer steel diameter.

Lower magnitude residual compressive stresses are observed in the Al₂O₃ compared to the SiC reinforcements. This is attributed to a substantially lower Young's modulus (E), see table 3, for Al₂O₃ compared to SiC. Even in the case of a

low CTE mismatch system with SiC reinforcement, namely 304/Al-SiC 30P, it still exhibits drastically higher residual compressive stresses in the reinforcement than a high CTE mismatch system reinforced with alumina, 304/Al-Al₂O₃ 46P. Furthermore, the lower stiffness of the Al₂O₃ results in less load taken on the reinforcement, thus as the loads balance, higher magnitude residual stresses are observed in the matrix.

Comparable magnitude residual stresses are observed in the 304/Al-SiC 30P and 304/Al-SiC 55P despite having drastically different CTE mismatches, 1.0 and 4.8 ppm/K respectively. When considering the different reinforcement percentages, 30 volume percent and 55 volume percent, it can be seen that less of the MMC material volume is in compression in the 30P case.

Figure 35. Neutron diffraction residual stress results in 304/Al-SiC 30P a) 1.0" outer steel diameter, b) 0.626" outer steel diameter.

While the magnitudes seem comparable for each phase, evaluating the composite material as a sum of its proportional components yields a composite stress nearly 200 MPa further in compression. The observed increase in the MMCs residual

compressive stress is a direct result of the additional confinement caused by the larger CTE mismatch between steel and MMC.

4.3 Theoretical Stress Predictions

Axisymmetric FEA models were created within the ABAQUS software to predict residual stresses forming within the macro composite materials systems based on the CTE mismatch. Figure 36 presents results of the model predicting residual stresses based on CTE mismatch. Linear quad elements were used with a characteristic length of ~ 0.15 mm resulting in 38,000 elements for the 1.0" steel geometry and 23,000 elements for the 0.625" case.

Figure 36. FEA models of 304SS/Al-SiC 55P (a) 1.0" steel OD uniform cylindrical geometry (b) 0.500" steel OD (left) and 0.300" steel OD (right) dog-bone geometries [80]. (c) Predicted confining pressure within the composite system; sign convention is negative for tension.

The lighter color observed in the gauge section of the 0.300" sample compared to 0.500" sample indicates a lower magnitude compressive stress is present. These thermo-mechanical simulations did not consider heat transfer and assumed a uniform temperature profile which decreased linearly from an assumed stress free temperature of 400°C to room temperature (25°C). The effective temperature drop is the difference between the temperature at which stresses begin forming in the materials and room temperature. This is not to be mistaken with the temperature difference between the fabrication temperature and room temperature.

A literature review was performed as a starting point to determine an estimated effective temperature drop for the macro composite materials system. Fitzpatrick *et al.* use an Eshelby type model to represent a 2124Al-SiC plate fabricated via powder metallurgy and hot iso-static pressing. Their model estimated an effective temperature drop between 175-350 °C in a composite annealed at 400 °C and heat treated at 505 °C for 2 hours [102]. Doré *et al.* determined a stress free temperature of 630 °C for aluminum alloy 6063 [112]. Colley *et al.* and Phillion *et al.* investigated AA5182 and reported a stress free temperature of ~570 °C and ~560 °C respectively [113], [114]. Based on these values an effective temperature drop of approximately 540 °C would be observed. However, these stress free temperatures are expected to be highly dependent on alloy composition and microstructure. As the literature review indicated a range of values for the effective temperature drop, a parametric study was performed to determine how the effective temperature drop influences the residual stresses observed in the macro composite materials systems. Figure 37a-c presents the model's

stress predictions on the 304SS/Al-SiC 55P system for initial temperatures of 400, 500, and 600 °C respectively.

Figure 37. Model predictions for residual stress vs. distance into steel shell and MMC core for initial temperatures of (a) 400 °C (b) 500 °C (c) 600 °C.

An initial temperature of 400 °C results in an effective temperature drop ~375 °C to room temperature (25 °C). It is evident that as the initial temperature is increased an increased magnitude residual stresses is observed; tensile in the steel shell and compressive in the MMC core. The predictions also show an increased separation of the radial component compared with the hoop and axial in the steel with an increased effective temperature drop. Similarly the axial component in the core shows increased separation relative to the hoop and radial components with increasing temperature. These trends are not unexpected since a materials expansion and contraction in three dimensions are governed by its Poisson's ratio. Figure 38 illustrates this observation in the steel shell of the 304/Al-SiC 55P sample as measured by neutron diffraction.

Figure 38. Measured residual stress vs. distance into steel shell and MMC core.

