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UNIVERSITY OF CALIFORNIA, SAN DIEGO

Aluminum-Tungsten Fiber Composites
With Cylindrical Geometry and
Controlled Architecture of Tungsten Reinforcement

A Thesis submitted in partial satisfaction of the requirements
for the degree Master of Science

in

Engineering Sciences (Mechanical Engineering)

by

Carl Joseph Lucchese

Committee in charge:

Professor Vitali Nesterenko, Chair

Professor Marc Meyers

Professor David Benson

2010

The Thesis of Carl Joseph Lucchese is approved and it is acceptable in quality and form for publication on microfilm and electronically:

Chair

University of California, San Diego

2010

Dedication

I would like to dedicate this work to my mother, Alice Paula Lucchese and father, Carl Joesph Lucchese Sr. for showing me the world and always encouraging me to do my best. It is to their patience, guidance, courage, and wisdom that I have come to reach this mile stone in my engineering career. Do or do not, there is no try.

Table of Contents

Signature Page.....	iii
Dedication.....	iv
Table of Contents.....	v
List of Abbreviations.....	viii
List of Symbols.....	ix
List of Diagrams.....	xi
List of Figures.....	xiii
List of Tables.....	xx
Acknowledgements.....	xxi
Abstract.....	xxii
Introduction.....	1
Section 1 – State of the Art of Metal Matrix Composites.....	3
1.1 – Current Application of Metal Matrix Composites.....	3
1.2 – Design Considerations and Liquid Phase Processing.....	5
1.3 – Solid Phase Processing and Powder Metallurgy.....	7
1.4 – Non-Powder Solid State Processing.....	9
Section 2 – Research Goals.....	12
2.1 – A Brief Review of the Combustion of Aluminum Particles.....	12
2.2 – DARPA Goals.....	16
2.3 – Why a Metal Matrix Composite as an Energetic Material?	18
Section 3 – Research Findings & Development.....	20

3.1 – The Concept and Configurations.....	20
3.2 – Construction of the Samples	24
3.3 – Planar Studies and Configuration 1.....	33
3.4 – Second Sample of Configuration 1.....	45
3.5 – Configuration 2 and the Prevention of “Brazilian” Fracture.....	53
3.6 – Annealing of the Aluminum.....	62
3.7 – Samples 3 & 4 of Configuration 1.....	64
3.8 – Samples 5 & 6 of Configuration 1 and Intermetallic Formation...	75
3.9 – Improved HIP Treatment of 200 um W Reinforcement.....	87
3.10 – HIP Results for Configuration 3.....	91
3.11 – Internal Pressurized Configuration 4 to Prevent Buckling of W Reinforcement.....	106
3.12 – Mechanical Testing Setup.....	112
3.13 – Tensile Test Validation	118
3.14 – Mechanical Strength of Samples of Configuration 1.....	121
3.15 – Mechanical Strength and Critical Strain of Configuration 3.....	127
Section 4 – Conclusions.....	137
Section 5 – Summary of Contributions.....	139
Section 6 – Future Research.....	140
Appendices.....	143
Appendix A - Tungsten Fiber Spacing.....	143
Appendix B – Sample Dimensions, Mass, and Densities.....	146
Appendix C – Summary of HIP Treatments.....	147

Appendix D – Finite Element Modeling.....	160
Appendix E – Compression Calculation	171
Appendix F – SEM Analysis of Sample 9.....	178
References.....	181

List of Abbreviations

Aluminum.....	Al
Aluminum Alloy 6061-T6.....	Al 6061-T6
Aluminum-Tungsten.....	Al-W
Brinell Hardness.....	HB
Brittle to Ductile Transistion Temperature.....	BDTT
Cold Isostatic Pressing.....	CIP
Defense Advanced Research Projects Agency.....	DARPA
Electro-Discharge Machine.....	EDM
Face Centered Cubic.....	FCC
Finite Element Method.....	FEM
Grams per Cubic Centimeter.....	g/cc
Hot Isostatic Pressing.....	HIP
Vickers Hardness.....	HV
Plastic Bonded Explosive.....	PBX
Polytetraflouroethylene.....	PTFE
Scanning Electron Microscope.....	SEM
Trinitrotoluene.....	TNT
Tungsten.....	W

List of Symbols

Change in Gibb's Free Energy at Standard Temperature and Pressure.....	ΔG^o
Change in Entropy at Standard Temperature and Pressure in a Chemical Reaction.....	ΔS_{rxn}^o
Change in Enthalpy at Standard Temperature and Pressure in a Chemical Reaction.....	ΔH_{rxn}^o
Area.....	A
Coefficient of Thermal Expansion.....	α
Radius.....	b
Plastic Boundary.....	c
Change in Length.....	δ
Young's Modulus of Elasticity.....	E
Strain.....	ε
Chemical Compound Constant.....	k_o
Original Length.....	L
Arbitrary Force.....	P
Interfacial Pressure.....	P
Airy Stress Function.....	ϕ
Activation Energy.....	Q_d
Gas Constant or Radius.....	R
Radius.....	r
Stress.....	σ
Temperature.....	T

Time.....	t
Shear Stress.....	τ
Poisson's Ration.....	ν
Thickness of Intermetallic Compounds.....	x
Tensile Yield Stress.....	Y

List of Diagrams

Diagram 1 – Sample prepared for CIP.....	35
Diagram 2 - HIP process for Sample 1 of Configuration 1.....	42
Diagram 3 - Successful HIP process for sample 2, Configuration 1.....	51
Diagram 4 – HIP process for Sample 1 of Configuration 2.....	58
Diagram 5 - HIP process for Samples 3 and 4 in Configuration 1.....	66
Diagram 6 - HIP process for Sample 6, Configuration 1 with thicker Al 6061-T6 inner tube and higher temperature.....	77
Diagram 7 - Improved HIP treatment applied to Configuration 3 samples.....	89
Diagram 8 - HIP treatment for the first pair of W layers for Samples 9-13, Configuration 3...	90
Diagram 9 - Cross section view of the assembly for Configuration 4 inside the rigid stainless steel tube for internally applied HIP pressure.....	109
Diagram 10 - Cross section view of the assembly for Configuration 4 inside the Al 6061-T6 jacket.....	109
Diagram 11 - Cross section of the assembly of Configuration 4 with the additional degassing tube for vacuum sealing.....	110
Diagram 12 - Sketch of Al-W composite specimen for tensile testing. The outer diameter of the gauge section in sample 5 was reduced to 9.16 mm to expose the W wire.....	117
Diagram 13 - Dimensions of specimen's steel clamp for tensile testing.....	118
Diagram 14 – Strain strain curve of the elastic range for 1018 steel samples. A strain gauge was used to measure strain.....	119
Diagram 15 – Strain strain curve of the elastic range for 1018 steel samples with 0.02% offset curve.....	120
Diagram 16 – Strain strain curve of the 1018 steel sample.....	120
Diagram 17 - Tensile test result for Samples 5 and 6, Configuration 1.....	122
Diagram 18 – Stress strain curve for Samples 11 and 13, Configuration 3.....	130

Diagram 19 – Stress strain curve for Samples 11 and 13, Configuration 3 with a theoretical modulus line represented in green.....	136
Diagram 20 – HIP 1: HIP process for Sample 1 of Configuration 1.....	147
Diagram 21 – HIP 2: Successful HIP process for sample 2, Configuration 1.....	148
Diagram 22 – HIP 3: Sample 1, Configuration 2 HIP process.....	149
Diagram 23 – HIP 4: HIP process for Samples 3 and 4 in Configuration 1.....	150
Diagram 24 – HIP 5: HIP process for Sample 6, Configuration 1 with thicker Al 6061-T6 inner tube. Sample 5, Configuration 1 was also present during this process but lost its vacuum seal.....	151
Diagram 25 – HIP 6: HIP process applied to Sample 5, Configuration 1 and Sample 7, Configuration 3. Sample 5 was successfully completed while Sample 7 only had two of the four W layers applied.....	152
Diagram 26 – HIP 7: HIP process applied to Sample 7, Configuration 3 to secure the final pair of W fiber layers.....	153
Diagram 27 – HIP 8: HIP process applied to Sample 8, Configuration 4.....	154
Diagram 28 – HIP 9: HIP process unsuccessfully applied to Sample 9, Configuration 3 due to vacuum seal failure.....	155
Diagram 29 – HIP 10: HIP process applied to Sample 10, Configuration 3 to secure the first pair of W fiber layers.....	156
Diagram 30 – HIP 11: HIP process applied to Samples 9, 11, 12 and 13, Configuration 3 to secure the first pair of W fiber layers.....	157
Diagram 31 – HIP 12: HIP process applied to Samples 9-13, Configuration 3 to secure the final pair of W fiber layers. The vacuum seal on Sample 12 failed.....	158
Diagram 32 – HIP 13: HIP process applied to Samples 9 and 12, Configuration 3. Sample 12 had the final two layers of W fibers applied. Sample 9 was HIP treated again to see if the interface gaps between the aluminum tubes that remained after the first successful HIP cycle could be closed in a densified sample.....	159
Diagram 33 - Stress vs Time from the course tensile model for several elements along the fracture plane.....	170

List of Figures

Figure 1 – Close up of plastic guide for assembling axial rods.....	25
Figure 2 – Mount for constructing plastic guides. Notice black plastic guide in center.....	25
Figure 3 – Mount for drilling plastic guide. Cover removed to show guide in center.....	26
Figure 4 – The sample after the second layer of W wire was applied.....	27
Figure 5 – Tungsten wire dispenser. Spring acts as clutch mechanism to maintain tension of W wire.....	27
Figure 6 – Tungsten wire dispenser. A guiding notch is seen as the thin groove cut in brass colored threaded bolt.....	28
Figure 7 – Al guide for third layer of W, second layer of W rods in axial direction.....	29
Figure 8 – Third layer of W rods applied. Notice remaining glue to keep rods and wire in place and Al guide disk. This layer was removed and redone with the forth layer of W wire.....	30
Figure 9 – Final assembly of sample with four layers of W wire, two Al tubes, and a steel core.....	30
Figure 10 - Sample 7, Configuration 3. Pre-HIP assembly.....	32
Figure 11 - Planar sample of a sheet of Al 6061-T6 being compressed into a W mesh (100, 0.0509 mm in diameter) in a CIP process at 345 MPa. Notice the deep penetration of W wires into the aluminum with some plastic deformation of the top of the W wires.....	34
Figure 12 – Cross sectional view of sample 1, Configuration 1 after EDM cut.....	36
Figure 13 – Sample 1, Configuration 1 demonstrate fracture and cracking of W wire in contact with W rods.....	37
Figure 14 – Axial W rods are seen coming out of the page, they press against the inner layer of Al 6061-T6 during a CIP process in Sample 1, Configuration 1.....	38
Figure 15 – A cross section with the hoop wires coming out of the page showing severe fracturing of radial wires with little deformation imparted to the axial rods.....	38
Figure 16 – A cross section with the hoop W wires coming out of the page showing the “Brazilian” fracture of hoop wire.....	39

Figure 17 – Sample 1, Configuration 1 after annealing and a second CIP process at 345 MPa. Notice the decreased distance between the axial W layers due to the fracture of the separating W wires.....	40
Figure 18 – Sample 1, Configuration 1 in a vacuum-sealed jacket of Al 6061-T6 for a HIP treatment.....	41
Figure 19 – Al 6061-T6 Jacket of Sample 1, Configuration 1 after the HIP treatment.....	42
Figure 20 – A cross section of Sample 1, Configuration 1 after a HIP process with the axial rods coming out of the page. The compact W structure probably prevents flow of the aluminum during HIP, creating a void.....	43
Figure 21 - A cross section of Sample 1, Configuration 1. Notice the voids between the layers of aluminum.....	44
Figure 22 – SEM image of a cross section of Sample 1, Configuration 1.....	44
Figure 23 - Sample 2 after a CIP process at 50 MPa. No plastic deformation or “Brazilian” fracture is observed in the contact region between W fibers.....	46
Figure 24 - Failed piece of Sample 2, Configuration 1 after 40 kHz sonication in acetone for 20 minutes.....	47
Figure 25 - Piece of Sample 2, Configuration 1 to be HIP treated after sonication in acetone for 20 minutes at 40 kHz.....	48
Figure 26 - Sample 2, Configuration 1 prepared inside of a Al 6061-T6 jacket for the failed HIP cycle. Notice the narrow vacuum tube and the geometrical discontinuity to the jacket cap that lead to the failure of the vacuum seal. The wall of the tube was pushed down into the large aluminum cap, leading to fracture.....	50
Figure 27 - Sample 2, Configuration 1. Notice full penetration of Al around the W rods and wires with no Al-W compound created.....	51
Figure 28 - Sample 2, Configuration 1. “Brazilian” fracture of W rods with aluminum filling the cracks in W observed after the HIP process.....	52
Figure 29 - Sample 2, Configuration 1. Buckling was observed in plane perpendicular to axis and in axial plane.....	52
Figure 30 - Aluminum alloy 6061 tubes used to assemble Sample 1, Configuration 2. The inner and outer diameters of the tubes from the smallest to largest are: 7.5 mm and 8.1 mm, 8.6 mm and 8.88 mm, 10.08 mm and 10.36 mm, 10.84 and 11.12 mm, and 11.6 and 12.2 mm.....	57
Figure 31 - Fully assembled Sample 1, Configuration 2 before vacuum encapsulation.....	57
Figure 32 - Sample 1, Configuration 2 prepared within an Al 6061-T6 vacuum-sealed jacket for a HIP process.....	58

Figure 33 - Sample 1, Configuration 2 after the HIP process. Notice the bump along the entire length of the sample.....	59
Figure 34 - Cross section of Sample 1, Configuration 1 after the HIP process. Notice the irregular W rod spacing and extreme W structure deformation at the top of the sample.....	60
Figure 35 - Sample 1, Configuration 2, optical image. Notice the aluminum fully penetrated the W structure but the individual layers of aluminum did not bond, leaving behind local voids.....	60
Figure 36 - Sample 1, Configuration 2, optical image. Notice the non-uniform spacing between the W rods.....	61
Figure 37 – The buckling region in Sample 1, Configuration 2, optical image.....	62
Figure 38 - Overall cross-section of Sample 3, Configuration 1. Notice buckled region at the bottom of the sample.....	67
Figure 39 - Overall cross-section of Sample 4, Configuration 1 with a thicker inner tube. Notice buckled region at the bottom of the sample.....	68
Figure 40 – Sample 3, Configuration 1, optical image. Notice the regularly spaced W rods and full penetration of aluminum inside the W structure.....	68
Figure 41 - SEM picture of Sample 4, Configuration 1 with a thicker inner tube to show full penetration of aluminum into the W structure.....	69
Figure 42 - Sample 4, Configuration 1 with a thicker inner tube, optical image to show the regularly spaced W rods.....	69
Figure 43 - SEM image of Sample 4, Configuration 1 to show the regularly spaced W rods...	70
Figure 44 - SEM image of Sample 3, Configuration 1. Aluminum particles are highlighted in red and Tungsten particles are highlighted in green. No diffusion of the elements resulting in Al-W compound is noticeable.....	71
Figure 45 - Sample 4, Configuration 1 with a thicker inner tube, optical image. Notice the contact welding between the W wire and rods.....	72
Figure 46 - Buckled region in Sample 3, Configuration 1, optical image.....	73
Figure 47 - SEM image of buckled region of Sample 3, Configuration 1.....	73
Figure 48 – Piece of Sample 3, Configuration 1 with an outer diameter of 9.13 mm to expose the outer buckled layer of W wire in the axial plane.....	74
Figure 49 – Piece of Sample 3, Configuration 1 with an outer diameter of 9.13 mm in the non buckled region to show the regularly spaced W wire.....	74

Figure 50 - Sample 5, Configuration 1, optical image after the first failed HIP process. The “Brazilian” fracture seen is probably due to the mechanical clamping during the assembly process.....	79
Figure 51 - Sample 6, Configuration 1, optical image. Notice the full penetration of the aluminum into the preserved W structure.....	80
Figure 52 – Buckled region observed in the lower region of Sample 6, Configuration 1, optical image. The W wires layers have buckled in the axial plane into the empty space.....	80
Figure 53 – Buckled region observed in the higher region of Sample 6, Configuration 1 under an optical microscope. The W wires layers have buckled in the axial direction into empty space.....	81
Figure 54 - A region of Sample 6, Configuration 1 with a thicker Al 6061-T6 inner tube corresponding to a top part of the sample place vertically in the HIP furnace. Notice the full penetration of aluminum into the W structure.....	82
Figure 55 - SEM image of another cross section also within the top region of Sample 6, Configuration 1 with a thicker Al 6061-T6 inner tube. Notice the void between the third and forth W layers. Aluminum seen inside the W structure is from the inner Al tube.....	83
Figure 56 - SEM image of an interface between the third layer of W rods and inner Al 6061-T6 tube from a top region in Sample 6, Configuration 1 with a thicker inner Al 6061-T6 tube. The W is seen in green and the aluminum is seen in orange. Notice the diffusion of W into the aluminum.....	84
Figure 57 - SEM image of Sample 6, Configuration 1. Notice the Al-W compound between the W rods, the frayed boundaries of the W rods due to the diffusion of the W into the aluminum, and the transport of an Al-W compound as the aluminum deformed around the W rod.....	85
Figure 58 - SEM image of Sample 6, Configuration 1. Notice the frayed edges of the W that suggests liquid aluminum was formed to promote a reaction between the materials..	85
Figure 59 - Sample 5, Configuration 1 after the successful HIP cycle. Notice the overall well preserved regular W structure and the buckled region in the lower portion of this cross section. Bucking of the W wire was in both the axial and radial directions.....	87
Figure 60 - Sample 7, Configuration 3, optical image after the first HIP process served to encapsulate two layers of W within the Al 6061 matrix.....	92
Figure 61 - Sample 7, Configuration 3, optical image after the first HIP process. Notice the plastically deformed W wire at the contact points with the W rods.....	92
Figure 62 - Sample 7, Configuration 3 under an optical microscope after the first HIP process. Notice the plastic deformation and resulting fracture of the W wire with the contact with W rods.....	93
Figure 63 - Sample 9, Configuration 3 after the first HIP treatment, optical image.....	94

Figure 64 - Sample 9, Configuration 3, optical image. Notice the two interfaces left behind between the aluminum layers after the second HIP treatment.....	96
Figure 65 - Sample 9, Configuration 3, optical image after a third HIP treatment with a fully densified structure. This cycle was applied to explore the interface gap left after the previous HIP cycles could be sealed. Notice the severe shear deformation and fracture of the W rods.....	97
Figure 66 - Sample 7, Configuration 3, optical image. Notice the overall well preserved W structure fully encapsulated within the aluminum alloy 6061 matrix.....	98
Figure 67 - Cross section of a piece of Sample 7, Configuration 3. Notice the disturbed W rod spacing in the vicinity of local buckling in the radial direction.....	99
Figure 68 - Sample 7, Configuration 3, optical image. Notice the region of localized buckling in the axial direction after the second HIP cycle.....	99
Figure 69 - Sample 7, Configuration 3, optical image. Notice the region of local buckling in the radial direction.....	100
Figure 70 - Two pieces each 25 mm in length of Sample 7. Notice the irregular spacing of the exposed W wire.....	101
Figure 71 - Sample 10, Configuration 3 after the second HIP treatment, optical image. Notice the voids between the W fibers.....	102
Figure 72 - Sample 7, Configuration 3, optical image. Notice the plastic indentation in the W wires due to the initial pressurization of the contacting W fibers. The motion of the W rods is due to the migration of aluminum into the W structure.....	103
Figure 73 - SEM image of Sample 7, Configuration 3. Al 6061 is seen in red and W is seen in green. Aluminum seen inside the W was deposited during sample polishing. A W rod is seen plastically deforming a contacting W wire.....	103
Figure 74 - Sample 7, Configuration 3, optical image showing the left over steel and the reacted layer of steel with aluminum below it after machining to an inner diameter of 7.5 mm.....	105
Figure 75 - Cross sectional view of Sample 7, Configuration 3 showing a thicker layer, perhaps as much as 200 um of steel left behind after machining the inner diameter to 7.5 mm.....	105
Figure 76 - Final vacuum sealed assembly of Sample 8, Configuration 4 before HIP Treatment.....	110
Figure 77 - Cross section of Sample 8, Configuration 4. Notice the fractured W wires to the left, causing the entire W structure to expand outward.....	112
Figure 78 - Sample 5, Configuration 1 prepared for tensile testing. Notice transverse buckling in the outermost exposed W wire layer.....	113
Figure 79- Geometry of Sample 5, Configuration 1 within the steel clamps. Only half of the steel clamps are shown.....	115

Figure 80 - Geometry of steel casing around the steel clamps and Sample 5, Configuration 1.	115
Figure 81 - Tensile test set up with steel clamps and casing holding Sample 6, Configuration 1.....	116
Figure 82 – Fracture surface due to tensile testing of Sample 6, Configuration 1. Notice the cone shape of the broken W rods, suggesting the W fibers supported the axial load until they fracture themselves, translating a load to the aluminum matrix.....	124
Figure 83 – Fracture surface due to tensile testing of Sample 5, Configuration 1. Notice the inter-planar fracture surface.....	125
Figure 84 - SEM picture of the fracture surface of sample 5, Configuration 1 after tensile testing.....	126
Figure 85 - SEM image of the fracture surface of Sample 6, Configuration 1.....	126
Figure 86 - SEM picture of a W rod fracture surface from sample 5, Configuration 1 after tensile testing.....	127
Figure 87 - Sample 10, Configuration 3 after mechanical testing. Exposed W wire was damaged in sample machining for mechanical test.....	130
Figure 88 - Sample 11, Configuration 3 after mechanical testing.....	131
Figure 89 – SEM image of Sample 7, Configuration 3 after the second HIP treatment. Notice the microscopic infiltration of aluminum into the surface of the W rod. The fracture of the W rod is attributed to the local clamping performed at the end of the sample during assembling of W reinforcement prior to HIP treatment.....	132
Figure 90 - Sample 13, Configuration 3 after mechanical testing. Exposed W wire was damaged in sample machining before testing.....	133
Figure 91 - Course solid type 1 model.....	166
Figure 92 - Fine solid type 1 model.....	166
Figure 93 - Tetrahedral type 15 model.....	167
Figure 94 - Fine model with initial tungsten rod fracture. Note failed elements are deleted in model.....	167
Figure 95 - Fine model with initial aluminum matrix failure. Also notice the slight necking of the aluminum matrix. Note failed elements are deleted in model.....	168
Figure 96 - Fine model with continued necking and failure of the aluminum matrix. Note failed elements are deleted in model.....	168
Figure 97 - Fine model with the outermost fibers of the aluminum matrix breaking last. Note failed elements are deleted in model.....	169

Figure 98 - Fine model when fully fractured. Note failed elements are deleted in model.....	169
Figure 99 – SEM line scan of the cross section of a W fiber exposed to all four HIP treatments. Notice the sharp boundary between the W fiber seen in gray and the aluminum matrix seen in black.....	179
Figure 100 – SEM line scan from Figure 99 of the aluminum. At a distance from the beginning of the scan larger than 60 um the peaks are thought to be noise.....	180
Figure 101 - SEM line scan from Figure 99 of the tungsten. At a distance from the beginning of the scan smaller than 60 um the peaks are thought to be noise.....	180

List of Tables

Table 1 - Energy of formation and entropy of elements at standard temperature and pressure [16].....	12
Table 2 – Summary of configurations. Additional information for Configurations 2 & 3 found elsewhere.....	23
Table 3 - Dimensions of all five Al 6061-T6 tubes for Configuration 2.....	24
Table 4 – Vickers Hardness Results after HIP treatment and aging Al 6061-T6 with 20 kgf...	64
Table 5 - Components for Sample 8, Configuration 4.....	108
Table 6 - Tungsten Layer 1 Spacing. 274 um (183* um) is the prescribed spacing.....	143
Table 7 – Tungsten Layer 3 Spacing. 128 um (183* um) is the prescribed spacing.....	144
Table 8 - Tungsten Layer 2 Spacing. 274 um is the prescribed spacing.....	145
Table 9 - Tungsten Layer 4 Spacing. 156 um is the prescribed spacing.....	145
Table 10 – Measured Sample dimensions, mass, and density.....	146
Table 11 – Material Properties for the FEM analysis.....	163
Table 12 – Fracture strength of FEM models.....	164

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ABSTRACT OF THE THESIS

Aluminum-Tungsten Fiber Composites With Cylindrical Geometry and Controlled Architecture of Tungsten Reinforcement

by

Carl Joseph Lucchese

Master of Science in Engineering Sciences (Mechanical Engineering)

University of California, San Diego, 2010

Professor Vitali Nesterenko, Chair

A aluminum matrix-W rod/wire structural material in support of DARPA initiative BAA 08-23 was developed and its density and mechanical strength ascertained, both being part of the DARPA matrices. Aluminum tubes and four 90 degree cross-ply tungsten fiber layers were arranged such that under extreme static pressure conditions the aluminum would viscoplastically flow into the tungsten arrangement to create a metal matrix composite. It was found that a cold isostatic process induced “Brazilian” fracture in the tungsten fibers and that the pressurization rate affected the onset of buckling of the tungsten fibers coiled in the hoop direction.

The best results were obtained with two treatments using a hot isostatic pressing process at 500 Celsius and 200 MPa, one treatment for each pair of tungsten fiber layers. No noticeable reaction between aluminum and tungsten was detected. No “Brazilian” fracture and reduced buckling of the tungsten wire in the axial and radial directions were achieved with this manufacturing process. The resulting samples in this successful approach had approximately the desired density of steel, 7.8 g/cc, and an average mechanical strength of 453 MPa, exceeding the DARPA phase 1 matrix of 345 MPa.

Introduction

What would a revolutionary reactive material look like, and how would it be produced? These are the questions that are posed by DARPA announcement BAA 08-23 [1] that determine the goals of this project. This material should have a density of 7.8 g/cc, and an ultimate tensile strength of at least 345 MPa for phase 1, and 690 MPa for phase 2. For safety concerns in handling the material, the composite constructed should only release the stored energy upon the application of a certain powerful stimulus [1].

A reactive material of this nature can be used to increase the power of an explosive while also being safe to store. By meeting Class 4.1 Defense Department Explosive Safety Board criteria, the composite will not explode in an accidental fire, and by being impact insensitive will not detonate when accidentally struck [1]. Such an explosive can be used on warships, being stored to create a safe ammunition magazine from inadvertent explosions the likes of which lead to the loss of the U.S.S Arizona at Pearl Harbor in 1941. Additionally, the reactive material can be used with any war vessel or soldier to provide extra security from the explosive accidentally harming the carrier. When loaded with a central charge and detonated, the cylindrical composite will greatly enhance the blast. Thus besides increased safety, a warhead with this material would be more powerful and efficient. Modern conventional warheads use steel as a casing to penetrate armor and carry the explosive without further aid to the explosive event. A reactive structural material would not only have the strength to penetrate armor and carry the explosive,

but would also participate in the energy release under extreme conditions of dynamic deformation. The method described here to build a tungsten fiber reinforced aluminum composite could also be adapted to other applications such as selective strengthening in an automotive engine block or ductile radiation shields [2, 3].

An approach that helps to meet the phase 1 goals of density and ultimate tensile strength uses two hot isostatic pressing (HIP) treatments with three aluminum alloy 6061-T6 tubes and four 90 degree cross-ply tungsten fiber layers. Two layers of tungsten fiber are secured against an inner aluminum tube and this assembly slides into an exterior aluminum tube. After encapsulating this structure into a vacuum-sealed aluminum jacket the specimen undergoes a hot isostatic pressing process to migrate the aluminum into the tungsten structure due to creep and plastic deformation. The specimen is relieved of pressure and cooled so that it may be machined into a cylinder, leaving a thin layer of aluminum on both sides of the tungsten structure. The process is repeated to increase the thickness of the tungsten fiber aluminum matrix metal composite. The hot isostatic pressing treatments involved a maximum temperature of 500 Celsius, a maximum pressure of 200 MPa, a pressurization rate below 2.5 MPa/min, and cooling under the maximum pressure. The resulting composite samples had densities approximately of 7.8 g/cc and an average maximum ultimate tensile strength of 453 MPa.

Section 1 -

State of the Art of Metal Matrix Composites

1.1 - Current Applications of Metal Matrix Composites

Over the past two decades metal matrix composites have grown from a concept in theoretical papers to materials used in a broad range of applications today. A 1975 study of such composites for wear applications showed that hard particles helped increase aluminum's wear resistance and soft particles would decrease such resistance [4]. Ceramic particles and fibers were also seen to increase tensile strength while reducing elongation of aluminum matrices [4]. Structural research with aluminum tungsten fiber composites have shown such improvements in the aluminum matrix as increased elastic modulus, strength, and damping capacity [5]. Aluminum alloys, including 6061-T6 are used in the automotive industry to selectively increase the strength in engine blocks, pistons, and drive shafts. Brake components commonly created by powder metallurgy are being implemented in many automobiles today, taking advantage of ductile metals mixed with particulates resistive to wear. Such brakes are even being considered for trains, and as a whole ground transportation as of 2005 makes up 69% of the metal matrix composite market [2]. Thermal management technology needed by the electronics industry make up 27% of the market, and the rest is taken up by the aeronautics industry [2]. Military applications also make up part of the market but due to confidentiality it is difficult to accurately measure the size of this market share in metal matrix composites.

Examples of other applications of metal matrix composites include radiation absorption, ductile magnets, and thermal expansion. Ceramics reinforcement, commonly in the form of ceramic B_4C fibers in a metal matrix are used as a radiation shield. It can sustain the thermal expansion loads from the nuclear reactor operation due to the ductile metal matrix while providing the necessary protection for employees operating the reactor with the boron-carbide particulates. Other radiation shielding composites have been achieved with powder metallurgy involving an aluminum matrix with tungsten or tantalum particles [3]. The powder metallurgy approach combined with a magnetic field could align magnetic particles inside a non-magnetic powder matrix such that a ductile and machinable magnetic metal matrix composite can be created [6]. Such research efforts have focused on aluminum and steel matrices with magnetic particles to be used as sensors in applications that may mechanically load the sensor such that a normal ceramic magnet would fail [6]. The electronic industry is in need of materials that can transport heat quickly from microchips in order to protect the microchips from premature failure. However substrates of varying metals causes thermal expansion stresses. To create a substrate that has a low coefficient of thermal expansion to match the silicon chips while transporting heat well, a composite of aluminum with silicon has been developed. The silicon is deposited in molten aluminum, mixed, and cooled to create a silicon structure within the aluminum to limit the thermal expansion of the composite while retaining an acceptable heat transfer coefficient [7].

1.2 - Design Considerations and Liquid Phase Processing

Metal matrix composites primary design advantage lies in their ability to have many properties be finely tailored. For example, by changing volume, morphology, and distribution of components, a wide amount of material properties can be obtained [2]. This ability to fine-tune their properties makes them ideal to design materials for unique and challenging purposes.

Metallic properties such as ductility and toughness can be blended with ceramics using their high strength and high modulus of elasticity to increase shear and compressive strength as well as reducing the composite's thermal coefficient of expansion [8]. Such physical properties can also be affected by dense dislocation regions at the composite interfaces created due to the thermal contractions, geometric constraints, and plastic deformation during the processing of the composite [5, 8]. Composites commonly include particles, fibers, and/or platelets inside a larger framework or matrix metal [8]. Particulates can change age times required for the matrix to harden. Additional intermetallic particulates can also diffuse and form within the composite's components during an aging process. The material behavior with temperature needs to be understood for each component to better predict the properties of the composite and the necessary processing [8]. Of particular interest to this research is that heat treatment after processing of the composite is not seen to affect the modulus of elasticity in aluminum matrix composites [8]. However, an increase in tensile strength was seen in composites made of tungsten and aluminum 2024 powders when the processed composite was treated to bring the aluminum into

the T6 condition [3]. In all metal matrix composite designs the cognizant engineers need to take into account the component's elastic modulus, tensile strength, density, melting temperature, chemical stability, thermal stability, coefficient of thermal expansion, geometry, and compatibility with the matrix material to create a successful material for their design function [8].

Liquid metal and powder metallurgy are the most common approaches to process metal matrix composites [2]. Stir casting involves uniform mixing of a molten metal and a ceramic or metal particulate before cooling [2]. Liquid metal infiltration involves a ceramic structure that is injected with liquid metal and then cooled to form the composite [2]. Problems however could include reinforcement motion and damage, coarsening of grain sizes, and undesired reactions with components [8]. Liquid processing does by its nature require a high temperature that can promote diffusion between the composite components. This diffusion interaction may result in the chemical reaction between the components forming a new and often undesirable compound. Thus, wetting behavior is of particular importance to any metal matrix composite made with components in the liquid phase as such behavior determines the shape and amount of reacted intermetallic compound in the matrix material [8]. To promote wetting of components in the liquid and solid phases agents or coatings are applied often resulting in reaction compounds at the interface regions of the components [8]. Interfacial cracks or voids between any of the components and intermetallic compounds could also become crack initiation sites, thus care must be

taken to control new compounds created in a high temperature, multiphase processes [8].

1.3 – Solid Phase Processing and Powder Metallurgy

Solid phase processing includes powder metallurgy and mechanical processing. Although all the components are solid high temperatures should still be applied, most often in a hot isostatic pressing treatment. That can promote reactions between components of a composite. Often solid state processing such as powder metallurgy includes the mixing of components, a cold isostatic pressing (CIP) process to plastically deform the components together, a degassing process to remove air from a porous composite, a hot isostatic process to densify the composite, and finally mechanical processing to make the composite brick of material a usable part [8]. The same steps are also applied even when the initial components do not include powders, but are mechanically constructed structures that are to be pressed together to form a single material or part [9, 10]. Such solid state processing is shown to be effective for this research to avoid reactions more commonly seen in processes with liquid phases.

In the case of powder metallurgy the morphology of the particles and porosity of the resulting material after processing greatly affect the dynamic mechanical properties [11]. In a study by Cai and Nesterenko using a CIP process with metallic powders and a polymer composite, it was found that composites with fine metallic particles, even with low density have a greater ultimate compressive strength than composites with course metallic particles [11]. Porous samples with course particles

had varying strengths, which can be expected when the ultimate compressive strength is greater than the densification pressure [11]. Gradual densification during the initial deformation may result in greater strength. The samples fractured through shear localization as was the case with the Polytetrafluoroethylene (PTFE) matrix fractured due to stress concentrations formed by the particles. The PTFE would not allow the coarse particles to move along micro fractures to form supporting force chains. For porous or dense samples with fine particles force chains are created as the metallic particles align to carry the load. Upon further deformation the chain breaks and the particles move due to the micro cracks. The moving metal particles eventually meet each other to reform new force chains, thus increasing the ultimate strength of the material. Eventually after enough iterations of this behavior, the force chains will fail to form, and the composite will fracture [11].

When these force chains are created, intense heat is generated within the composite due to friction between particles and the breaking and creation of atomic bonds [12]. In dynamic compression, such as impact with a rigid wall, the relative densities between the particles in the composite cause each particle to move with respect to one another rather than traveling together as a single body. Heavy tungsten particles for example in a PTFE matrix move through the matrix imparting their kinetic energy to the matrix in the form of heat [12]. If additional metallic particles are present in the composite, such as aluminum the thermal energy can be transferred from the PTFE to the aluminum particles. Once fracture of the matrix has been achieved these hot aluminum particles would be ejected into the oxygen rich air.

Larger tungsten particles as expected impart more heat to the PTFE matrix but fail to form force chain, lending to a decreased dynamic strength [12]. Such large W particles acted as stress concentrations inside the PTFE matrix causing shear 45 degree macrocracks to form under the compressive loading. Small tungsten particles impart enough heat to increase the temperature of the PTFE matrix to create hot aluminum particles while attaining a high dynamic strength due to the creation of force chains [12]. Fractured samples showed debonding between components of the composite as well as fracture of the PTFE matrix [12]. This is to be expected as interfaces between particles like grain boundaries do not have the strength of the individual grains or crystals. With such work in powder metallurgy to create a metal matrix composite capable of releasing hot aluminum particles when a dynamic stimulus is applied, the compressive impact load in this case, the starting point for this research is determined.

1.4 - Non-Powder Solid State Processing

For the reactive material being developed, there is interest in avoiding powder metallurgy and instead use metallic plates and fibers to construct a metal matrix composite. Powder formed metals often have some level of bulk distributed porosity higher than in solid metals cooled from a melt. Such work also follows the steps outlined for solid-state processing with a focus on the HIP treatment. As in powder metallurgy the powder must first undergo a CIP treatment to plastically deform the particles together into a structure such that a vacuum can be formed within the structure. By not using a powder the separate CIP treatment may or may not be

necessary before densifying the structure together into a composite material with a HIP treatment. The CIP treatment can be incorporated into the HIP cycle.

Studies conducted to measure the growth of the bi-metallic compound formed between steel fibers in an aluminum matrix initially in the form of plates used HIP treatments of 70 MPa, 500 to 575 Celsius to fully migrate aluminum around the steel fibers [10]. The spacing of the fibers, 150 μm in diameter, was between 40 and 150 μm with the thickness of the aluminum plates being 100 μm [10]. Magnetic composites of an aluminum matrix with steel particles had HIP cycles of 79 to 95 MPa at 540 to 610 Celsius conducted within a magnetic field [6]. To fully densify these composites moderately high pressures were needed with the aluminum matrix at a temperature just below its melting point. The high temperatures promote diffusion, thus a reaction to form the bi-metallic compound, and decrease the mechanical strength of the plates such that they will plastically flow around the steel fibers. However pressing the plates into a sheet of fibers did create issues such as residual porosity, fiber fracture, residual stresses in the fibers and matrix, fiber swimming, and matrix contamination [9].

It was found that compound formation around the fibers was not only dependent on time and temperature for diffusion but pressure and fiber spacing. As pressure is increased more contact is made between the components of the composite, creating interfaces at high pressures and subsequently high temperatures. The increased contact and temperature at the interface regions of the composite components promotes diffusion between those components. As spacing was increased

the consolidation time decreased [9]. The spacing in this particular study was 150 to 230 um between steel fibers in an aluminum matrix. An equation to describe the thickness of the intermetallic compound with respect to time was found to be [9]:

$$x = k_o \exp\left(\frac{Q_d}{2RT}\right) \sqrt{t} \quad (1)$$

Where k_o and Q_d are constants unique to chemical compound being created, Q_d in particular being the activation energy due to diffusion. Other variables from the equation above are the gas constant, R , temperature, T , and time, t . From here we see time and activation energy are directly related to the thickness of the compound where as temperature has an inverse relationship to the compounds thickness. All compound growth was dominated by diffusion mechanisms [10].

Section 2 –

Research Goals

2.1 - A Brief Review of the Combustion of Aluminum Particles

In the pursuit of creating a new reactive material knowledge of the basic chemistry and physics of the proposed design is required. It is known that aluminum has an extremely favorable enthalpic reaction with oxygen [13, 14, 15] and it is this reaction that is used as a basis to create a metal matrix composite material for the purpose of releasing a high amount of energy. The reaction of interest is presented below with values for the enthalpy and entropy tabulated in Table 1 at standard temperature and pressure.



$$\Delta H_{rxn}^{\circ} = -3339.6 \text{ kJ/mol} \quad (3)$$

Table 2 - Energy of formation and entropy of elements at standard temperature and pressure [16].

	Energy of Formation at STP (kJ/mol)	Entropy (J/K-mol)
Al (solid)	0	28.3
Al ₂ O ₃ (solid)	-1576.4	50.99
O ₂ (gas)	0	205

From elementary thermal dynamics the relation of Gibb's Free Energy show:

$$\Delta S_{rxn}^o = 2 * 50.99 - (4 * 28.3 + 3 * 205) \frac{J}{mol - K} = -626.22 \frac{J}{mol - K} \quad (4)$$

$$\Delta G^o = \Delta H_{rxn}^o - T \Delta S_{rxn}^o \quad (5)$$

$$\Delta G^o = -3339.6 \frac{kJ}{mol} + 298K * 626.22 \frac{J}{mol - K} = -3.2 * 10^6 \frac{J}{mol} \quad (6)$$

$$\Delta G_{equilibrium}^o = 0 \Rightarrow \frac{\Delta H_{rxn}^o}{\Delta S_{rxn}^o} = 5.3 * 10^3 K \quad (7)$$

The reaction of aluminum with oxygen to form alumina is very exothermic and spontaneous up to temperatures of approximately 5,000 Celsius as shown in the Equations 2-7. To release a large amount of energy due to this reaction the amount of aluminum in contact with oxygen has to be maximized by using small particles of aluminum. The oxygen from the surrounding air is sufficient. The question is, however, just how do small particles of aluminum combust in air?

Aluminum particles when exposed to oxygen in the air spontaneously form an outer layer of alumina. The thickness of such a layer has been measured to be 2-3 nm in sputtering experiments [17]. Once the alumina shell is created, heat from the reaction to form the alumina flows into the body of the aluminum particle. If the particle were small enough, this heat would significantly increase the temperature of the entire particle. Larger particles need to be preheated such that the amount of energy exothermically released is enough to sustain the temperature of the particle. Due to the significant difference between the coefficients of thermal expansion for alumina and aluminum, mechanical stresses will be developed within this aluminum

particle and the thin alumina shell. Since the coefficient of expansion for aluminum is three times greater than the coefficient of expansion for alumina, the aluminum particle will stretch out the alumina shell, causing the shell to fracture and crack open. Once fractured more fresh surfaces of aluminum will be exposed to the air and the process repeats until most of the aluminum within the original particle has reacted to form alumina [14]. Eventually the aluminum/alumina particle reaches vaporization temperature and may become a hot gas. As seen with high-speed photography aluminum does indeed burn both on the surface of the particles and as a gas [14]. If multiple aluminum particles were of the correct diameter in the correct concentration the hot alumina gas would form a rapidly expanding shock wave or explosion.

Of critical importance is the formation of the alumina shell. As seen in a sputtering process the oxide layers of 2-3 nm thick of alumina appear nearly instantaneously [17]. With greater heating of the aluminum body the thickness of the alumina is increased, strongly indicating this is a diffusion-limited process [17]. With this experiment the activation energy of face centered cubic (FCC) aluminum with thermally induced oxygen was calculated to be 131.2 kJ/mol for lattice diffusion and 69.1 kJ/mol for grain boundary diffusion [17]. The activation energy was measured in the range of 423 - 573 K to be in the range of 19 - 23.4 kJ/mol [17]. Due to the excited ionic state of the oxygen atoms in the sputtering process, the activation energy measured in this experiment is far below the activation energy of alumina with thermally excited oxygen in both atomic and ionic forms as would be expected when using natural air [17].

Actual experiments with aluminum particles show particles greater than 30 μm burn due to the previously mentioned diffusion process but other particle sizes may burn by different mechanisms [13]. The time it takes for the particle to completely combust is directly related to the initial diameter of the aluminum particle, and the combustion time is independent of temperature [13]. Ignition of the aluminum particles is, however, dependent on the oxidizer's composition and the temperature of the particles. For aluminum particles ignition temperatures are seen to range between 600 and 2,050 Celsius [14]. Aluminum powders were tested to show the difference in combustion behavior with larger aluminum particles ($>20 \mu\text{m}$) that followed a diffusion-limited model or Beckstead correlation [13]. It was also seen that nano-sized aluminum particles ignite and burn at temperatures below what is necessary for micro sized aluminum particles [13]. The burn rate of nano-sized particles was seen to be related to particle diameter, pressure, and oxidizer content, similar to micro-sized aluminum particles but by different rates [13].

However, nano-sized aluminum particles do not show a significant increase in blast performance compared to micro-sized aluminum particles when mixed with other explosive powders [15]. A study found that mixing aluminum powders of varying diameters with TNT and PBX gives mixed results [15]. The nano-sized aluminum powders (100-200 nm in diameter) decreased the velocity of detonation in PBX but increased the velocity in TNT when replacing 12 μm diameter aluminum particles [15]. No increase in pressure was seen in aquarium tests and no increase was

seen in air-blast tests with the nano-sized aluminum particles over the micro-sized aluminum particles [15].

It is our goal to research a material that will store solid aluminum such that under a certain stimulus it is released at high a temperature in particulate form with fresh surfaces. The resulting particle size should be of the order of 30 microns at temperatures exceeding 600 Celsius. Preheating the aluminum as a solid or in particle form before being dispersed is critical to ignite the aluminum particles sooner, rather than being heated due to friction and contact with hot gases from an internal explosive. Additionally the preheating of the aluminum particles would aid the diffusion process of ambient oxygen in the air into the aluminum particles while maintaining the thermal stresses within the particles such that the alumina surface continues to crack, exposing more aluminum for continued exothermic reactions. These chain reactions within each aluminum particle would eventually lead the particles to vaporize and as a hot gas expand explosively.

2.2 - DARPA Goals

Processing and application of metal matrix composites have been met with several challenges such as struggles to find a comprehensive solution to strength, density, and reactivity. Previous work with composites, metallurgy, failure mechanisms, modeling and simulation, self propagating high temperature synthesis, energetics, and material processing technology have been accomplished, helping to shape the current DARPA program. Achievements in metal matrix composite

strength, material failure mechanism characterization, and combustion behavior of metallic particles have greatly advanced this field of research, but none of the work has brought the knowledge together to find a comprehensive solution. The goal of this study is to create a composite that is a comprehensive solution of high mechanical strength, high energy density, and a self-controlled mechanism to release the energy upon command [1].

These ultimate goals laid out by DARPA are broken down into two phases. The phase one goals are [1].

1. Produce samples of at 250 g.
2. A reactive material with an ultimate tensile strength of 345 MPa.
3. The density of steel, 7.8 g/cc.
4. Impact insensitivity and a blast performance at least twice that of an inert case. Meaning the sample should not release energy when impacted upon, but only under the desired powerful stimulus.

Such a material can lead the way to develop similar composites for other applications as well as provide their safe storage and delivery. By design, since the material will only discharge its energy upon the use of a particular stimulus such as a detonation of traditional explosive applied in a unique fashion design accidental impact or fire will not cause the material to discharge, thus avoiding potential to cause severe damage to carrier.

As described in section 1.4, powder metallurgy will not be pursued to create the metal matrix composite. To achieve the desired energy density as much aluminum as possible should be present within our material with very little if no voids or porosity commonly found in powder formed materials. As such, the approach used for aluminum and steel fiber composites [9-10] to plastically deform aluminum into a fiber structure will be followed in our research with a tungsten mesh incorporated into a aluminum matrix. No liquid phase processing will be pursued to minimize the creation of intermetallic compounds due to the desire to have as much aluminum as possible to react with the ambient oxygen. Though such metal fiber composites have been known, none have been developed for the purpose of a reactive structural material using aluminum and tungsten fibers with highly controlled architecture in cylindrical geometry. Additionally the challenge of deforming cylinders with incorporated fiber structure as we desire to create, instead of pressurizing plates into the fibers, has not be addressed in previous work.

2.3 - Why a Metal Matrix Composite as an Energetic Material?

Due to the sensitive nature of weapon systems current explosive technology cannot be fully obtained or described. However in the search of using aluminum powder as an explosive it has been successfully mixed into other powder or liquid explosives to indeed increase their power [15]. In these applications the aluminum powder is heated to ignition temperature by interacting with the hot gas from the detonation. As can be expected, not all of the aluminum powder would successfully ignite and would instead be jettisoned with the blast wave. Studies like those for

Defense Research and Development in Canada indicate only so much aluminum powder of any size can be added to an explosive before performance is decreased [15]. The goal by creating a new solid material to transport and store the aluminum powder is to maximize the amount of aluminum that would ignite in an explosive event.

Of course aluminum casing can be used as well, but as expected not all of the pure aluminum case would fracture into the necessary particle sizes to ignite within the required time period to increase the power of the blast wave. Also aluminum cases do not have the density and strength of steel cases to both successfully store and transport the explosive payload. This is where a metal matrix composite can be applied to create a material that would have a high strength for storage and transport but would also fracture into an array of small particles under the action of a traditional explosive to increase the amount of energy released.

Section 3 –

Research Findings & Development

3.1 - The Concept and Configurations

The solution presented here to address the DARPA announcement BAA 08-23 [1] involves tungsten fibers (W) arranged in a highly controlled cylindrical structure within an aluminum alloy 6061-T6 (Al 6061-T6) matrix. The choice of aluminum as the metal matrix material is to take advantage of the spontaneous exothermic reaction with oxygen when aluminum will be pulverized by detonation of traditional explosive. Tungsten fibers are to provide mechanical strength to help the composite attain a tensile strength similar to steel as well as provide a mechanism for which to disperse the aluminum in an array of small particles.

The tungsten fibers are arranged in a wrapped mesh structure to form a cylinder. Aluminum alloy 6061-T6 fills all the remaining space within the mesh structure, leaving a hole in the center of the composite to maintain the hollow cylinder shape. In the center of this cylinder the explosive can be placed. Upon detonation a blast wave is imparted to the inside surface of the cylinder. As seen in metal particle composites in PTFE the varying densities between the components will causes the components to exert forces between one another, even moving with respect to one another [12]. The concept here is that the entire tungsten mesh structure with its greater density moves outward at a much slower speed than the aluminum matrix. The

aluminum matrix is to be heated by the multiple shock and release waves within the composite imposed by the blast and the resulting reflections from the inner and outer surfaces of the cylinder. With the heating of the aluminum causing the matrix metal to lose strength and become softer the aluminum is expected to move with a greater speed away from the detonation products through the tungsten structure. The mesh structure should create a torturous path for the heated aluminum causing it to plastically deform, increase in temperature, and fracture into jets, and later into hot particles.

Thirteen samples of different Configurations using tungsten fibers of 200 μm in diameter were assembled. Of the four Configurations of aluminum tubes and tungsten fibers, three were externally pressurized and one internally pressurized. Each Configuration was composed of an internal Al 6061-T6 tube followed by four layers of tungsten fibers in alternating directions. The tungsten fiber layer contacting the internal aluminum tube lies in the axial direction with the final layer of tungsten wire being wrapped around the specimen in the hoop direction. An external Al 6061-T6 tube is then slid into place around the entire tungsten fiber structure. The Configurations vary in initial spacing between the tungsten fibers, number of fibers in each direction, number of aluminum tubes within the entire pre-pressurized assembly, and pressurization direction. All the specimens were 30% tungsten by volume, half the total volume of tungsten in each direction, axial and hoop. Tables 2-3 detail the Configurations. Configuration three involves two pressurization cycles, one for each pair of tungsten layers. An interim external Al 6061-T6 tube of the following

dimensions: 9.3 mm internal diameter and 11.1 mm external diameter is used for first HIP treatment. Excess aluminum is machined away and the final two layers of tungsten and Al 6061-T6 tube are applied. Configuration two involves placing a thin Al 6061-T6 tube between each layer of tungsten fibers as described in Table 3.

Table 2 – Summary of configurations. Additional information for Configurations 2 & 3 found elsewhere.

Configuration	1	2	3	4
Internal Al 6061-T6 Tube Dimensions (mm)	ID = 7.5 mm OD = 8.1 mm	ID = 7.5 mm OD = 8.1 mm	ID = 6.896 mm OD = 8.5 mm	ID = 7.5 mm OD = 8.1 mm
External Al 6061-T6 Tube Dimensions (mm)	ID = 9.8 mm OD = 11.1 mm	ID = 11.6 mm OD = 12.2 mm	ID = 10.1 mm OD = 11.1 mm	ID = 10.1 mm OD = 11.1 mm
Number of Axial Tungsten Rods – 1 st Layer	54	54	71	71
Number of Axial Tungsten Rods – 3 rd Layer	87	87	78	78
1 st Layer Tungsten Rod Spacing (um)	274	274	183	183
2 nd Layer Tungsten Wire Spacing (um)	274	274	242	242
3 rd Layer Tungsten Rod Spacing (um)	128	128	183	183
Fourth Layer Tungsten Wire Spacing (um)	156	156	120	120
Pressurization	External	External	External	Internal

Table 3 - Dimensions of all five Al 6061-T6 tubes for Configuration 2.

Al 6061-T6 Tube / Property	1	2	3	4	5
ID (mm)	7.5	8.6	10.08	10.84	11.6
OD (mm)	8.1	8.88	10.36	11.12	12.2

Section 3.2 - Construction of the Samples

To construct the first sample of Configuration 1 an Al 6061-T6 tube measuring approximately 130 mm in length and 0.75 mm in thickness was obtained. The tube was etched in a solution of nitric acid (75% by volume) and hydrofluoric acid (25% by volume) for approximately 133 minutes to the desired thickness of 0.3 mm with an inner diameter of 7.5 mm and an outer diameter of 8.1 mm. Inside of this Al 6061-T6 tube a 01 steel rod, 7.49 mm was inserted such that the ends of the steel rod extended out of the tube to be gripped by various clamps to assemble the tungsten fiber layers. For samples with a inner diameter of 6.896 mm a steel mandrel of 6.883 mm in diameter was used. A total of 54 tungsten rods, 200 um in diameter from AM Systems was then placed parallel to the tube with a circumferential separating distance of 264 um, close to our desired distance of 274 um. To assemble this first layer a plastic guide was created as seen in Figure 1. In Figures 2 and 3 the mount to manufacture the guides is shown. These guides tightly fit around the aluminum tube and hold the two opposite ends of the rods over the entire sample length.



Figure 1 – Close up of plastic guide for assembling axial rods.

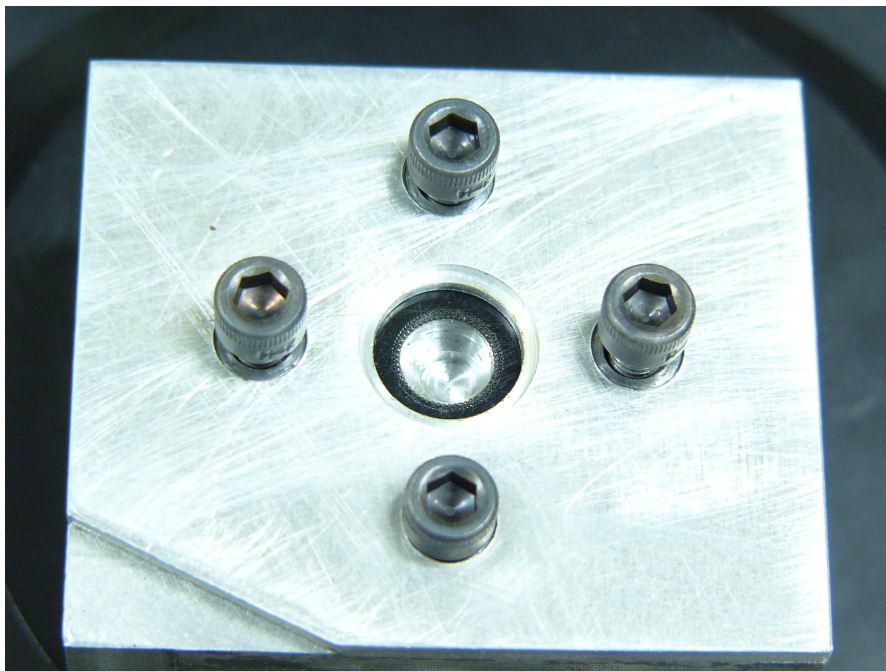


Figure 2 – Mount for constructing plastic guides. Notice black plastic guide in center.

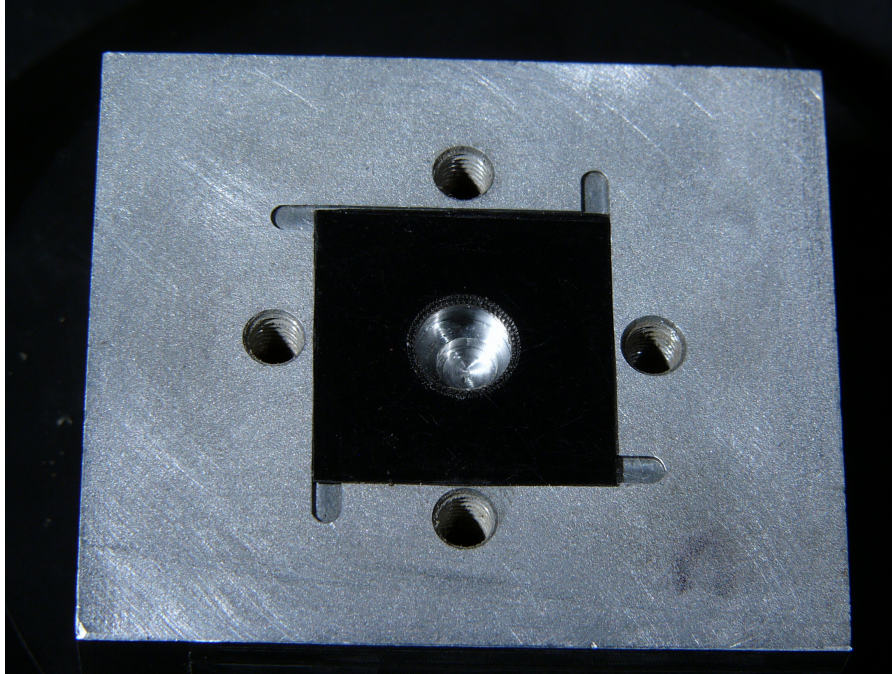


Figure 3 – Mount for drilling plastic guide. Cover removed to show guide in center.

A second layer of W wire was then wrapped around the sample in the hoop direction with a spacing of 250 μm , slightly denser than our target of 274 μm . The completed second layer can be seen in Figure 4. This wrapping process consisted of a spool of Alfa Aesar 200 μm in diameter W wire on a spring clutch as seen in Figure 5. This approach was designed and implemented by Kevin Gott. The wire would pass through a narrow notch seen in Figure 6, which acts as a guide to the sample. The sample is driven to rotate and translate in the axial direction to wrap the W wire around the sample. Due to this wrapping process the axial wires of the first layer became twisted around the aluminum tube. To keep the radial wire tight around the sample the wire was fixed in place with superglue at the ends.



Figure 4 – The sample after the second layer of W wire was applied.

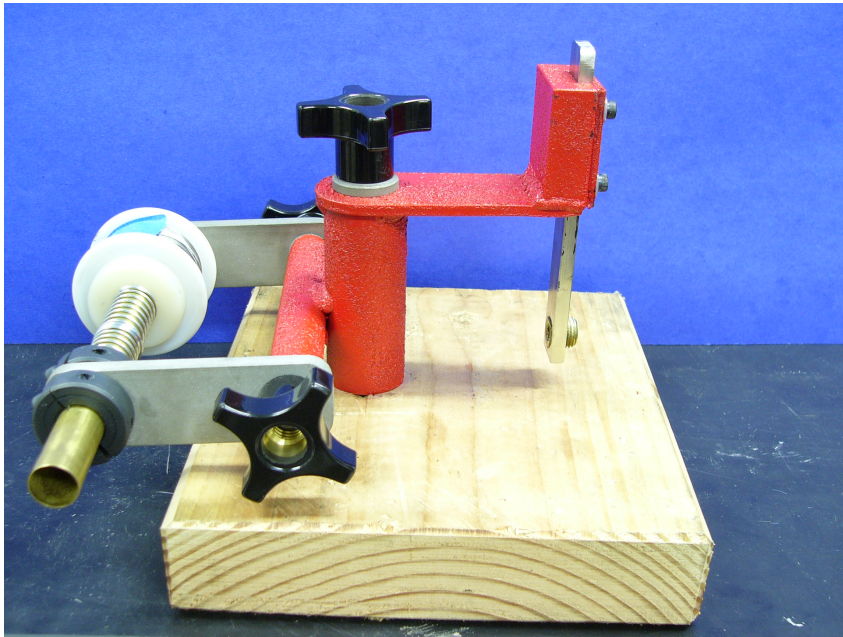


Figure 5 – Tungsten wire dispenser. Spring acts as clutch mechanism to maintain tension of W wire.

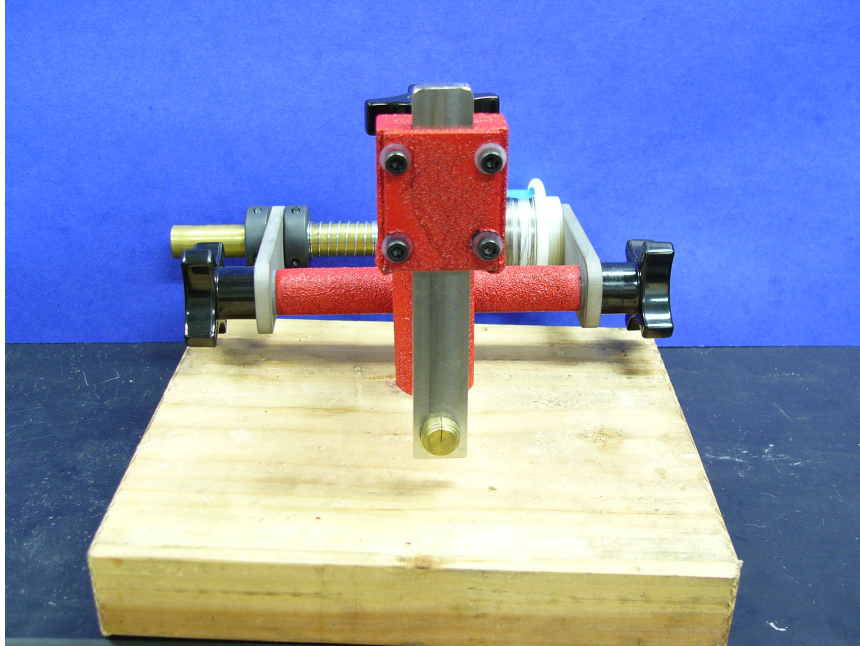


Figure 6 – Tungsten wire dispenser. A guiding notch is seen as the thin groove cut in brass colored threaded bolt.

The third layer of W wire, second in the axial direction, was applied at the same time as the forth layer, second in the radial direction, to avoid this warping effect. This axial layer was kept in place with rigid aluminum disks created in the same manor as the plastic guides as seen in Figure 7, which pass through holes for the wire pattern, and the radial wire was kept in place with superglue at the ends. A total of 87 W rods also from AM Systems was used for the third layer as seen in Figure 8, and a Alfa Aesar W wire was used for the forth layer. The spacing of the third W layer, second in the axial direction was an average of 123 μm , slightly smaller than our desired 128 μm , and the spacing of the forth layer, second in the radial direction was an average of 190 μm , wider than our target of 156 μm . To construct both layers at the same time one side of the sample had all the layers fixed, the axial layer with an

aluminum guide and the hoop layer with superglue. The second axial guide was placed close to the fixed guide grabbing the W rods to make a local region of straight rods right in front of the wire to be wrapped. As the W wire was being wrapped, the second aluminum guide was gently pushed by hand to straighten the rods beneath and ahead of the wrapping layer. After all layers of W wire were applied an outer tube of Al 6061-T6 was slid into place around the sample to a loose fit as seen in Figure 9. The outer tube was the same length as the sample with a thickness of 0.65 mm, an inner diameter of 9.8 mm, and an outer diameter of 11.1 mm.



Figure 7 – Al guide for third layer of W, second layer of W rods in axial direction.

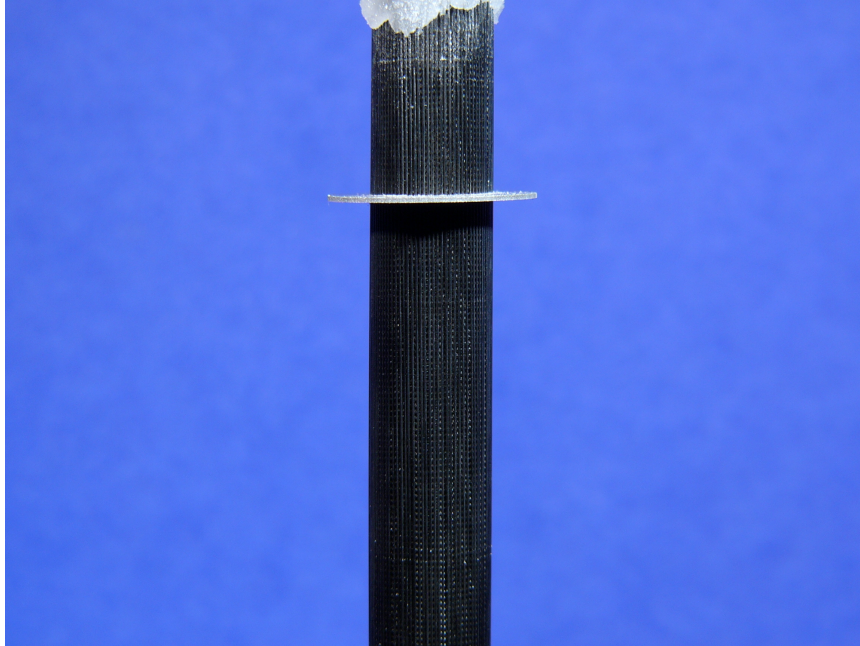


Figure 8 – Third layer of W rods applied. Notice remaining glue to keep rods and wire in place and Al guide disk. This layer was removed and redone with the forth layer of W wire.



Figure 9 – Final assembly of sample with four layers of W wire, two Al tubes, and a steel core.

The most successful samples with respect to the DARPA matrices were made with Configuration 3. With samples in this Configuration the first W rod and second W wire layers were constructed simultaneously as described for the third and fourth layers of W in sample 1 of Configuration 1. Aluminum disks were used to keep the axial rods in place and all layers were kept in place by superglue. No superglue was allowed to be within the final assembly. After the external Al 6061-T6 tube was slid over the assembly the ends of the tube were then mechanically clamped to squeeze the structure together. The superglue areas of the samples would be outside the clamped external aluminum tube, and would be then cut away. This simple clamping of the ends of the sample at room temperature was seen to cause local fracturing of the W rods and wires in the clamped region but allowed the entire assembly to be stable for the degassing and HIP treatment. No CIP process was used for this solid-state Configuration. The sample was then placed inside an Al 6061-T6 capsule or jacket. A vacuum was created of at least 20 microns of mercury within the capsule and sealed with a weld. Figure 10 shows sample 7, a sample of Configuration 3 in a sealed aluminum jacket ready for a HIP treatment.



Figure 10 - Sample 7, Configuration 3. Pre-HIP assembly.

With a successful HIP treatment to create an aluminum composite with two layers of W fibers construction of the next pair of W fibers proceeded as discussed with aluminum guides and simultaneous assembly. To prepare this sample for the construction of the last pair of W layers the ends of the samples were removed and polished for microstructure observation using optical microscope. Using an optical microscope shallow tooling holes were located in the center of the mandrel and W structure. In the event the centers were not in the same location the center of the W structure was marked. Using the shallow holes at the ends, the sample was mounted in tooling knobs to have the outer diameter machined to this new centerline between the tooling holes. The outer diameter is machined to the closest nominal size such that the sample can be easily mounted into a collet grip. Within the grip of the collet a center

hole is drilled through the length of the mandrel large enough to accommodate a wire for an electro-discharge machine (EDM) process. With an EDM the inner diameter of the mandrel is cut to 6.35 mm. A new stainless steel mandrel, 6.34 mm in outer diameter is then inserted inside the previous mandrel. This means there is a gap of at most 0.01 mm between the mandrels and a thickness of approximately 270 μm of 01 steel left behind. With the new mandrel the last pair of W layers are applied with a crimping aluminum tube, and compressed together at the ends of the sample. As discussed the sample is vacuum encapsulated for a second HIP treatment.

3.3 - Planar Studies and Configuration 1

Samples of this Configuration were used to study and solve the various issues with fracture of the W fibers, buckling of or within the assembly, and the formation of intermetallic compounds. Preliminary studies with sheets of Al 6061-T6 did not show signs of fracturing W wire when pressed against a rigid steel plate at 345 MPa as seen in Figure 11. But “Brazilian” fracture of the W rods in the earliest cylindrical samples was observed. Sample 1 in Configuration 1 was constructed exactly as described in section 3.2, but underwent a CIP process with the intention to plastically deform the components of the structure together in preparation for a HIP process.

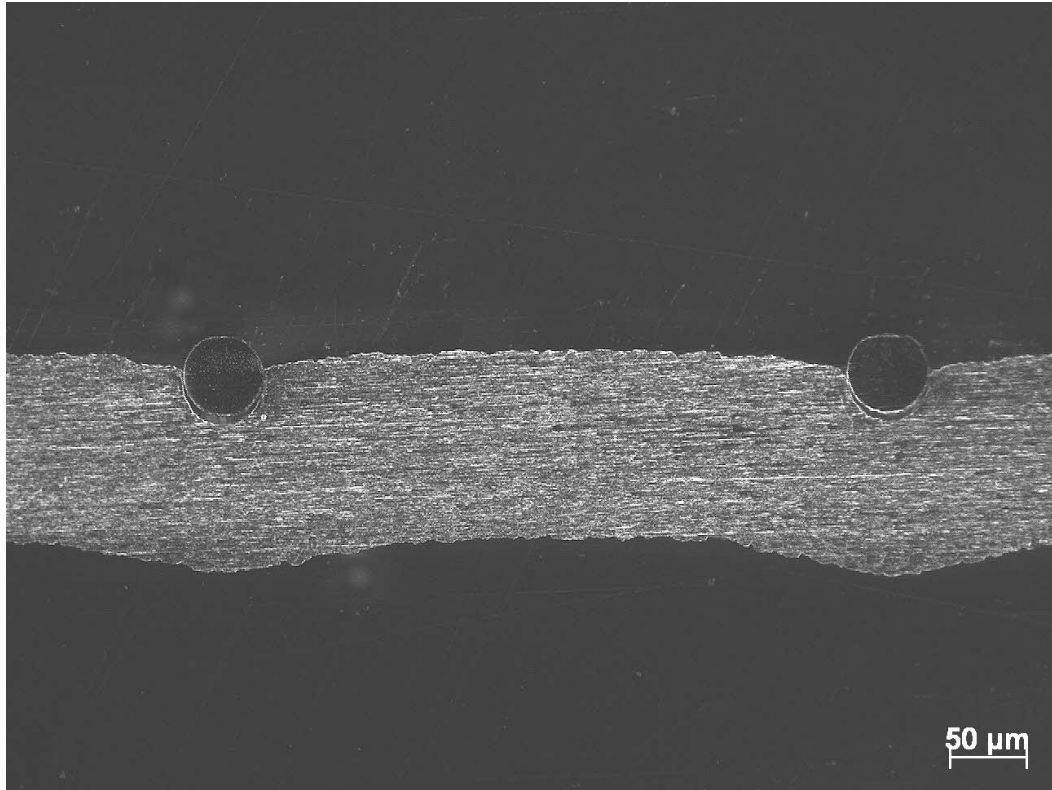


Figure 11 - Planar sample of a sheet of Al 6061-T6 being compressed into a W mesh (100, 0.0509 mm in diameter) in a CIP process at 345 MPa. Notice the deep penetration of W wires into the aluminum with some plastic deformation of the top of the W wires.

This sample 1 was then prepared for the CIP process as seen in Diagram 1. A steel rod with an outer diameter of 7.4 mm to form a tight fit to the inner diameter of the sample was gently tapped into the core. Then the sample was placed in a Tygon tube of an inner diameter of 12.7 mm and a thickness of 1.6 mm. To get the sample into the tube, grease (Lubristeal Stopcock Grease) needed to be applied. Rubber stoppers and steel plugs were placed at each end and tightened with steel clamps. The ready to be CIP treated sample in the rubber jacket was approximately 216 mm in length.

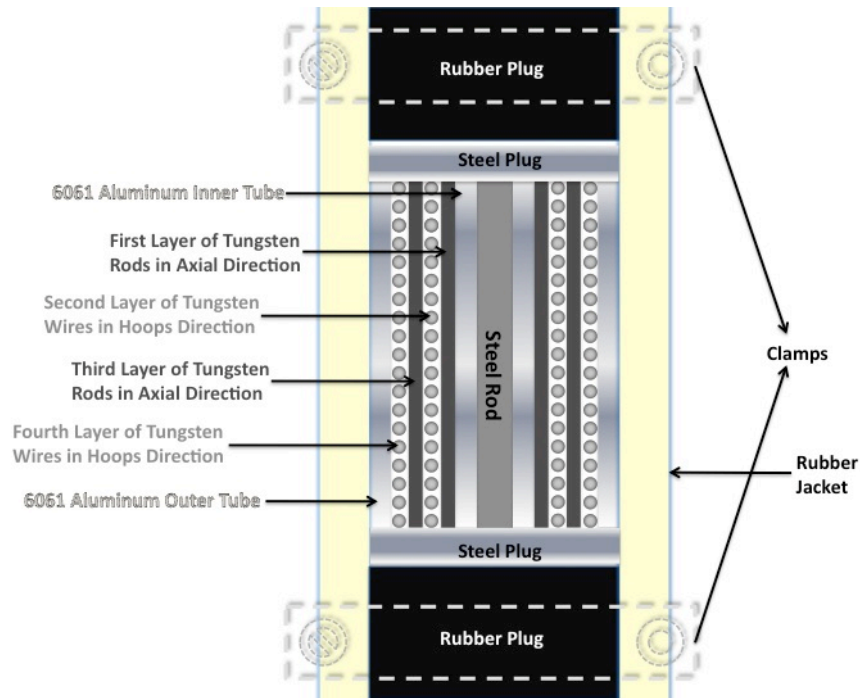


Diagram 1 – Sample prepared for CIP.

The sample then underwent a CIP process at 345 MPa for 30 minutes. After the process the pressure was reduced to atmospheric pressure for approximately five minutes and the sample removed from the pressure vessel. The Tygon jacket was removed from the sample with a scalpel. Approximately thirteen millimeters of material was removed from both sample ends to effectively cut off the areas with the superglue and collapsed deformation. Another 0.3 mm specimen was cut off to observe under an optical microscope. The cuts were made with an EDM. A cross section of the sample after the cuts were made can be seen in Figure 12. An average outer diameter of 10.8 mm was measured after the CIP process.



Figure 12 – Cross sectional view of sample 1, Configuration 1 after EDM cut.

Under observation with an optical microscope the deformation of the layers in the sample can be clearly seen. Of most importance was the existence of the cracks and fractures in the W wire in areas they make contact with each other as seen in Figure 13. The W wire penetrated the aluminum layers as seen in Figure 14 an average of 33.3 μm with a range of 13 μm to 50 μm and a standard deviation of 8.6 μm . The W rods penetrated the contacting W wire an average of 29.6 μm with a range of 13 μm to 50 μm and a standard deviation of 11.2 μm . Tungsten wire penetration is restricted to the axial rods going into the hoop wires. The axial rods show little

evidence of being plastically deformed by the hoop wires. As seen in Figure 15 most of the radial wires have cracked or fractured in a “Brazilian” mode as seen in Figure 16.

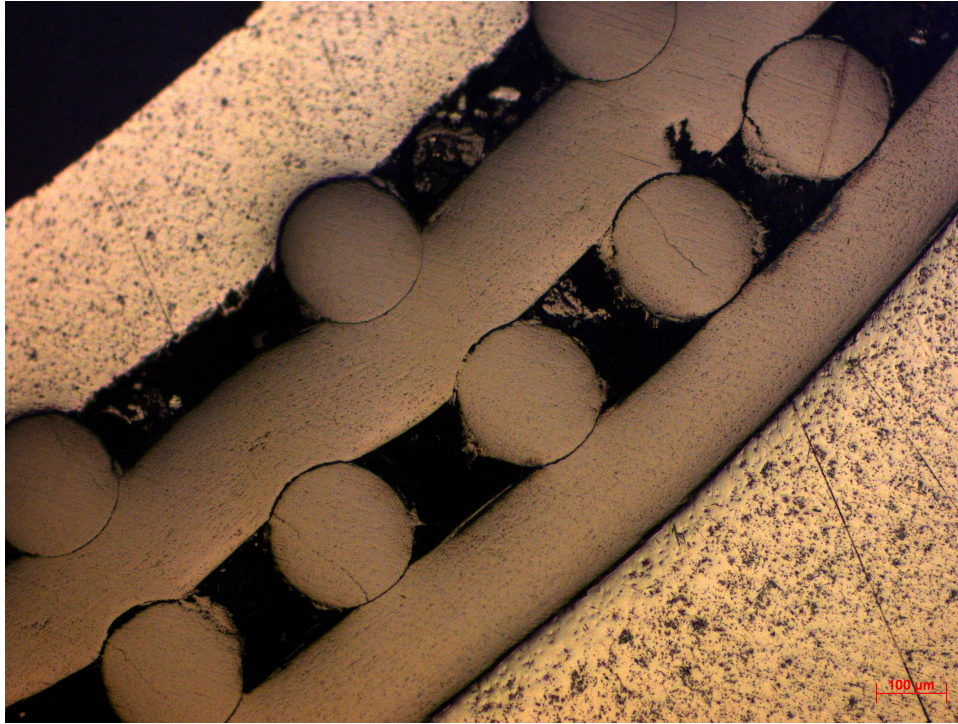


Figure 13 – Sample 1, Configuration 1 demonstrates fracture and cracking of W wire in contact with W rods.

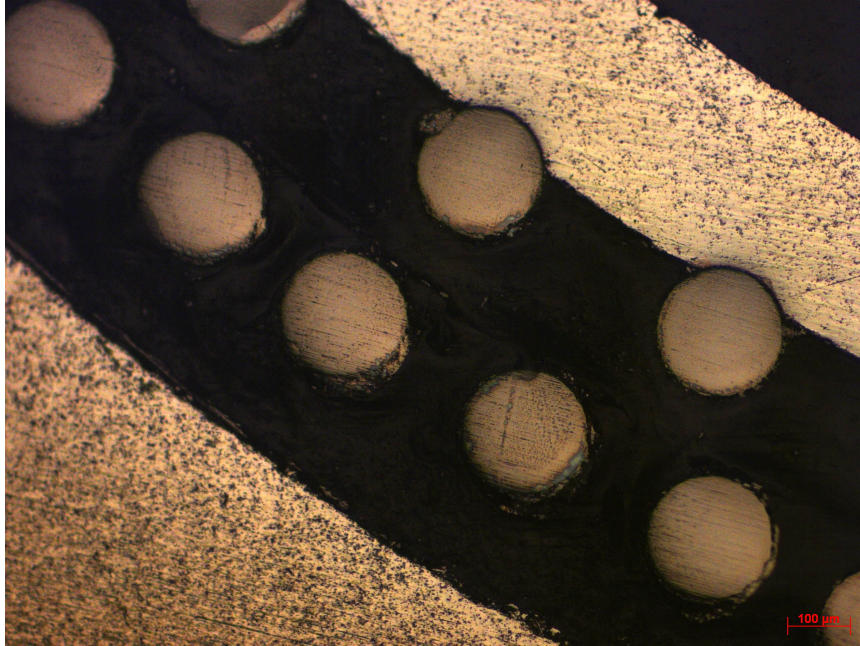


Figure 14 – Axial W rods are seen coming out of the page, they press against the inner layer of Al 6061-T6 during a CIP process in Sample 1, Configuration 1.

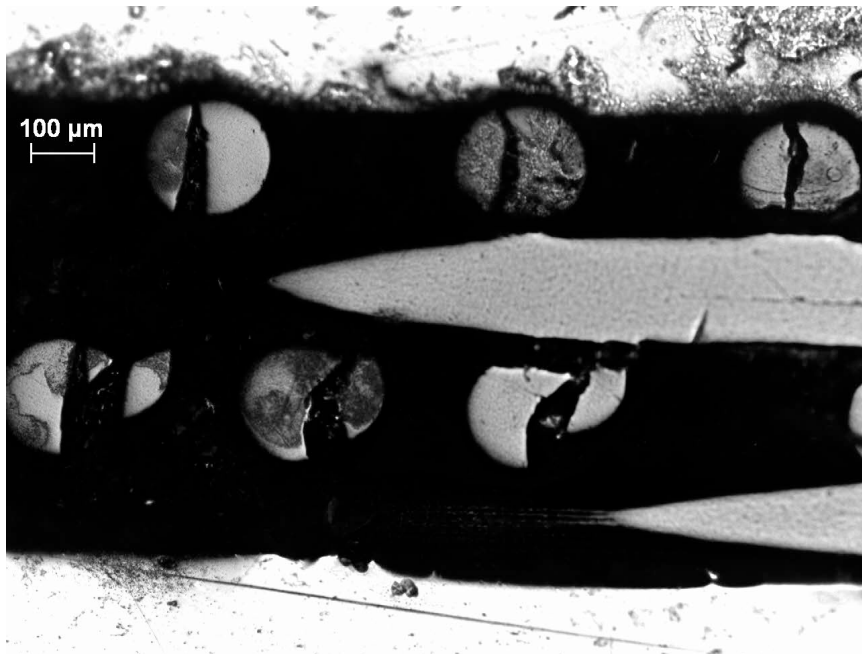


Figure 15 – A cross section with the hoop wires coming out of the page showing severe fracturing of radial wires with little deformation imparted to the axial rods.

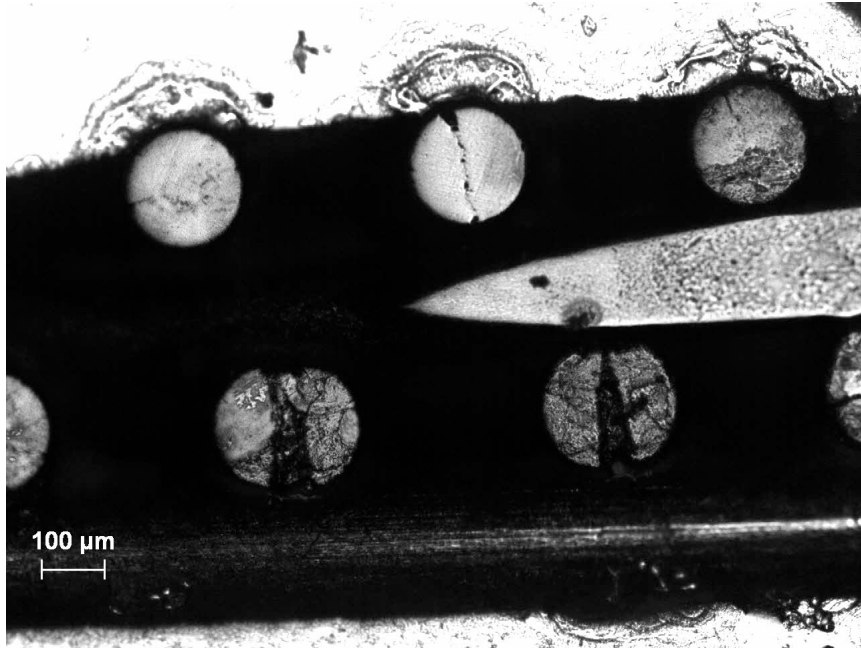


Figure 16 – A cross section with the hoop W wires coming out of the page showing the “Brazilian” fracture of hoop wire.

In an attempt to mitigate the fracturing observed in the first sample under CIP the sample was annealed to see if a softer aluminum would result in a more favorable CIP results. The annealing process was at 413 degrees Celsius for three hours with a slow cool down time period. Using the same procedure as described earlier, the sample was prepared for a CIP process at 345 MPa. After this second CIP process the outer diameter decreased from 10.8 mm to 10.6 mm. Unfortunately most of this deformation appeared not to be due to aluminum migration but from further fracturing of the W wires as seen in Figure 17. Thus, a CIP process did not ensure full density of composite and resulted in multiple fractures of W rods. For this reason a HIP process was pursued.



Figure 17 – Sample 1, Configuration 1 after annealing and a second CIP process at 345 MPa. Notice the decreased distance between the axial W layers due to the fracture of the separating W wires.

To prove that a HIP process can soften the aluminum layers such that they will viscoplastically flow around the W wires and W rods, sample 1 of Configuration 1, after 2 subsequent CIP treatments was prepared for a HIP process by placing the structure inside a Al 6061-T6 jacket. A vacuum was pulled and sealed with a weld as seen in Figure 18. The temperature and pressure history during the HIP treatment are presented in Diagram 2. The initial outer diameter of the jacket around the piece was measured to be an average of 14.82 mm, and after the HIP treatment the outer

diameter was measured to be an average of 14.67 mm. The deformation of the jacket around sample 1 can be seen in Figure 19.