The experimental results presented in figure 38 seem to fall somewhere between the predictions from the 500 and 600 °C initial temperature models, indicating an effective temperature drop likely between 475 and 575 °C. Since the predicted stresses appear to be very sensitive to the temperature change, it will be necessary to more accurately determine the exact effective temperature for each system. Exact initial stress formation temperatures could be measured via neutron diffraction by heating a macro composite sample near the melting point of the matrix and subsequently cooling while measuring strain. Additional equipment exists such as Gleeble® thermal-mechanical simulators which subject a sample to an identical thermal cycle as they experience during fabrication. Using a dilatometer and strain gauge they can directly measure the temperature at which stresses begin forming within the material [7], [79], [115], [116].

Once an accurate effective temperature drop is known for each macro composite materials system, the models can be updated and used to predict the residual stress in the Nitronic/Mg-B₄C system. Boron has an extremely high neutron absorption cross-section and thus the strain in the B₄C cannot be measured via neutron diffraction. However, our experimental data and the simulations suggest that the benefits and property enhancements from the encapsulation are largely independent of the interfacial phase structure (Chapter 5) between encapsulating materials and MMCs and are predominantly determined by the residual stresses arising from the CTE mismatch. We anticipate it would be reliable to theoretically predict the residual stress in B₄C should that becomes necessary for design, manufacturing or other purposes in application. Furthermore, residual stress conditions could be predicted in macro composite materials systems consisting of more complex geometries.

4.4 Conclusions

1. Neutron diffraction can be used as an accurate and reliable method for measuring bulk residual tensile and compressive stresses within the steel encapsulated metal matrix composites.
2. Low magnitude residual stresses within the steel are observed in the A36/Al-SiC systems which exhibit a low CTE mismatch between outer steel and MMC core. Conversely, the steel in the 304SS/Al-SiC systems have significantly higher residual stresses due to a large CTE mismatch between adjacent materials.

3. The confinement of the 0.626" 304SS on the Al-SiC MMC results in a 50 % more compressive stress in both the matrix and the reinforcement as compared with the same system surrounded by A36. The 1.0 inch thick surrounding steel results in a residual compressive stress in the MMC core approximately 15 % greater than the 0.626 inch steel for the 304/Al-SiC system.
4. The higher CTE mismatch offered by the 304 SS vs. the A36 steel surrounding the Al-Al₂O₃ 46P system results in over 100 % increases in tension in the steel and nearly 100 % increase in compression in the Al₂O₃.
5. The systems which exhibit the highest residual compressive stress in the reinforcement particles are the high CTE mismatch (4.8 ppm/K) 304/Al-SiC 55P systems with .626" and 1.0" steel OD demonstrating averages stresses of approximately -500 and -600 MPa respectively.
6. The FEA models created within the ABAQUS software predict a strong temperature dependence on residual stress calculations within the macro composite materials systems which exhibit a high CTE mismatch. Furthermore, comparable residual stress conditions are observed between the model and neutron diffraction measurements when an effective temperature drop between 475 and 575 °C is implemented. A residual tensile stress of approximately 300 MPa is observed in the steel shell and a compressive stress of approximately -200 MPa in the MMC core is observed under these conditions.

Chapter 5

INTERFACIAL CHARACTERIZATION

5.1 Sample Preparation

Sample preparation, specifically proper mounting, grinding, and polishing techniques are extremely important for many advanced characterization techniques like SEM, EBSD, TEM, etc. After the fracture surface of each of the tensile specimens had been extensively evaluated they were sectioned for further investigation. Each specimen had a minimum of three sections transverse to the direction of testing made within the gauge length. Specimens were cross sectioned using a wet diamond cut-off saw with a feed speed of 0.05 mm/s to minimize deformation. This allowed for material both within and away from the necked region to be characterized as evident from figure 39.

Figure 39. Schematic of tensile specimen indicating loading direction and typical cross section locations [117].

The resulting cross sections were mounted in Bakelite and incrementally ground with silicon carbide paper from 320 to 1000 grit. Subsequently, they were polished using 3, 1, and 0.3 micron diamond slurries and final polished with 0.05 micron colloidal silica in a Buhler VibroMet2 vibratory polisher. This resulted in a mirror like finish which allowed for microscopic inspection of the surface. Figure 40 depicts a typical resulting cross section after mounting, grinding, and polishing.

Figure 40. Section of a macro composite tensile specimen mounted in Bakelite.

5.1.1 Mounting

After sectioning a sample, the user must decide if the sample should be mounted, based on its size and shape and the characterization techniques to be performed. Samples which are too small or awkwardly shaped to manually polish often benefit from mounting. Typical mounting materials include phenolics, acrylics, epoxies, and diallyl phthalates. Each has its own benefit and application for properties such as edge retention, hardness, clarity, etc. [118]. Some mounting materials even contain electrically conductive additives for ease of imaging in the SEM.