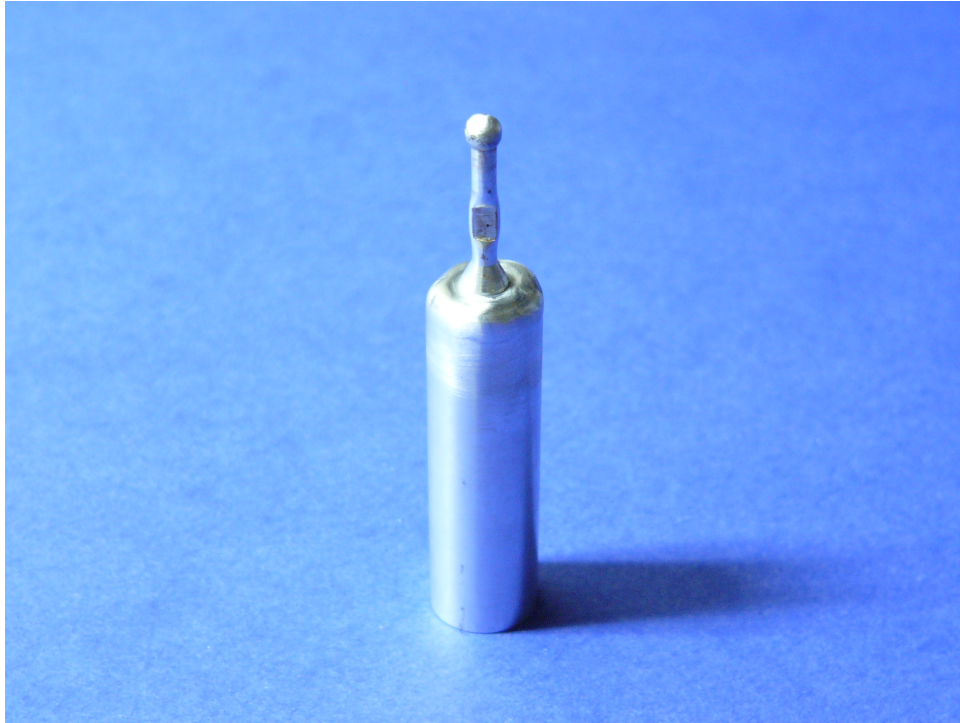


Figure 18 – Sample 1, Configuration 1 in a vacuum-sealed jacket of Al 6061-T6 for a HIP treatment.

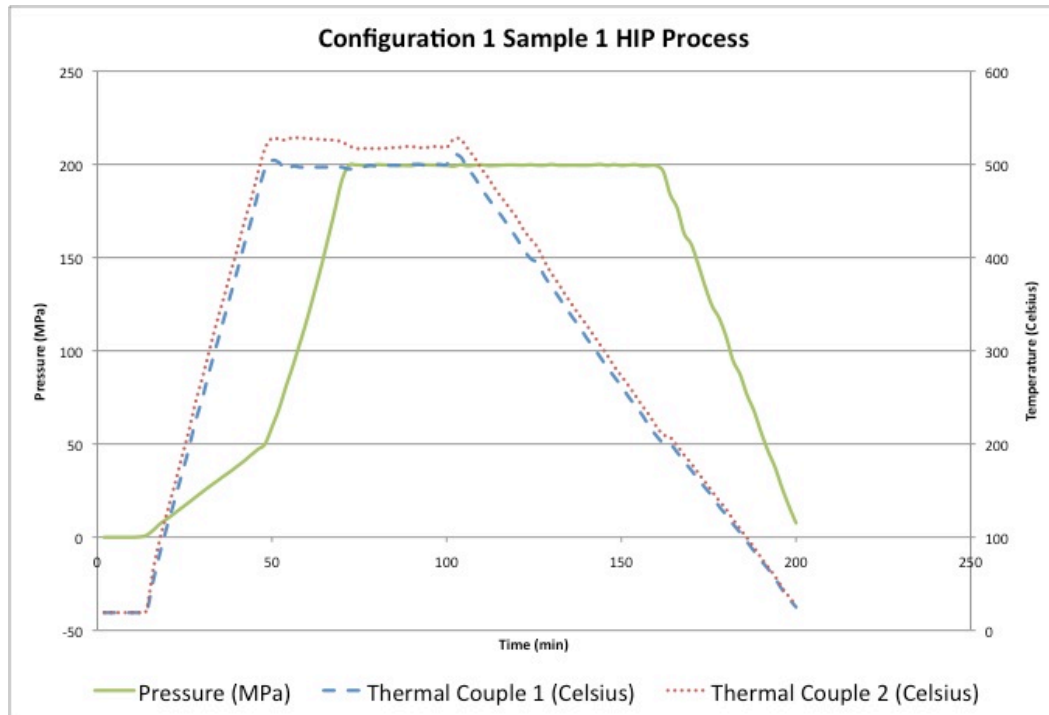


Diagram 2 - HIP process for Sample 1 of Configuration 1.

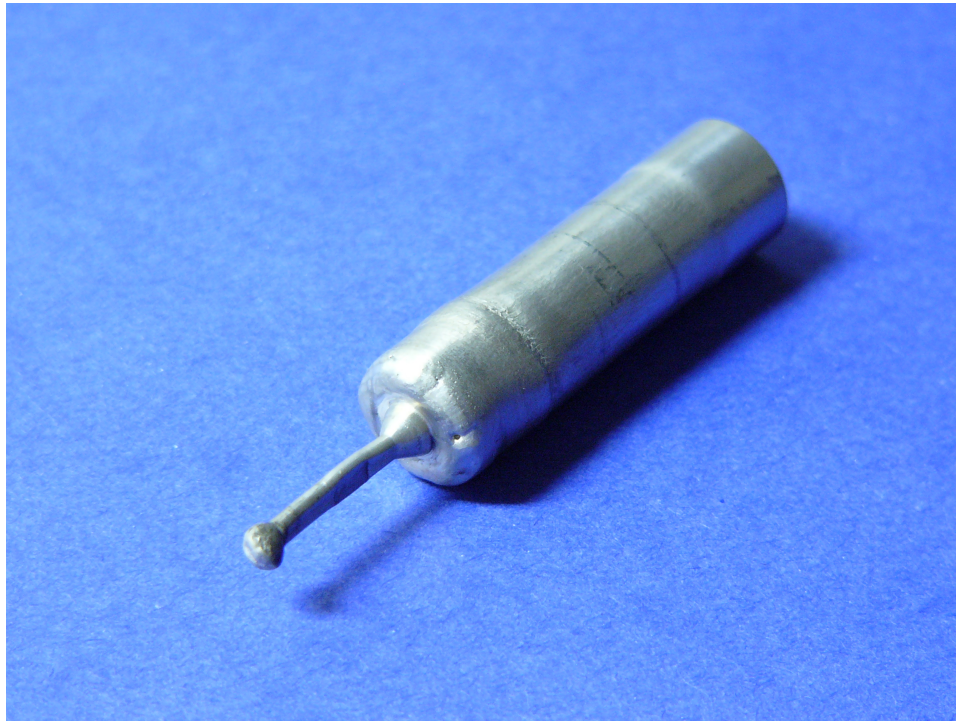


Figure 19 – Al 6061-T6 Jacket of Sample 1, Configuration 1 after the HIP treatment.

Cross sections were cut with an EDM and microstructures observed by an optical microscope. It was seen that the aluminum fully penetrated the W structure, even inside the cracks in the W fibers as seen in Figure 20. However small voids are seen between the aluminum layers in places where the layers failed to bond together as seen in Figures 21 and 22. This approach proved that a HIP process could migrate the aluminum such that an aluminum matrix would fully surround the W fibers, even in regions where the W layers were in contact. No reaction product between W and Al 6061-T6 was observed with a scanning electron microscope (SEM).

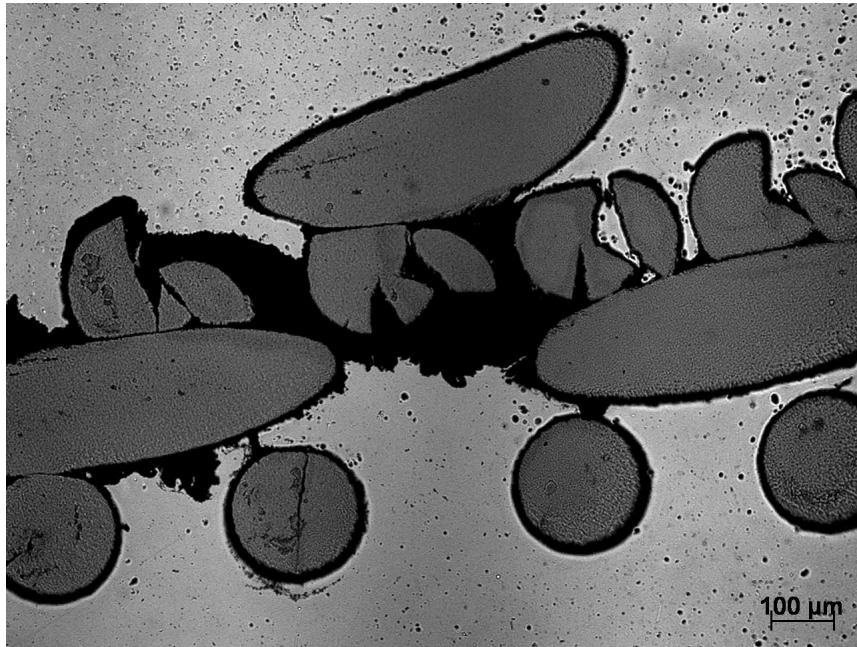


Figure 20 – A cross section of Sample 1, Configuration 1 after a HIP process with the axial rods coming out of the page. The compact W structure probably prevents flow of the aluminum during HIP, creating a void.

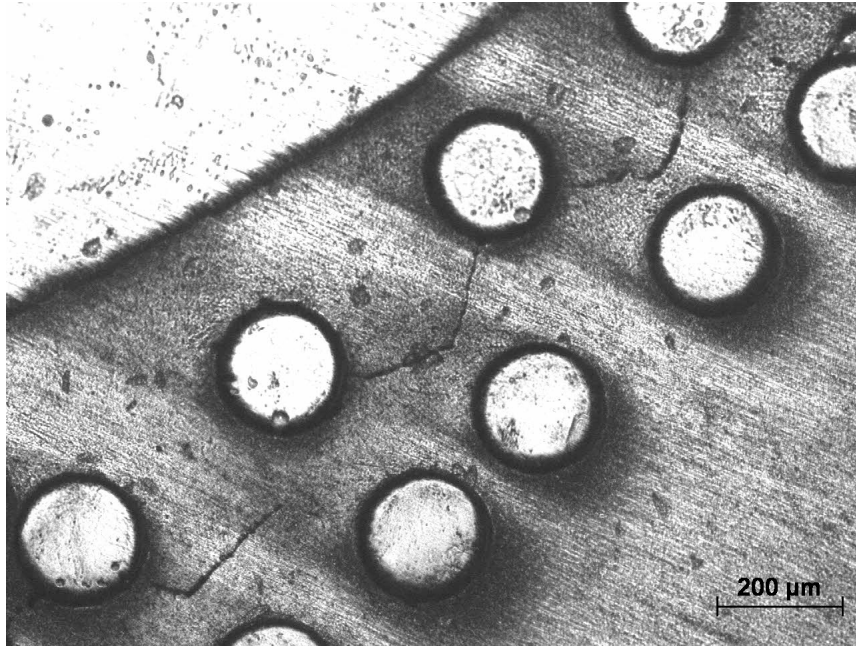


Figure 21 - A cross section of Sample 1, Configuration 1. Notice the voids between the layers of aluminum.

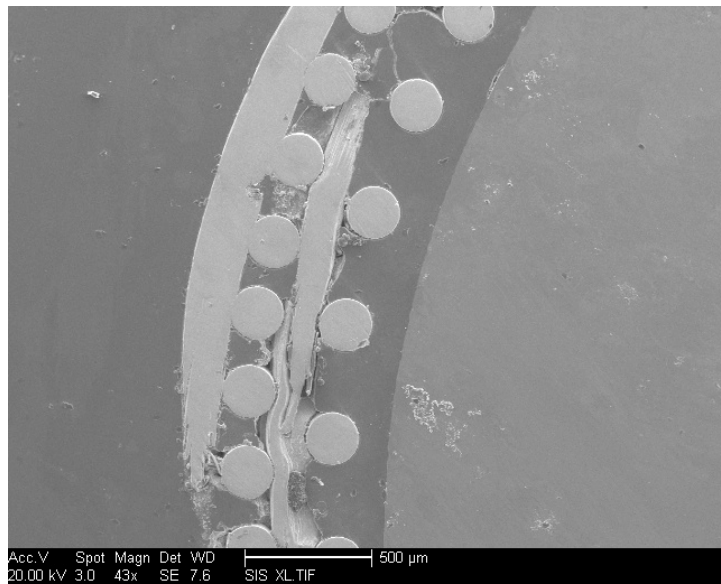


Figure 22 – SEM image of a cross section of Sample 1, Configuration 1.

3.4 - Second Sample of Configuration 1

A second sample in Configuration 1 was successfully created, and underwent multiple CIP processes. Both CIP processes were done at low pressures to avoid fracture of the W fibers, 20 MPa and 50 MPa, and resulted in no visible deformation of either component material. The sample was cleaned using sonication in acetone, resulting in severely dislocating the W structure in two of the three pieces of this second sample. After cleaning, the single remaining piece was encapsulated and unsuccessfully HIP treated due to a failed vacuum seal. The sample was cut out of the aluminum jacket and machined to fit into another aluminum jacket. A second successful HIP process was completed on sample 2 similar to sample 1. Again the Al 6061-T6 was seen to fully penetrate the W structure with no reactions occurring with the W. However, now with the hoop W wires preserved from fracture, buckling was observed in these W wires, distorting the entire W structure. This sample was machined to an inner diameter of 7.5 mm, an outer diameter of 9.57 mm with a density of 7.88 g/cc.

The second sample in Configuration 1 was prepared similarly as described in section 3.2. The spacing of all four W layers was built to closely match the desired geometry for Configuration 1. With the sample successfully built it was prepared as previously described in section 3.3 for a CIP process.

Sample 2 in Configuration 1 was CIP treated twice at different pressures, 25 MPa and 50 MPa. Before pressurizing the outer diameter of sample 2 was 11.17 mm,

and after pressurizing at 20 MPa the outer diameter was 11.11 mm. Further pressurizing at 50 MPa brought the outer diameter to 10.88 MPa. No plastic deformation of either component material is observed in the region around the W structure. Additionally no fracture of the W rods is seen in Figure 23. Thus, the pressure to cause “Brazilian” fracture in the W must be between 50 MPa and 350 MPa, and more studies must be conducted to find this critical pressure.

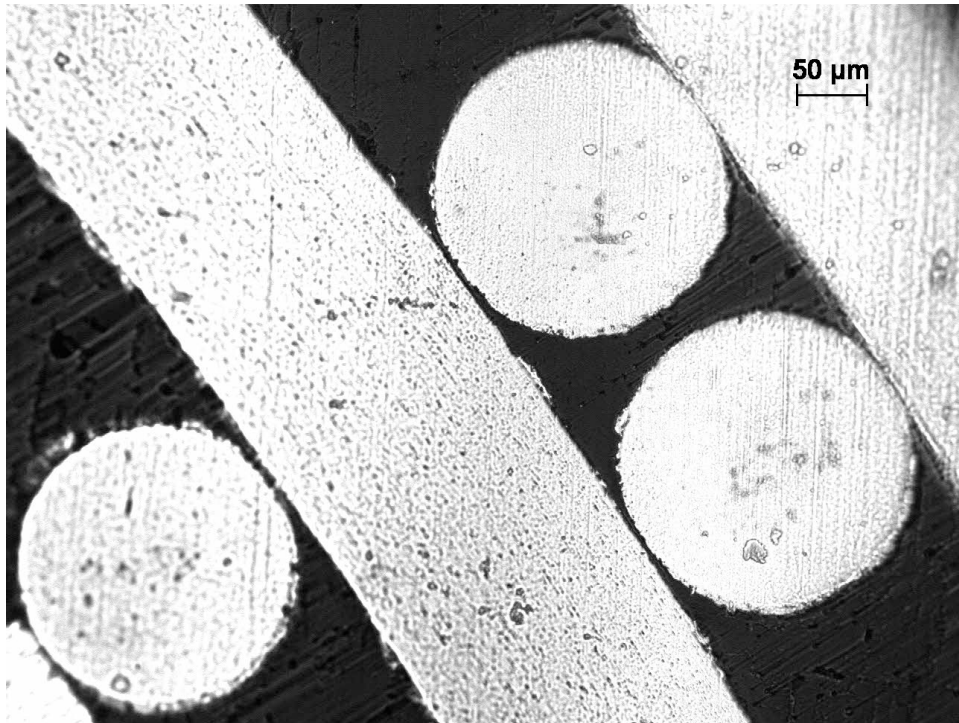


Figure 23 - Sample 2 after a CIP process at 50 MPa. No plastic deformation or “Brazilian” fracture is observed in the contact region between W fibers.

After the CIP processes sample 2 was divided into three approximately equal in length pieces. In an effort to polish the cross sections of the pieces for photography sonication cleaning at 40 kHz in acetone was attempted for over thirty minutes.

Sonication was thought to be necessary to remove small particles from the sample cross section and interior. Unfortunately two of the three pieces of sample 2 had unacceptable migration of the W structure as seen in Figure 24 after 20 minutes. The only surviving piece of sample 2 as seen in Figure 25 was prepared for a HIP process identically as sample 1. The partially exposed W wire in the outer hoop layer on this piece had to cut flush to the cross section before encapsulation.



Figure 24 - Failed piece of Sample 2, Configuration 1 after 40 kHz sonication in acetone for 20 minutes.



Figure 25 - Piece of Sample 2, Configuration 1 to be HIP treated after sonication in acetone for 20 minutes at 40 kHz.

Sample 2 underwent a nearly identical HIP process as sample 1 as seen in Diagram 3. Minor modifications were made to heat and cooling rates to minimize time at 500 degrees Celsius, thus decreasing the risk of reaction between W and Al 6061-T6. The first HIP treatment ended in failure as the vacuum seal broke prior or during the process, leading to uniform pressurization inside and outside the sample and thus preventing aluminum migration. The walls of a small tube as seen in Figure 26 used to create the vacuum seal is believed have been pushed into the Al 6061-T6 jacket due to the annealing of the aluminum jacket and thus decreasing the mechanical strength of the jacket. The external pressure caused a critical mismatch in strain where the thick jacket cap connected to the thin tube. The tubes outer diameter decreased more than the inner diameter of the aluminum jacket's cap, and under this high

pressure the tube was then pushed into the jacket, creating a region of high strain and subsequent fracture. The sample was mechanically removed from its encapsulating aluminum jacket, and placed inside a new Al 6061-T6 jacket. The second HIP attempt was a success. As observed in sample 1 no reaction was detected in sample 2 between the W and Al 6061-T6 with an SEM. Also, as seen in sample 1 complete penetration of the W structure was accomplished in sample 2 as seen in Figure 27. Voids observed are believed to be due to the underlying W wire close to the plane under observation. Fracture of some W rods due the HIP cycle were observed in these cross sections as seen in Figure 28. Of great concern for this sample and HIP method is the observed buckling of the W wires as seen in Figure 29. This is believed to be due to the external pressure applied to the wires as the outer Al 6061-T6 tubes plastically flows around the wires. In the beginning of the HIP cycle the W wire does not have adequate support from underneath or from the deforming Al layer and buckles. Buckling is observed in the plane of the cross section of the sample and in the planes parallel to the axis of the sample, into the empty space available in the W structure.

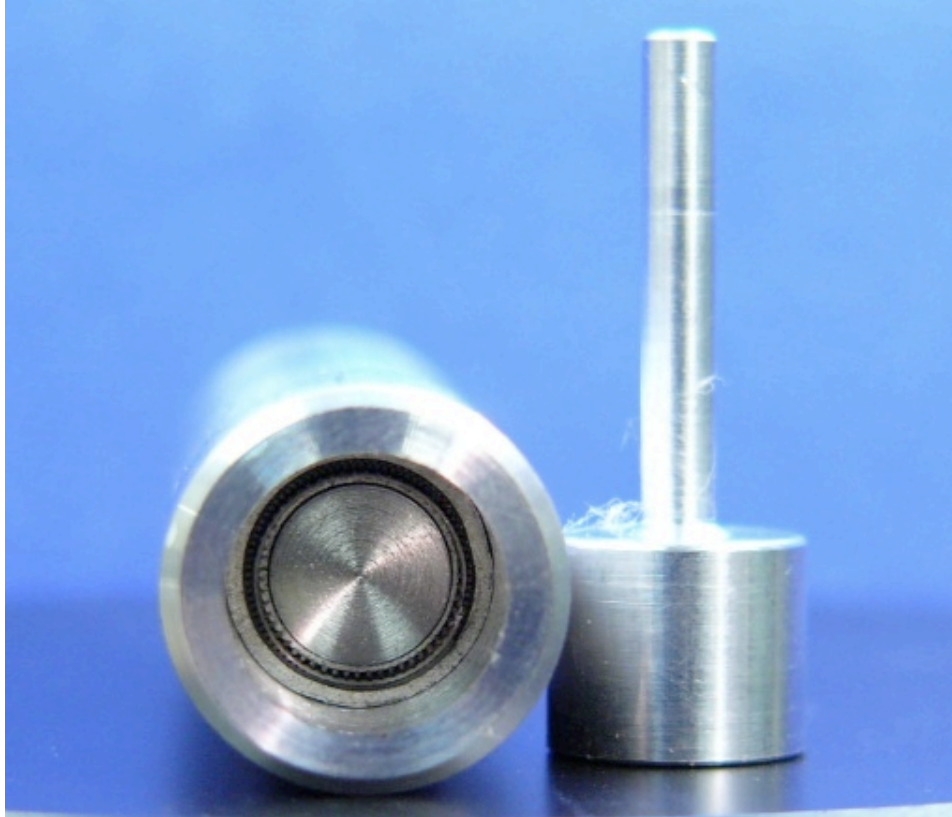


Figure 26 - Sample 2, Configuration 1 prepared inside of a Al 6061-T6 jacket for the failed HIP cycle. Notice the narrow vacuum tube and the geometrical discontinuity to the jacket cap that lead to the failure of the vacuum seal. The wall of the tube was pushed down into the large aluminum cap, leading to fracture.

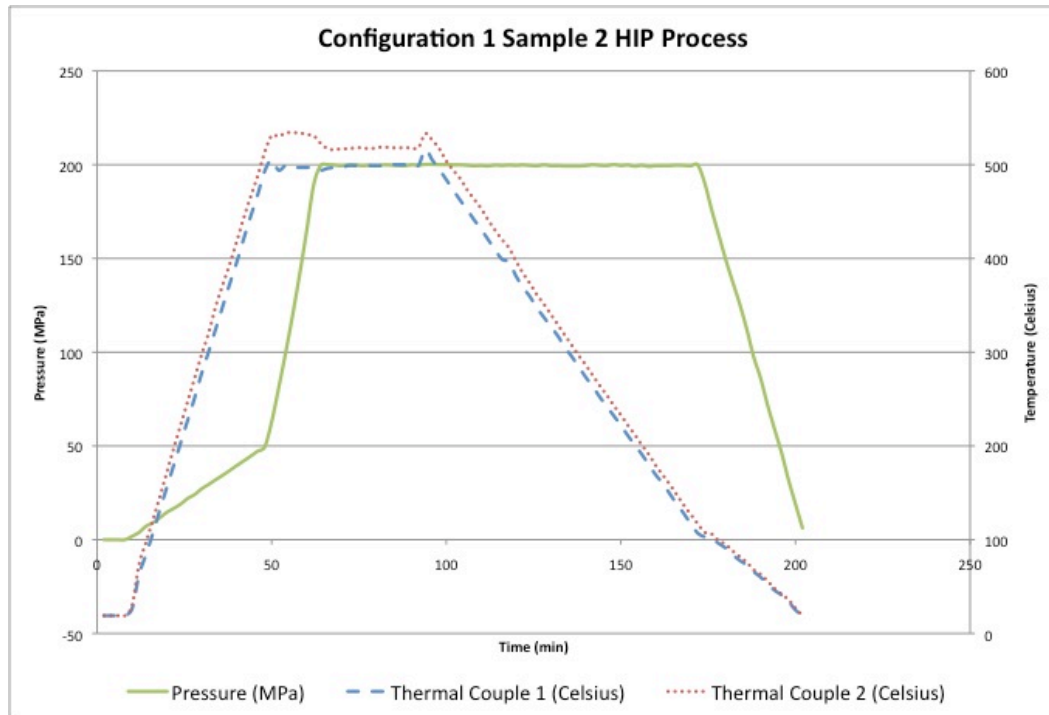


Diagram 3 - Successful HIP process for sample 2, Configuration 1.

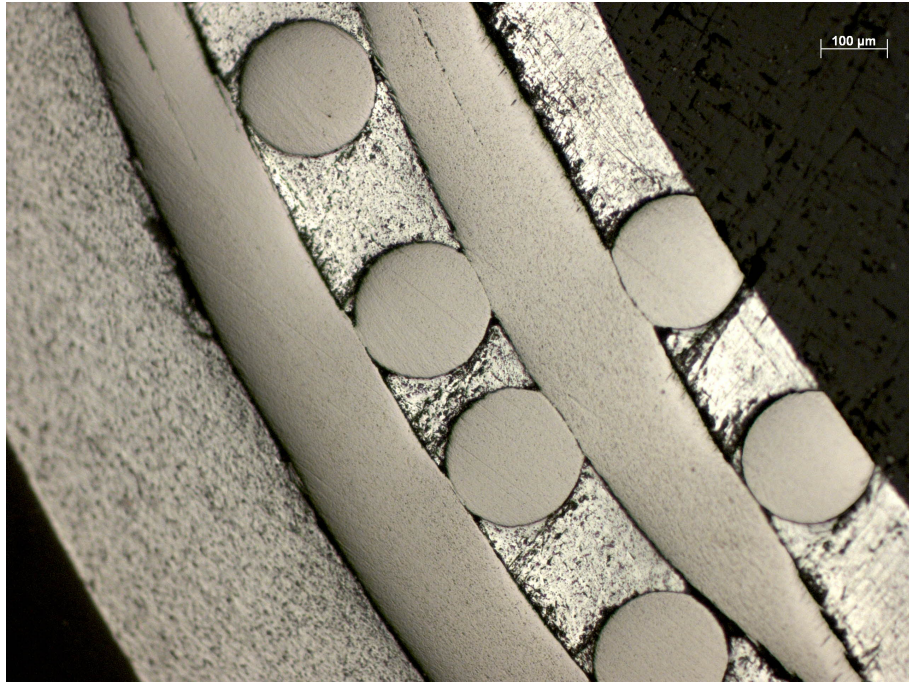


Figure 27 - Sample 2, Configuration 1. Notice full penetration of Al around the W rods and wires with no Al-W compound created.

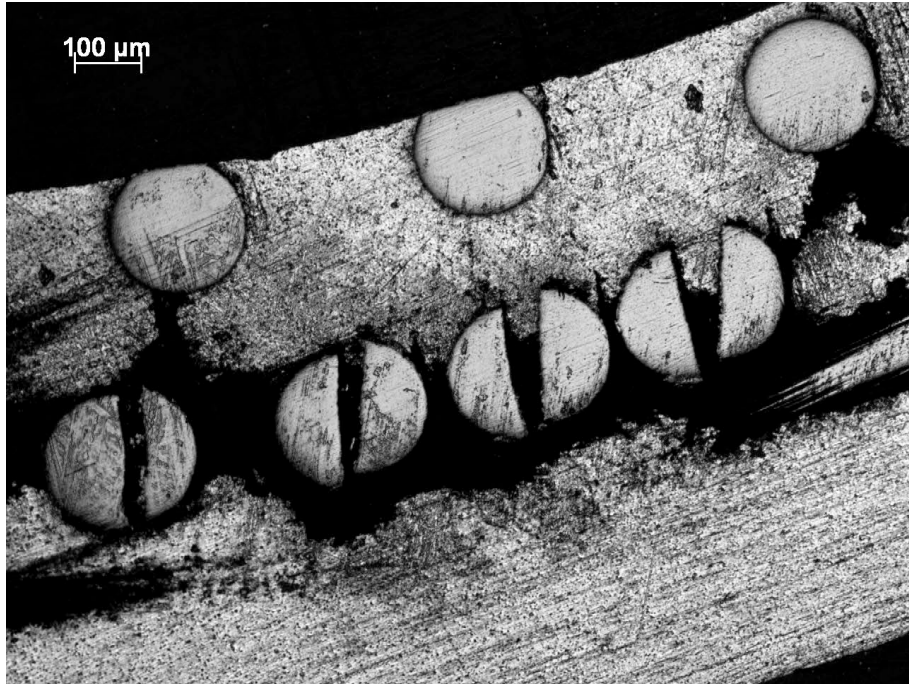


Figure 28 - Sample 2, Configuration 1. “Brazilian” fracture of W rods with aluminum filling the cracks in W observed after the HIP process.

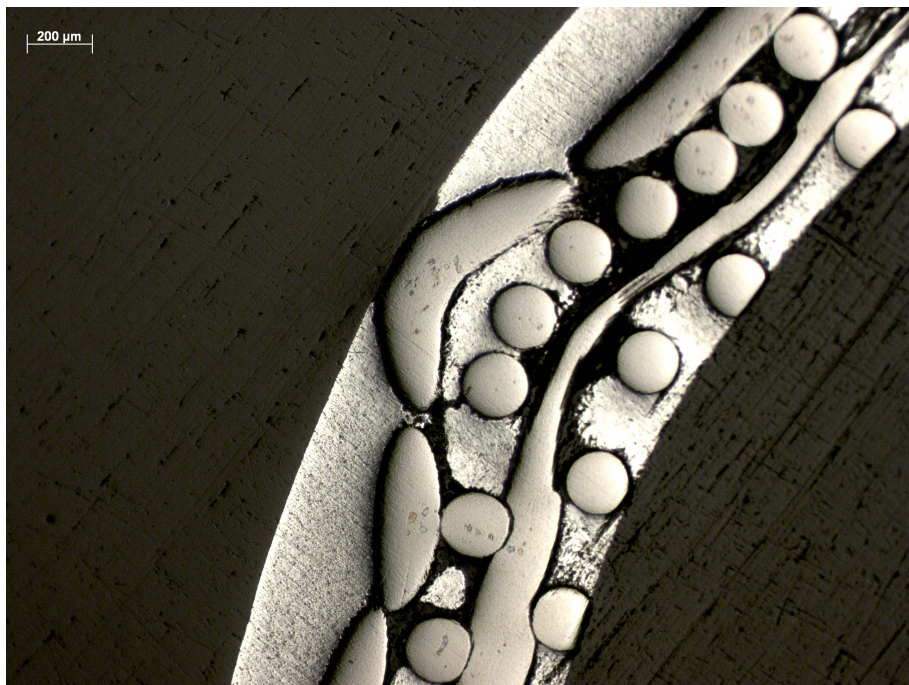


Figure 29 - Sample 2, Configuration 1. Buckling was observed in plane perpendicular to axis and in axial plane.

After the successful HIP process it can be seen in Figures 27-29 that sample 2 in Configuration 1 was machined to the desired geometry to achieve a density close to that of steel, 7.8 g/cc. The final dimensions of sample 2 were a length of 44.02 mm, an inner diameter of 7.5 mm, and an outer diameter of 9.57 mm. These dimensions result in an outer layer of aluminum, approximately 230 μm thick to accommodate the buckle in the outermost W wire. Large deviations in the spacing, as listed in appendix A, of sample 2 of Configuration 1 could be due to the displacement of W fibers during sonication cleaning in acetone, and buckling of the W during HIP.

It is thought that if this sample were to be actually tested with an inner explosive the outer layer of Al will fail to heat up significantly and spall off the sample in large chunks not suitable to react with the oxygen in the air. More aluminum should be on the inside surface of the sample such that the aluminum will heat up due to the reflecting shock waves and being moved through the W structure to be shredded into the desired small particles. Aluminum outside of the W structure is wasted material. Buckling interferes with machining of the part, but may not be a problem for the performance.

3.5 - Configuration 2 and the Prevention of “Brazilian” Fracture

Due to the issue of fracturing in the W rods and wires Configuration 2 was designed and the use of a CIP process abandoned. Based on the experience of the first two samples, it was observed that plastically deforming the structure provided stabilization for a degassing process was not only detrimental but also unnecessary.

Mechanical clamping of the ends of the sample or preliminary CIP may be needed to resist motion of the W fibers (e.g. due to the non-uniform thermal expansion of all the components) to keep the W architecture intact during the HIP process. Degassing is also not expected to be an issue for these components or parts, unlike powders for which in solid state processing a degassing process would remove some of the powder if not CIP treated beforehand.

Application of high pressures while the structure is hot may allow the W to be more ductile since the brittle to ductile transition temperature (BDTT) for W is in the range of 97 to 197 Celsius for single crystalline tungsten [18]. It is not obvious that the fracture toughness or BDTT changes greatly for poly crystalline W [19]. Unfortunately the greatest ductility for single crystalline tungsten occurs at the BDTT since the BDTT in W is strongly related to dislocation mobility. The movements of dislocations need energy, leaving less energy at the crack tip, and the dislocations act as singularity points to shield the crack from loading. The area above the BDTT plasticity is thought to restrict dislocation motion, thus lowering the fracture toughness [18]. The low ductility and low toughness of W fibers at room temperature definitely aided in the fracturing of the W rods and wires along with the stress concentrations due to the point contact of the W fibers with each other, and the “Brazilian”-like loading between the W rods and contacting W wires. Pressurizing the components in a HIP process should help avoid fracturing the W fibers due to the fibers being more ductile at 500 Celsius but mostly due to the encapsulation effect of the W rods and wires by the Al matrix.

In “Brazilian” fracture the ideal opposite point loading on the fiber cross section allows the fiber to fail much earlier than expected due to the resulting tensile stresses developed in the center plane. It is thought that as the loads are spread out over an area the resulting magnitude of the central tensile stresses can be reduced. Thus Configuration 2 was developed to place ductile aluminum between all the W layers. It was thought the aluminum would deform around the fibers during pressurization, providing an encapsulating effect and helping to avoid the point loads conducive to “Brazilian” type fracture.

To process a sample of Configuration 2, many of the manufacturing techniques and dimensions discussed in section 3.2 were used. To help prevent stress concentrations from forming where the W wires and rods contacted each other, thus contributing to the fracture of the W fibers, layers of aluminum would be placed between each layer of W. These layers of Al 6061-T6 were meant to plastically deform around the nearby W fiber to carry enough of the pressure load to reduce the load on the W, thereby preventing fracture and possibly even buckling in the W mesostructure. In total five Al tubes were machined and etched to the proper dimensions to fit in between a now slightly expanded W structure as can be seen in Figure 30. The inner diameters and outer diameters of the Al 6061 tubes from the inside to the outside of the complete structure were as follows: 7.5 mm and 8.1 mm, 8.6 mm and 8.88 mm, 10.08 mm and 10.36 mm, 10.84 and 11.12 mm, and 11.6 and 12.2 mm. Each layer of Al 6061-T6 and W was applied in successive order with the first Al 6061-T6 tube being fitted to a stainless steel core in preparation for a HIP

process of an outer diameter of 7.4 mm. The W was locked into place with superglue as the next aluminum tube was applied. This superglue region was removed after the assembly was completed and the outermost aluminum tube was mechanically clamped. The mechanical clamping moved the layers of W rods from the prescribed locations, thus changing initial spacing. Mechanical agitation also played a roll in distorting each layer of W during the construction process since only mechanical clamping was keeping the fibers in place. The final result of this process to build sample 1 of Configuration 2 can be seen in Figure 31.

This sample was directly prepared for a HIP process similar to what was done for samples 1 and 2 of Configuration 1. An Al 6061-T6 jacket was made to encapsulate the sample in which a vacuum of four microns was drawn before being mechanically sealed and welded. The sample of Configuration 2 to be HIP treated can be seen in Figure 32. As can be seen in Diagram 4 that the HIP process was slightly adjusted. Pressure was initially raised to 10 MPa in an attempt to keep the structure tight as the temperature was increased that would cause the structure to expand, thus possibly allowing further deterioration the initially assembled W architecture. This pressure was below the pressures observed for sample 2 in Configuration 1, 20 MPa and 50 MPa that proved no fracturing within the W fibers would be induced. Pressure and temperature were increased independently, 20 Celsius/min and 6 MPa/min respectively. Thermal couples within the furnace (at the bottom and middle) controlled the HIP cycle and demonstrated the fluctuations in temperature. It is not believed these fluctuations caused any significant effect on the specimen.



Figure 30 - Aluminum alloy 6061 tubes used to assemble Sample 1, Configuration 2. The inner and outer diameters of the tubes from the smallest to largest are: 7.5 mm and 8.1 mm, 8.6 mm and 8.88 mm, 10.08 mm and 10.36 mm, 10.84 and 11.12 mm, and 11.6 and 12.2 mm.



Figure 31 - Fully assembled Sample 1, Configuration 2 before vacuum encapsulation.



Figure 32 - Sample 1, Configuration 2 prepared within an Al 6061-T6 vacuum-sealed jacket for a HIP process.

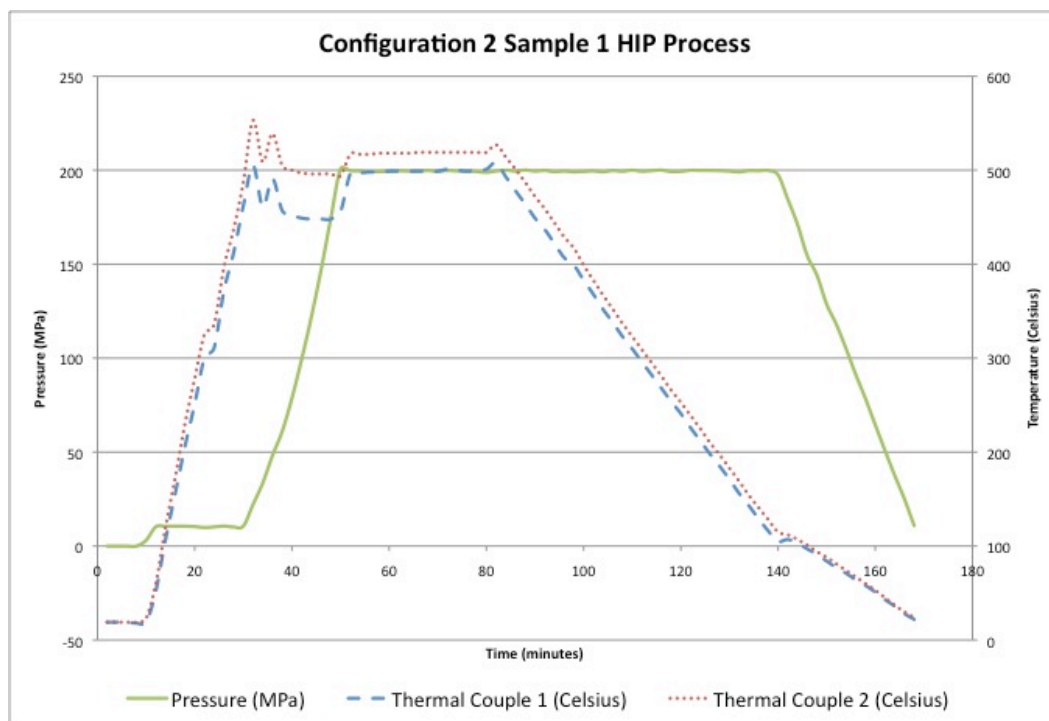


Diagram 4 - HIP process for Sample 1 of Configuration 2.

Although the HIP process was successful, the results of the composite's structure were disappointing. After the HIP process a severe bump was seen running the entire length of the sample (Figure 33). The average diameter of the HIP treated sample not including the bump was 14.38 mm with the bump averaging an outer diameter of 15.84 mm. With a cross sectional cut, as can be seen in Figure 34 this bump was a result of severe buckling of the W wires and subsequently of the aluminum tubes. Although aluminum was forced through the entirety of the W structure the aluminum layers did not completely bond to one another during the HIP process, and in some cases voids can be seen (Figure 35). Additionally the W rod architecture and spacing was largely disturbed in this Configuration probably due to buckling of the aluminum tubes and W wires distorting the entire W structure. This irregular spacing of the W rods can be seen in Figures 34-36.



Figure 33 - Sample 1, Configuration 2 after the HIP process. Notice the bump along the entire length of the sample.



Figure 34 - Cross section of Sample 1, Configuration 1 after the HIP process. Notice the irregular W rod spacing and extreme W structure deformation at the top of the sample.

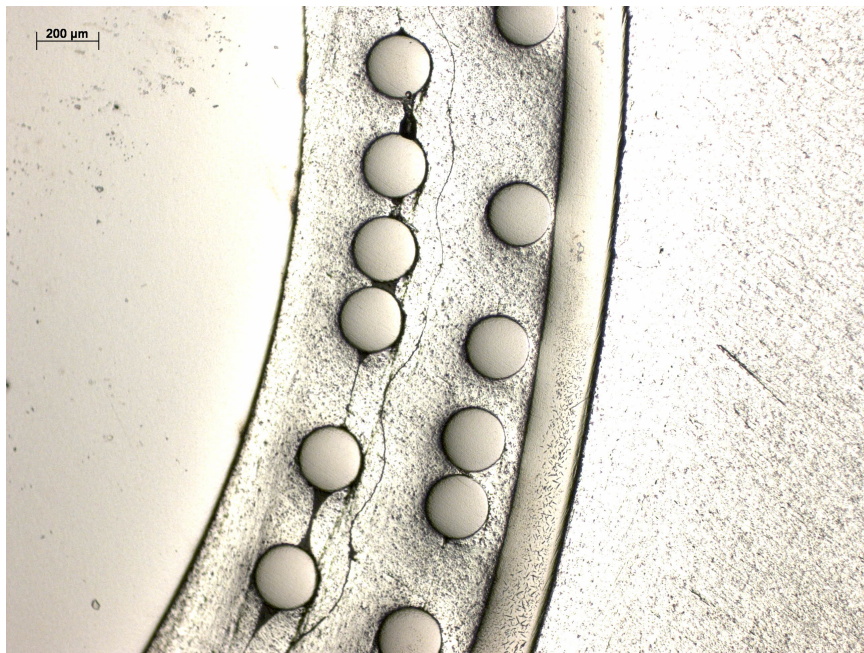


Figure 35 - Sample 1, Configuration 2, optical image. Notice the aluminum fully penetrated the W structure but the individual layers of aluminum did not bond, leaving behind local voids.

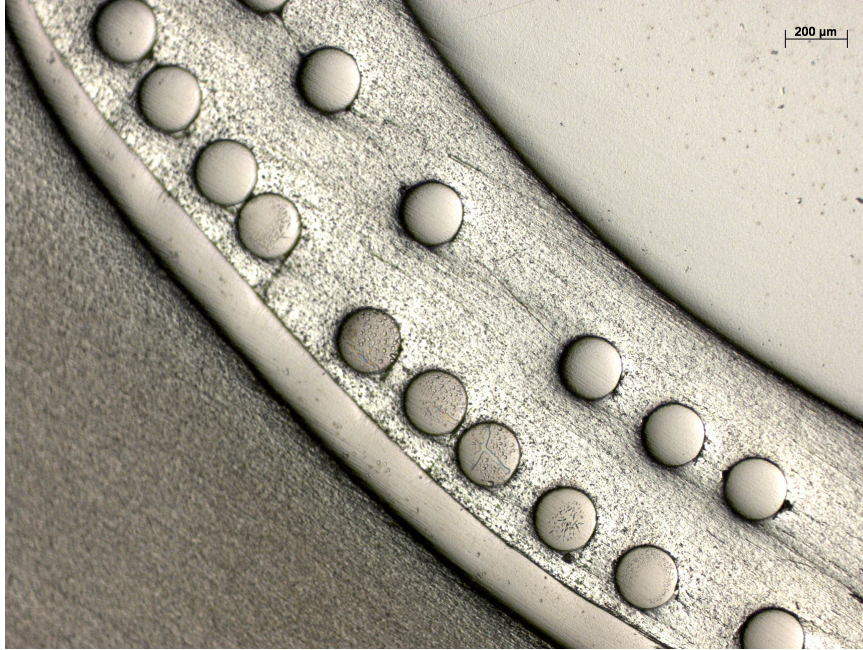


Figure 36 - Sample 1, Configuration 2, optical image. Notice the non-uniform spacing between W rods.

Although Configuration 2 prevented fracture of the W fibers their buckling was exacerbated as seen in Figures 34 and 37. The Al 6061-T6 layers buckled in addition to the W wires. It is believed that this buckling was enhanced by the soft, hot aluminum layers failing to support the W wires while the external pressure was rapidly increased. The aluminum did not have enough time to viscoplastically flow into the W structure to support it before high pressures were applied. The W wires in the hoop direction were overloaded during the rapid pressure increase, resulting in all the layers buckling. This Configuration of Al 6061-T6 layers between the W layers shows that support for the W structure is weaker during the HIP process as compared to Configuration 1 where the W layers directly load each other. The aluminum layers between the individual W layers introduce more open space conducive for buckling of W wires in the hoop direction.

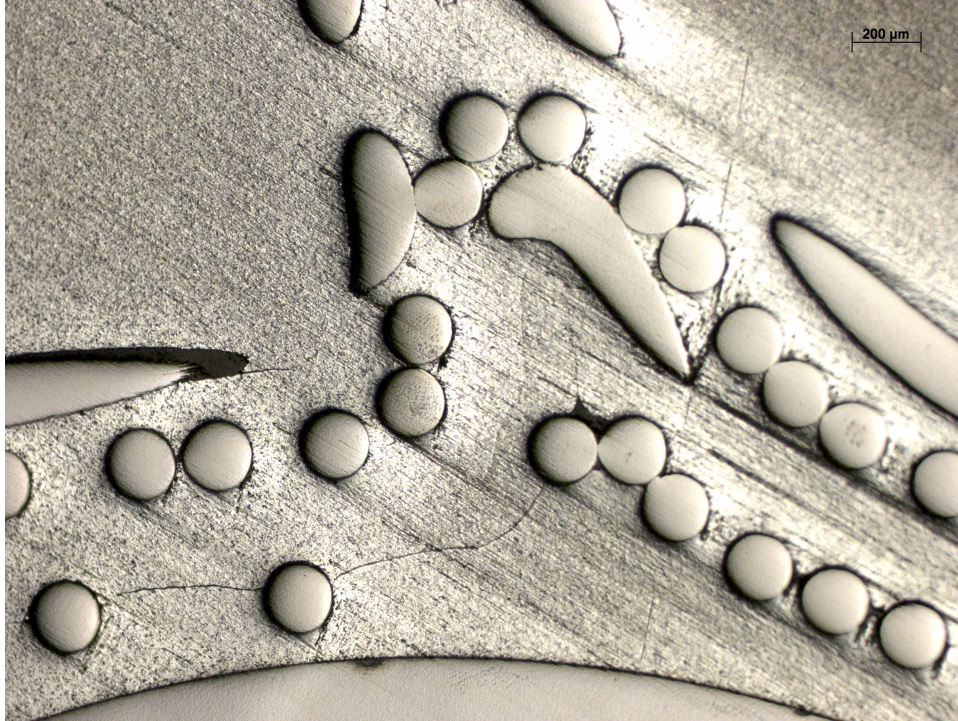


Figure 37 – The buckling region in Sample 1, Configuration 2, optical image.

Section 3.6 - Annealing of the Aluminum

It was expected that at 500 Celsius the Al 6061-T6 tubes would lose their initial mechanical strength and stiffness due to annealing. It has been measured in a study of aircraft landing gear made of aluminum alloy 6061-T6 that flash annealing of the metal can occur [20]. From this study it can be seen that after a single exposure of intensely hot gas at 482.2 Celsius, the yield strength of the rectangular Al 6061-T6 tube dropped from approximately 270 MPa to 50 MPa [20]. The samples were allowed to cool before undergoing tensile testing to ascertain mechanical strength. Elongation was seen to exceed 20% after approximately 12 minutes at 482.2 Celsius

[20]. Our HIP treatments reach a maximum temperature of 500 Celsius and were held at the maximum temperature for at least one hour. Since aluminum conducts heat very quickly and our total time at the maximum temperature far exceeds the time necessary to flash anneal Al 6061-T6 it was expected that the aluminum tubes would viscoplastically deform at the minimum pressure possible to completely penetrate the W structure with minimal disturbance to the structure.

The Vickers hardness measurement of the aluminum alloy 6061-T6 tube sample was 957 MPa in the as received condition and decreased to 407 MPa after a HIP treatment very similar to what is shown in Diagram 2, proving our HIP cycle does anneal the Al 6061-T6 as listed in Table 4. Tube 1 was not cooled slowly but quenched by cooling under pressure then releasing pressure quickly. The aging applied here to a piece of sample 1 of Configuration 2 at 160 Celsius for 18 hours in a vacuum furnace, however, did not return the aluminum alloy to the T6 condition but did succeed in increasing the hardness of the matrix metal. The softening of the aluminum matrix is critical for the formation of our composite since the reduced mechanical strength of the aluminum matrix would cause its viscoplastic deformation at lower pressures. With reduced pressures required to migrate the aluminum through the W structure the W fibers themselves would have to carry less load, and less load will reduce the risk of bucking and fracture of the W fibers. Additionally it is thought that a weaker aluminum tube would deform to a greater degree with increased pressure to push aluminum into all of the available space within the W fiber structure.

Table 4 – Vickers hardness Results after HIP treatment and aging Al 6061-T6 with 20 kgf.

Sample / Hardness	Location	Vickers Hardness (HV 20)	Hardness (MPa)
Al 6061-T6 Tube as Received.	Cross Section	97.6	957.13
Al 6061-T6 Tube 1 After HIP (Diagram 2)	Cross Section	55.4	543.29
Al 6061-T6 Tube 2 After HIP (Diagram 2)	Cross Section	41.5	406.98
Sample 1 Configuration 2 After HIP (Diagram 4)	Al 6061 Jacket Cross Section	46.5	456.01
	Al 6061 Inside Tube Cross Section	47.7	467.78
Sample 1 Configuration 2 After HIP (Diagram 4) and Aging at 160 Celsius for 18 hrs.	Al 6061 Jacket Cross Section	56.6	555.06
	Al 6061 Inside Tube Cross Section	57.0	558.98

3.7 - Samples 3 & 4 of Configuration 1

A third and forth sample in Configuration 1 was successfully created, and underwent a single HIP process. The HIP was successful; resulting in a well-preserved W structure, full penetration of the aluminum in the W structure, no fracture of the W rods and no reaction between the W and Al 6061-T6. However due to

externally pressurizing these samples buckling of the W wire was induced. Sample 3 in Configuration 1 was machined to the desired dimensions of two 25 mm pieces, each with inner diameters of 7.42 mm. The outer diameters and densities of the pieces varied with one sample having an outer diameter of 9.28 mm and a density of 8.45 g/cc and the other sample measuring 9.13 mm and 8.67 g/cc respectively. Sample 4 in Configuration 1 was machined to the desired dimensions of an inner diameter of 7.46 mm, outer diameter of 9.67 mm, and a density of 7.87 g/cm³.

The third and fourth samples in Configuration 1 were prepared identically as described for sample 2 in Configuration 1, but with the fourth sample having one difference. The inner Al 6061-T6 tube was thicker with an inner diameter of 6.985 mm as opposed to 7.5 mm while maintaining the same outer diameter of 8.1 mm. This extra thickness of the inner Al 6061-T6 tube in sample 4 was done to see if more aluminum can be left on the interior of the final cylindrical sample. The spacing of all four W layers was built to closely match the desired geometry for Configuration 1. With the samples successfully built, it was prepared as previously described for a HIP process with a smaller diameter of steel core to support sample 4.

Samples 3 and 4 underwent a different HIP process than the previous samples as seen in Diagram 5. To keep the W structure tight with the aluminum tubes under heated conditions the pressure was first raised to 20 MPa. As seen from the CIP experiments on sample 2, 20 MPa does not induce plastic deformation or fracture in any of the components in the composite. Temperature was then increased to 500 Celsius while at this low pressure. Unlike the previous samples that raised pressure at

a rate of 6 MPa/min, samples 3 and 4 in Configuration 1 were pressurized at lower rate of pressurization, 1.5 MPa/min to the same maximum pressure of 200 MPa. This was to allow the Al 6061-T6 to viscoplastically flow into the W structure at the lowest possible pressure, thus allowing the W wire to be supported by the aluminum matrix at higher pressures, thus possibly avoiding buckling and fracture during the penetration. Ductility in the W fibers should also help distribute the pressure loads to prevent “Brazilian” fracture. The initial outer diameters of the Al 6061-T6 jacket was an average of 15.84 mm and 15.87 mm for samples 3 and 4 respectively, and after the HIP process the outer diameters decreased to a range between 14.82 mm and 15.37 mm. The wider diameters were due to a buckle induced in the W wire layers.

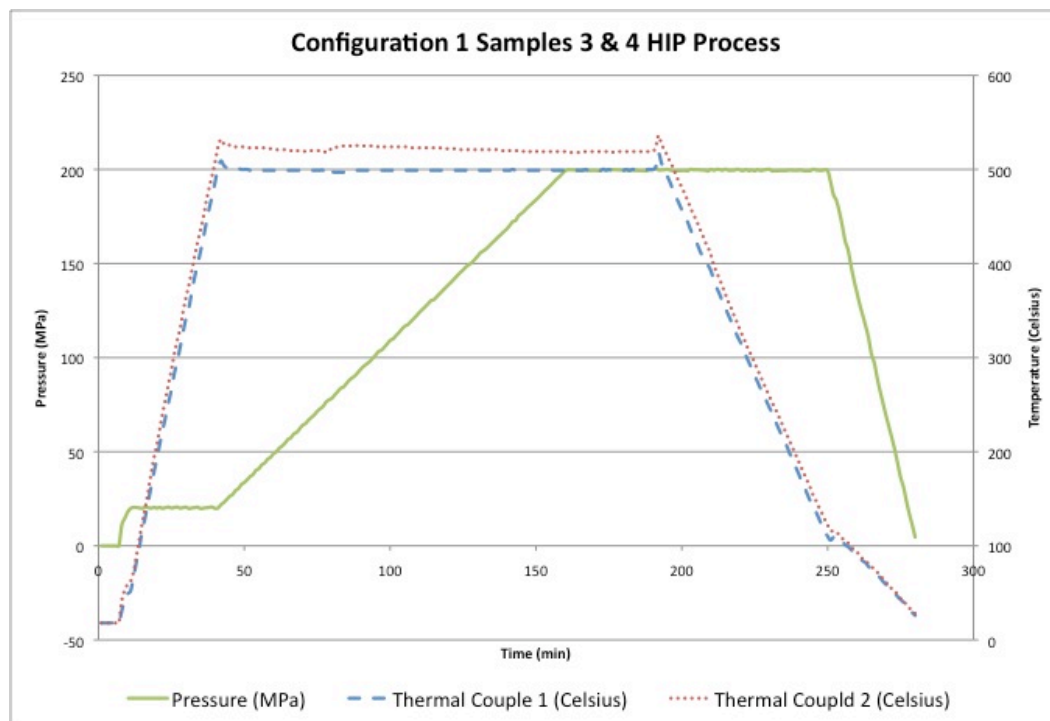


Diagram 5 - HIP process for Samples 3 and 4 in Configuration 1.

Overall samples 3 and 4 were a relative success as seen in Figures 38 and 39. Aluminum fully penetrated the W structures with no voids visible in the sample cross sections as seen in Figures 40 and 41. No fracturing of the W rods was seen, and the W structure was remarkably preserved as seen in Figures 42 and 43. The spacing of the rods was a much greater improvement from the previous samples as tabulated in appendix A. This is further evidence that a CIP treatment is actually harmful to the creation of this composite, and should be avoided. The non-uniform flow of aluminum into the W structure or the non-uniform thermal expansion of the structure changes the W structure's spacing. However the slow flow of aluminum into the W structure is better suited to not disturbing the W rod spacing and protecting the W wire from severe buckling as seen in the sample of Configuration 2.

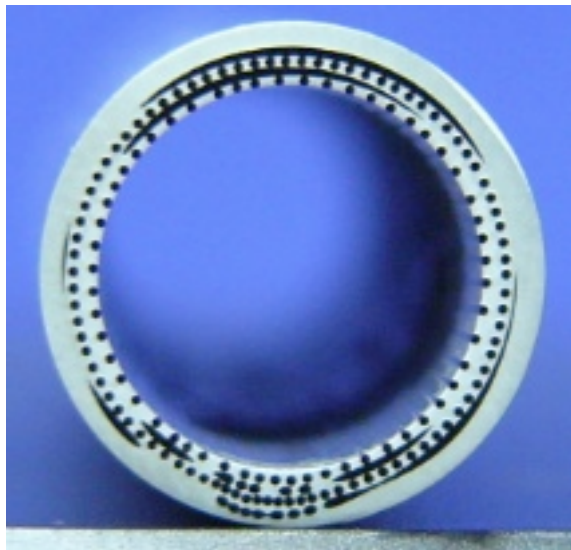


Figure 38 - Overall cross-section of Sample 3, Configuration 1. Notice buckled region at the bottom of the sample.



Figure 39 - Overall cross-section of Sample 4, Configuration 1 with a thicker inner tube. Notice buckled region at the bottom of the sample.

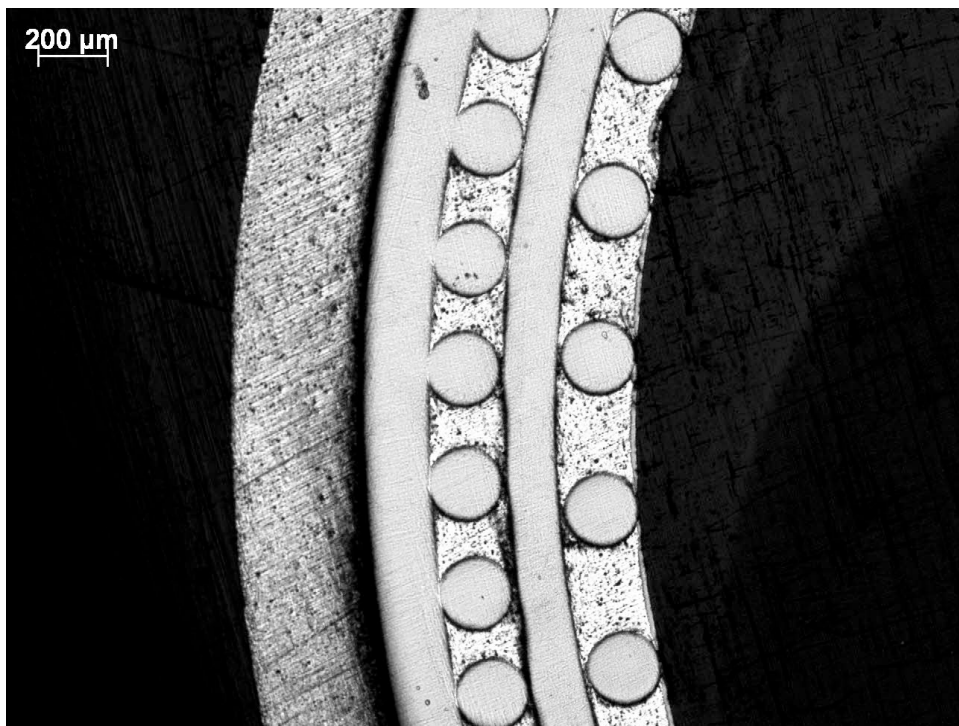


Figure 40 – Sample 3, Configuration 1, optical image. Notice the regularly spaced W rods and full penetration of aluminum inside the W structure.

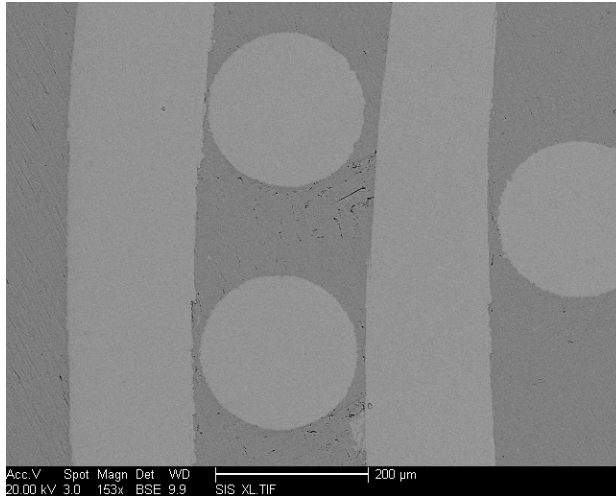


Figure 41 - SEM picture of Sample 4, Configuration 1 with a thicker inner tube to show full penetration of aluminum into the W structure.

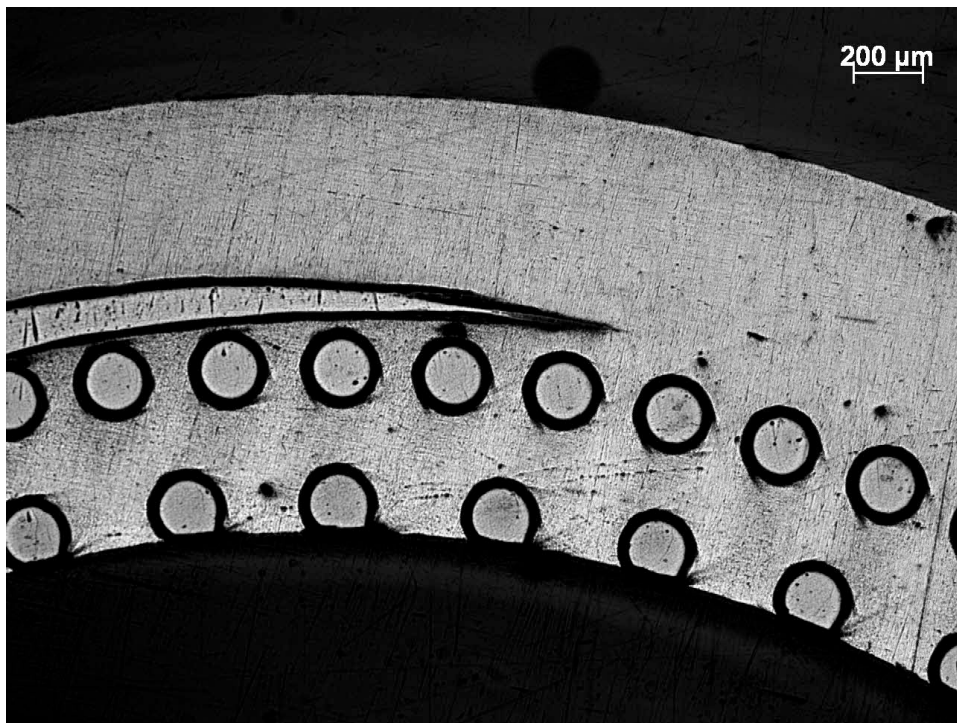


Figure 42 - Sample 4, Configuration 1 with a thicker inner tube, optical image to show the regularly spaced W rods.

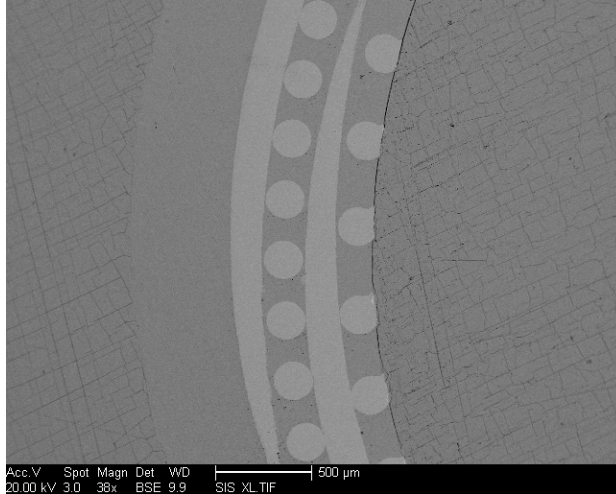


Figure 43 - SEM image of Sample 4, Configuration 1 to show the regularly spaced W rods.

No reaction is seen in these samples as proven by an SEM image of sample 3 seen in Figure 44. Since both samples were HIP treated simultaneously it is assumed that if one sample did not show evidence of reaction between the W and aluminum components, the same should valid for the other sample. However in sample 4 localized bonding between the W fibers was noticed as seen in Figure 45. Intense pressure accompanied intense heat created by friction from the motion of the W fibers with respect to one another could of welded the W together. This sort of welding can only help our structure stay together during the HIP and mechanical testing. However such welding of the fibers may also create areas of stress concentrations within the composite leading to early failure upon loading.

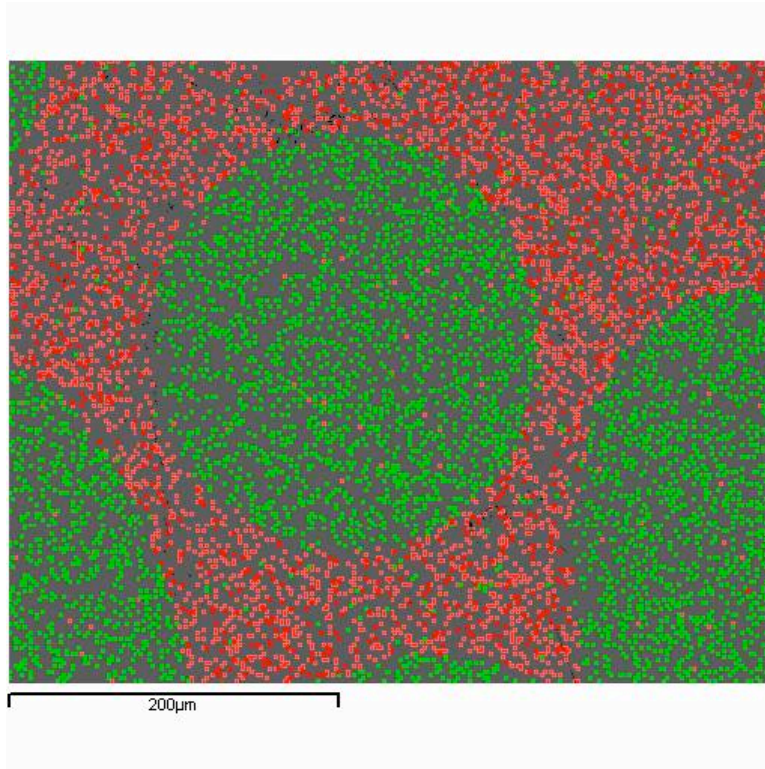


Figure 44 - SEM image of Sample 3, Configuration 1. Aluminum particles are highlighted in red and Tungsten particles are highlighted in green. No diffusion of the elements resulting in Al-W compound is noticeable.



Figure 45 - Sample 4, Configuration 1 with a thicker inner tube, an optical image. Notice the contact welding between the W wire and rods.

Buckling of the W wires is observed in both samples (as seen in Figures 46 and 47) both in the axial direction where the wire would see free space, and the radial direction with the heated aluminum layers above. Evidence of buckling in the axial direction can be seen in Figure 48 with Figure 49 as comparison of an ideal cross section. It is felt the buckling of the W wires is unavoidable during a externally HIP treated process since the pressure required to plastically flow the heated Al 6061-T6 through the W fiber structure is greater than the critical pressure needed to buckle the W wire coils.

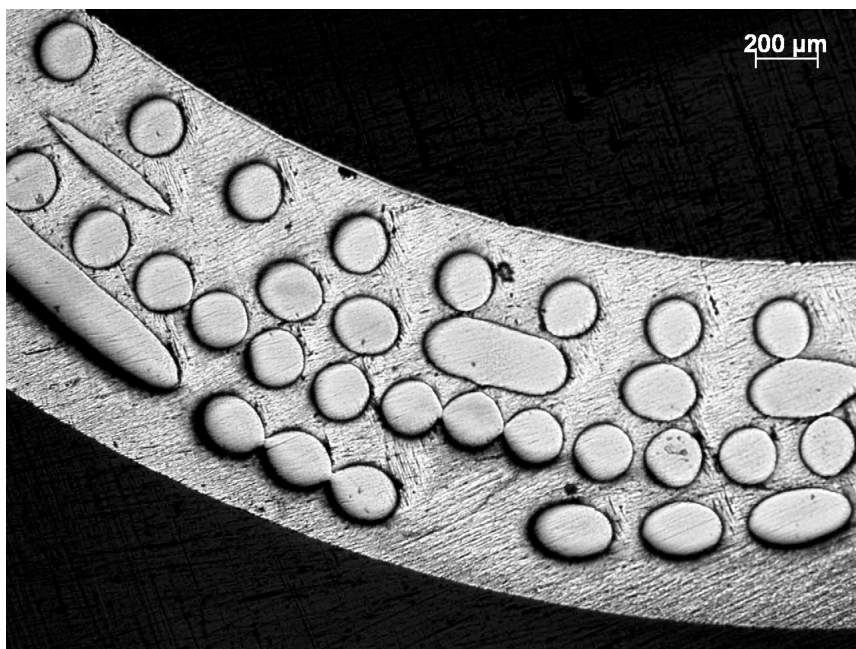


Figure 46 - Buckled region in Sample 3, Configuration 1, optical image.

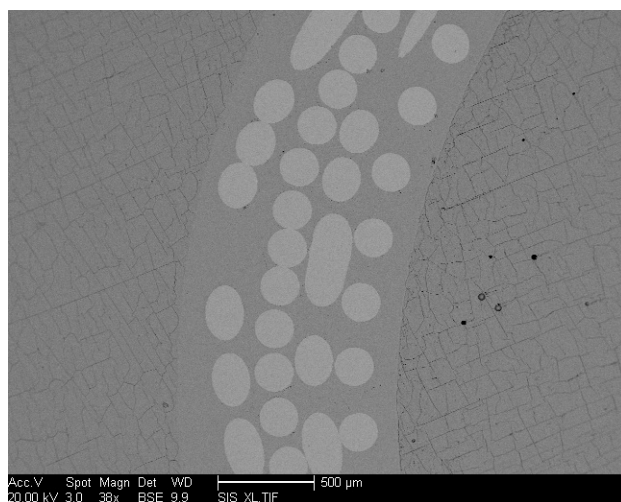


Figure 47 - SEM image of buckled region of Sample 3, Configuration 1.

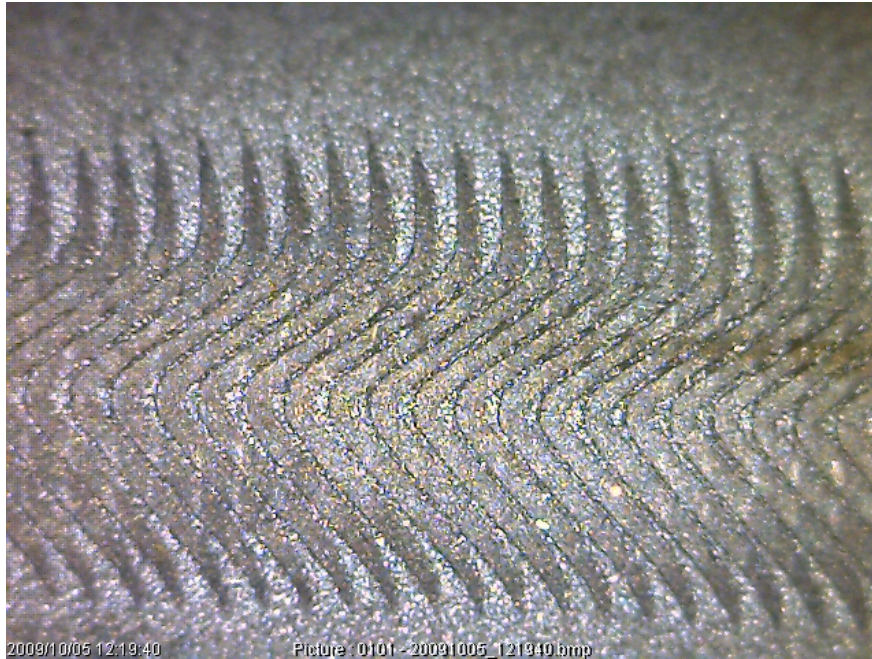


Figure 48 – Piece of Sample 3, Configuration 1 with an outer diameter of 9.13 mm to expose the outer buckled layer of W wire in the axial plane.

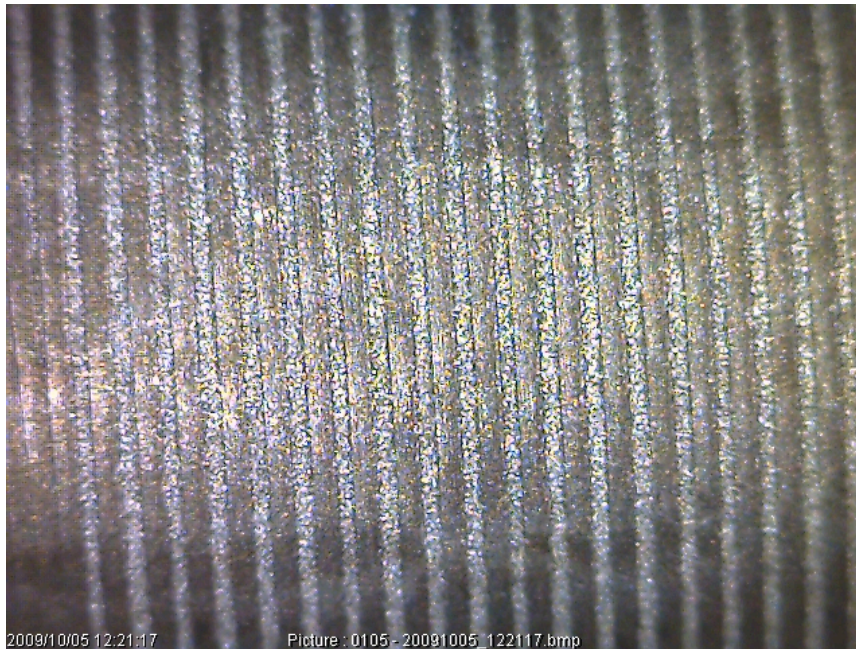


Figure 49 – Piece of Sample 3, Configuration 1 with an outer diameter of 9.13 mm in the non buckled region to show the regularly space W wire.

No difference is seen between samples 3 and 4 of Configuration 1 though the thickness of the inner Al 6061-T6 tube was different. It was hoped that sample 3 would gain more support from the steel core to prevent buckling, and that the extra thickness of aluminum on sample 4 would leave a thin aluminum layer between the inner diameter at 7.5 mm and the inner boundary of W structure. Both samples instead buckled, and some aluminum on the inside of the samples was removed anyway to reach the desired density. The radial displacement of the W structure toward the center of the samples appears to be the same. To gain extra aluminum on the inside of the cylindrical samples, an extra thickness needs to be created with the increase in the outer diameter of the inner Al 6061-T6 tube. Now that “Brazilian” type fracture has been mostly prevented in the Al-W composites due to the slow pressurization rate implemented in the HIP treatment extra space must be eliminated in the initial structure to prevent buckling caused by external pressure.

3.8 – Samples 5 & 6 of Configuration 1 and Intermetallic Formation

Two additional samples of Configuration 1 were created to gather preliminary data on the ultimate tensile strength of the composite. The fifth and sixth samples of Configuration 1 were created. Sample 5 underwent two HIP processes, whereas sample 6 was HIP treated successfully on the first attempt. The first HIP was unsuccessful for sample 5 due a failed vacuum seal. No Al 6061 migrated into the W structure, and no fracture or buckling of the W was observed due to this first failed

HIP process. This HIP cycle used to treat the sample was similar to the HIP cycle applied to sample 4, but with a maximum temperature of 540 Celsius to enhance Al penetration into W architecture at low pressure. Sample 6 resulted from this HIP treatment with a well-preserved W structure, regions of full penetration of the Al 6061-T6 into the W structure, and no fracture of the W rods. But localized regions of Al-W compound were found in sample 6. The Al-W compound expanded within the W structure, leaving behind voids as well. Due to the high temperature of this HIP cycle and heat released from friction between all the components intense local temperatures may have been created, thus promoting diffusion between the W and Al. Due to the failed vacuum in sample 5 the lack of pressurized interfaces between the components prevented diffusion between them, and thus the formation of a reaction product. The second HIP cycle performed identically to the process applied to sample 4 at 500 Celsius was successful for sample 5. However buckling of the W wire was noticeable in both samples after their relative successful HIP treatments.

The sixth sample in Configuration 1 was prepared identically as described for sample 4, and the fifth sample in Configuration was prepared identically as described for sample 3. With the samples successfully built they were prepared as previously described for a HIP process.

Samples 5 and 6 underwent the same HIP process that was applied to samples 3 and 4 of Configuration 1 as can be seen in Diagram 6. A higher maximum temperature was applied in hope of making the Al 6061-T6 layers softer so that they would viscoplastically deform at lower pressures that would also not buckle the W

wires. The initial outer diameter of the aluminum jacket for sample 6 was 19.09 mm, and after the HIP process the outer diameter decreased to an average of 18.4 mm. The larger aluminum alloy 6061-T6 jacket for the HIP process as compared to sample 4 was due to the sample failing to fully insert into the smaller jacket used for sample 4. With sample 6 jammed inside the first Al 6061-T6 jacket a second Al 6061-T6 jacket was created to encapsulate both the sample and the first jacket. The initial outer diameter of the aluminum jacket for sample 5 was an average of 15.82 mm, and after the HIP process the outer diameter decreased to an average of 15.6 mm. A vacuum of 1 micron was created and sealed inside the jackets, first mechanically and then with a weld.

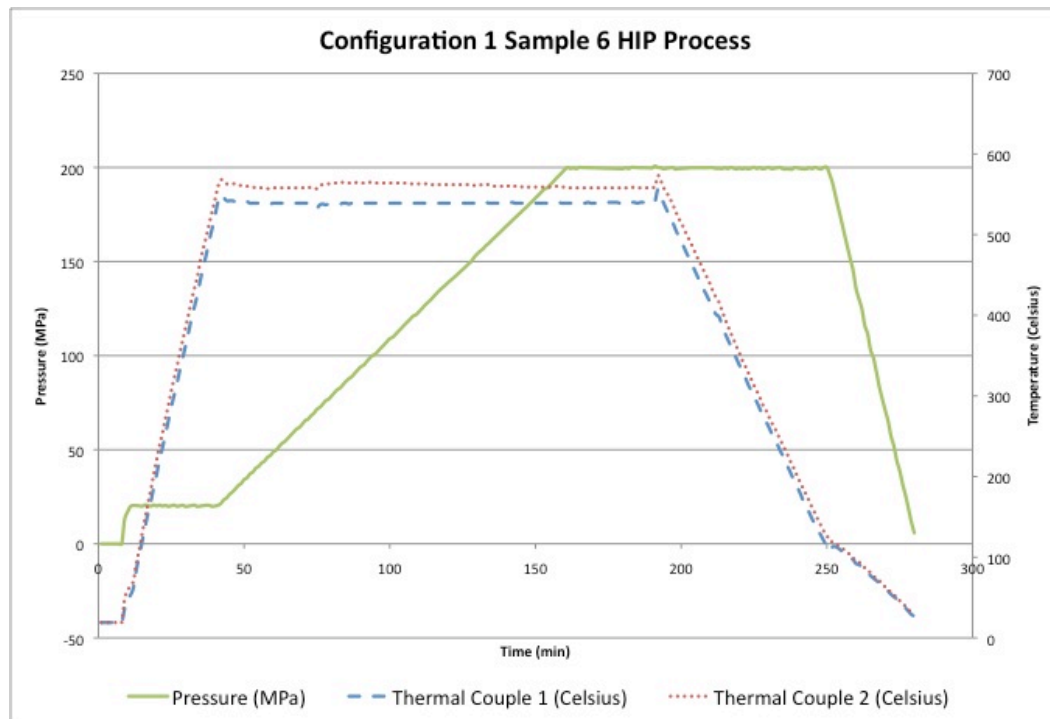


Diagram 6 - HIP process for Sample 6, Configuration 1 with thicker Al 6061-T6 inner tube and higher temperature.

The first HIP process for samples 5 and 6 had mixed results. For sample 5 the vacuum seal broke, leaving the W structure intact with no plastic deformation or fracture. However fracture is observed in Figure 50 due to the clamping of the ends of the sample in the assembling process. When the layers of W were applied, they were mechanically clamped into place and an audible snap was heard. Since the mechanical clamping occurred at the ends of the sample during fabrication, it is expected in the same region of the sample that “Brazilian” fracture could be found. Since the clamp has a fixed displacement, any cracks induced into the W rods should have a limited length, not extending through the length of the rods. With the fractures localized to the ends of the sample the final product can be simply cut 3-5 mm of the end zones to discard this flaw. Proof of such restricted crack growth comes from observing cross sections, as seen in Figures 52-54, taken further away from the ends of the samples showing no fracture of the W rods.

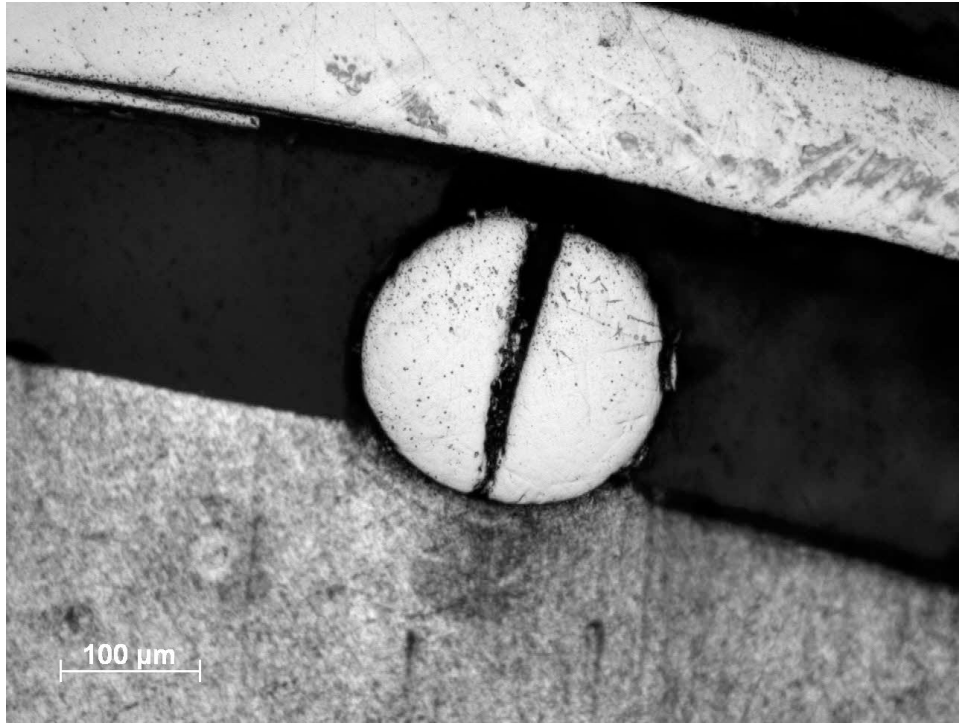


Figure 50 - Sample 5, Configuration 1, optical image after the first failed HIP process. The “Brazilian” fracture seen is probably due to the mechanical clamping during the assembly process.

The region of sample 6 located lower in the furnace that was at a temperature of 560 Celsius shows that aluminum fully penetrated the W structure without causing fracture or leaving behind voids as can be seen in Figure 51. A buckle however is still observed as seen in Figure 52, even at these elevated temperatures. This buckle is observed throughout the entire length of the sample as seen in Figure 53 from a cross section selected from the highest region of the sample region which was HIP treated at 540 Celsius. Also within this high region of sample 6 a large void was found between the third and forth layers of W.

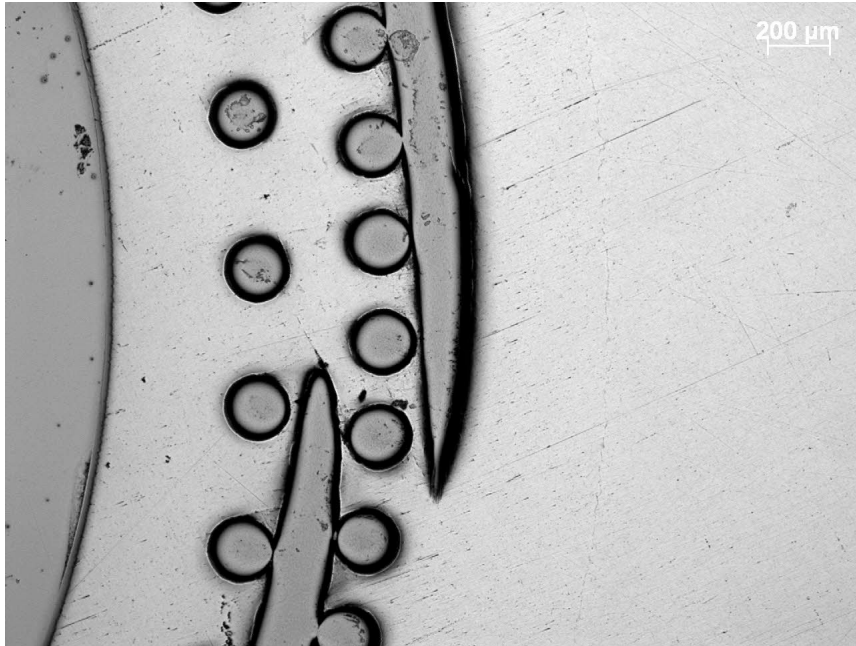


Figure 51 - Sample 6, Configuration 1, optical image. Notice the full penetration of the aluminum into the preserved W structure.