5.1.2 Grinding and Polishing

Following sectioning and mounting of the sample, it is typically ground to remove the larger marks left from cutting and level the sample surface. Grinding uses abrasives bonded or fixed to paper or a platen. Grinding is generally performed with some form of lubricant, typically water, for heat dissipation and removal of swarf. Following grinding, polishing is performed utilizing free abrasives suspended on a cloth. Numerous cloth types exist and are selected based on the material and process. For example, cotton is used for rough polishing of metals, napless cloth for coarse or fine polishing with diamond, and velvet cloth for final polishing of soft materials [119]. Abrasive size is decreased with each step of the polishing process until the final polish is performed. Typical abrasive size for the final polish is below 1 micron which is substantially smaller than the reinforcement particles used in this project and includes abrasives like alumina (Al_2O_3), silica (SiO_2), and diamond.

5.2 SEM Characterization

A minimum of three samples from each macro composite tensile bar were prepared as outlined above. A focus was placed on the behavior and interaction between the steel shell and MMC core. The combination of these dissimilar materials, specifically iron and aluminum, often results in the formation of intermetallic compounds. Inherent to the processing temperatures, hold times, and diffusion coefficients of the constituents, the observed intermetallic layers varied significantly between the macro composite systems. As the intermetallic regions can exhibit poor mechanical properties, specifically ductility, compared with the parent alloys, it was important to perform a thorough investigation.

A focus was placed on the behavior and interaction between the steel shell and MMC core. A reaction layer forms between the materials based on the chemistry of the phases in contact, diffusion coefficients, time, temperature, and concentration gradients among other factors. The reaction layer between the materials not only varies by thickness but also shape. The top darker region is the MMC, the intermediate brighter region the reaction layer, and the bottom brightest region the steel in each case. The reaction layer observed in figure 41(a): A36/Al-Al₂O₃ 46P, exhibits irregular dendritic like structure with an average thickness of about 250 μ m. Figure 41(b): 304SS/Al-SiC 55P, shows a much more uniform reaction layer with average thickness of about 170 μ m. This reaction layer appears to be much more brittle as compared with 5a. Figures 39c and 39d show the Nitronic/Mg-B₄C 45P interface. The images illustrate a reaction layer thickness of about 10 μ m. Additional

images can be found in appendix B for the interfacial regions of the remaining macro composite materials systems.

Figure 41. Backscatter electron micrographs of the interface between steel and MMC. (a) 100x magnification of A36/Al-Al₂O₃ (b) 100x magnification of 304/Al-SiC (c) 100x magnification of Nitronic/Mg-B₄C (d) 1000x magnification of Nitronic/Mg-B₄C [117].

Table 7 presents the average reaction layer thickness of each of the macro hybridized materials systems as measured in the SEM. The interfaces in the A36/Al-SiC 30P, A36/Al-SiC 55P, 304/Al-SiC 30P, and Nitronic/Mg-B₄C 45P samples varied from having no SEM detectible reaction layer to having some thin discontinuous areas of reaction thus no XRD intermetallic peaks were observed on these samples. The

thinner reaction layers are likely due to the substantially shorter processing times associated with the 30P process and the fact that Mg does not react with Fe.

Table 7. List of macro hybridized materials systems with resulting average reaction layer thickness.

Steel Encapsulant	Matrix Material	Reinforcement	Reinforcement percentage	Average Reaction Layer Thickness (μm)
A36	Al-10Si	SiC	30 vol%	Max 3.5
A36	Al-10Si	SiC	55 vol%	Max 15
A36	Al-4Mg	Al_2O_3	46 vol%	250
304SS	Al-10Si	SiC	30 vol%	30
304SS	Al-10Si	SiC	55 vol%	140
304SS	Al-4Mg	Al_2O_3	55 vol%	160
Nitronic® 50	Al-10Si	SiC	55 vol%	190
Nitronic® 50	Mg AZ91E	B_4C	45 vol%	10

The Iron-Aluminum phase diagram is shown in figure 42 with the steel/MMC processing temperature conditions superimposed. As evident from figure 42, the formation of each of the following intermetallic phases is possible: FeAl, FeAl₂, Fe₂Al₅, FeAl₃ (or Fe₄Al₁₃). A literature review was performed to determine which intermetallic compounds were most probable under these conditions. Kajihara prepared a diffusion couple of Al/Fe/Al via a diffusion bonding technique and anneals at 823 and 913 K. At both temperature only Fe₂Al₅ forms, indicating it has a higher rate of formation than FeAl, FeAl₂, and FeAl₃ at these temperatures [120]. Maitra and Gupta report on the formation of Fe₂Al₅, FeAl and Fe₃Al between a diffusion couple of pure Fe and a high purity Al-Si eutectic alloy annealed at temperatures ranging

from 600-900 °C [121]. Kobayashi reports on hot dipping steel into a molten aluminum bath for 300s for temperatures ranging from 973-1273K and observing FeAl, Fe₂Al₅ FeAl₃ at the interface layer [122]. It is obvious all four intermetallic phases can form at processing temperatures associated with the fabrication of these macro composite materials systems so multiple characterization methods were sought to accurately determine which intermetallic compounds have formed.