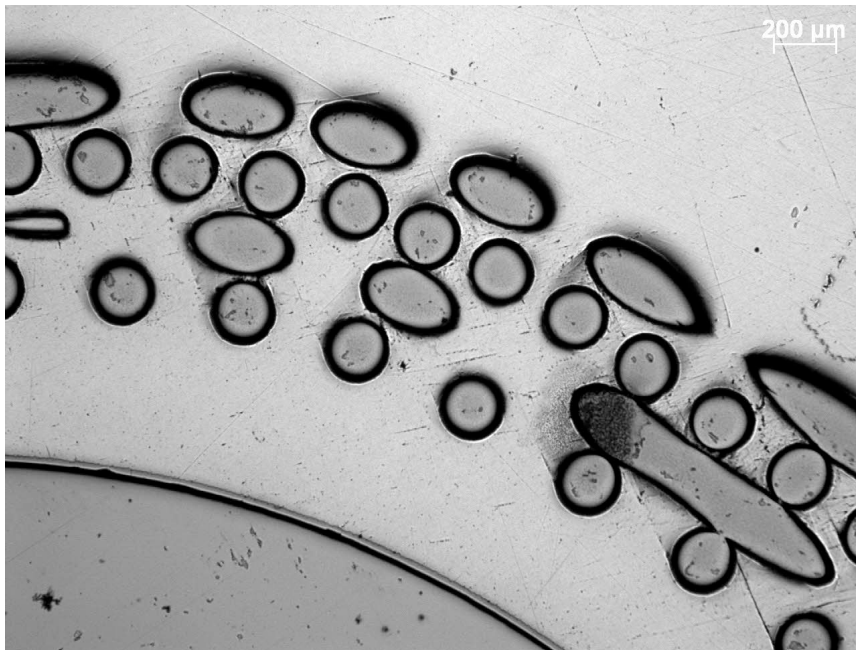


Figure 52 – Buckled region observed in the lower region of Sample 6, Configuration 1, optical image. The W wires layers have buckled in the axial plane into the empty space.

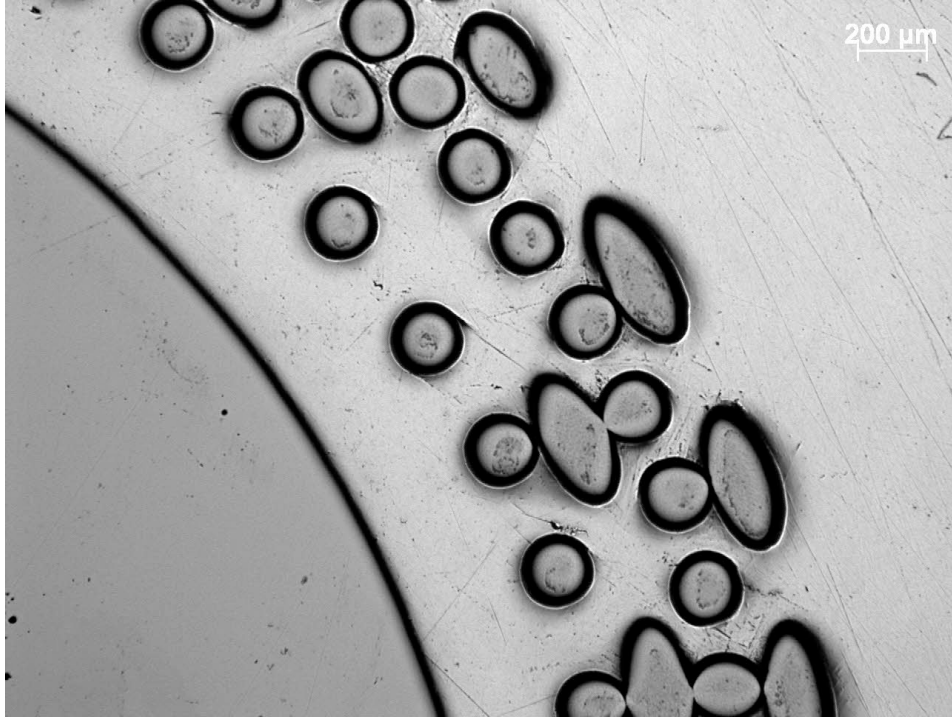


Figure 53 – Buckled region observed in the higher region of Sample 6, Configuration 1 under an optical microscope. The W wires layers have buckled in the axial direction into empty space.

Spacing between axial W rods within sample 6 was more consistent but further from our desired spacing as compared to samples 3 and 4 as presented in appendix A. Samples 4 and 6 were initially built from the same structure that was cut into two pieces. Thus, it can be concluded that a greater maximum HIP temperature, as seen with sample 6 compared to sample 4, resulting in softer aluminum moving into the W structure at a lower pressure does not significantly affect the overall W rod displacement.

Sample 6 has evidence that large voids can be created between the W layers. Several cross sections were sliced from the highest region of sample 6, with respect to

the HIP furnace, to see that within millimeters of each other these voids can appear and disappear. Figure 54 shows a region of aluminum fully penetrating the W structure and Figure 55, taken from a close by cross section, shows a void between the third and forth layers of W. An Al-W compound forming to expand against the aluminum layers could create these voids.

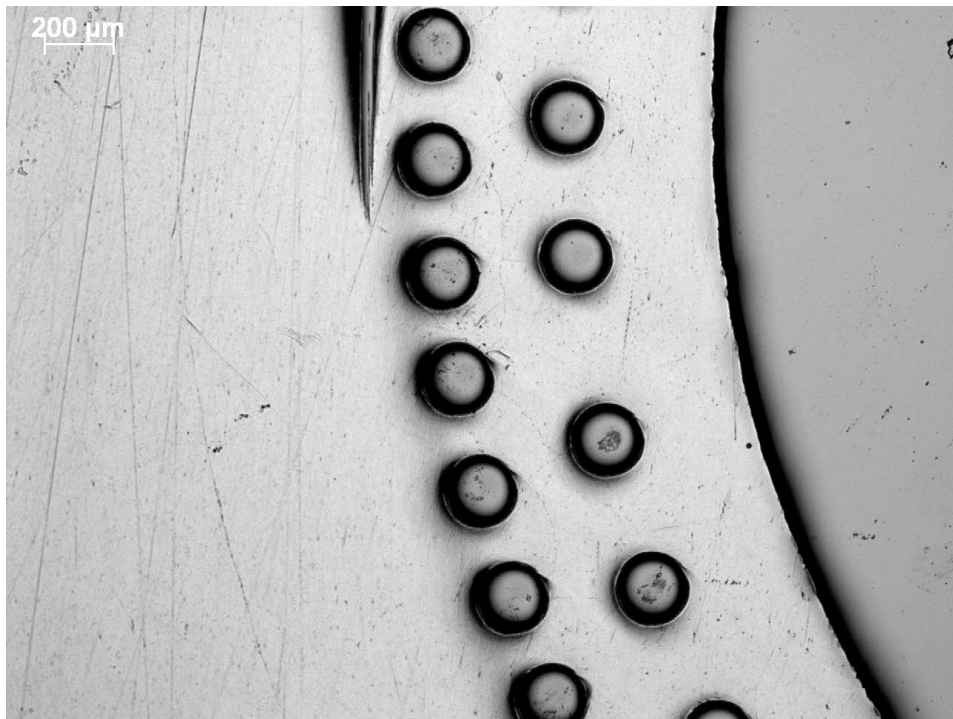


Figure 54 - A region of Sample 6, Configuration 1 with a thicker Al 6061-T6 inner tube corresponding to a top part of the sample placed vertically in the HIP furnace. Notice the full penetration of aluminum into the W structure.

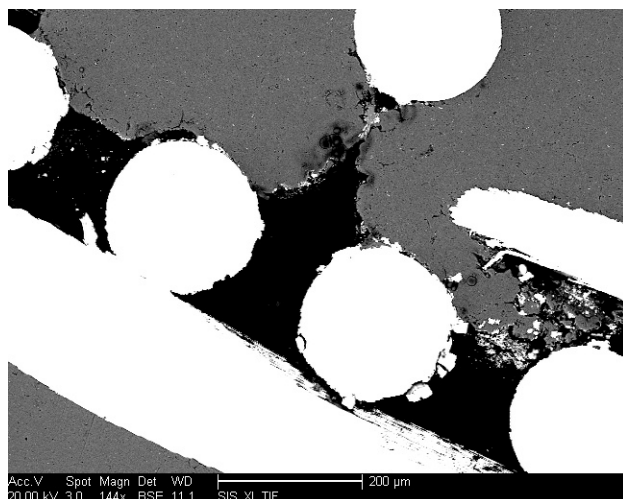


Figure 55 - SEM image of another cross section also within the top region of Sample 6, Configuration 1 with a thicker Al 6061-T6 inner tube. Notice the void between the third and forth W layers. Aluminum seen inside the W structure is from the inner Al tube.

It was observed under SEM that inside the region with a void W and Al 6061 reacted to form a compound as could be seen in Figure 56. This reaction is believed to be due to the intense heat from friction between all the components and the high temperature of the HIP treatment, 540 Celsius. As can be seen in Figure 57 aluminum from the inner layer moved past the W rods dragging reacted compound with it. Frayed boundaries between the W and aluminum further suggest the W began to diffuse into the aluminum to form a compound. As can be seen in Figures 57 and 58, an Al-W compound formed due to one of the materials becoming molten from the intense heat created by friction to fill the space between W rods and the aluminum layers, preventing the outermost layer of aluminum to penetrate the outer wrapping of W. Keeping the maximum temperature of the HIP treatment below 520 Celsius, and minimizing the time of the HIP cycle at this maximum temperature should reduce or

eliminate the creation of a Al-W compound. If possible, keeping the W fibers from moving with respect to one another should prevent hot spots forming due to friction.

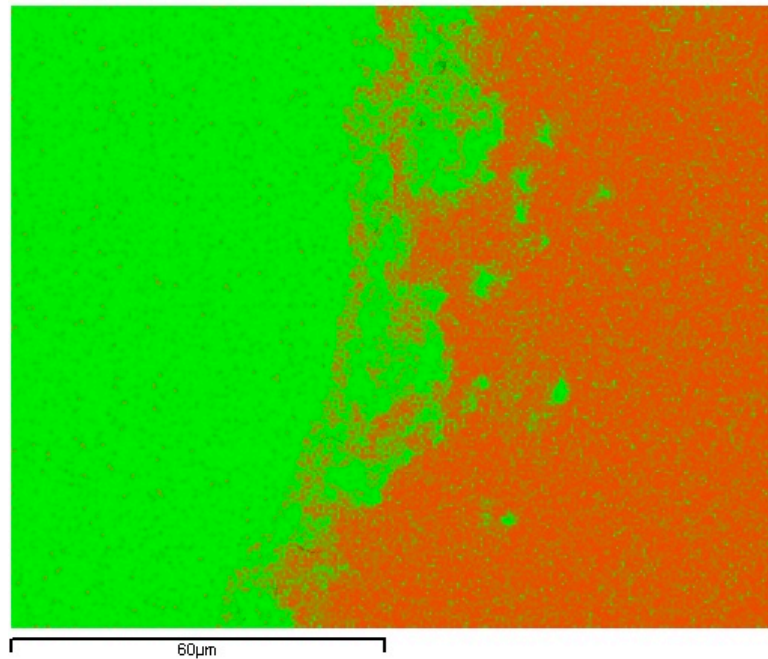


Figure 56 - SEM image of an interface between the third layer of W rods and inner Al 6061-T6 tube from a top region in Sample 6, Configuration 1 with a thicker inner Al 6061-T6 tube. The W is seen in green and the aluminum is seen in orange. Notice the diffusion of W into the aluminum.

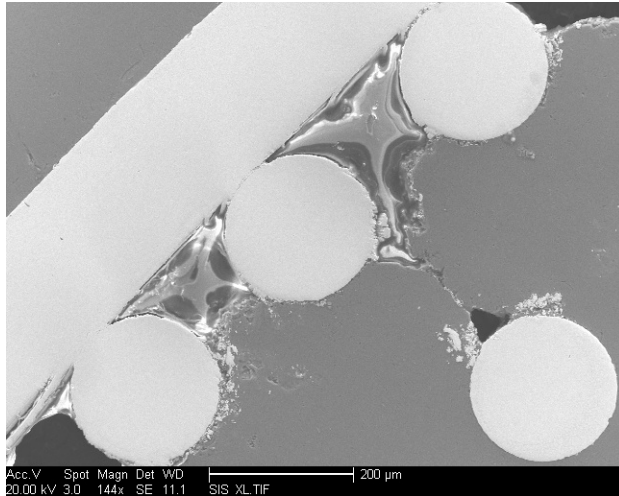


Figure 57 - SEM image of Sample 6, Configuration 1. Notice the Al-W compound between the W rods, the frayed boundaries of the W rods due to the diffusion of the W into the aluminum, and the transport of an Al-W compound as the aluminum deformed around the W rod.

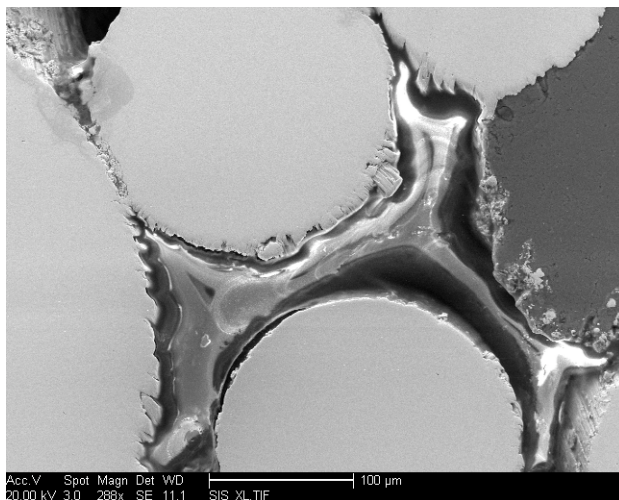


Figure 58 - SEM image of Sample 6, Configuration 1. Notice the frayed edges of the W that suggests liquid aluminum was formed to promote a reaction between the materials.

Sample 5 was machined and prepared for the second HIP treatment as described previously. The HIP cycle applied to the sample, however, was identical to the treatment given to sample 3 with the nominal temperature in the furnace to be at

500 Celsius. The initial outer diameter of the sample was an average of 19.02 mm with the Al 6061-T6 jacket before the HIP process and 18.35 mm after.

Figure 59 shows the second HIP cycle was successful, with the results closely match what is seen in samples 3 and 4. Aluminum was fully migrated into the W structure without fracturing the W fibers and buckling of the W wire was induced in the radial and axial directions. It is suspected sample 5 may have regions of an Al-W compound due to being treated in the 540 Celsius HIP cycle with sample 6. Due to lack of pressurized contacts between the W structure and surrounding Al 6061-T6, it is thought that reactions between the components during the first failed HIP cycle were kept to a minimum.

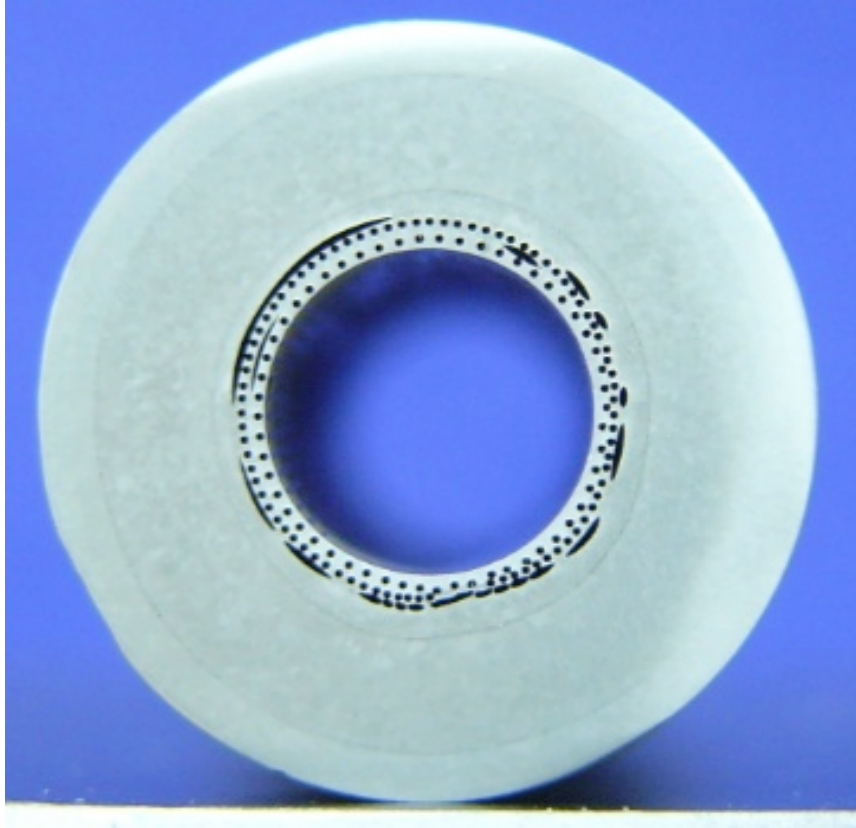


Figure 59 - Sample 5, Configuration 1 after the successful HIP cycle. Notice the overall well preserved regular W structure and the buckled region in the lower portion of this cross section. Bucking of the W wire was in both the axial and radial directions.

Section 3.9 – Improved HIP Treatment of

200 μ m W Reinforcement

With the first two tungsten layers applied and sealed into place by an interim Al 6061-T6 tube, the samples of Configuration 3 were successfully degassed inside Al 6061-T6 jackets a HIP treatment was applied. The vacuum within the samples was commonly close to five microns or better. The HIP process was applied twice, once

for each pair of W layers as is seen in Diagram 7. Before the temperature was raised, the pressure was increased to 20 MPa to squeeze the structure together such that it maintained its arrangement while thermally expanding during temperature rise. Temperature was increased such that the controlling thermal couple located in the vertical center of the furnace of the HIP machine was set at 500 Celsius. A second thermal couple closer to the heating plate, set 50 mm below the controlling thermal couple commonly measures temperatures up to 20 Celsius greater than the controlling thermal couple. Pressure was then increased slowly over two hours to 200 MPa such that the aluminum tubes were at a uniform temperature and slowly flows into the W structure, minimizing distortion to the W structure. The maximum temperature of 500 Celsius and maximum pressure of 200 MPa was held for 30 minutes. The samples were then cooled under pressure to assure extremely tight interlocking interfaces between the components of the composite. Finally pressure was released and the temperature was brought down to room temperature.

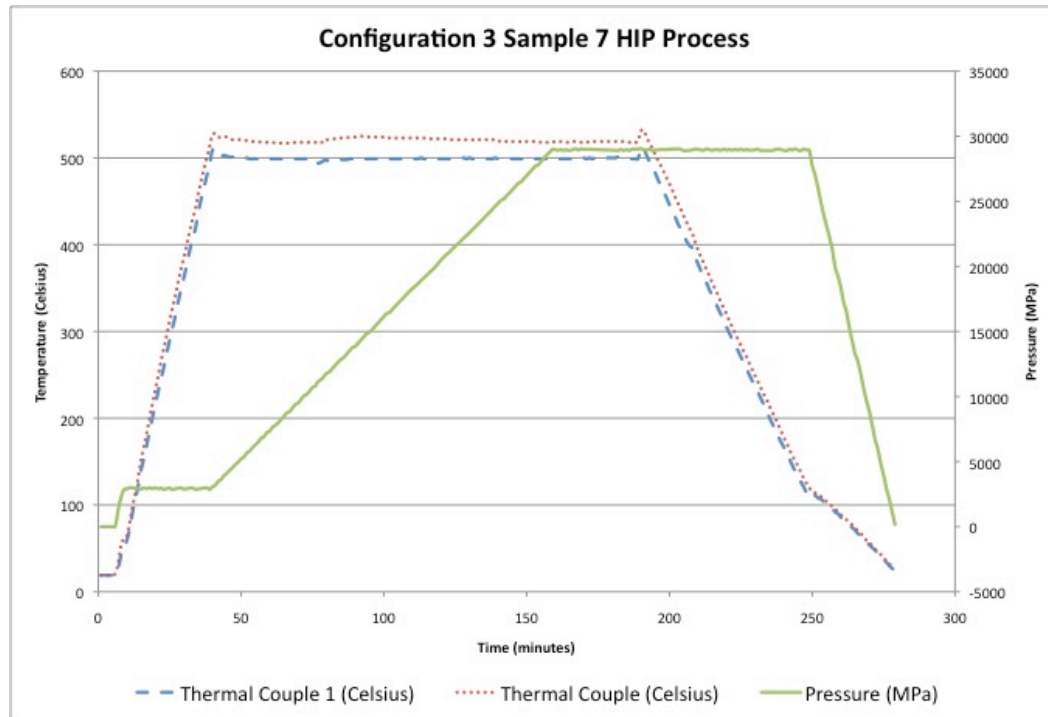


Diagram 7 - Improved HIP treatment applied to Configuration 3 samples.

Only one of the samples in Configuration 3 received the exact same HIP treatment for both pairs of W layers. Five other samples of Configuration 3 were created, but with a different HIP cycle for the first pair of W layers in an attempt to minimize the time at maximum temperature to minimize the formation of reaction compound. As was seen in section 3.8 reaction product between aluminum and tungsten can be created at the elevated temperatures during the HIP process. Since the creation of the reaction product is diffusion controlled, and thus related to time, minimizing the time at these elevated temperatures should reduce the amount of reaction product. For a sample in Configuration 3 made with two HIP treatments as seen in Diagram 7, the components of the composite experienced the maximum

temperature of 500 Celsius for a total of 5 hours. To reduce this time the HIP cycle seen in Diagram 8 was applied to samples 9-13 in Configuration 3. By increasing the pressurization rate from 1.5 MPa/min to 2.5 MPa/min and decreasing the soak time at maximum temperature and pressure from 30 minutes as seen in Diagram 7 a soak time of 10 minutes the overall time the components of the composite experienced the maximum temperature was reduced to 3.9 hours. Although it would take a greater period of time to form a compound between aluminum and tungsten at the temperatures in the HIP treatments it has been seen in liquid metal studies of Al-W composites that intermetallic compounds were created in 50 minutes at 670 Celsius [21].

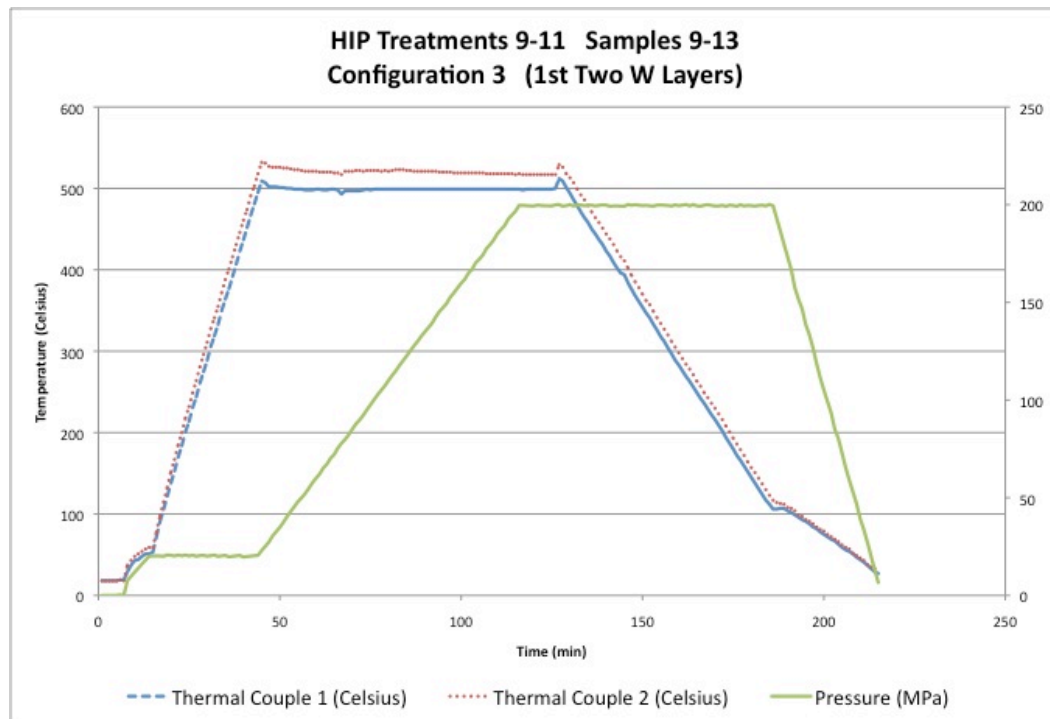


Diagram 8 - HIP treatment for the first pair of W layers for Samples 9-13, Configuration 3.

Section 3.10 - HIP Results for Configuration 3

The HIP process, as can be seen in Diagram 7 for sample 7, the first sample of Configuration 3 was a success. The outer diameters of the two pieces of the specimen were 15.9 mm and 15.875 mm initially and shrunk to 15.51 mm and 15.495 mm respectively. Full penetration of aluminum was achieved into the W structure with minimal disturbance of the W structure with no signs of strong buckling or widespread fracture. The most preserved W rod spacing of any Configuration can be seen in Figure 60. Due decreasing the amount of free space with in the W structure buckling was kept to a minimum; greatly preserving the initially prescribed W rod spacing. Plastic deformation was also observed at the contact of W wire with W rods, and in some places fracture of the W wire was observed at these contact areas (Figures 61 and 62). This type fracture is localized, and it is not expected to have a significant impact on the properties of interest: density and axial tensile strength. No “Brazilian” fracture was observed in the W rods. This plastic deformation with little fracture in the W rods suggests that for only two layers the initial pressure of 20 MPa was close to but less than the critical pressure for widespread fracture in the W components. The plastic deformation of the W structure is desirable to keep the entire structure together during an explosive event. However, such plastic deformation between the fibers can give rise to stress concentrations within the material during a multi axial loading event that can lead to premature failure of the material.

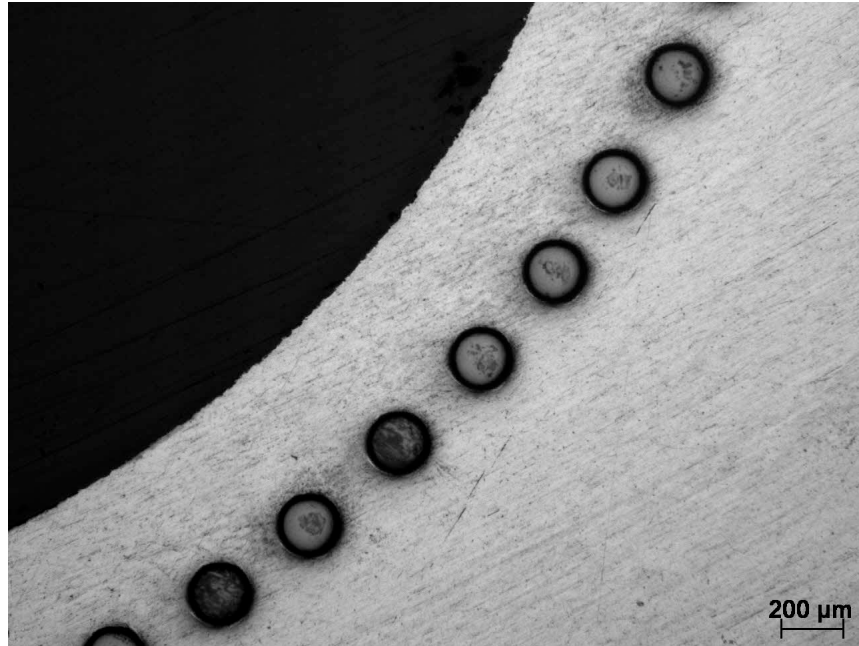


Figure 60 - Sample 7, Configuration 3, optical image after the first HIP process served to encapsulate two layers of W within the Al 6061 matrix.

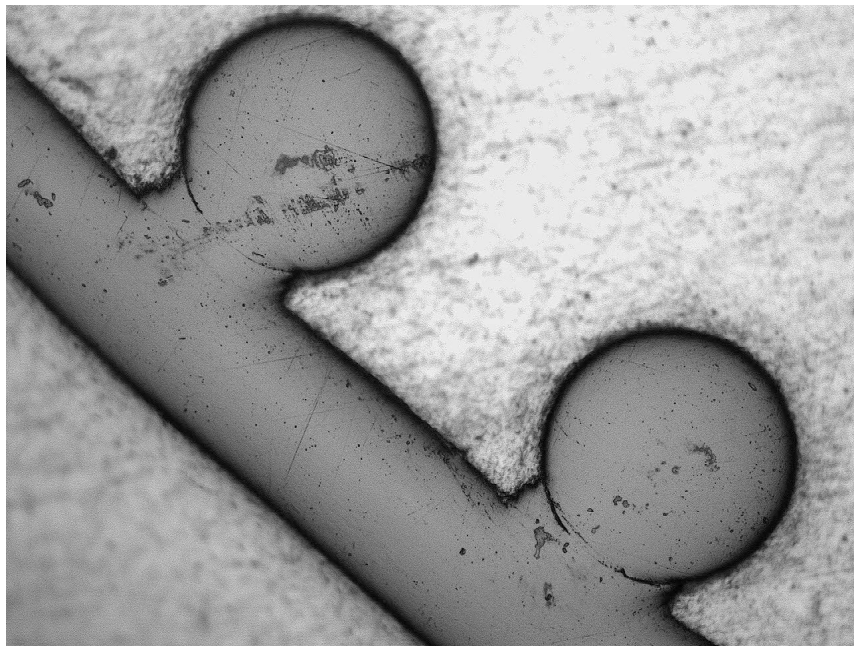


Figure 61 - Sample 7, Configuration 3, optical image after the first HIP process. Notice the plastically deformed W wire at the contact points with the W rods.

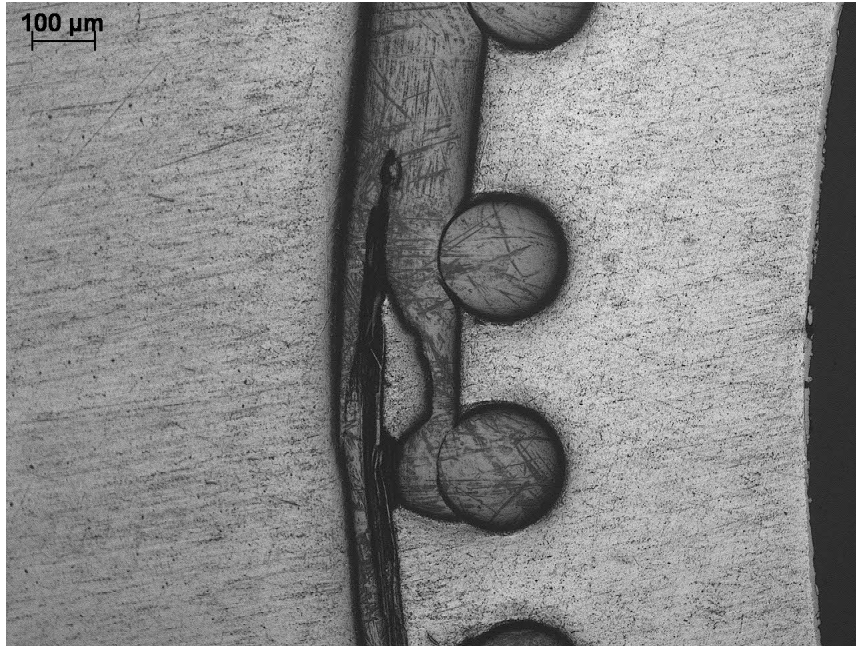


Figure 62 - Sample 7, Configuration 3, optical image after the first HIP process. Notice the plastic deformation and resulting fracture of the W wire with the contact with W rods.

Although the other samples in Configuration 3 underwent the HIP treatment seen in Diagram 8 for the first two layers of W fibers, the results were very similar to what was seen in sample 7. Samples 9-13 also demonstrated that the aluminum tubes become fully integrated into the W structure with no widespread fracture or buckling of the W fibers while not plastically deforming the W wire. The prevention of such deformation is thought to be due to the use of a thicker aluminum jacket. With the extra thickness of the aluminum jacket carrying more of the external pressure load less force was transmitted through the W structure. The average outer diameter of samples 9-13 was 19 mm before the HIP treatment and 18.7 mm afterwards. However with the use of a reduced time at maximum pressure, an unbonded region was left behind

between the two aluminum tubes. With the elastic relaxation of the structure after the HIP treatment, this interface expanded creating the small gaps seen in Figure 63.

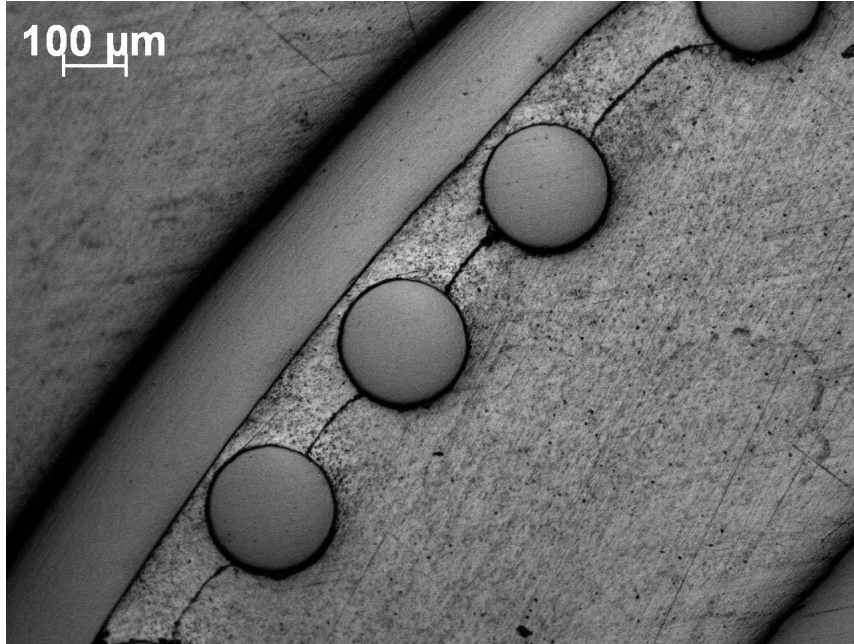


Figure 63 - Sample 9, Configuration 3 after the first HIP treatment, optical image.

After the first successful HIP cycle samples 7 and 9-13 had their outer diameters machined such that a layer approximately 300 μm thick of aluminum remained outside the outermost layer of W wire on which the last two layers of W fiber were applied. Once constructed, the samples were encapsulated inside Al 6061-T6 jackets and HIP treated identically as before. The second HIP process applied to the samples of Configuration 3 can be seen in Diagram 7. The initial outer diameters of the jacketed pieces of sample 7 were both 15.83 mm, which changed to 15.39 mm and 15.43 mm. Again thicker jackets for samples 9-13 were used with initial outer

diameters averaging 19.1 mm decreasing to 18.7 mm. This HIP process was a success, resulting in penetration of aluminum into all the available space within the W structure with no reaction between W and aluminum. Samples 9-13, however, did not successfully bond the layers of aluminum together as would have been expected with the HIP process seen in Diagram 7.

In samples 9-13 two interfaces were left behind within the aluminum matrix. These interfaces formed a cylindrical surface encompassing the W rods as seen in Figure 64. It is thought that in these samples the W structure carried the pressure load such that the aluminum within the structure was shielded and did not flow into the aluminum below. Without a pressurized interface between the aluminum layers no diffusion occurred to bond the layers. This shield phenomena may have also contributed to the failure of the bonding of the aluminum between the third and fourth layers of W fibers. Elastic relaxation opened the unbonded interfaces after the HIP treatment. These interface voids seen in Configuration 3 as well as sample 1 of Configuration 2 found in section 3.5, suggest that as the number of aluminum layers is increased in an externally pressurized specimen, the chance of creating voids between the layers increases as well. Following the successful construction of the composite, sample 9 was vacuum encapsulated again to see if an additional HIP cycle, seen in Diagram 7, would close the interface gaps. But due to the shielding effect of the W structure the interfaces were left intact. In fact, additional HIP treatments on fully densified samples could introduce severe shear deformation and fracture into the W rods as shown in Figure 65. This sort of fracture was the result of voids existing in a

nearly densified sample. Also, the replacement of the initial steel mandrel as discussed in section 3.2 could also leave behind a 10 μm gap within the steel components. The structure moves into the empty space and with tungsten's low strain to failure fractured with the shearing of the aluminum matrix. Sample 7 that was treated with the longer HIP cycles had complete bonding between the aluminum layers with no fracture of the W rods as seen in Figures 66-69. Other issues such as local buckling within the Al-W composite, however, were found in all samples of Configuration 3.

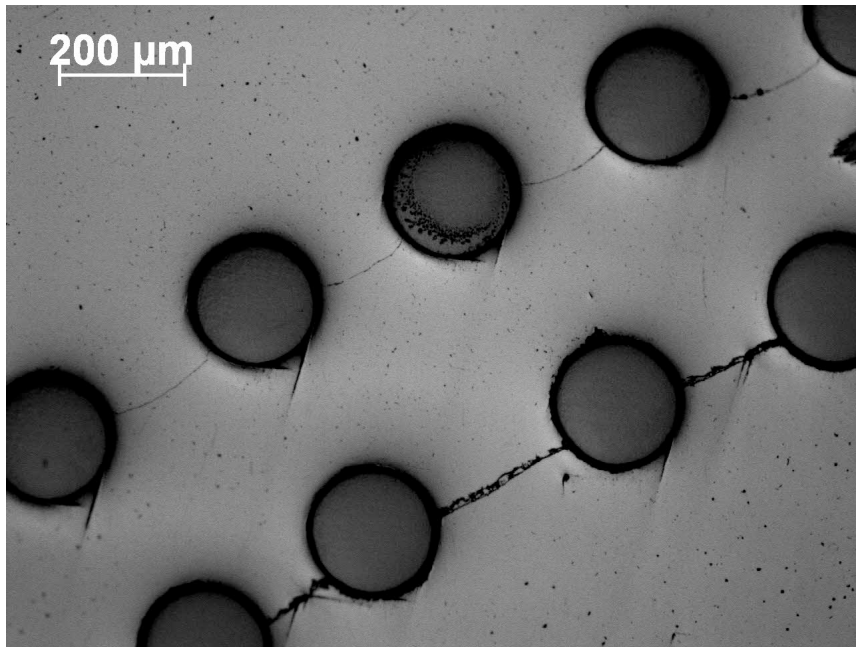


Figure 64 - Sample 9, Configuration 3, optical image. Notice the two interfaces left behind between the aluminum layers after the second HIP treatment.

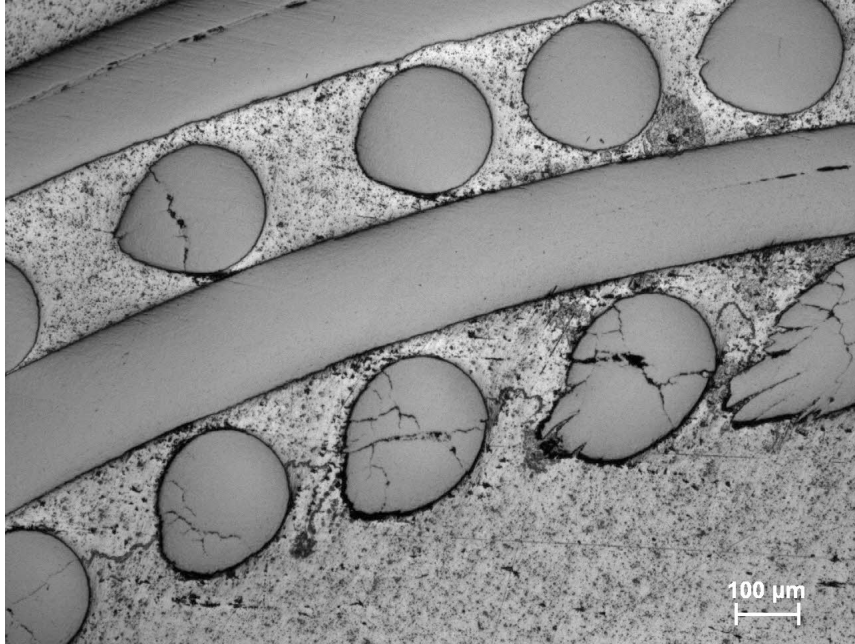


Figure 65 - Sample 9, Configuration 3, optical image after a third HIP treatment with a fully densified structure. This cycle was applied to explore the interface gap left after the previous HIP cycles could be sealed. Notice the severe shear deformation and fracture of the W rods.

Sample 7, as well as other samples of Configuration 3 had local buckling and regions of well preserved and disturbed W structure. Overall the spacing of the W rods and the W wire, as well as the entire W arrangement was practically ideal inside the aluminum alloy 6061 matrix as can be seen in Figure 66. However when sample 7 was further divided into four pieces, it was observed that this spacing could vary within 25 mm of axial length as evidenced in Figure 67. It is unknown if the W rods fractured to slide along a circumference to new locations or if the rods bend in the hoop direction.

The regions of disturbed spacing within the first layer of W rods is observed in a region of local buckling in all samples of Configuration 3. Other examples of local

buckling both within the plane of a cross section and along the axis of the sample can be seen in Figures 68 and 69. It is felt that due to the nature of external pressurization the W wire buckling is extremely difficult to prevent but these regions of buckling are restricted to local areas in Configuration 3. Both the aluminum tubes and tungsten wires are at risk of buckling, especially with the excess of free space within the W structure. Having less free space to buckle due to using two HIP cycles for two layers at a time helped prevent buckling throughout the entire structure, but did not fully prevent this phenomenon. In regions of local buckling severely undesirable distortion of the W structure resulted. Note that the first layer of W wire appears to have buckled as well, suggesting the encapsulated W layers inside the aluminum matrix do not fully support the W layers above them during the HIP process and can be deformed themselves.