Figure 42. Iron-Aluminum Phase diagram illustrating steel/MMC encapsulation processing temperature range [123].

5.3 Energy Dispersive Spectroscopy

EDS is a technique which allows for the determination of a materials chemical composition. High energy electrons from the beam in an SEM bombard the target material which then emits characteristic X-rays produced by each element within the

material [124]. The EDS software analyzes the energy spectrum and determines the abundance of each of the specific elements.

Backscattered electron imaging was the first step for identifying an additional phase present at the steel/MMC interface based on a contrast difference. EDS was then used as a means of identifying which elements were present within that phase. Table 8 presents the material chemistries for the steels and matrix alloys. Evident from the figure are high concentrations of Chromium and Nickel in both of the stainless steels. The AZ91E has approximately 9 weight percent aluminum and the Al-10Si alloy has nearly 10 weight percent silicon. These alloying elements come into play when the interfacial region is characterized. Interaction between these species occurs as the atoms interdiffuse during and after processing. EDS is used to qualitatively determine which elements are present in select areas on the samples. The focus here is on the interface between the steel shell and MMC core.

Table 8. Alloy chemistries within the macro composite systems.

Element	A36	304SS	Nitronic®50	MgAZ91E	Al-4Mg	Al-10Si
C	0.25-0.29	0.08 max	0.06 max	-	-	-
Cu	0.20 max	-	-	0.003	-	0.2
Cr	-	18.0-20.0	20.5-23.5	-	-	-
Mn	1.03 max	2.00 max	4.0-6.0	0.22	-	0.1
Mo	-	-	1.5-3.0	-	-	-
Ni	-	8.0-12.0	11.5-13.5	-	-	-
N	-	0.1 max	0.3 max	-	-	-
P	0.04 max	0.045 max	-	-	-	-
Si	0.28 max	0.75 max	1.00 max	0.035	-	8.5-9.5
S	0.05 max	0.03 max	-	-	-	-
Ti	-	-	-	-	-	0.2
V	-	-	0.10-0.30	-	-	-
Zn	-	-	-	0.63	-	0.05
Al	-	-	-	8.25	Balance	Balance
Fe	Balance	Balance	Balance	0.014	-	0.2
Mg	-	-	-	Balance	4	0.45-0.60

Sources: Sources: M Cubed Technologies (MCT) [81], directly from the manufacturer's websites or datasheets [125], or MatWeb [126].

Figure 41 presents EDS phase maps of the A36/Al-Al₂O₃ 46P system. On the left hand side of the figure is the steel material (light gray), in the middle of figure is the reaction zone (medium gray), and on the right side is the MMC (dark gray with white particulates).

Figure 43. EDS phase map of A36/Al-Al₂O₃ 46P system.

Consistent with the low alloy nature of the A36 steel and Al-4Mg matrix alloy, only iron and aluminum appear to be present in the reaction layer. Oxygen also appears to be present throughout with a high concentration in the reinforcement particles as expected of alumina (Al₂O₃). None of the trace elements were in significant concentration thus do not appear in the elemental list. Figure 44 presents the EDS maps of the Nitronic/Mg-B₄C 45P system.

Figure 44. EDS maps of the Nitronic/Mg-B₄C 45P system (a) 500X magnification (b) 1500X magnification.

There appears to be a uniform band of aluminum present in the reaction layer between the Nitronic steel and MgAZ91 matrix. This aluminum is diffusing out of the matrix material and reacting with the steel. The EDS maps indicate there is a high concentration of iron in the reaction layer but also nickel and chrome are observed. These elements are present in the steel and react with the aluminum contained within the Mg AZ91E at elevated temperatures. Formation of ternary, FeNiAl₉, and numerous binary compounds, CrAl₄, Cr₄Al₉, NiAl₃, and Ni₂Al₃ etc. are possible at processing temperatures [127].

5.4 X-ray Phase Identification

XRD was performed on a Bruker D8 X-ray diffraction system in conjunction with the DIFFRAC.SUITE software. The target X-ray material was Copper with a 1.5418 angstrom wavelength operating at 40 kV and 40mA. The monochromatic Cu $K_{\alpha 1}$ line is isolated by the Vario monochromator at the X-Ray tube. A LynxEye position sensitive detector is included which permits up to $4^{\circ} 2\theta$ of diffracted beam to be measured continuously while scanning. A continuous 2θ scan mode was applied from 20 - $120^{\circ} 2\theta$ with an increment of 0.05° .