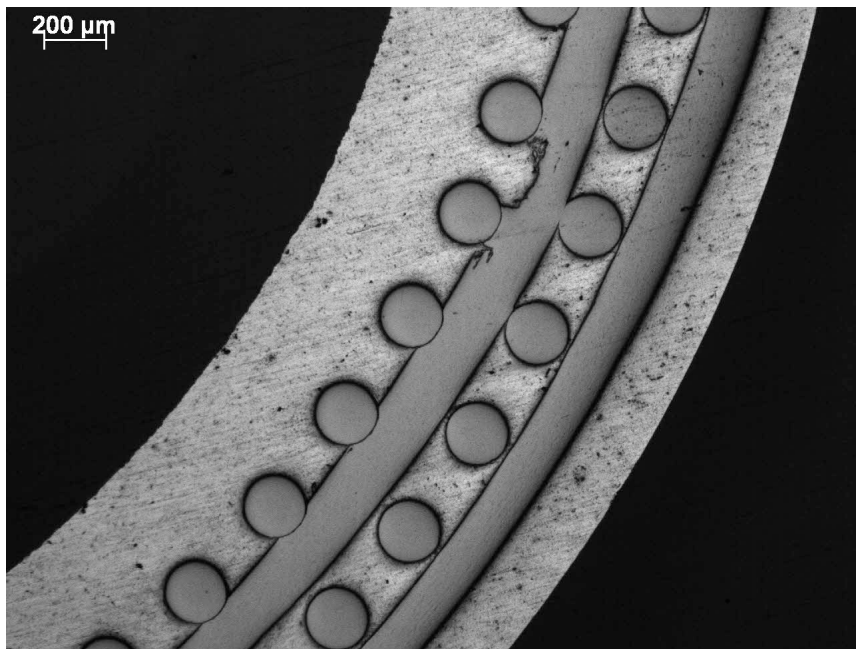


Figure 66 - Sample 7, Configuration 3, optical image. Notice the overall well preserved W structure fully encapsulated within the aluminum alloy 6061 matrix.

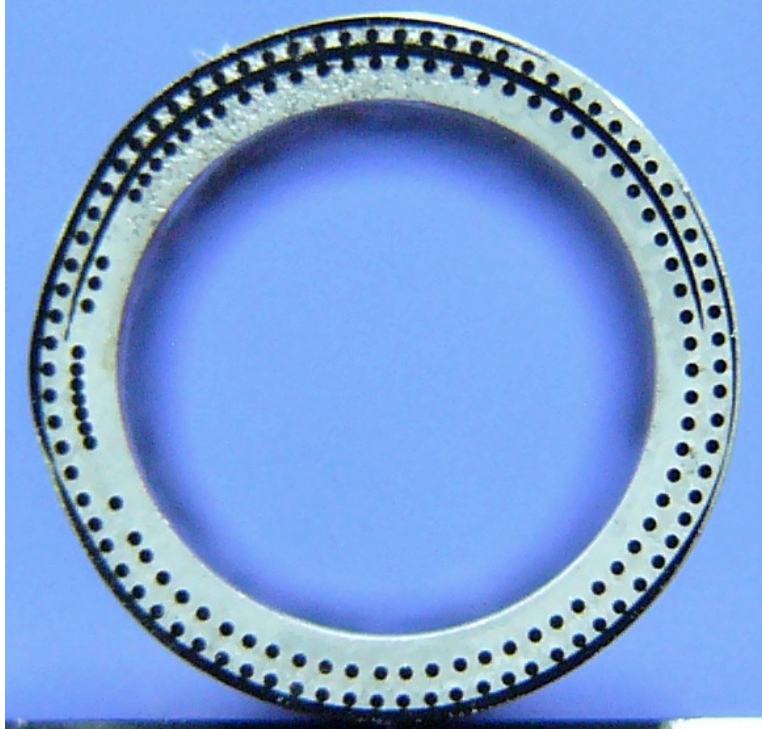


Figure 67 - Cross section of a piece of Sample 7, Configuration 3. Notice the disturbed W rod spacing in the vicinity of local buckling in the radial direction.

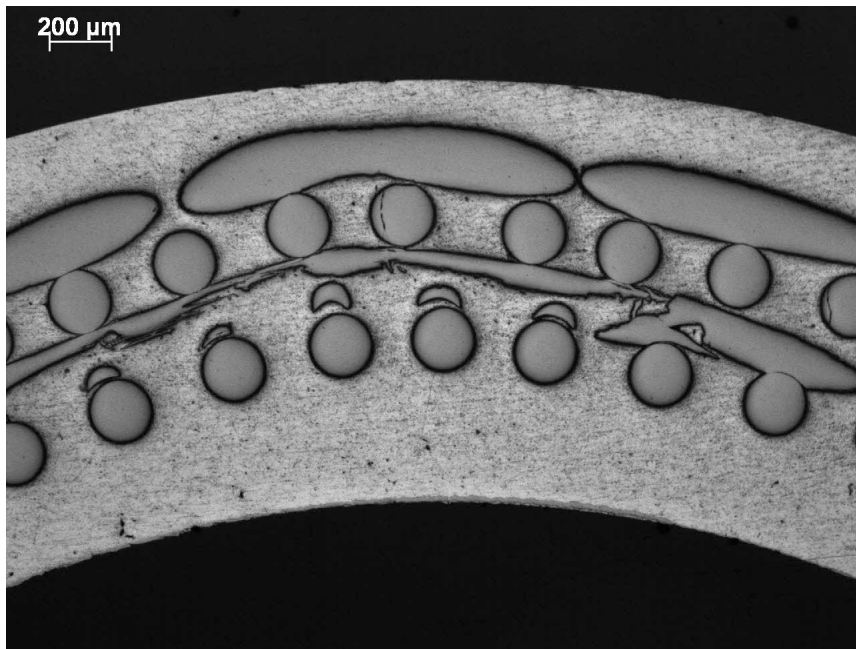


Figure 68 - Sample 7, Configuration 3, optical image. Notice the region of localized buckling in the axial direction after the second HIP cycle.

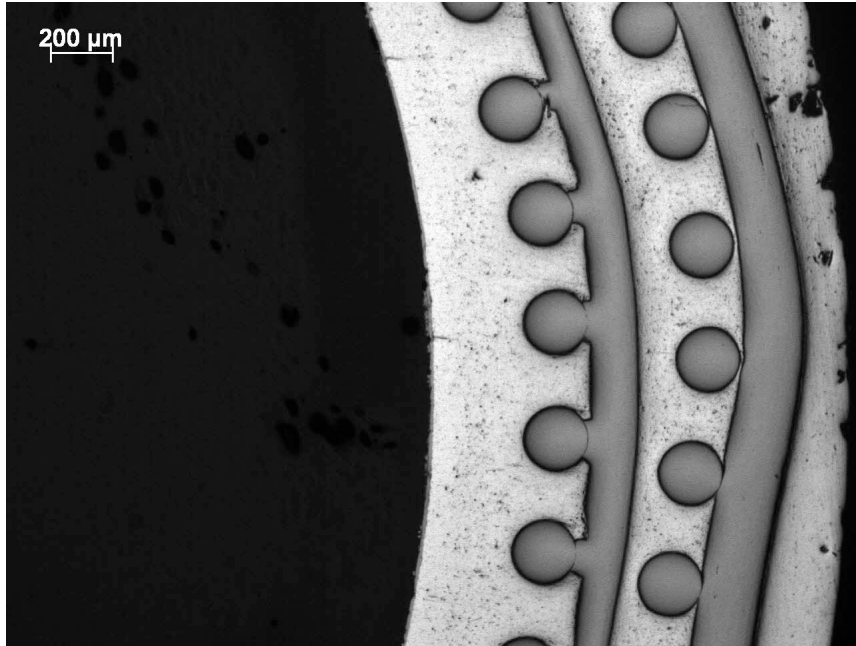


Figure 69 - Sample 7, Configuration 3, optical image. Notice the region of local buckling in the radial direction.

Irregular spacing is also observed in the outer layer of W wire. Once exposed due to machining to required density, it can be seen in Figure 70 that the spacing between the coils of W wire is not uniform. This sort of flaw can exist within any of samples of any Configuration, and suggests there is a limit to the benefit of initially pressurizing before heating the samples in the HIP process. Reduction or elimination of the initial pressurization within the HIP process may reduce or prevent the occurrence of this flaw in the W wire spacing. It is thought that although the spacing between the fibers within any layer is not ideal the spacing is adequate enough such that the random flaws are self-correcting for the purpose of shredding the aluminum matrix into micrometer sized particles under explosively applied load.



Figure 70 - Two pieces each 25 mm in length of Sample 7. Notice the irregular spacing of the exposed W wire.

Other material flaws within these samples of Configuration 3 are small voids between the aluminum layers and the W rods (Figure 71). Figure 72 shows how W rods could be displaced during the transport of aluminum as the sample was pressurized to 200 MPa at 500 Celsius. The W rods plastically deformed the contacting W wire during the initial 20 MPa pressurization at room temperature. This shows that plastically deforming the W structure may not assure that the structure stays intact during the HIP process. Evidence of W rod motion further supports a theory presented in section 3.7 that friction between the W entities causes areas of extreme heat and possibly reactions with the aluminum matrix and W fibers. As confirmed by SEM in Figure 73 no reaction was found in the cross sections of sample 7 and no reaction compound is assumed to be in the other samples of this Configuration due to their decreased time at maximum temperature. Sample 9,

however, did experience a total time of approximately 9 hours and no intermetallic compound was found within the final cross sections.

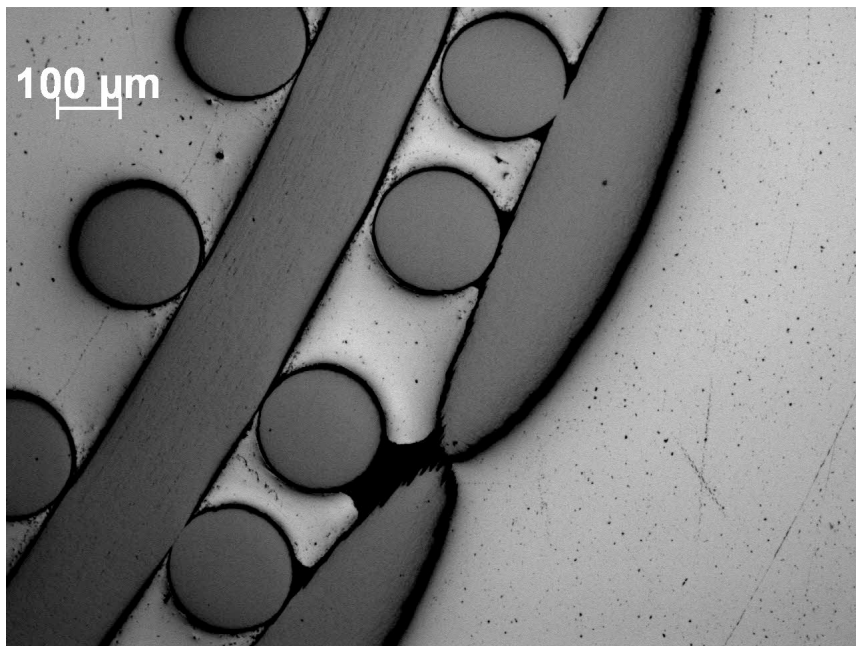


Figure 71 - Sample 10, Configuration 3 after the second HIP treatment, optical image. Notice the voids between the W fibers.

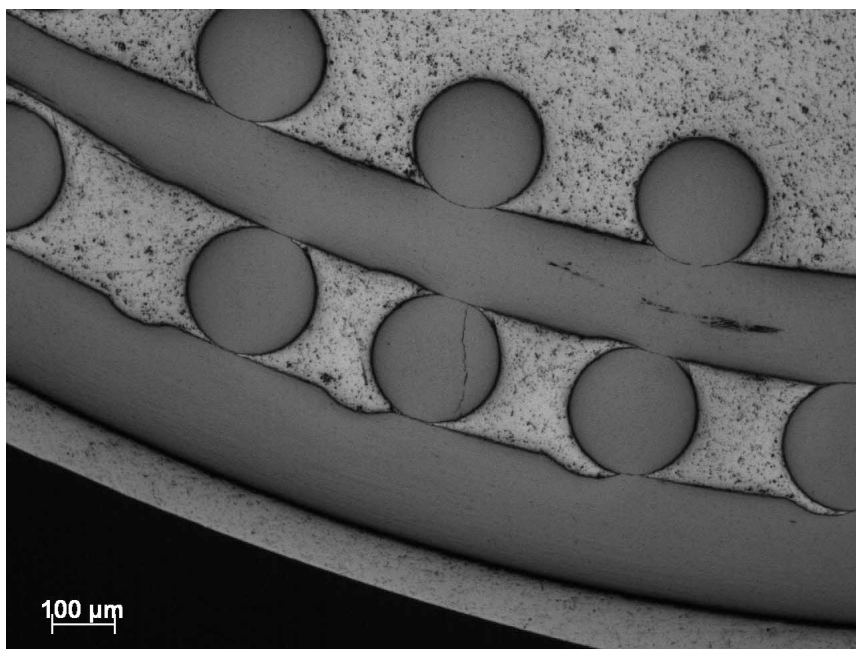


Figure 72 - Sample 7, Configuration 3, optical image. Notice the plastic indentation in the W wires due to the initial pressurization of the contacting W fibers. The motion of the W rods is due to the migration of aluminum into the W structure.

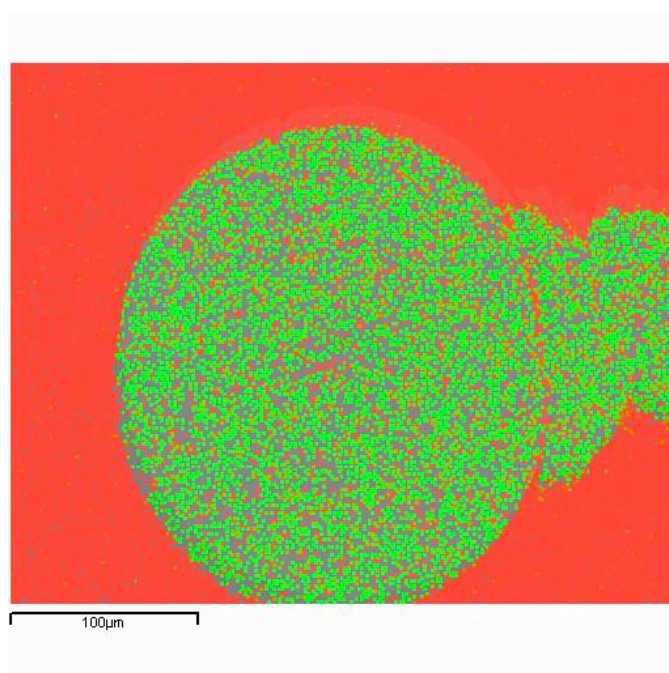


Figure 73 - SEM image of Sample 7, Configuration 3. Al 6061 is seen in red and W is seen in green. Aluminum seen inside the W was deposited during sample polishing. A W rod is seen plastically deforming a contacting W wire.

As seen in Figures 74 and 75 a layer of steel was left behind on the interior of the sample. Figure 74 shows also a reaction layer between the steel and aluminum similar to what seen in the consolidation studies of steel fibers with aluminum in section 1.4. A reacted layer between the steel and aluminum is inevitable during our HIP process due to the applied pressures and temperatures. Further work with multiple HIP cycles to build the composite should take into account the loss of aluminum from the inner diameter due to the subsequent reactions with the steel and the removal of that material with the steel core if it is to be replaced by more steel to help construct the next W layers. Thus the desired amount of aluminum left on the interior of the sample determines the size of the steel mandrel and how many HIP cycles can be used to apply the W layers. In the later samples of Configuration 3, 9-13 the reduction in overall HIP treatment time allowed the reacted layers of steel to be thin enough that it was fully removed when machining the desired inner diameter of 7.5 mm. Due to the non-uniformity of the composite structure, steel or its compound with aluminum was either absent (Figure 67) or as thick as 200 μm as seen in Figure 75 in various regions of sample 7.

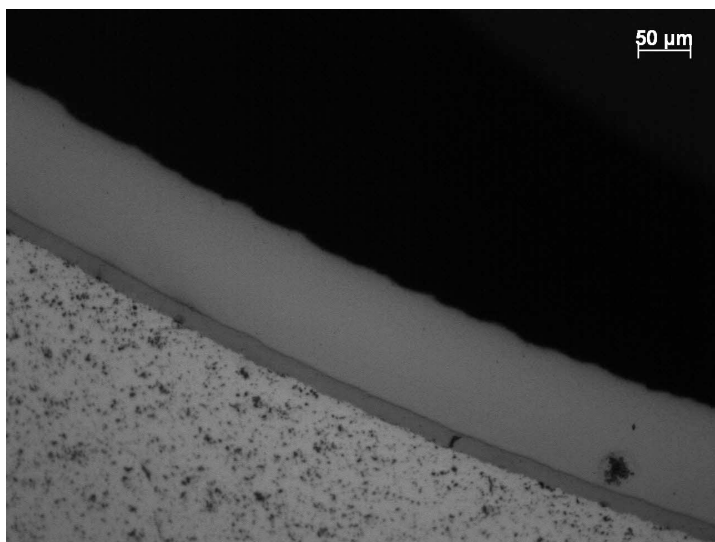


Figure 74 - Sample 7, Configuration 3, optical image showing the left over steel and the reacted layer of steel with aluminum below it after machining to an inner diameter of 7.5 mm.

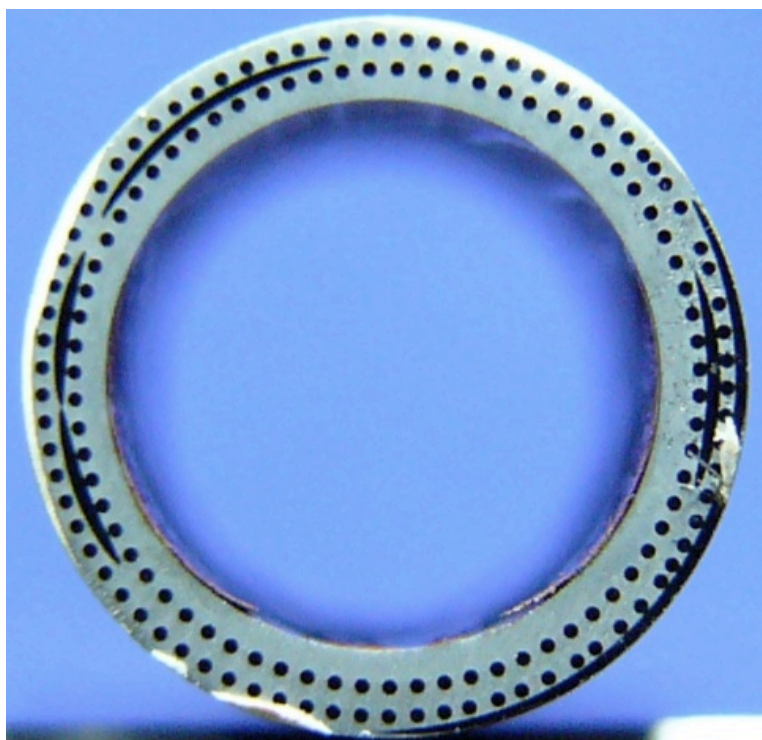


Figure 75 - Cross sectional view of Sample 7, Configuration 3 showing a thicker layer, perhaps as much as 200 μm of steel left behind after machining the inner diameter to 7.5 mm.

3.11 - Internally Pressurized Configuration 4 to Prevent Buckling of W Reinforcement

The slow pressurization rate indicated that “Brazilian” type fracture of the W fibers could be avoided but externally pressurizing the assembly still left specimens with buckled regions. Since buckling is an unstable deformation whose occurrence can appear at lower compressive loads it is of practical interest to avoid such compressive loading completely. Pressurizing from the inner surface of the cylinder would expand the inner aluminum tube into the W structure, keeping the W wires in tension. With all the components of the composite in tension buckling should be eliminated.

Sample 8 in Configuration 4 was successfully created with a single HIP process. With this Configuration pressurized from the interior surface of the cylinder Al 6061-T6 fully penetrated the W structure with no fracture of the W rods. No reaction compound created between the components. However stretching the W wires into a soft, hot outer aluminum tube resulted in fracturing of W wires, severely distorting the local W structure. The HIP cycle used to treat this sample was similar to the HIP cycles applied to sample 7. The sample was machined to a length of 90.12 mm, an inner diameter of 7.17 mm and an outer diameter of 10.47 mm. The resulting density of sample 8 was 6.03 g/cc due to the aluminum left in the interior of the sample and from the extra aluminum that flowed in the volume created by the fractured hoop W wires, expanding the entire W structure.

The eighth sample in Configuration 4 was prepared similar to what has been described previously. The concentric components are listed in Table 5. The aluminum tubes and W structure are prepared in the same fashion as described for sample 2, but with different spacing for W components. Instead of using a central steel mandrel the inner aluminum tube is used instead. This inner Al 6061-T6 tube had a length such that the ends would be outside the aluminum jacket. A stainless steel cylinder approximately 10 mm in thickness is slid around the assembly as seen in Diagram 9. As seen in section 3.10, an undesired compound can be created between the steel and aluminum, and by the use of stainless steel this compound can be prevented from being created in this HIP process. The assembly was then slid into an Al 6061-T6 tube that would be capped to form a jacket. The Al 6061-T6 caps were welded into place as seen in Diagram 10. To pull vacuum an additional aluminum cylinder was welded onto the outside of the aluminum jacket from which a tiny hole, 30 μm in diameter was drilled concentrically to the samples outermost Al 6061-T6 tube as seen in Diagram 11. A vacuum of 6 microns was created and sealed first mechanically, and finally with a weld. The final assembly prepared for the HIP treatment can be seen in Figure 76. The degassing tube is at a 30-degree angle to the axis of the assembly in order to fit inside the HIP furnace.

Table 5 - Components for Sample 8, Configuration 4.

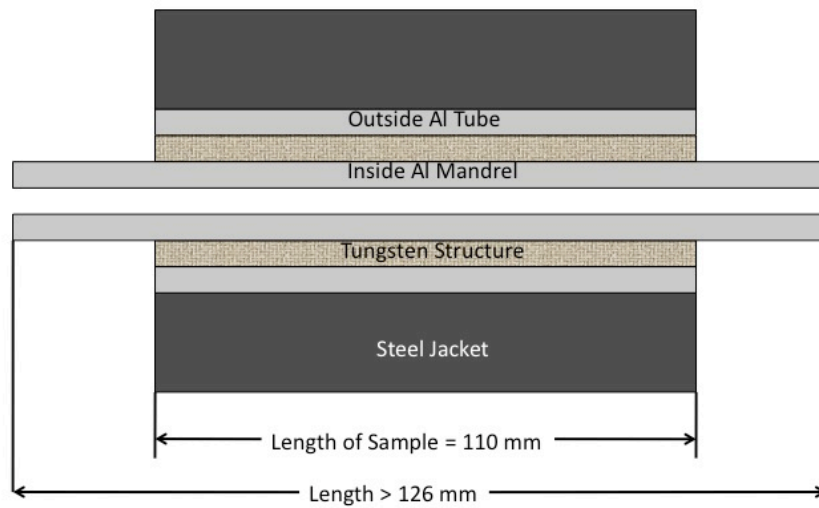
DARPA Internal HIP Geometry

Property/ Part	Inside Al Mandrel	Tungsten Structure	Outside Al Tube	Steel Jacket	Outside Al Jacket	Al End Caps x 2
Color Key						
Material	Al 6061	200 um (dia) W rods and wires	Al 6061	Stainless Steel 303	Al 6061	Al 6061
Length (mm)	> 126	110	110	110	124	6
Inner Diameter (mm)	2.36	8.5	10.1	11.1	31.75	8.5
Outer Diameter (mm)	8.5	10.1	11.1	31.75	43.75	31.75

Note: Geometry is made up of concentric cylinders.

Excess length of inside Al mandrel is removed after final welding.

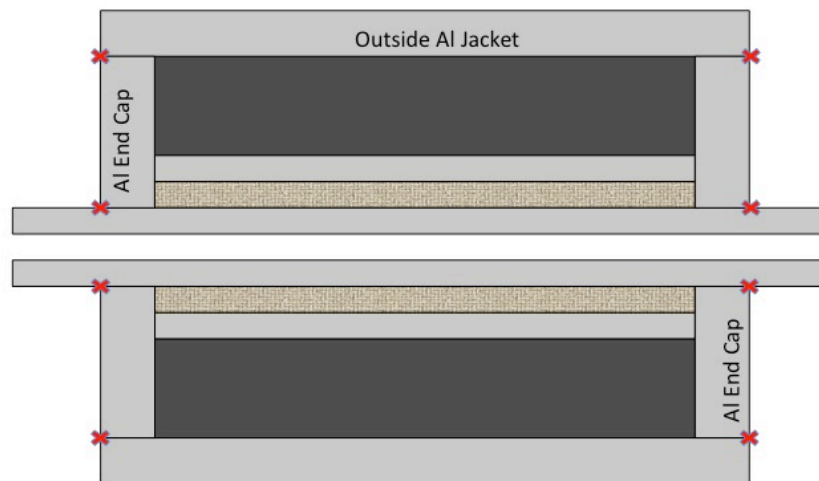
DARPA Internal HIP Sample and Steel Confinement



Note: Not Drawn to Scale.

Diagram 9 - Cross section view of the assembly for Configuration 4 inside the rigid stainless steel tube for internally applied HIP pressure.

DARPA Internal HIP Sample and Al 6061 Jacket Assembly



Note: Not Drawn to Scale.
Edge welds marked with a red 'x'.

Diagram 10 - Cross section view of the assembly for Configuration 4 inside the Al 6061-T6 jacket.

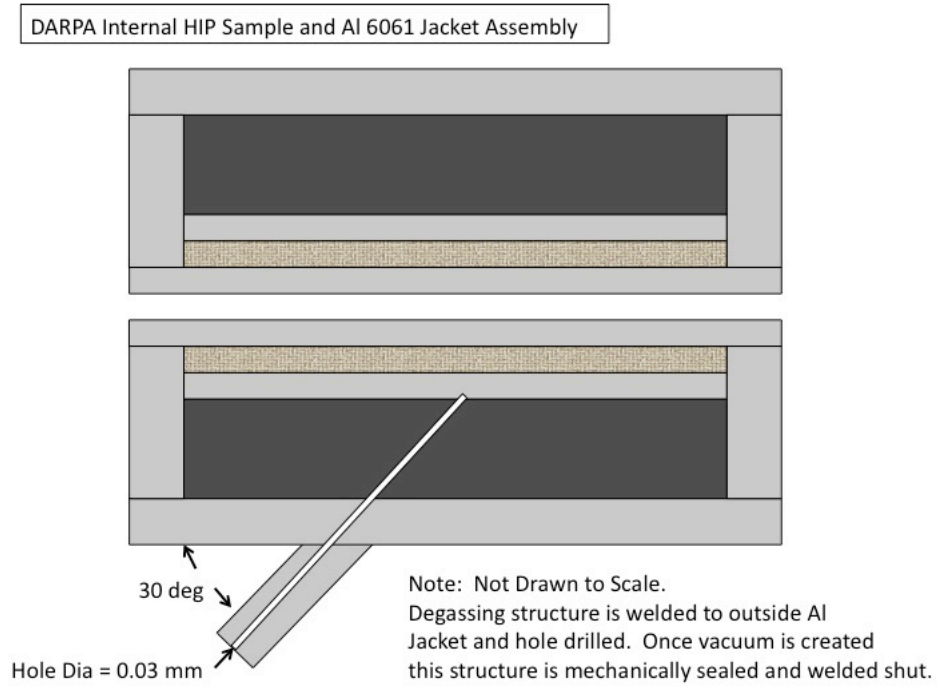


Diagram 11 - Cross section of the assembly of Configuration 4 with the additional degassing tube for vacuum sealing.



Figure 76 – Final vacuum sealed assembly of Sample 8, Configuration 4 before HIP treatment.

Sample 8 underwent the same HIP process that was applied to samples 3 and 4 of Configuration 1 (Diagram 9). This HIP treatment of sample 8 had mixed results. Although the Al 6061 fully penetrated the W structure without forming an Al-W compound or fracturing the W rods the W wires were stretched and fractured. As can be seen in Figure 77, the outermost aluminum tube did not restrict the displacement of the W wire caused by internal pressure, resulting in fracture of W wire in tension. Once fractured, the entire W structure expanded and aluminum flowed to fill the space within the W structure.

Internally pressurizing the composite has the best potential to avoid all the flaws experienced with external pressurization; “Brazilian” fracture, buckling, and compound formation. Improvements to Configuration 4 include the reduction in the thickness of the outermost Al 6061-T6 tube, and reduction of the internal diameter of the stainless steel tube. This would result in the steel resisting the displacement of the W wire, avoiding fracture of the wire. “Brazilian” fracture could be induced into the W wire instead but it is thought that with a thin layer of aluminum between the outermost W wire and the steel the aluminum would plastically deform to distribute the load to the wire thus avoiding “Brazilian” fracture. Care must be taken to make sure enough volume of aluminum is available to flow into the W structure without causing fracture of the aluminum tubes, resulting in the loss of vacuum. Thus for samples with more than four layers of W challenges remain to be solved to scale up the design of Configuration 4.



Figure 77 - Cross section of Sample 8, Configuration 4. Notice the fractured W wires to the left, causing the entire W structure to expand outward.

3.12 – Mechanical Testing Setup

Samples 5 and 6 in Configuration 1 were prepared for mechanical tensile testing as can be seen in Figure 78. Sample 5 in Configuration 1 was machined to an average outer diameter of 9.16 mm and an inner diameter of 7.5 mm inside a 15 mm gauge section. The gauge section was slightly ellipsoidal in shape due to the buckle present in the W wires with major and minor diameters of 9.3 mm and 9.1 mm respectively. The outermost W wire layer was gently exposed. Overall length of

sample 5 at this point for tensile testing was 59.69 mm. The outer diameter of sample 6 was 9.7 mm with an inner diameter of 7.45 mm in the gauge region of 10 mm.

Overall sample 6 was 43.9 mm in length before mechanical testing.



Figure 78 - Sample 5, Configuration 1 prepared for tensile testing. Notice transverse buckling in the outermost exposed W wire layer.

The samples were placed between steel clamps machined to fit to its geometry as can be seen in Figure 79. These clamps were then installed within a steel casing as seen in Figure 80 and attached to the tensile machine through a pin connection (Figure 81). The dimensions of the samples and steel clamps could be found in Diagrams 12 and 13 respectively.

In order to measure the strength of this metal matrix composite several issues had to be addressed. Achieving the desired density in the gauge region was critical, otherwise the information collected would be for a different composite. However, the cylindrical samples could not be supported directly through pin connections since the material properties throughout the sample would be anisotropic. Machining through the aluminum matrix and W fibers could introduce unwanted stress concentrations with the pin connection itself being another source of premature failure. By leaving behind part of the aluminum jacket to be machined into a shoulder clamps was used to hold the composite samples. The clamps would transmit load axially and in the radial direction into the sample due to their 15-degree slope. To support the samples locally in the region of the clamps steel plugs were inserted inside the central hole of the samples such that the sample would not be squeezed or multi-axially loaded. This sort of design was felt to minimize the creation of stress concentrations while adequately loading the samples in the axial direction.

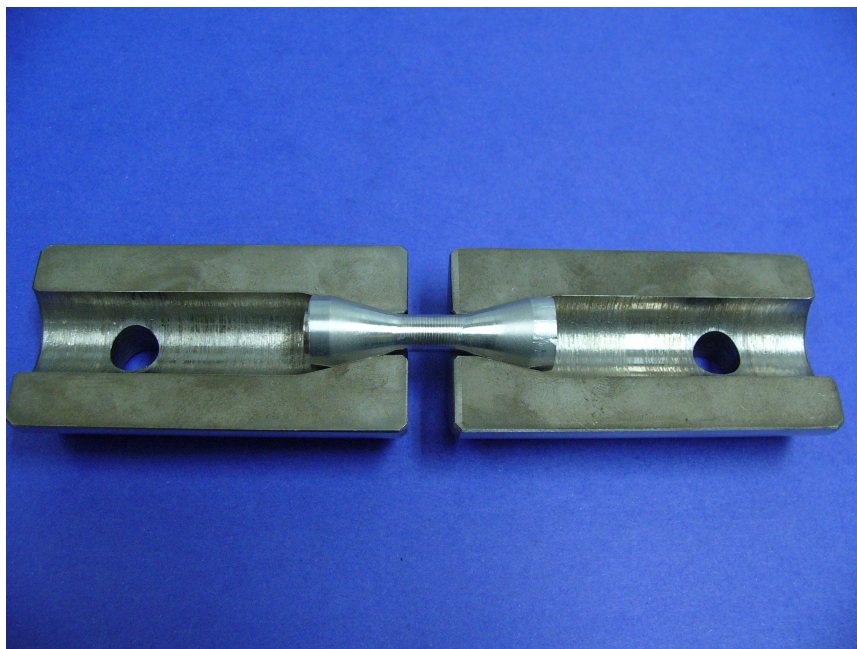


Figure 79– Geometry of Sample 5, Configuration 1 within the steel clamps. Only half of the steel clamps are shown.

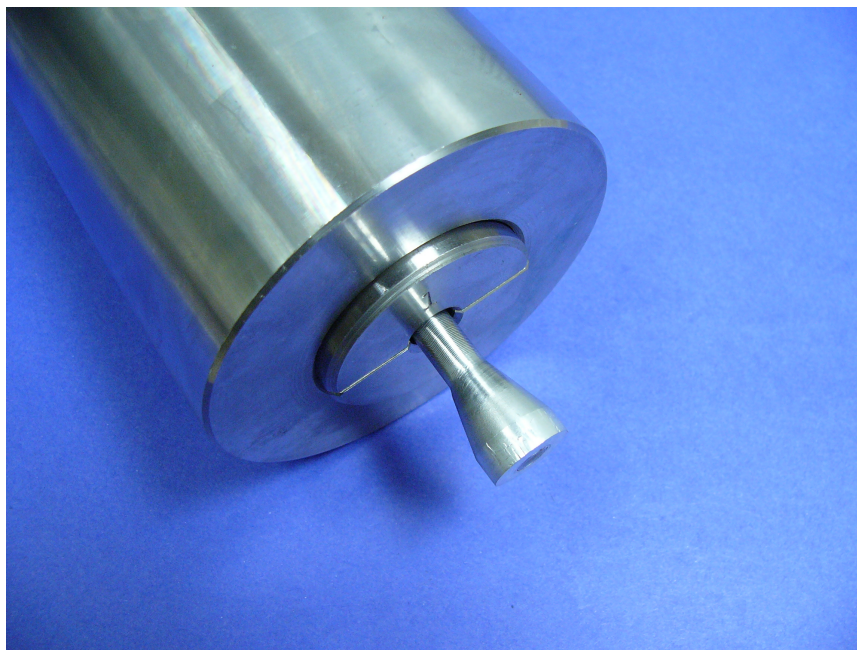
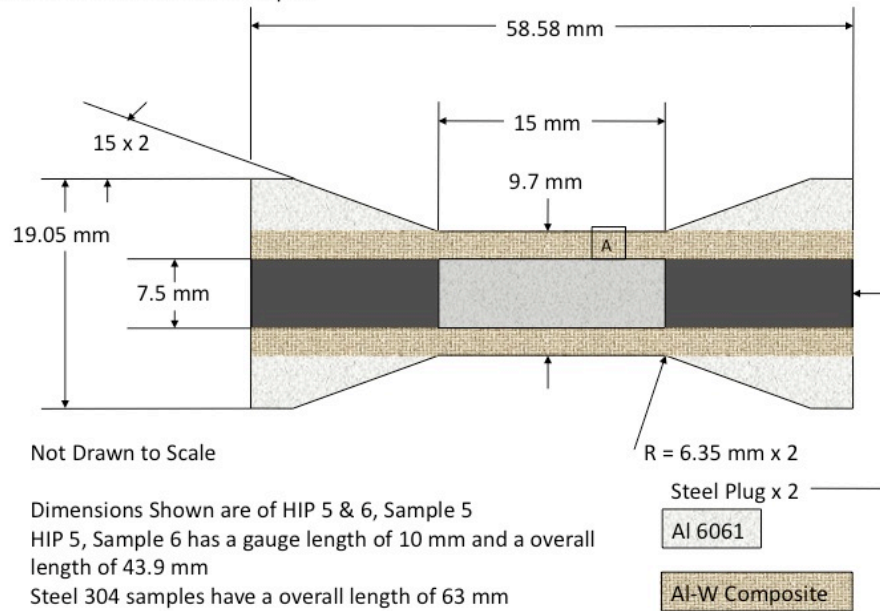


Figure 80 - Geometry of steel casing around the steel clamps and Sample 5, Configuration 1.



Figure 81 - Tensile test set up with steel clamps and casing holding Sample 6, Configuration 1.

DARPA
Mechanical Tensile Test Samples



DARPA Mechanical Test Sample
View A

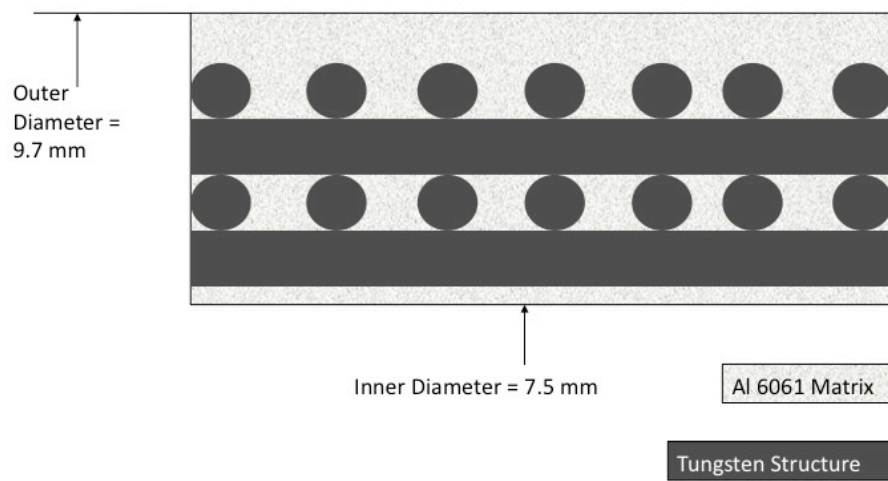


Diagram 12 - Sketch of Al-W composite specimen for tensile testing. The outer diameter of the gauge section in sample 5 was reduced to 9.16 mm to expose the W wire.

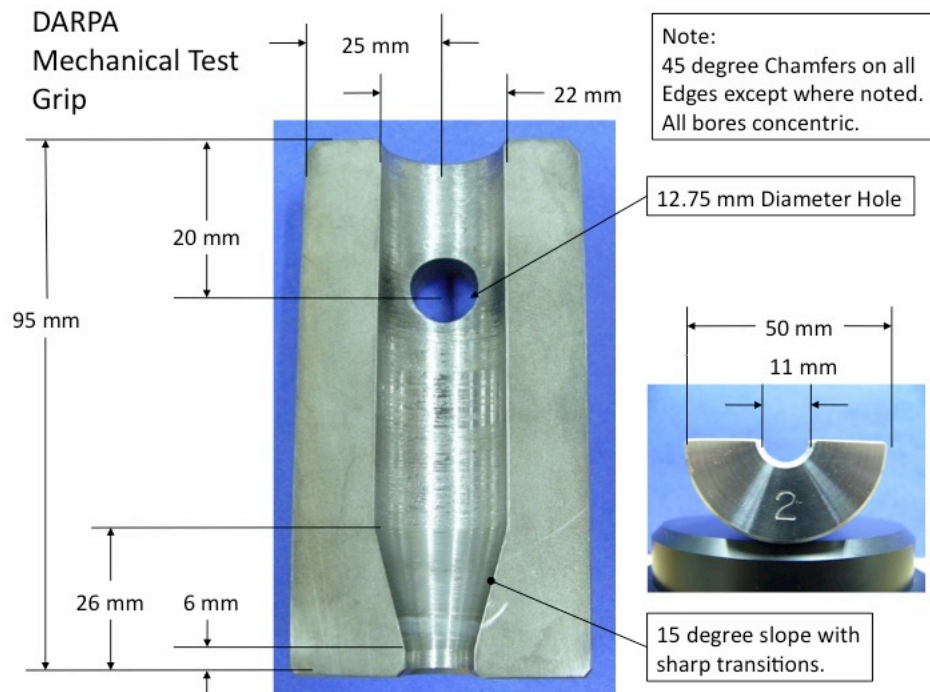


Diagram 13 - Dimensions of specimen's steel clamp for tensile testing.

3.13 – Tensile Test Validation

Three samples of 1018 Steel with hardness 219 HB were machined as described in section 3.12 and Diagram 12 and underwent destructive tensile testing. The goal of the studies was to validate our testing setup and procedure for our composite. As seen in Diagram 14, the elastic modulus of all three samples closely matched the documented modulus of 205 GPa. The average ultimate tensile strength measured, 675 MPa is close to the documented value of 634 MPa for 1018 steel with hardness 197 HB close to the value of the tested samples. The average 0.02% yield strength was measured to be 512 MPa and is illustrated in Diagram 15. Elongation to

failure was measured to be an average of 15% as seen in the stress strain curve for the samples in Diagram 16. It is felt that due to the possible heat treatment of the steel that the strength was increased while sacrificing ductility. Overall the values obtained for these 1018 steel samples match the documented data, thus validating our tensile test findings.

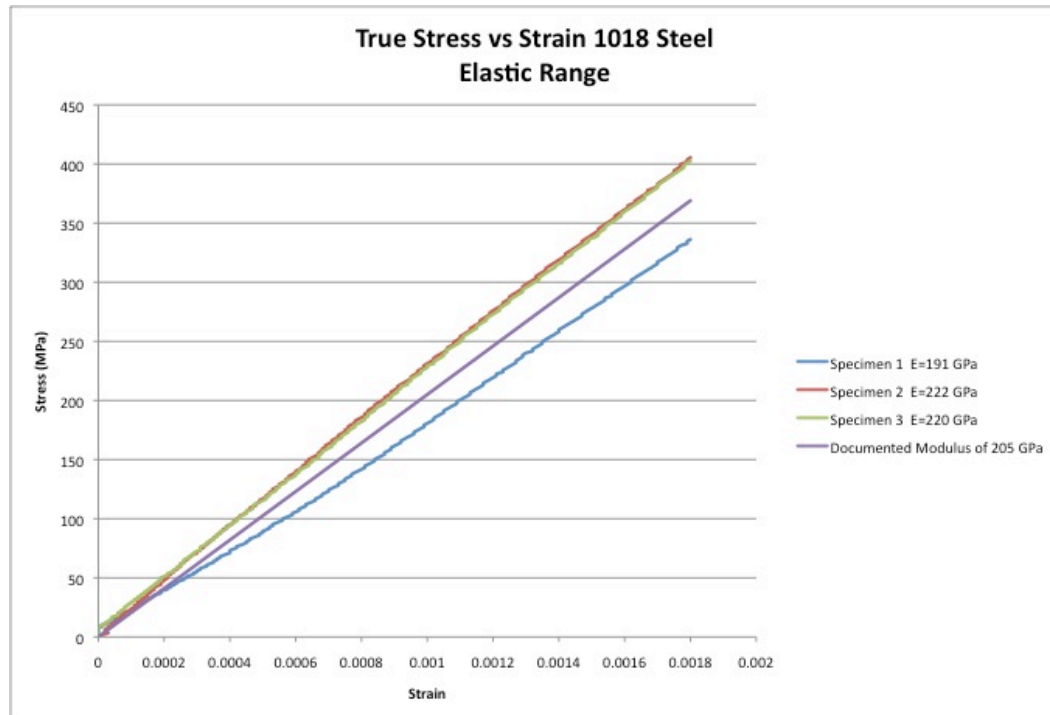


Diagram 14 – Strain strain curve of the elastic range for 1018 steel samples. A strain guage was used to measure strain.

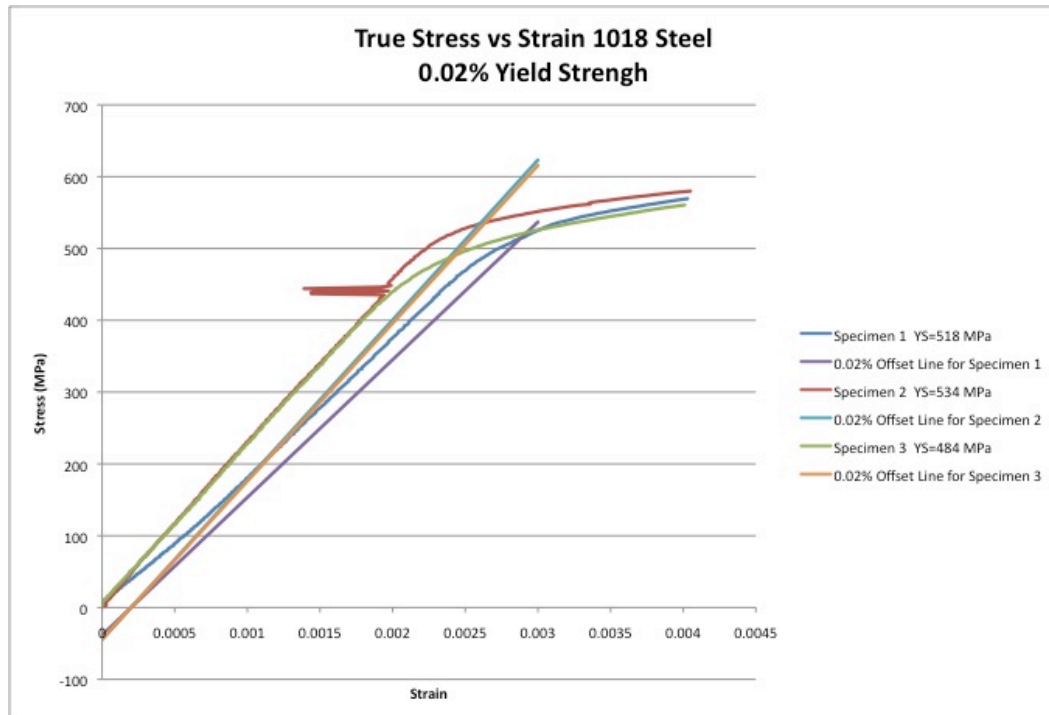


Diagram 15 – Strain strain curve of the elastic range for 1018 steel samples with 0.02% offset curve.

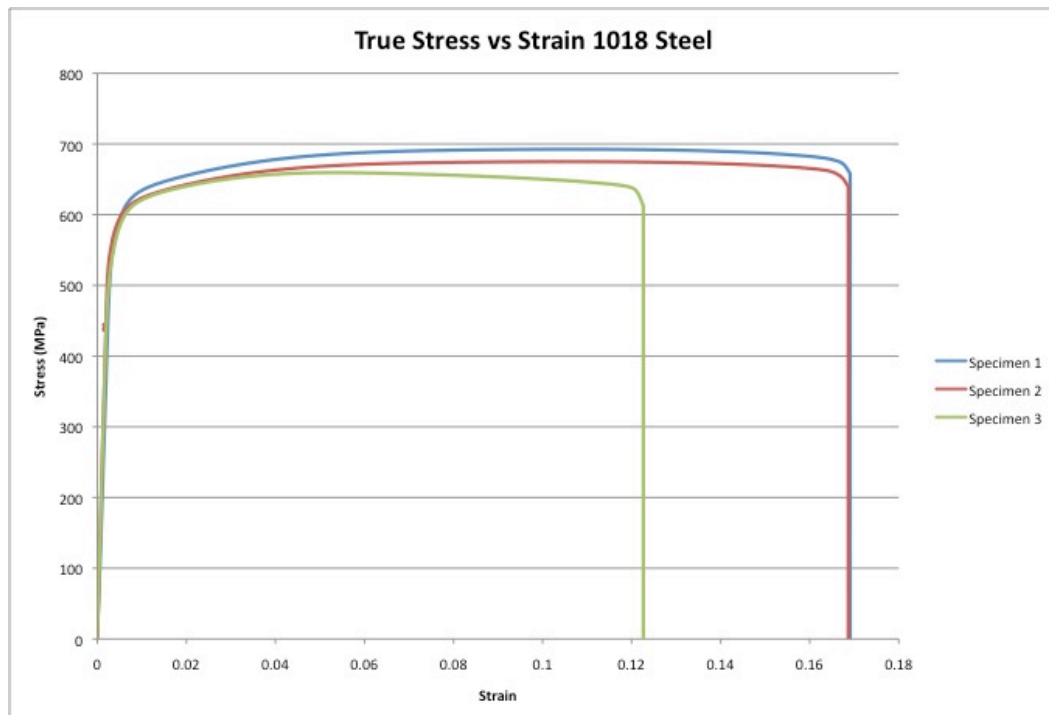


Diagram 16 – Strain strain curve of the 1018 steel samples.

3.14 – Mechanical Strength of Samples of Configuration 1

Few composite samples were prepared for mechanical testing. It was observed that sample 6 fractured at 408 MPa in a brittle fashion within the shoulder region, a stress concentrator. The density of this loaded region was found to be 7.34 g/cc. Sample 5 also fractured in a brittle fashion within the gauge section with an ultimate tensile strength of 647 MPa and a density of 9.37 g/cc. Overall the samples fractured in a brittle fashion.

The successful tensile tests of samples 5 and 6 showed that this Al-W metal matrix composite behaves in a brittle fashion at room temperature. A maximum of stress of 408 MPa was reached before the sample fractured at a shoulder with sample 6. An ultimate tensile stress, as seen in Diagram 17, of 648 MPa was measured in sample 5, which fractured in the gauge section. These stress values exceed the ultimate tensile strength of the Al 6061-T6 alloy of 310 MPa and the phase one goal of 345 MPa. It should be noted that after the HIP treatment the aluminum matrix is no longer in the T-6 condition as discussed in section 3.6. The strain measured in these tests should be disregarded since it is the head displacement of the tensile machine without a correction for the elastic displacement of the machine itself. The outer and inner diameter of the gauge regions did not change which is to be expected during brittle fracture. Sample 6 fractured at a stress concentration and the composition of sample 5 had a greater density than we desired. By adding an additional area of Al 6061-T6 to sample 5 to bring the density to 7.8 g/cc, the ultimate tensile strength of the composite

with density 7.8 g/c can be estimated. This data from both samples represents minimum values of ultimate tensile strength for the composite.

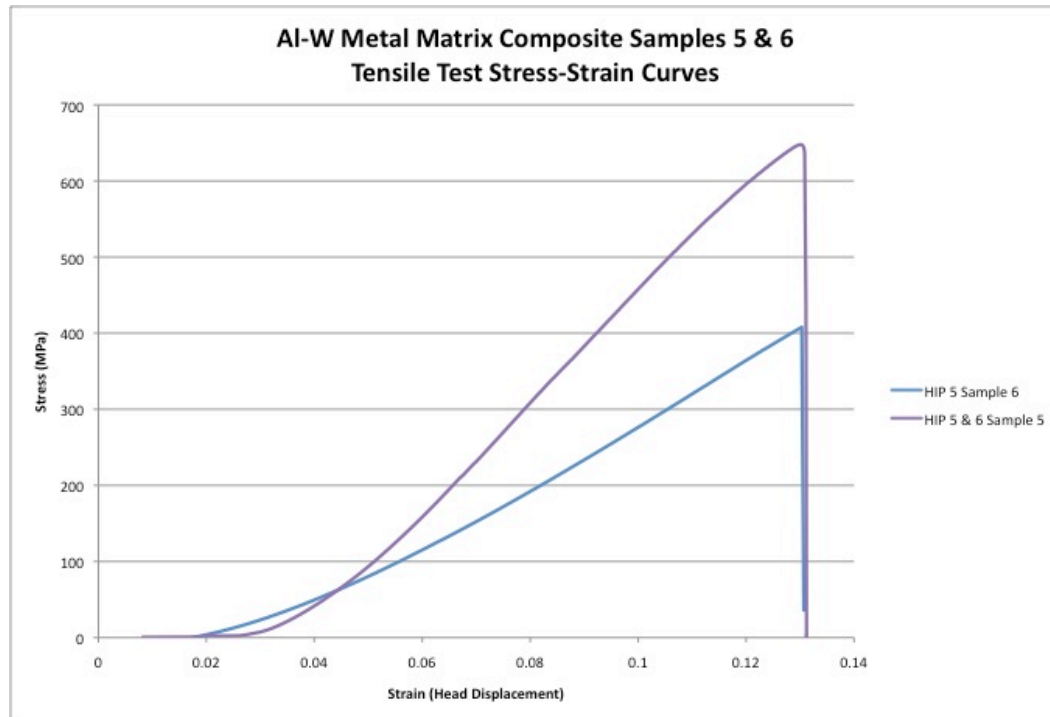


Diagram 17 - Tensile test result for Samples 5 and 6, Configuration 1.

Further observation of the fracture surfaces of samples 5 and 6 in Configuration 1 after the tensile test under an SEM reveals the mechanism in which the components interacted to support the tensile load. Figures 82 and 83 show the fracture surfaces of the samples 6 and 5 respectively. Notice the cone shape of the W rods suggesting the rods carried the load until ultimately fracturing in a plastic fashion, thus transferring an overload stress to the Al 6061 matrix. As seen in Figure 84, the aluminum matrix has a dimpled surface, indicative of a ductile material fully

supporting a load plastically before ultimately fracturing. It is also seen that most the W rods fractured in the same plane as the aluminum matrix, suggesting fiber pullout was minimized. Figure 85 shows the extent of fiber pullout seen in the samples.

If the components move with respect to one another they will not interact to behave as a single material, but as separate parts. Since the thermal expansion coefficient of aluminum is four times that of W, as the composite cooled during the HIP process, the aluminum matrix squeezed the W fibers. This contraction is so intense, the aluminum in the vicinity of the fiber would plastically deform but maintain an extremely tight interface. This tight interface would also include the interlocking surfaces of the components. Due to this thermal contraction and the flow of the plastic aluminum into the W structure the dislocation density within the aluminum matrix is quite high [5]. The dislocation density in W fiber aluminum composites was measured to be on the order magnitude of 10^{11} to 10^{12} per square meter [5]. These dislocations increase the mechanical strength of the composite [18]. Figure 86 shows radial cracking throughout the W rods. These cracks could result from those residual thermal stresses being released upon tensile fracture of the cross section and also due to local bending of W rods during the test.

It is thought the W wire in the hoop direction does not significantly contribute to the axial strength of this composite, so the buckled wire should have no effect on the tensile strength. Supporting evidence for this was seen in that both samples where the fracture surfaces did not stay between two W wire coils but jumped between layers defined by the W coils as observed in Figure 83. As expected, delamination between

the W wire and the aluminum matrix would occur before the fracture of the W wire as seen in Figures 84 and 85. The W wire would carry load to immediate failure as the composite matrix fractured. The buckled W on the other hand may have carried axial loads, thus contributing to the strength, but this contribution is felt to be minor compared to the W rods. From samples 5 and 6 confidence was gained that the goal of an ultimate tensile strength of 345 MPa can be approached with the current geometry. Further studies with Configuration 3 were pursued due to this Configurations superiority over other Configurations in minimizing flaws such as buckling and compound formation.



Figure 82 – Fracture surface due to tensile testing of Sample 6, Configuration 1. Notice the cone shape of the broken W rods, suggesting the W fibers supported the axial load until they fracture themselves, translating a load to the aluminum matrix.

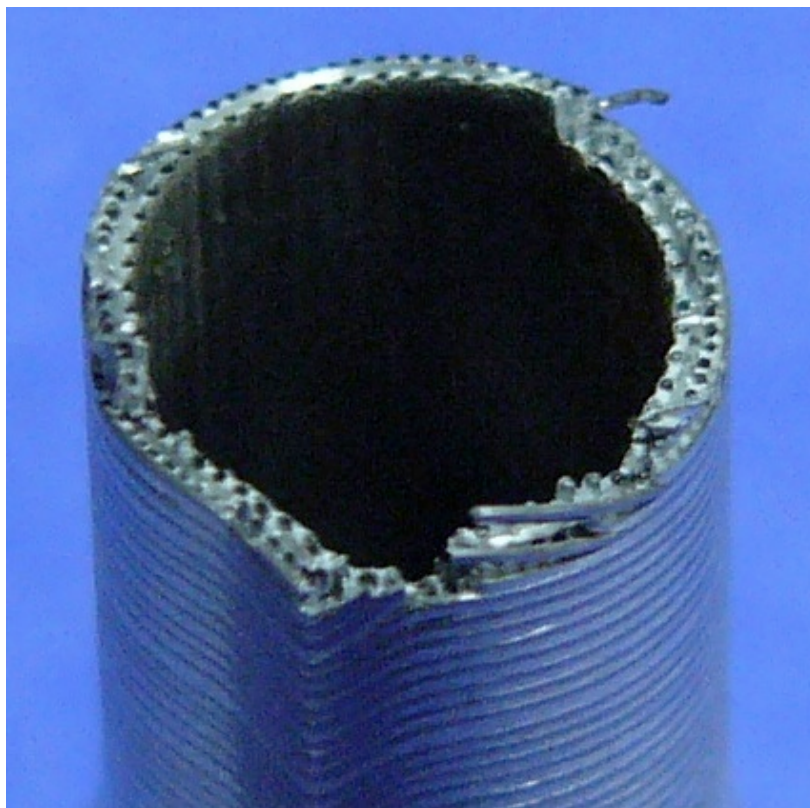


Figure 83 – Fracture surface due to tensile testing of Sample 5, Configuration 1. Notice the inter-planar fracture surface.

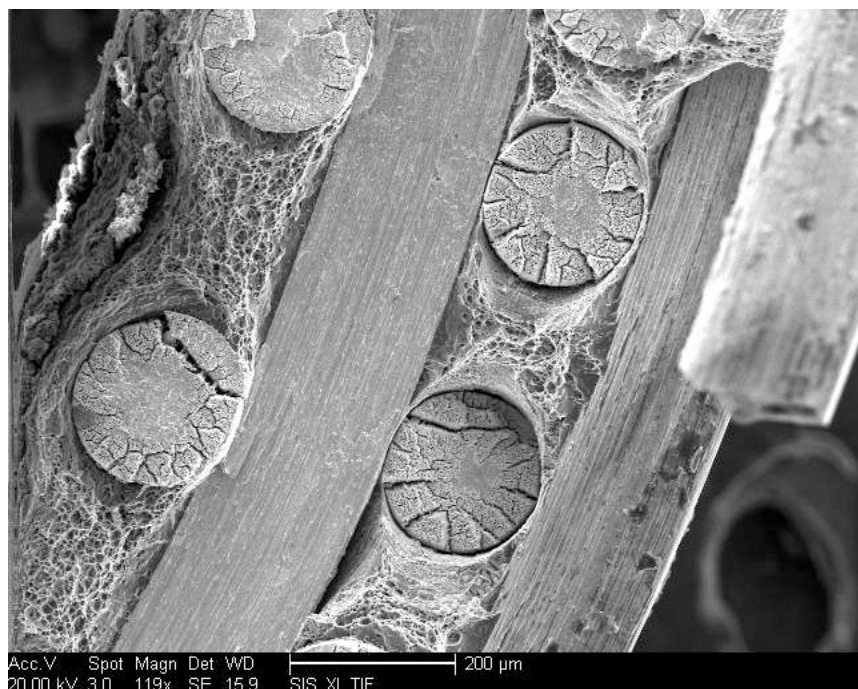


Figure 84 - SEM picture of the fracture surface of sample 5, Configuration 1 after tensile testing.

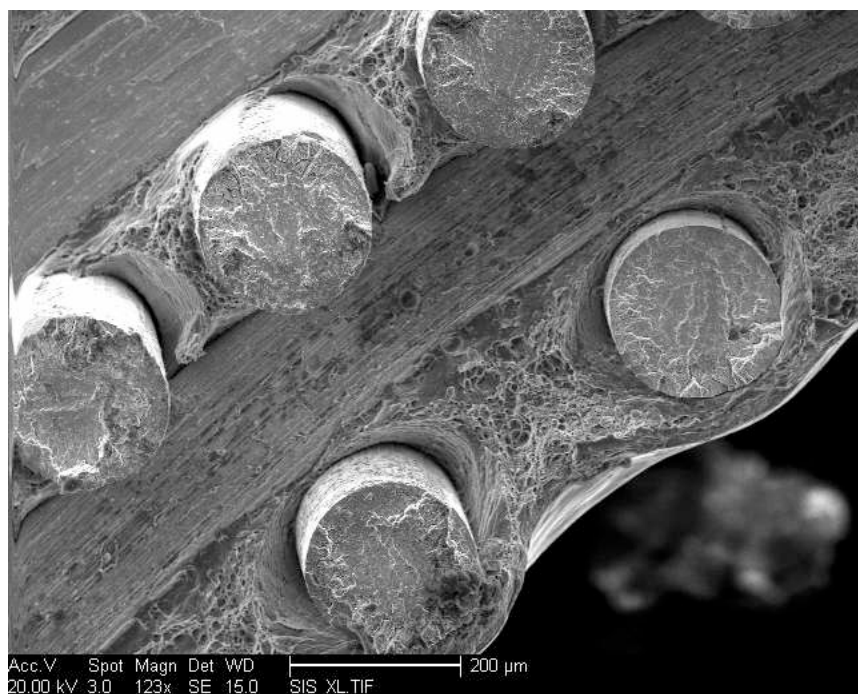


Figure 85 - SEM image of the fracture surface of Sample 6, Configuration 1.

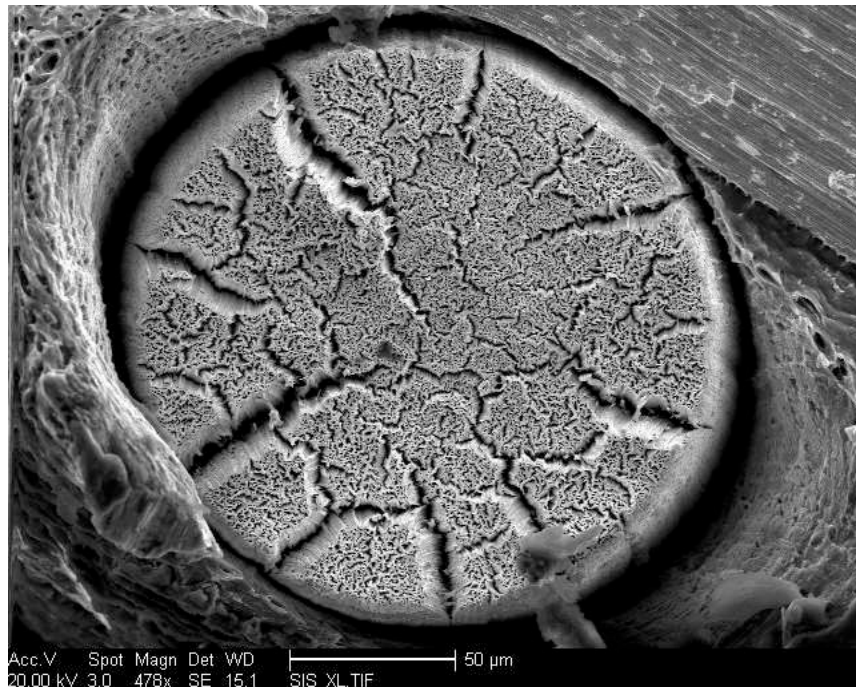


Figure 86 - SEM picture of a W rod fracture surface from sample 5, Configuration 1 after tensile testing.

Section 3.15 – Mechanical Strength and Critical Strain of Configuration 3

Even with flaws in sample 7 in Configuration 3 important DARPA goals for density and strength were achieved. Excess aluminum alloy 6061 is removed such that the outermost W wire layer is exposed. This aluminum, if left intact would not pass through the W structure during the planned explosive event, thus fracturing away from the composite region in large particles. Aluminum is desired on the inside of the cylindrical samples, as it is this inner aluminum that will be extruded through the W structure to break up into small hot aluminum particles. Since the removal of

aluminum is the method of choice to tune the desired density similar to the density of steel, the amount of aluminum alloy 6061 inside of the W structure varies with each sample. Ideally if the W structure does not move during the HIP treatment, it is expected a layer of aluminum 300 μm in thickness should be left behind. The density of the pieces of sample 7 for example ranged between 7.48 g/cc and 7.69 g/cc (target density of 7.8 g/cc).

Samples 10, 11 and 13 were prepared and tested in a tensile test as described in section 3.12. The ultimate tensile strength of Configuration 3 was measured to be an average of 453 MPa, exceeding the DARPA phase 1 requirement of 345 MPa. The gauge region of length 30.86 mm for sample 10 had an inner and outer diameter of 7.45 mm and 9.8 mm respectively. With an outer diameter of 9.8 mm, the outermost W wire layer was exposed, and in some places damaged in the cutting process as seen in Figure 87. Sample 11 also had a gauge length of approximately 30 mm with an inner and outer diameter of 7.5 mm and 9.97 mm respectively. Sample 13 had an inner and outer diameter of 7.5 mm and 10.11 mm respectively inside a gauge region of approximately 25 mm.

Similar to the results seen in Configuration 1 in section 3.14 the samples fractured in a brittle fashion with no measurable change in diameter after fracture. The ultimate tensile strength of sample 10 was measured as 475 MPa. The stress-strain curves obtained with samples 11 and 13, which broke inside the gauge region had a strain to failure of approximately 1.2% as can be seen in Diagram 18.

For samples 10 and 11 approximately 10%-20% of the W fibers pulled out of the aluminum matrix as seen in Figures 87 and 88. This is thought to be due to voids left behind after the second HIP process in the contact region between the W fibers seen in Figure 71 to weaken the interface of the fibers with the matrix. If the W fibers were fully surrounded by the aluminum matrix, the aluminum would squeeze the fibers during the cool down portion of the HIP treatment due to the thermal expansion coefficient of aluminum being four times greater than tungsten. This difference in contraction of the W fibers and surrounding aluminum matrix would deform both the aluminum and tungsten, leaving behind an interface region with intense pressures and stresses. Additionally when voids are left behind between the aluminum tubes as seen in Figures 63 and 64 this interface pressure can be reduced between the fibers and the composite. Without knowing the coefficients of friction between the W fibers and the aluminum matrix and the W fibers with themselves it is difficult to compare how each interface resists mechanical loading. However, it can be assumed that the coefficients of friction at the interfaces, aluminum and tungsten as seen in Figure 89, and the W fibers with themselves, are high due to microscopic interlocking created by the HIP processes. With a weak interface between some of the fibers and the composite matrix in samples 10 and 11, fracture occurred along the interface with the application of the tensile load, separating the fiber from the composite and preventing them from partaking the tensile load.

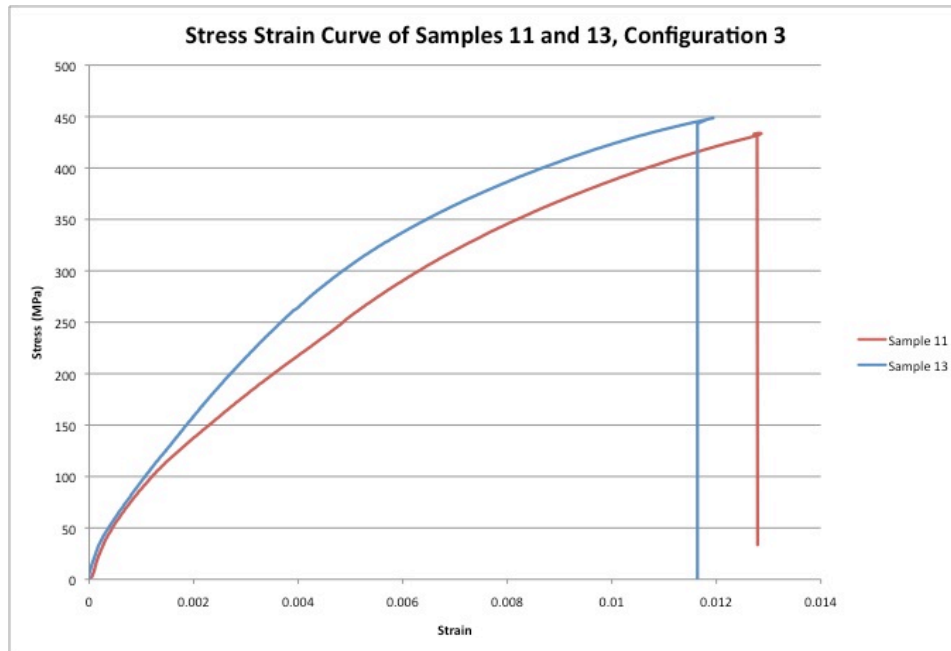


Diagram 18 – Stress strain curve for Samples 11 and 13, Configuration 3.

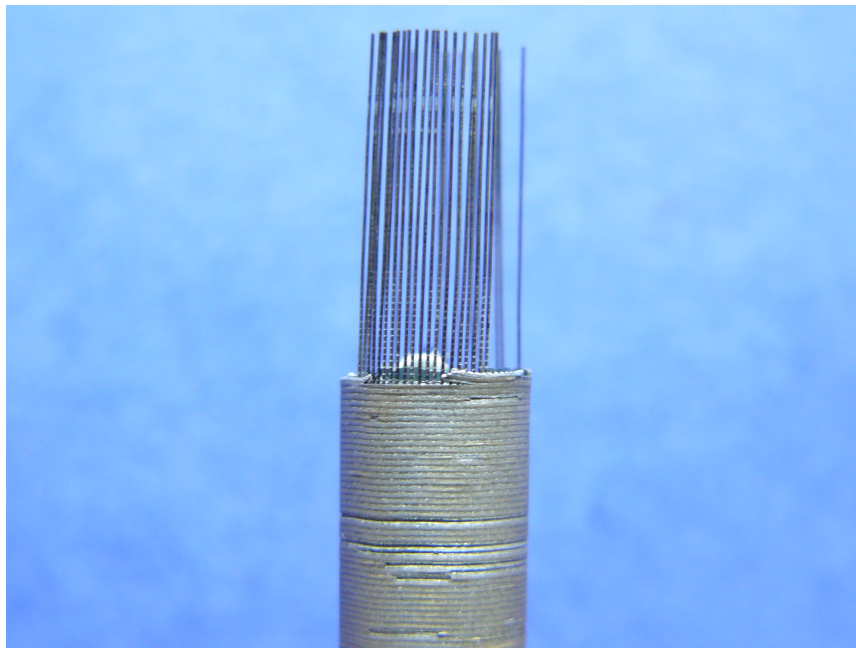


Figure 87 - Sample 10, Configuration 3 after mechanical testing. Exposed W wire was damaged in sample machining for mechanical test.



Figure 88 - Sample 11, Configuration 3 after mechanical testing.

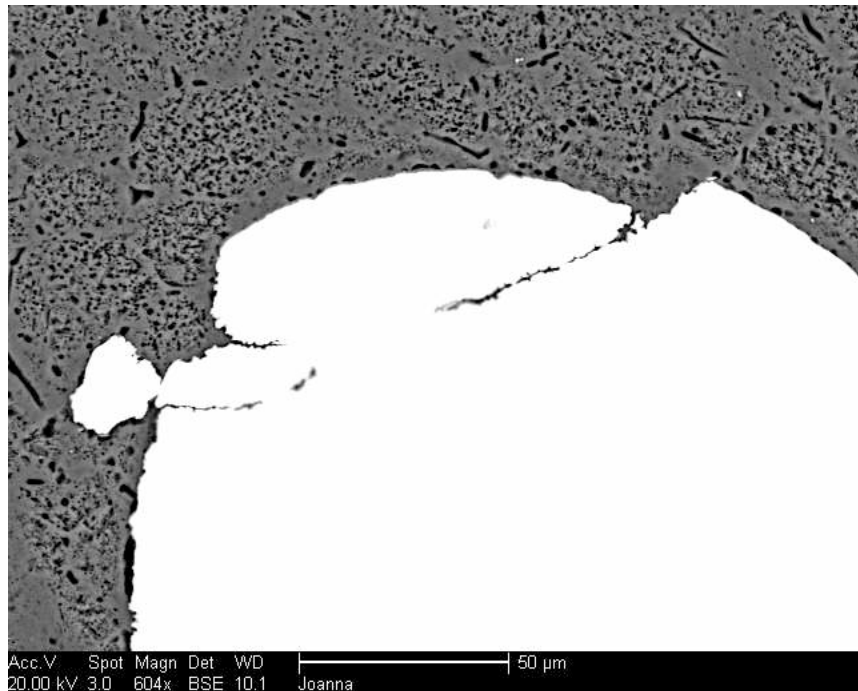


Figure 89 – SEM image of Sample 7, Configuration 3 after the second HIP treatment. Notice the microscopic infiltration of aluminum into the surface of the W rod. The fracture of the W rod is attributed to the local clamping performed at the end of the sample during assembling of W reinforcement prior to HIP treatment.

However sample 13 did not fail with fibers being pulled from the matrix as shown in Figure 90 and yet had the same flaws in the form of voids. It is thought that perhaps due to mechanical locking with the heavily deformed aluminum tubes and plastically deformed W wires, that the rods were kept in place. This locking however did not help the tensile strength of sample 13, 449 MPa overcome the strength of sample 10, 475 MPa. It is thought that other flaws such as delamination between the W wires and aluminum matrix and stress concentrations such as broken W wires introduced in the machining process contributed to the lower strength. Differences in the density between the samples, and thus the amount of aluminum in each sample also explain the variation in strengths. Future studies with samples of consistent

density and structure with minimal flaws such as voids may result in ultimate tensile strength greater than presented here. However even with these flaws this aluminum tungsten fiber composite obtains a robust strength exceeding the DARPA phase 1 requirement of 345 MPa.



Figure 90 - Sample 13, Configuration 3 after mechanical testing. Exposed W wire was damaged in sample machining before testing.

From the stress strain curves obtained from samples 11 and 13, the elastic modulus can be found and compared to the predicted elastic modulus. To find the theoretical modulus of this composite, we will assume the cross section resisting the tensile load is exactly 15% tungsten with the remainder being Al 6061-T6. For an arbitrary cross sectional area, A , an arbitrary gauge length, L and an arbitrary applied force, P with the moduli of the components being 69 GPa and 400 GPa for the Al 6061-T6 and tungsten respectively, the effective elastic modulus of this tungsten fiber composite can be predicted by the following analysis. Note the modulus of the aluminum matrix is expected to remain constant although the overall strength of the matrix was reduced due to the HIP treatments.

$$\sigma = E\varepsilon = \frac{P}{A} \quad (8)$$

$$\varepsilon = \frac{\delta}{L} \quad (9)$$

$$\delta = \frac{PL}{AE} \quad (10)$$

$$P = P_{Al} + P_W \quad (11)$$

Equations 8 and 9 are combined to derive Equation 10, and from Equation 10 the following can be derived. The deflection of the composite and the components is assumed to be the same.

$$P_{Al} = \frac{\delta A_{Al} E_{Al}}{L} \quad (12)$$

$$P_W = \frac{\delta A_W E_W}{L} \quad (13)$$

Using Equations 10-13 the following can be derived.

$$E = \frac{PL}{A\delta} = \frac{(P_{Al} + P_W)L}{(A_{Al} + A_W)\delta} = \frac{\left(\frac{\delta A_{Al} E_{Al}}{L} + \frac{\delta A_W E_W}{L}\right)L}{(A_{Al} + A_W)\delta} \quad (14)$$

$$E = \frac{A_{Al} E_{Al} + A_W E_W}{A_{Al} + A_W} \quad (15)$$

Introducing the relative values of cross sectional area of Al and W equally 0.85 and 0.15 correspondingly obtain the elastic modulus of this metal matrix composite.

$$E = (0.85 * 69 + 0.15 * 400) GPa = 119 GPa$$

By plotting a line with a slope of 119 GPa passing through the origin in Diagram 19 we see that the elastic region of our composite either is extremely small, 0.1% strain, or the composite displays nonlinear elastic behavior. Further tensile tests that release and reload the samples are required to ascertain the full mechanical behavior of this composite, including the onset of plasticity. For example, the shape and behavior of the stress strain curve is expected to be dominated by the tungsten fibers, meaning the fibers themselves would have a stress strain curve similar to the curve for our composite. The nonlinear behavior observed could be determined by the plastic behavior of the tungsten fibers, yet dominated by the plastic deformation of the

aluminum decreasing the cross sectional area. However the W fibers may restrict the aluminum from shrinking as expected explaining why no change in the cross sectional area in the region of fracture is observed. In all cases a complicated nonlinear interaction between the components of our composite dictate the mechanical behavior.

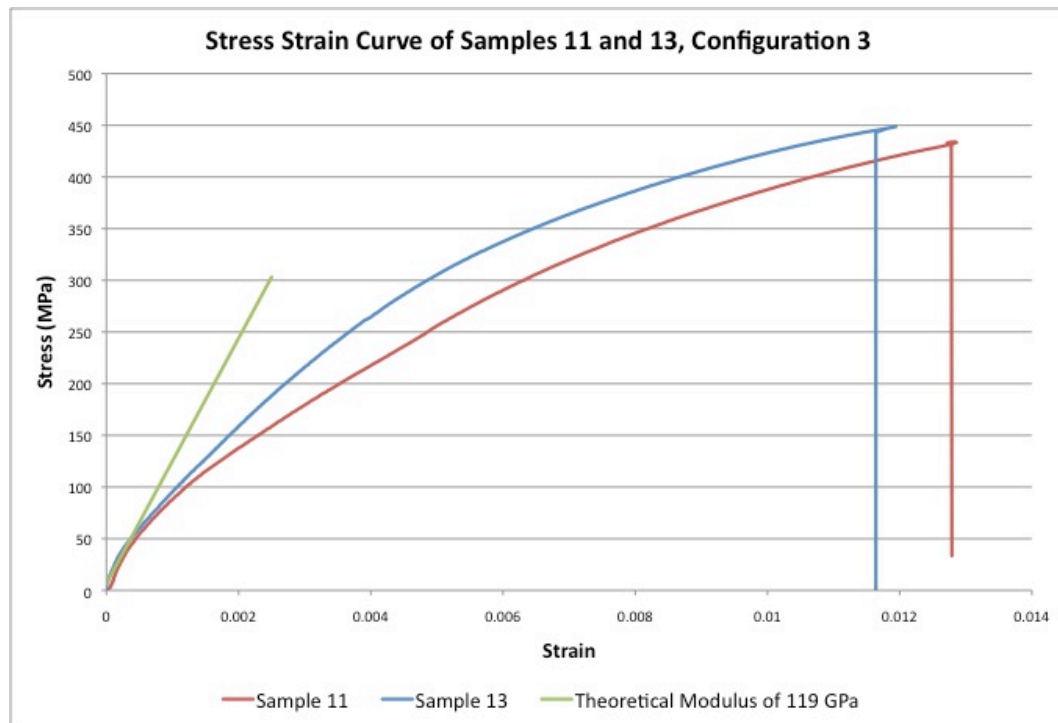


Diagram 19 – Stress strain curve for Samples 11 and 13, Configuration 3 with a theoretical modulus line represented in green.

Section 4 –

Conclusions

1. It was established that the best Configuration, in terms of cost and preparation time, to create a new composite material satisfying DARPA announcement BAA 08-23 [1] phase 1 goals is Configuration 3 with two HIP treatments, one for each pair of tungsten fiber layers. Aluminum alloy 6061-T6 is the matrix metal.
2. The best HIP treatment to apply twice to a specimen in Configuration 3 is seen in Diagram 7.
3. Pressurization must be slow, 1.5 or 2.5 MPa/min to prevent “Brazilian” type fracture in the tungsten fibers. Applying a maximum pressure of 200 MPa assures the aluminum tubes will flow in the tungsten structure. Cooling under pressure assures the components of the composite will stay in contact without moving with respect to one another due to elastic relaxation.
4. In any HIP process used to create this composite the temperature must first be raised to 500 Celsius. This assures the aluminum alloy 6061-T6 tubes will be as soft and weak as possible while staying in the solid state. Temperatures approaching 550 Celsius can promote the diffusion of tungsten within the aluminum and intermetallic reaction.

5. To prevent collapse or buckling of the aluminum tubes and tungsten fibers space needs to be minimized within the initial assembly when pressurizing externally. Thus Configurations 1 and 2 should be avoided.
6. The formation of an intermetallic compound between aluminum and tungsten can be avoided if the total time at maximum temperature, while pressurized within a HIP process is below 9 hours and 500 Celsius respectively.
7. A CIP process is unnecessary, even harmful to the creation of this composite with the Configurations studied here.
8. Additional HIP treatments with densified samples and interface gaps should be avoided. The gaps may not be eliminated and there is a risk for fiber deformation and fracture.
9. Sonication treatments of assembled structures should remain below two minutes or completely avoided.

Section 5 -

Summary of Contributions

1. A new material satisfying the density, and ultimate tensile strength of phase 1 requirements of DARPA announcement BAA 08-23 [1] has been created. A tungsten fiber structure within two aluminum alloy 6061-T6 tubes arranged in Configuration 3 with the two HIP treatments described by Diagram 7 would create such a composite. The resulting material will have a density approximately of 7.8 g/cc and an ultimate tensile strength of 453 MPa.
2. Demonstrated a that cylindrical tubes from Al 6061-T6 can be pressed into a prearranged structure of W rods and fibers with controlled spacing.

Section 6 -

Future Research

1. More studies of Configuration 3 need to be accomplished to see if the elimination of flaws such as voids and leftover interfaces between the aluminum tubes result in the prevention of fiber pull out and increase in ultimate tensile strength.
2. More Configurations similar to Configuration 4 should be developed to fully explore the costs and benefits of internally applying pressure to the assembly. This concept may provide a means to completely avoid the issues of “Brazilian” type fracture in the tungsten fibers, buckling of the tungsten wires in the hoop direction, and a reaction compound between the components.
3. This study focused on small-scale specimens that did not meet the final size requirements of DARPA announcement BAA 08-23 [1]. Issues in scaling up the design, either with larger components and/or more tungsten fiber layers need to be addressed. Understanding how long the components can be pressurized and heated up to 500 Celsius without causing a reaction compound to be created is needed.
4. Characterizing the difference in scaling up the design is required. For example in studies done to characterize the damping capacity of aluminum tungsten fiber composites, it was found that at the same volume fraction of the components the change of diameter of the fibers had a pronounced effect.

Damping capacity was found to increase with tungsten fiber diameter and reduced volume fraction of tungsten [5]. Different properties could be obtained when scaling up by increasing the number of W layers from increasing the size of the W fibers.

5. Replacement of the tungsten fibers with a more ductile metal with similar strength and density should be considered. Fibers made of a tungsten-rhenium composite show potential to increase strength and ductility in this composite.
6. Studies to see if aging of the composite's aluminum matrix back to the T6 condition can be done to see if the tensile strength of the composite can be increased as seen in composites with tungsten and aluminum powders [3]. To return the aluminum to the T6 condition, a specimen needs to be heated to 530 Celsius and held at that temperature for 3 hours. The specimen should then be quenched to room temperature before being heated to and held at 160 Celsius for 18 hours [22]. Such aging could relax the residual stresses induced from cooling the composite under pressure, and lead to looser interface between the components and perhaps fiber pull out when a tensile load is applied.
7. Reduction in the maximum pressure as seen in our preferred HIP cycles in Diagram 7 should be explored. Similar results for aluminum plates being compressed around steel fibers have been achieved with maximum pressures of 70 MPa [10].
8. The initial pressurization in our preferred HIP treatments as seen in Diagram 7 of 20 MPa may be unnecessary. Future samples can easily determine if this is true to optimize the HIP treatment.

9. To minimize the diffusion of aluminum into tungsten changing the pressurization rate and soak time in the HIP processes applied to a Configuration 3 specimen should be investigate. The goal is finding the minimum time to migrate the aluminum through the tungsten structure and bonding the aluminum layers together.
10. Can the process described here be used to develop another metal matrix composite to suit another purpose?

Appendices

Appendix A – Tungsten Fiber Spacing

Table 6 - Tungsten Layer 1 Spacing. 274 μm (183* μm) is the prescribed spacing.

Configuration/Sample/Notes	Average (μm)	Maximum (μm)	Minimum (μm)	Mode (μm)	Standard Deviation (μm)
1 / 1 / CIP 345MPa	262.4	388	118	263	53.8
1 / 2 / CIP 50MPa	283.9	477	118	277	51.4
1 / 2 / HIP 2	228.2	492	38	262	70.1
2 / 1 / HIP 3 (Non Buckled Region)	257.5	800	15	92	172.1
1 / 3 / HIP 4 (Non Buckled Region)	257.8	415	148	277	37.7
1 / 4 / HIP 4 (Non Buckled Region)	258.3	308	104	277	27.6
1 / 5 / HIP 5 (failed)	291.1	323	246	277	19.3
1 / 5 / Post Tensile Test (Non Buckled Region)	276.1	308	215	277	16.6
1 / 6 / HIP 5 (Non Buckled Region)	257.3	323	200	262	20
1 / 6 / Post Tensile Test (Non Buckled Region)	270.1	354	215	277	21.8
3 / 7 / HIP 6* (Layers 1-2)	172.4	246	108	169	17.8
3 / 10 / HIP 10* (Layers 1-2)	185.6	462	31	169	57.3
3 / 9 / HIP 11* (Layers 1-2)	177.2	723	15	169	91.9
3 / 11 / HIP 11* (Layers 1-2)	178.9	600	0	185	77
3 / 12 / HIP 11* (Layers 1-2)	177.7	446	46	169	51.8
3 / 13 / HIP 11* (Layers 1-2)	175	462	46	185	49.6

Table 7 – Tungsten Layer 3 Spacing. 128 μm (183* μm) is the prescribed spacing.