XRD was performed on the interface between each of the hybridized materials systems. Each system consists of a unique combination of steel and MMC resulting in considerably different reaction layers based on the interaction of the constituents. Table 8 shows the elemental composition of the steels and MMC matrix alloys used in this work, indicating significant amounts of alloying elements are present, specifically Chromium, Nickel, and Manganese, within the stainless steels.

Due to the variation in the reaction layer thicknesses, XRD was only able to index intermetallic phases in the hybridized materials systems which exhibit a sufficiently thick reaction layer. Figures 45a and 45c provide schematics of the measurement orientations and the resulting intermetallic layers imaged via SEM are shown in 45b and 45d respectively. In some cases transverse sectioning was not ideal for optimizing the thickness of the intermetallic layer for measurement. In these cases, axial cuts were made through the steel just outside the interface of the MMC. Subsequently the steel material would be polished away in order to increase the

exposed area of intermetallic being scanned compared with the surrounding materials. This helped to maximize the area of intermetallic which would be exposed to the X-ray beam. However, some of the adjacent material was also bombarded with X-rays and thus their peaks are present in the scans.

Figure 45. (a) Transverse cross-section X-ray configuration and (b) resulting intermetallic layers. (c) Axial cross section X-ray configuration and (d) resulting intermetallic layer.

Figures 46 and 47 show SEM micrographs of the reaction layers measured and their corresponding X-ray diffractograms (figure 48 and figure 49 respectively).

Figure 46. SEM micrograph of A36/Al-Al₂O₃ interface.

Figure 47. SEM micrograph of Nitronic/Al-SiC interface.

Figure 48. X-ray diffractogram A36/Al-Al₂O₃ interface [117].

Figure 49. X-ray diffractogram of Nitronic/Al-SiC interface [117].

Evident from the diffractograms, two intermetallic phases form at the A36/Al-Al₂O₃ interface, namely Fe₄Al₁₃ and Fe₂Al₅. Some minor peaks also identify Al₂O₃ which is consistent with the reinforcement particles in the MMC. Only one intermetallic phase, Fe₂Al₅, forms in the Nitronic/Al-SiC system. The structure of the aluminum rich phase Fe₄Al₁₃, as determined by X-ray diffraction is monoclinic, space

group B2/m and with lattice parameters $a = 1.5489$ nm, $b = 0.8083$ nm, $c = 1.2476$ nm.

The Fe_2Al_5 is orthorhombic, with lattice parameters $a = 0.7675$ nm, $b = 0.64030$ nm, $c = 0.4203$ nm. The identified Fe-Al intermetallics formed, $\text{Fe}_4\text{Al}_{13}$ and Fe_2Al_5 , are consistent with previous literature where Al rich phases are typically observed below processing temperatures of 1000°C and Fe rich phases above [7], [128].

5.5 Electron Backscatter Diffraction (EBSD)

EBSD is an advanced characterization technique used in conjunction with an SEM. EBSD works by diffracting an electron beam off of a crystalline material and collecting the resulting Kikuchi pattern on a phosphor screen. From the Kikuchi patterns, consisting of specifically oriented and shaped bands, detailed information about the crystalline structure can be obtained. For instance, the bands correspond to specific planes in the material and the angles between them relate to the angles between the planes. In addition, the observed intensity correlates to the type of atom and the width of the bands correlates to the spacing of atoms [129]. Figure 50 presents (a) raw and (b) indexed Kikuchi patterns for SiC.

Figure 50. Kikuchi pattern of 4H SiC a) raw b) indexed Kikuchi pattern.

Special consideration of the sample configuration within the SEM is worth noting. As the electron beam comes down the vertical column and the phosphor screen is typically placed in a horizontal configuration, a near 90° angle exists between the two. To ensure the maximum amount of electrons contact the screen following diffraction, the sample is typically tilted to 70° from the horizontal.

Following proper sample preparation and positioning in the specimen chamber, EBSD has many applications. A properly indexed Kikuchi pattern allows for orientation imaging of the sample, measuring variation in grain orientation down to less than 0.25° [129]. It can identify the different phases present in the sample including the different crystal structures that exist in each of the seven main crystal systems. Furthermore it can be used for texture analysis, to determine residual strain within deformed materials, characterize special boundaries such as twins and grain boundary misorientation among other applications [130].

As two intermetallic phases were identified within the A36/Al-Al₂O₃ system, EBSD was utilized to determine the approximate location and distribution of phases

within the interfacial region. Figure 51(a) shows an SEM micrograph of the interface observed in the A36/Al- Al_2O_3 system and figure 51(b) the corresponding EBSD phase map.

Figure 51. Interface of A36/Al- Al_2O_3 (a) SEM micrograph (b) EBSD phase map[117].

The red color on the left represents ferrite, the green section Fe_2Al_5 , the yellow section $\text{Fe}_4\text{Al}_{13}$, and a small blue area on the right the alumina (Al_2O_3) reinforcement. Consistent with the initial interface and concentration gradients, Fe_2Al_5 has a higher

percentage of iron than $\text{Fe}_4\text{Al}_{13}$ and thus is closer to the ferrite side of the figure (left). Kobayashi, talking about diffusion bonding and hot dip coating in an Fe/Al system, indicates a FeAl_3 layer is initially formed at the interface between Fe and Al by iron diffusion into the aluminum layer followed by a Fe_2Al_5 layer at the interface between the FeAl_3 and steel [122]. As the concentration gradient changes at the interface the ratio between iron and aluminum varies. Intermetallic phases form based on this ratio according to the phase diagram presented in figure 42. Differentiated intermetallic layers were only observed in the A36/Al- Al_2O_3 system as the other materials only exhibit one intermetallic phase (Fe_2Al_5) rather than the two observed here. Akdeniz *et al.* observed a similar columnar structure and formation of Fe_2Al_5 and FeAl_3 at the interface between a low carbon steel and pure Al processed at temperatures between 750 and 850 °C [131]. Similarly Bouché *et al.* reported on the formation of Fe_2Al_5 and FeAl_3 with similar morphology in a commercially pure solid Fe liquid Al couple at temperatures between 700 and 900 °C [132]. The author continues to report initial intermetallic formation occurs via an interface reaction then growth takes place without significant dissolution in the liquid. Once the thickness is large enough the two phases grow via diffusion and their dissolution can be observed [132].

5.6 Interdiffusion

Diffusion is the motion or transport of matter: gas, liquid, or solid, from one point to another point via the thermal motion of atoms or molecules. Diffusion in solid materials is prevalent in science and engineering for many processes including

phase transformations, dissolution and precipitation of second phases within a material, homogenization of alloys, high temperature creep, and heat treatments etc. [133]. Diffusion is governed by Fick's second law, the non-steady state diffusion equation (19):

$$\frac{\delta C}{\delta t} = D \left(\frac{\delta^2 C}{\delta x^2} + \frac{\delta^2 C}{\delta y^2} + \frac{\delta^2 C}{\delta z^2} \right) \quad (19)$$

where C is the concentration (mol/m³), t is time (s), D the diffusion coefficient (m²/s), and x, y, z the lengths (m) in a Cartesian coordinate system.

Multidirectional diffusion between two species of a binary A-B system is known as interdiffusion. The binary system considered for this research is the Iron-Aluminum pair. Diffusion of the substitutional solutes in this case occurs via the vacancy rather than interstitial mechanism based on a comparable size of iron and aluminum atoms. An atom is able to move through the lattice by making a series of jumps into nearby vacancies. Let us consider the simplified situation of one dimensional diffusion between the two materials. Figure 52(a) illustrates the initial condition between iron and aluminum exhibiting a lattice of each atom with a linear interface between the materials.

Figure 52. Binary diffusion system of iron and aluminum (a) Initial state at time $t = 0$ (b) Final state after some time, t .

After some time both species will diffuse at a rate proportional to their intrinsic diffusion coefficients, D_a and D_b respectively. The diffusion of both species results in the movement of both iron and aluminum atoms and the creation of a heterogeneous region between the two shown in figure 52(b). Since the diffusion rates are not the same for each species, iron and aluminum, there is an apparent net mass flow in the direction of the higher diffusion rate. This results in shrinking of the diffusion couple on one side and swelling on the other, an observation called the Kirkendall effect [133]. This phenomenon was originally observed in 1947 on the behavior between Zinc and alpha-Brass and coined by Ernest Kirkendall [134]. Typically the Kirkendall effect is observed by placing some form of inert marker at the initial interface of a diffusion couple. Throughout the interdiffusion experiment, both components are allowed to diffuse whereas the inert markers will remain in place. In the case of the steel/MMC interface the reinforcement particles behave like the inert markers. They are evenly distributed throughout the matrix, to include the steel/MMC interface, and they are too large to diffuse thus remain in their initial position.

Considering the special case of one-dimensional diffusion, equation (19) from above reduces to equation (20):

$$\frac{\delta C}{\delta t} = D \left(\frac{\delta^2 C}{\delta x^2} \right) \quad (20)$$

Solving for the concentration as a function of length and time (x and t respectively) yields equation (21):

$$C(x, t) = C_0 \operatorname{erfc} \left(\frac{x}{2\sqrt{Dt}} \right) \quad (21)$$

where C_0 is the initial surface concentration and erfc is the complimentary error function. From the denominator in equation (21) is a term called the bulk diffusion length ($\sqrt{Dt}$) and represents the characteristic distance an atom or molecule travels in the x direction after a time t [133].

By extrapolating measured data points for the impurity diffusion of aluminum in α (ferrite) and γ iron (austenite) from Bergner and Akimova respectively, diffusion coefficients for the range of processing temperatures can be obtained [135]. Similarly, the diffusion coefficient of iron in aluminum is calculated utilizing an Arrhenius equation of impurity diffusion in Al [133]. The results of these extrapolations are plotted in figure 53 below along with the maximum fabrication temperature range outlined in yellow.

Figure 53. Diffusion coefficient vs. Temperature for each binary diffusion couple.

Evident from figure 53, three binary diffusion couples exist and exhibit a range of diffusion coefficients. Iron into aluminum has the highest value at all temperatures, followed by aluminum into γ -iron (austenite) and lastly aluminum into α -iron (ferrite). Utilizing the bulk diffusion length from equation (21) in conjunction with the fabrication time/temperature profile, approximate reaction layer thicknesses can be predicted for each of the macro composite materials systems. The majority of the reaction layer forms during the interaction between the solid steel shell (held at processing temperature) and the molten MMC. In liquids, the rate of diffusion is on the order of fractions of millimeters per second. In solids, at temperatures near their melting points, the diffusion rate is on the order of one micrometer per second and quickly drops to nanometers per second as the temperature reaches half of the melting point [133]. Table 9 presents the expected reaction layer thicknesses for each system

which result from the solid state interdiffusion portion of the reaction and includes the average reaction layer thickness measured via SEM for comparison.

Table 9. Predicted vs. measured reaction layer thicknesses for each macro composite materials system.

Composite system	Steel diameter	Predicted Reaction Layer Thickness (μm)		Measured Reaction Layer Thickness (μm)	CTE Mismatch
		Fe $\rightarrow$ Al	Al $\rightarrow$ Fe		
Nitronic/Al-SiC-55P	0.3	46	3	136	4.3
	0.5	46	3	235	
Nitronic/Mg-B ₄ C-45P	0.3	5	0.3	10	-0.4
	0.5	5	0.3	10	
304SS/Al-Al ₂ O ₃ -55P	0.3	46	3	136	5.4
	0.5	46	3	170	
A36/Al-Al ₂ O ₃ -55P	0.3	46	1	192	0.5
	0.5	46	1	251	
304SS/Al-SiC-55P	0.3	46	3	123	4.8
	0.5	46	3	156	
A36/Al-SiC-55P	0.3	46	1	0	-0.1
	0.5	46	1	18.8	
304SS/Al-SiC-30P	0.3	0	0	44.5	1
	0.5	0	0	0	
A36/Al-SiC-30P	0.3	0	0	3.4	-3.9
	0.5	0	0	0	

As the diffusion couples have different rates of diffusion into one another, the contributions are listed separately. The reaction layer thickness will result from some sort of mixture of the two, but not necessarily a sum of the two lengths. Three of the systems with the lowest measured reaction layers, A36/Al-SiC-30P, 304SS/Al-SiC-30P, and Nitronic/Mg-B₄C-45P also have three of the thinnest predicted thicknesses. This is likely due to several factors during fabrication. First, the 30P samples are allowed to cool immediately after the MMC is cast into the steel cavity. Also the rate

of diffusion drops quickly with decreasing temperature so the cooling portion has little effect on the diffusion length. Finally, these samples have low or negative values for CTE mismatch resulting in limited residual stress. Furthermore, for the Nitronic/Mg-B₄C-45P system, as magnesium does not react with iron, the aluminum from the matrix is diffusing to the interface and reacting with iron. As aluminum is only an alloying element, with a significantly lower concentration (9 wt%), a substantially thinner reaction layer is observed even with the extended hold times associated with the 45P process. In the cases where the measured reaction layer thicknesses were substantially higher than the predicted results, specifically Nitronic/Al-SiC-55P, 304SS/Al-Al₂O₃-55P, and A36/Al-Al₂O₃-55P the CTE mismatch in each of those systems is larger than 4. The high magnitude residual stresses resulting from the high CTE mismatch is likely to support diffusion by maintaining intimate contact between the surfaces. Another factor affecting the diffusivity is the hydrostatic pressure. Equation (25) and the relationship between the diffusion coefficient and hydrostatic pressure are deduced following equations, (22), (23), and (24).

$$\Delta G = \Delta H - T\Delta S = \Delta E - T\Delta S + \rho\Delta V \quad (22)$$

where G is Gibbs free energy, H is activation enthalpy of diffusion, S is entropy, T is temperature, E is internal energy, ρ is hydrostatic pressure, and V is activation volume. The activation enthalpy can be isolated as follows:

$$\Delta H = \Delta E + \rho\Delta V \quad (23)$$

Plugging the activation enthalpy term into the Arrhenius relation for diffusion in solids (24) allows for a relationship to be seen between the diffusion coefficient and the enthalpy term.

$$D = D^0 \exp\left(-\frac{\Delta H}{k_B T}\right) \quad (24)$$

where D is the diffusion coefficient, D^0 is the pre-exponential or frequency factor, and k_B is the Boltzmann constant.

$$D = D^0 \exp\left(-\frac{\Delta E + \rho \Delta V}{k_B T}\right) \quad (25)$$

It is shown that diffusivity tends to decrease as the pressure increases. The term $\rho \Delta V$ can be almost negligible at ambient temperature in solids but can become significant at high pressures [133]. Werner and Mehrer showed the diffusivity of radiotracer ^{198}Au in Au single crystals to reduce to half under a pressure of 600 MPa and a constant temperature of 800K [136]. Unfortunately diffusion studies on aluminides are limited due to the lack of suitable radiotracers for Al. The radiotracer method is the standard technique and a direct method for measuring self and solute diffusion. It can cover a range of diffusivities, is element selective and is highly sensitive due to the use of nuclear counting facilities [133]. In addition, secondary ion mass spectrometry, Auger electron spectroscopy, and electron microprobe analysis are profiling techniques for diffusion studies.

Although the exact effect the residual stress has on the diffusivities of each diffusion couple is unknown some qualitative observations can be made. First, the cores of the macro composite systems are put into compression. Thus a pressure is exerted on the MMC inhibiting its diffusion which may result in decreased aluminum diffusivity into steel. On the other hand, neutron diffraction confirms no significant compression is observed in the steel phase so it is assumed no restrictions would be placed on the diffusivity of the steel as a result of pressure. However, as diffusivity is strongly temperature dependent, it is not known how much of an effect the pressure will have since residual stresses start forming at temperatures below processing conditions. At these decreased temperatures the rates of diffusion may have fallen below any sort of meaningful level, thus eliminating any contribution resulting from a residual stress.

5.7 Conclusions

1. The thickness and morphology of the intermetallic reaction layers varied drastically between each macro composite materials systems. The A36/Al-SiC 30P system exhibits a thin discontinuous reaction layer with maximum thickness of about $3.5\mu\text{m}$ whereas the A36/Al- Al_2O_3 exhibits a dendritic like reaction layer with an average thickness of approximately $250\mu\text{m}$.
2. XRD analysis and microstructural characterization suggest Fe_2Al_5 and $\text{Fe}_4\text{Al}_{13}$ are present in the reaction layer in the A36/Al- Al_2O_3 system. Conversely only Fe_2Al_5 is observed in the Nitronic/Al-SiC and 304/Al-SiC systems.

3. EDS and EBSD confirm the A36/Al-Al₂O₃ interface exhibits a two phase intermetallic region consisting of Fe₂Al₅ and FeAl₃ with a relatively smooth boundary layer between the two phases.
4. Three primary diffusion couples were evaluated based on the intermetallic phases indexed via XRD, namely Al- α Fe, Al- γ Fe, and Fe-Al. Based on the diffusion coefficients of these three couples over the temperature range associated with the processing conditions, the diffusion of iron into aluminum seems to dominate.
5. Significantly larger reaction layers were measured for each of the macro composite materials systems as compared to the lengths predicted with the bulk diffusion length term.

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Appendix A

X-RAY SCANS OF TENSILE SPECIMENS

Figure A1. A36/Al-Al₂O₃ 55P macro composite materials systems.

Figure A2. A36/Al-SiC 30P macro composite materials systems.

Figure A3. A36/Al-SiC 55P macro composite materials systems.

Figure A1. 304SS/Al-SiC 55P macro composite materials systems.

Figure A5. (Samples1-3) Nitronic/MgB₄C 45P (Samples4-6) Nitronic/Al-SiC 55P macro composite materials systems.

Appendix B

**STEEL/MMC INTERFACE OF MACRO COMPOSITE MATERIALS
SYSTEMS**

Figure B1. SEM micrograph of steel/MMC interface of A36/Al-SiC 30P macro composite materials system.

Figure B2. SEM micrograph of steel/MMC interface of A36/Al-SiC 55P composite materials system.

Figure B3. SEM micrograph of steel/MMC interface of 304/Al-SiC 30P composite materials system.

Figure B4. SEM micrograph of steel/MMC interface of 304/Al-Al₂O₃ 46P macro composite materials system.

Appendix C

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