Configuration/Sample/Notes	Average (μm)	Maximum (μm)	Minimum (μm)	Mode (μm)	Standard Deviation (μm)
1 / 1 / CIP 345MPa	114.2	200	75	106	22.9
1 / 2 / CIP 50Mpa	195	1769	0	0	268
1 / 2 / HIP 2	97.1	846	0	46	99.3
2 / 1 / HIP 3 (Non Buckled Region)	97.7	477	0	31	98.9
1 / 3 / HIP 4 (Non Buckled Region)	116.2	277	0	123	35
1 / 4 / HIP 4 (Non Buckled Region)	112	369	15	123	39
1 / 5 / HIP 5 (failed)	141.2	492	15	154	60.1
1 / 5 / Post Tensile Test (Non Buckled Region)	132.8	185	77	138	21.1
1 / 6 / HIP 5 (Non Buckled Region)	114.9	154	92	108	11.5
1 / 6 / Post Tensile Test (Non Buckled Region)	123.6	138	92	123	9.2
3 / 7 / HIP 7* (Layers 3-4)	167.3	415	0	169	51.5
3 / 10 / HIP 12* (Layers 1-2)	167.2	477	31	169	91.9
3 / 9 / HIP 12* (Layers 1-2)	174.4	131	0	169	70.6
3 / 11 / HIP 12* (Layers 1-2)	176.8	308	77	169	27.1
3 / 12 / HIP 13* (Layers 1-2)	176.6	508	0	169	71.1
3 / 13 / HIP 12* (Layers 1-2)	176.4	400	31	185	58.6

Table 8 - Tungsten Layer 2 Spacing. 274 μm is the prescribed spacing.

Configuration/Sample/Notes	Average (μm)	Maximum (μm)	Minimum (μm)	Mode (μm)	Standard Deviation (μm)
1 / 5 / Post Tensile Test (Non Buckled Region)	216.1	277	138	246	43.6
1 / 6 / Post Tensile Test (Non Buckled Region)	288.1	831	0	123	205.4

Table 9 - Tungsten Layer 4 Spacing. 156 μm is the prescribed spacing.

Configuration/Sample/Notes	Average (μm)	Maximum (μm)	Minimum (μm)	Mode (μm)	Standard Deviation (μm)
1 / 5 / Post Tensile Test (Non Buckled Region)	127.3	200	15	185	55.5
1 / 6 / Post Tensile Test (Non Buckled Region)	183.5	692	0	15	208.9

Appendix B – Sample Dimensions, Mass, and Densities

Table 10 – Measured Sample dimensions, mass, and density.

Sample Number	Configuration	Inner Diameter (mm)	Outer Diameter (mm)	Length (mm)	Mass (g)	Density (g/cc)
2	1	7.5	9.57	44.02	9.65	7.88
3	1	7.54	9.98	53.27	12.9	7.08
3-1	1	7.43	9.28	25.46	5.3	8.45
3-2	1	7.42	9.13	25.51	5	8.67
4	1	7.46	9.67	54.65	12.3	7.87
5	1	7.5	9.16	15 (gauge section)	-	9.37 (gauge section)
6	1	7.45	9.7	10 (gauge section)	-	7.36 (gauge section)
7-1	3	7.5	9.95	61.02	15.4	7.48
7-2	3	7.5	9.86	61.36	15.3	7.47
7-1-A	3	7.5	9.91	24.84	6.25	-
7-1-B	3	7.47	9.95	24.92	7.23	-
7-2-A	3	7.54	9.9	25.03	6.2	7.59
7-2-B	3	7.51	9.89	24.85	6.3	7.69
8	4	7.17	10.47	90.12	26.2	6.03
9-A	3	7.48	9.93	25.2	6.28	7.45
9-B	3	7.49	9.96	25.14	6.33	7.53
10	3	7.54	9.8	30 (gauge section)	-	-
11	3	7.5	9.97	30 (gauge section)	-	-
11-A	3	7.5	9.91	24.5	6.1	7.42
12-A	3	7.51	9.94	25.24	6.22	7.44
12-B	3	7.51	9.93	25.11	6.28	7.46
13	3	7.5	10.11	25 (gauge section)	-	-

Appendix C – Summary of HIP Treatments

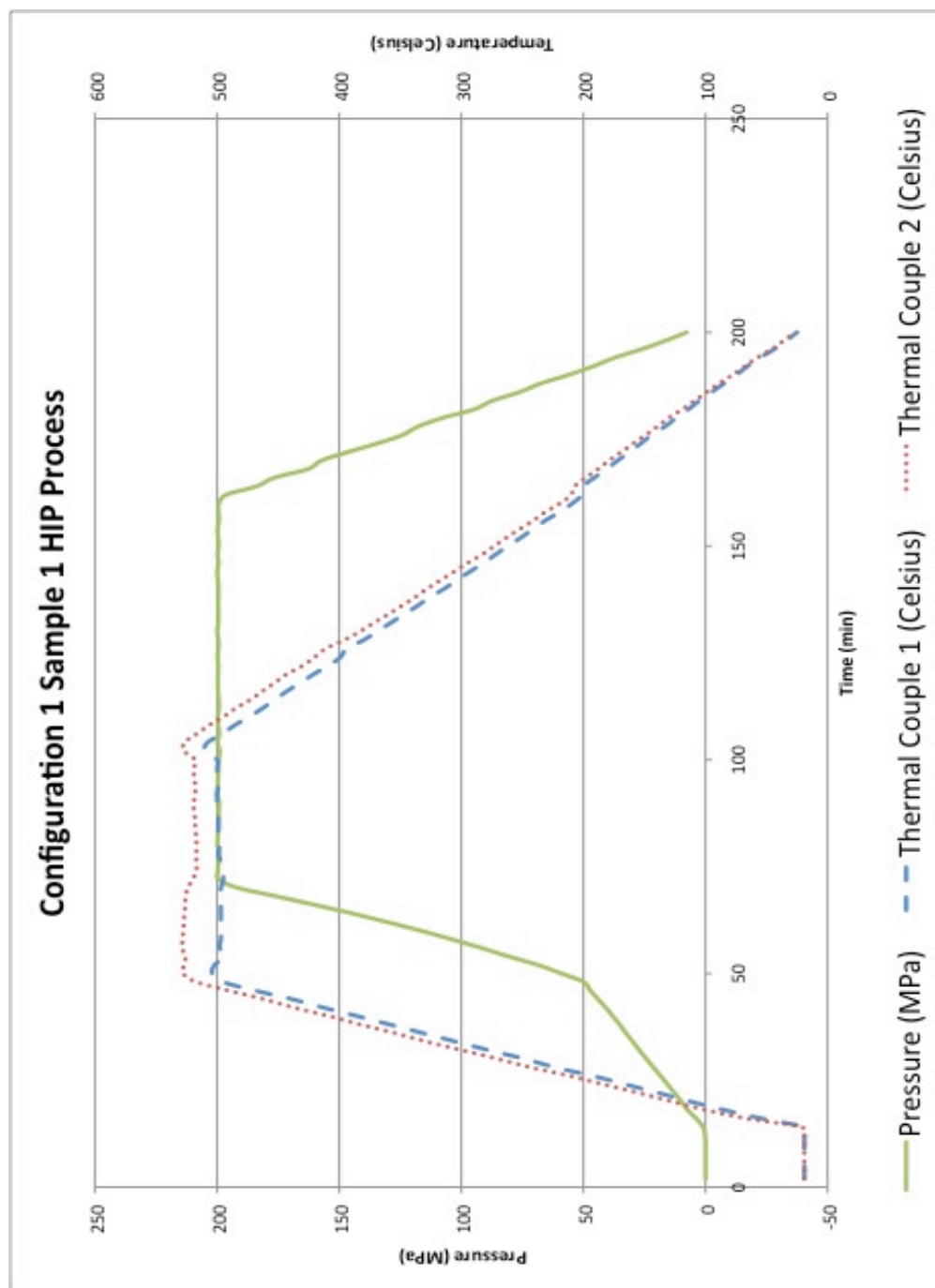


Diagram 20 – HIP 1: HIP process for Sample 1 of Configuration 1.

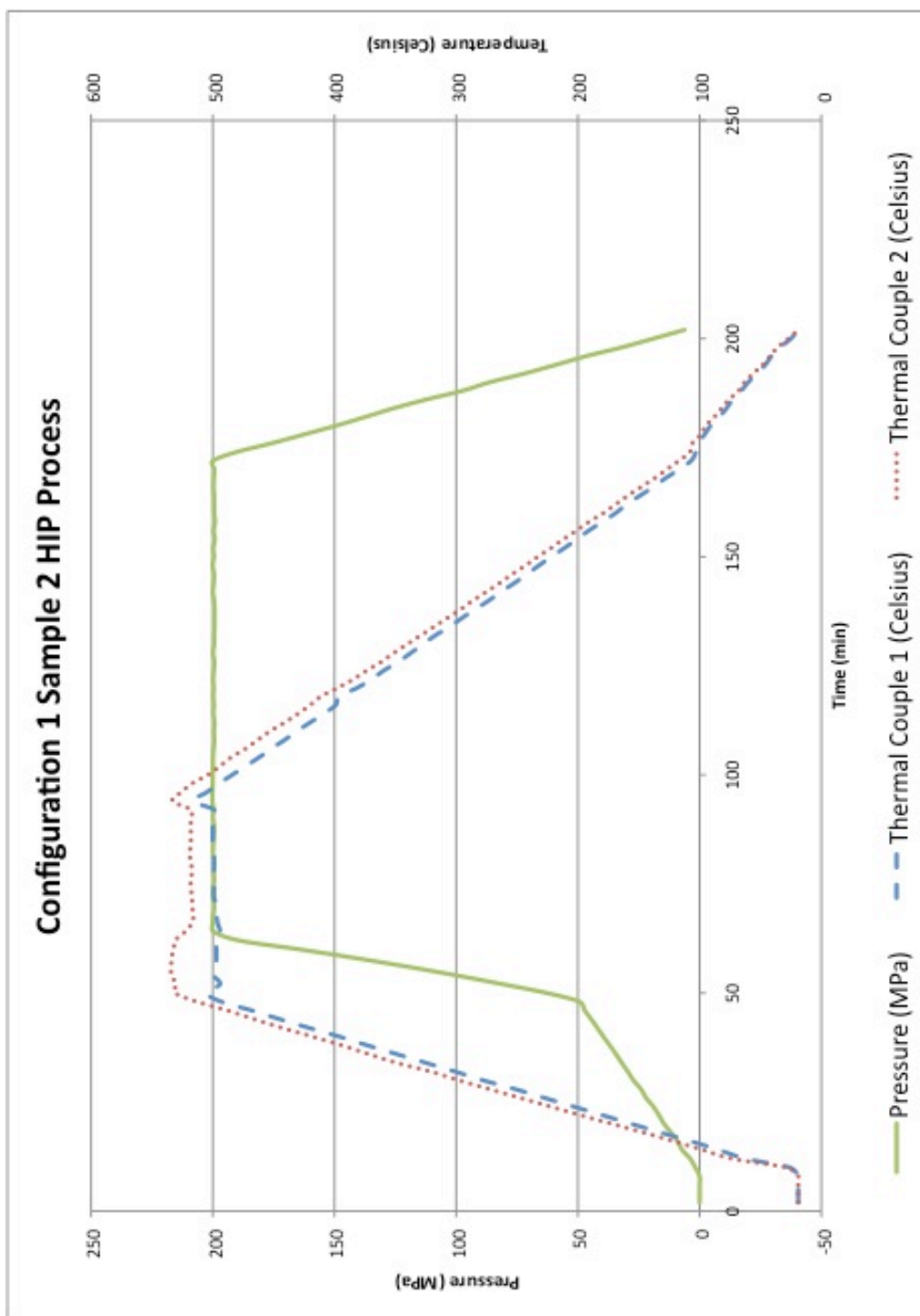


Diagram 21 – HIP 2: Successful HIP process for sample 2, Configuration 1.

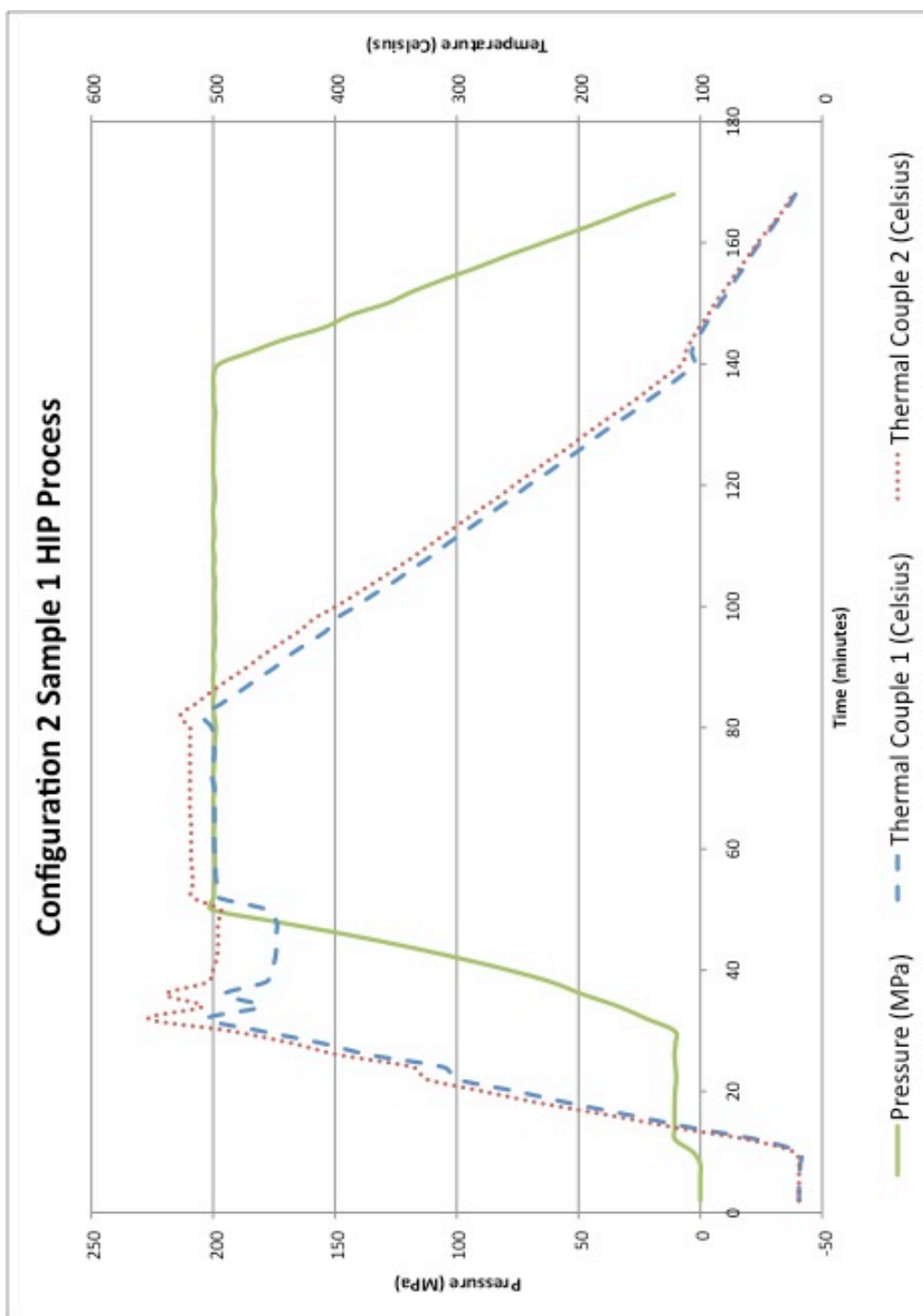


Diagram 22 – HIP 3: Sample 1, Configuration 2 HIP process.

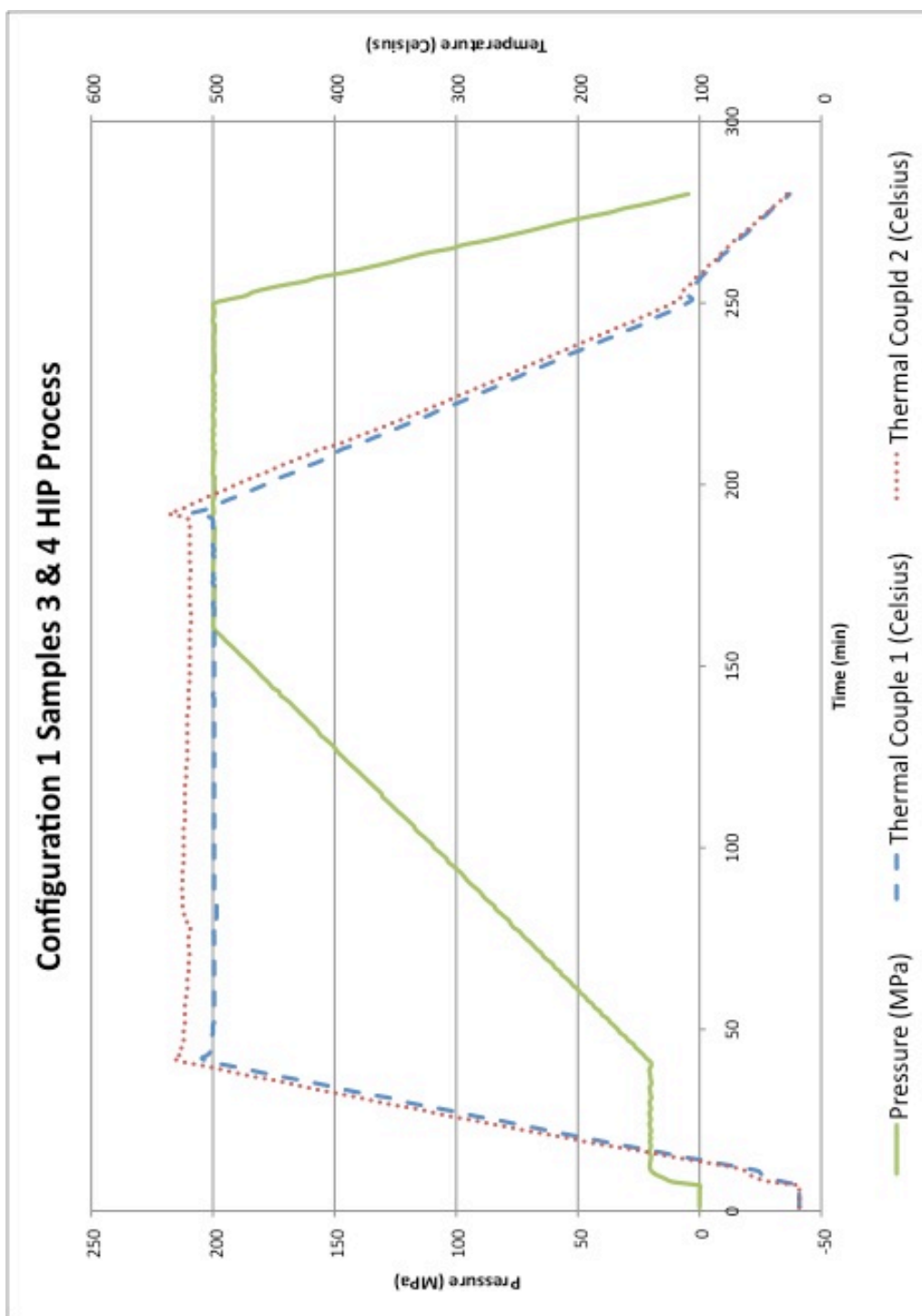


Diagram 23 – HIP 4: HIP process for Samples 3 and 4 in Configuration 1.

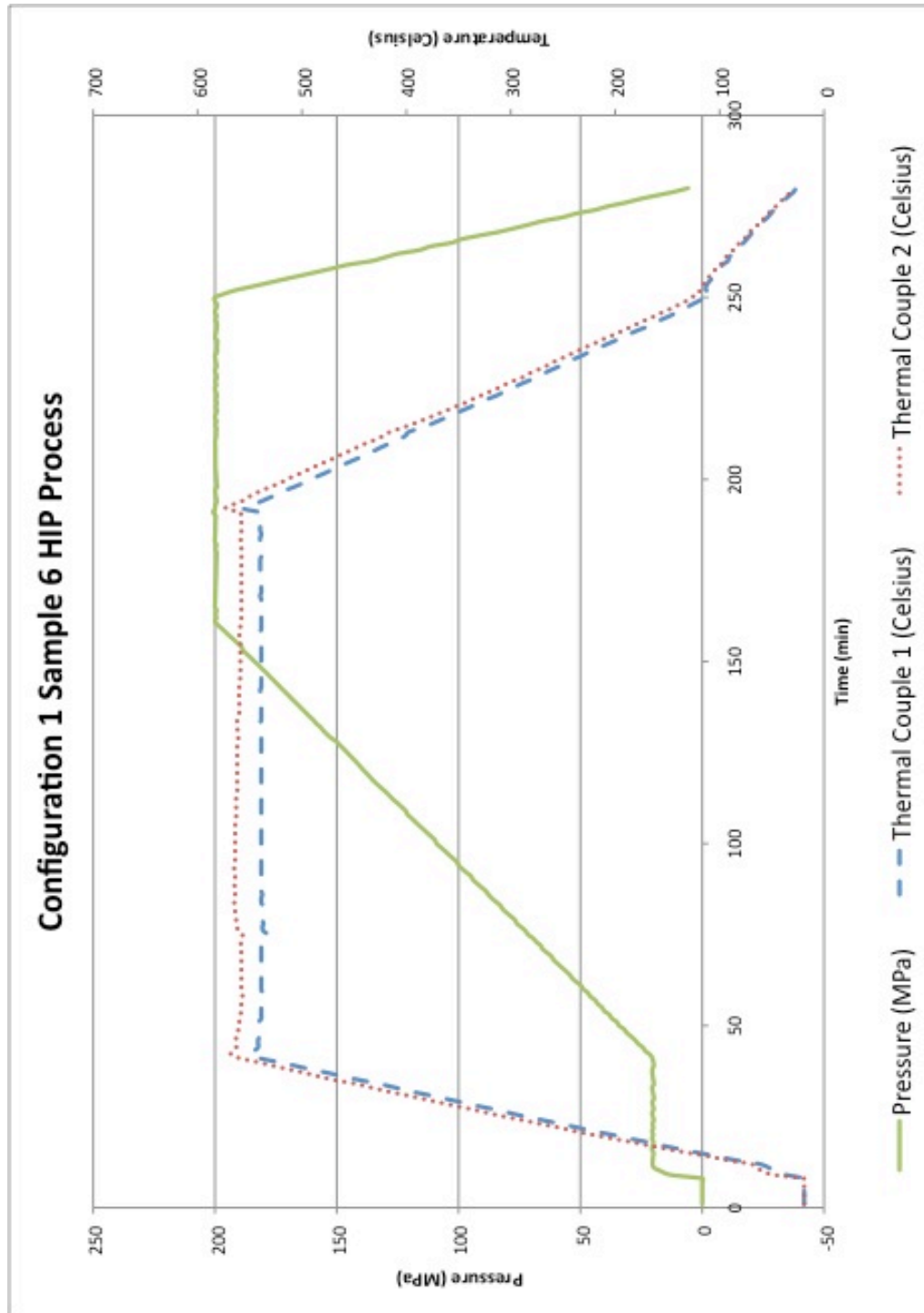


Diagram 24 – HIP 5: HIP process for Sample 6, Configuration 1 with thicker Al 6061-T6 inner tube. Sample 5, Configuration 1 was also present during this process but lost its vacuum seal.

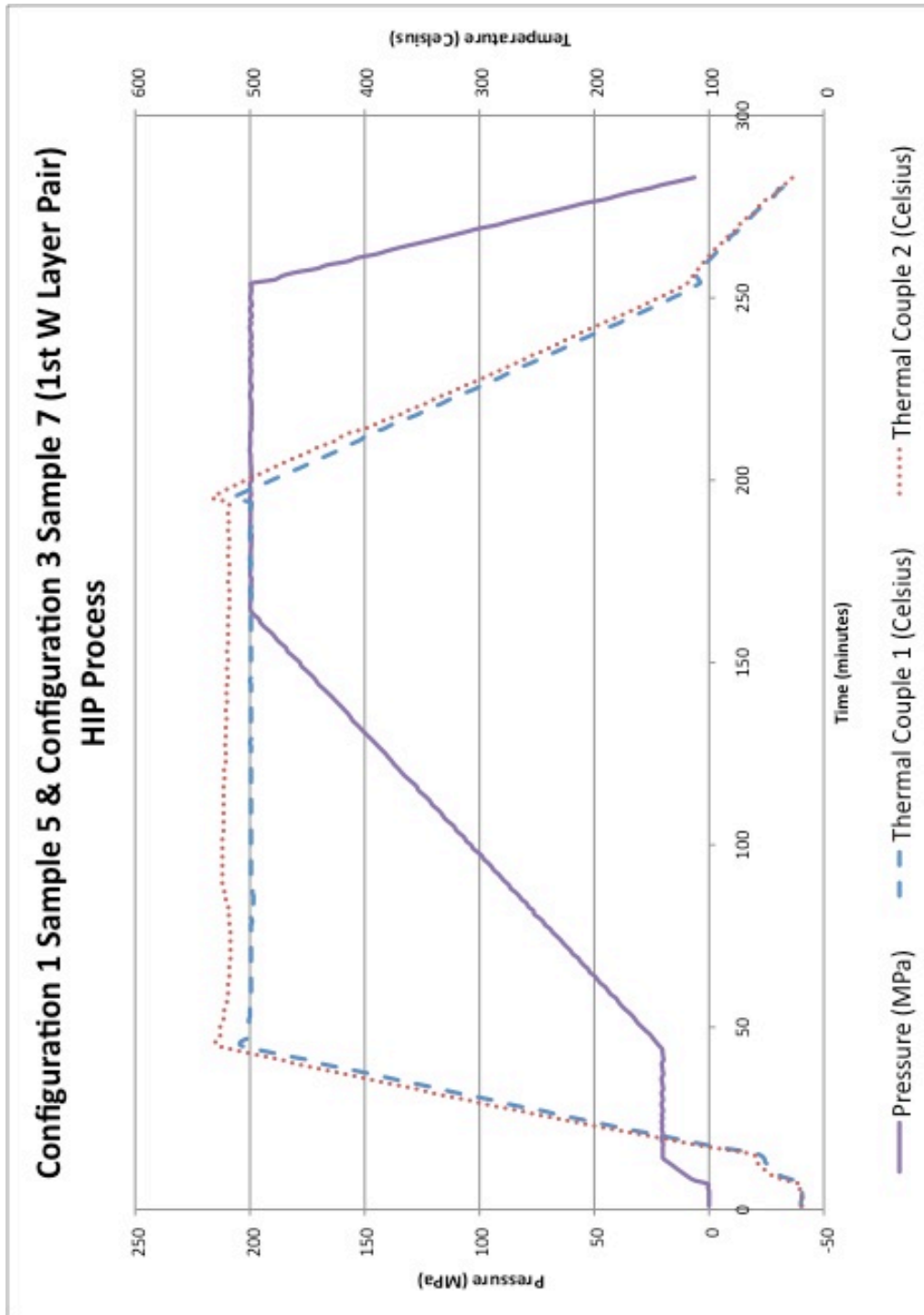


Diagram 25 – HIP 6: HIP process applied to Sample 5, Configuration 1 and Sample 7, Configuration 3. Sample 5 was successfully completed while Sample 7 only had two of the four W layers applied.

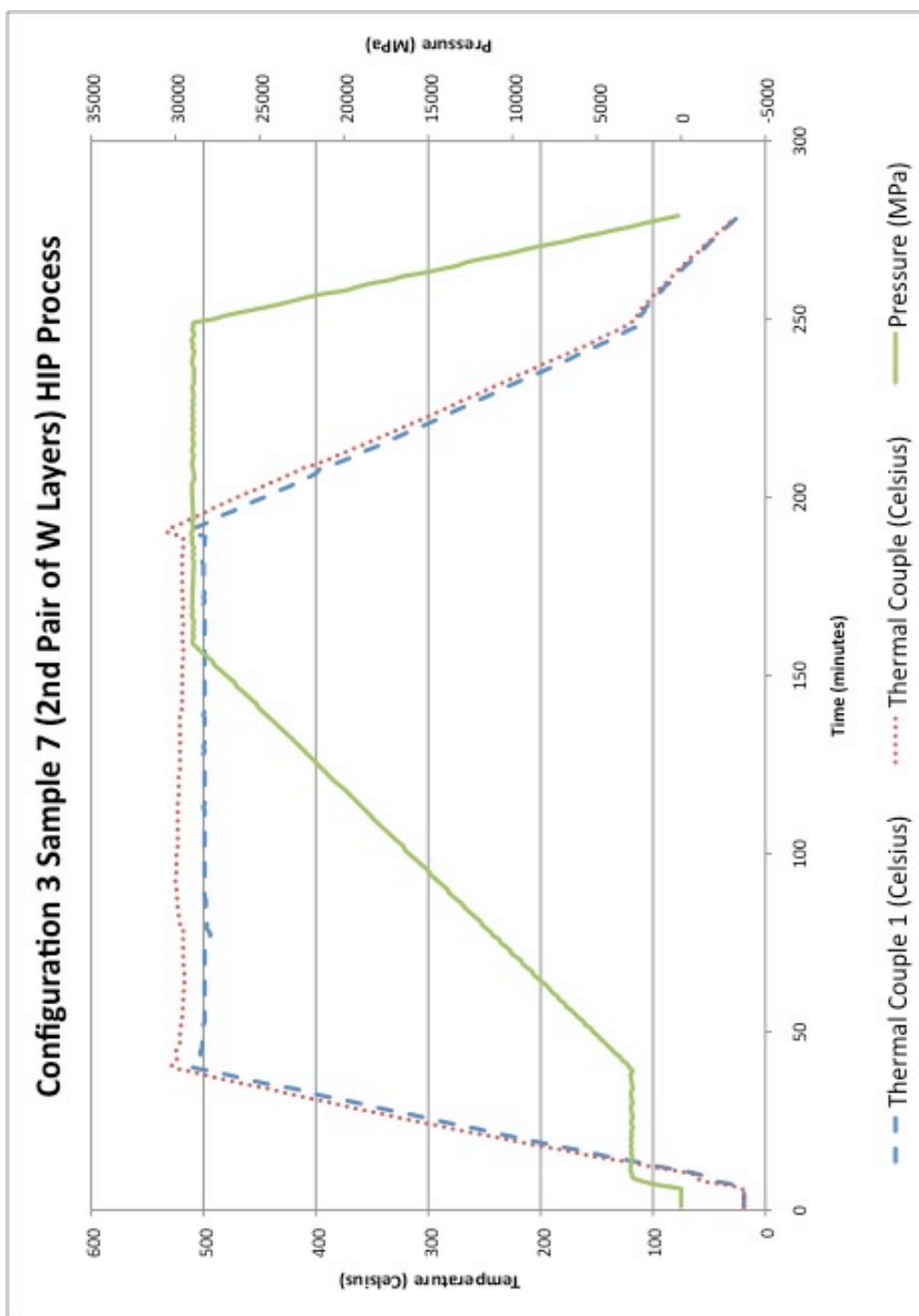


Diagram 26 – HIP 7: HIP process applied to Sample 7, Configuration 3 to secure the final pair of W fiber layers.

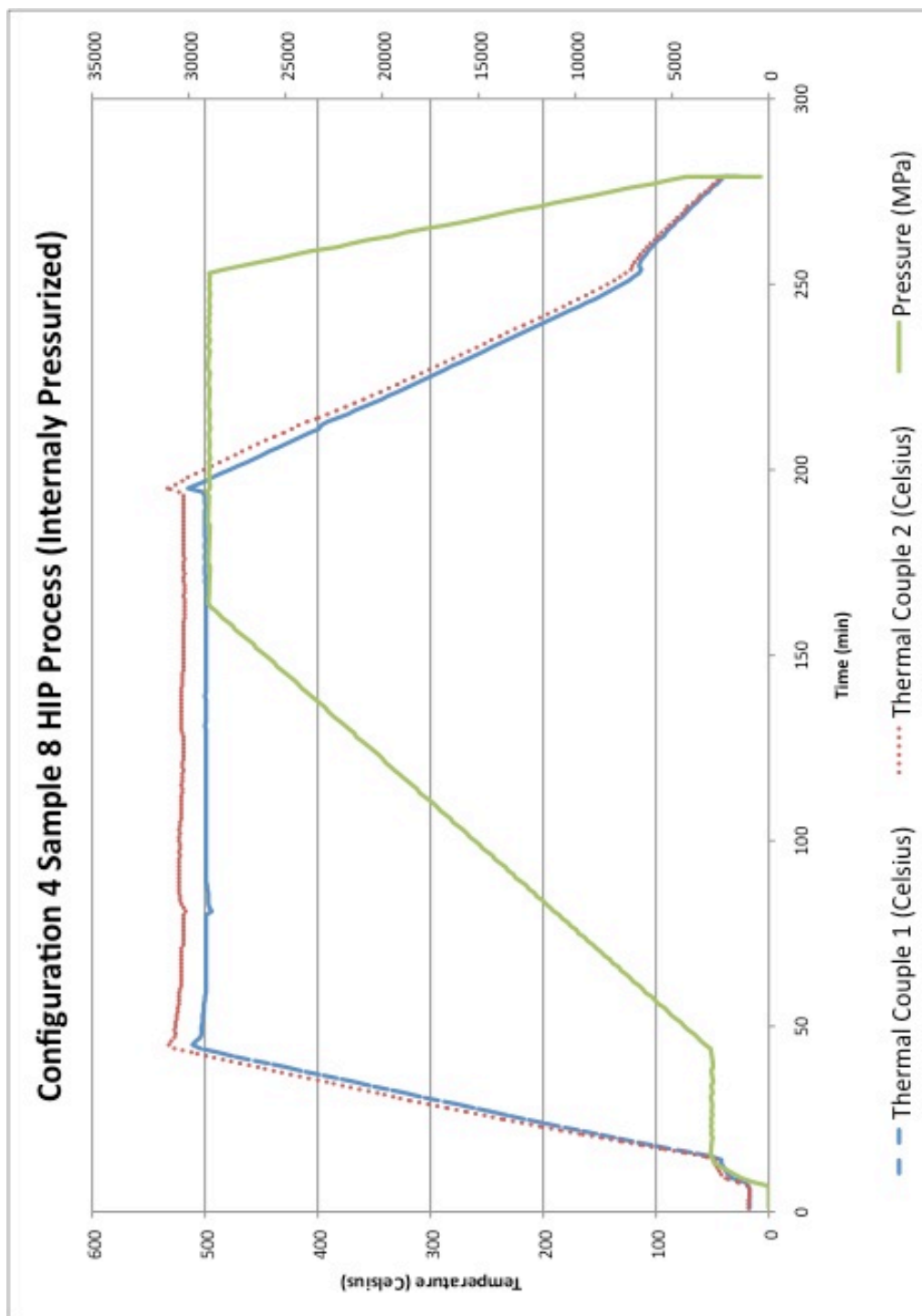


Diagram 27 – HIP 8: HIP process applied to Sample 8, Configuration 4.

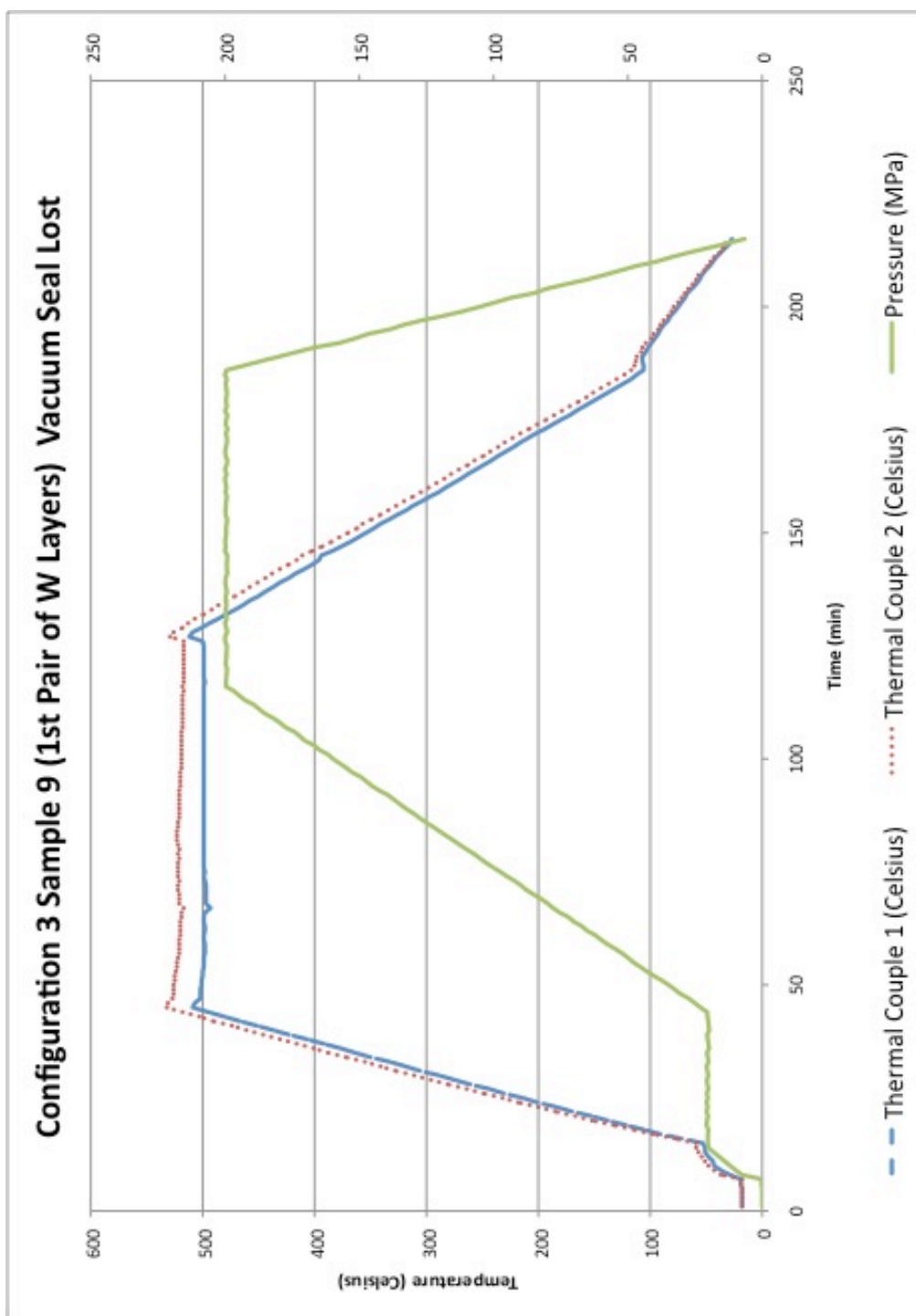


Diagram 28 – HIP 9: HIP process unsuccessfully applied to Sample 9, Configuration 3 due to vacuum seal failure.

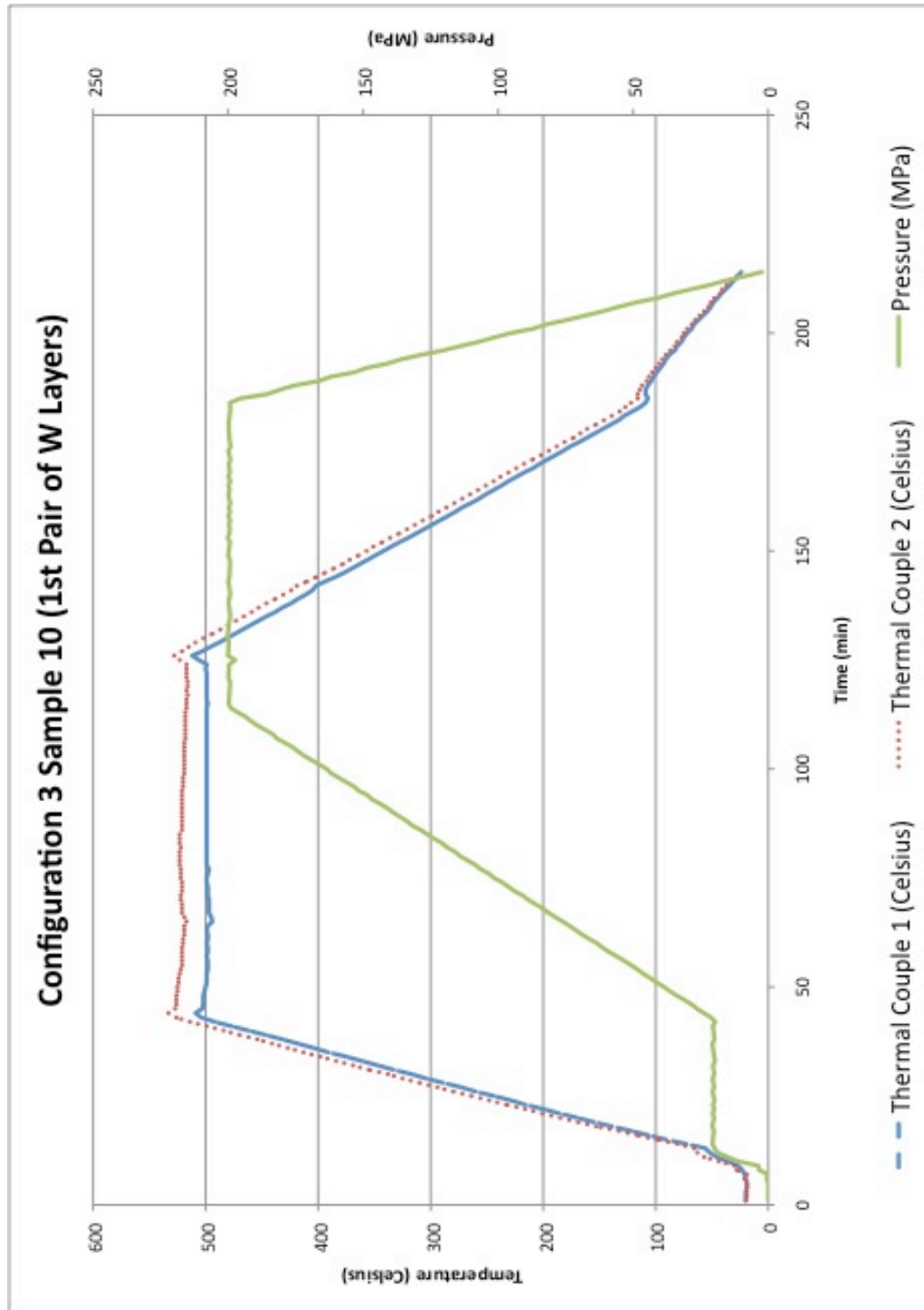


Diagram 29 – HIP 10: HIP process applied to Sample 10, Configuration 3 to secure the first pair of W fiber layers.

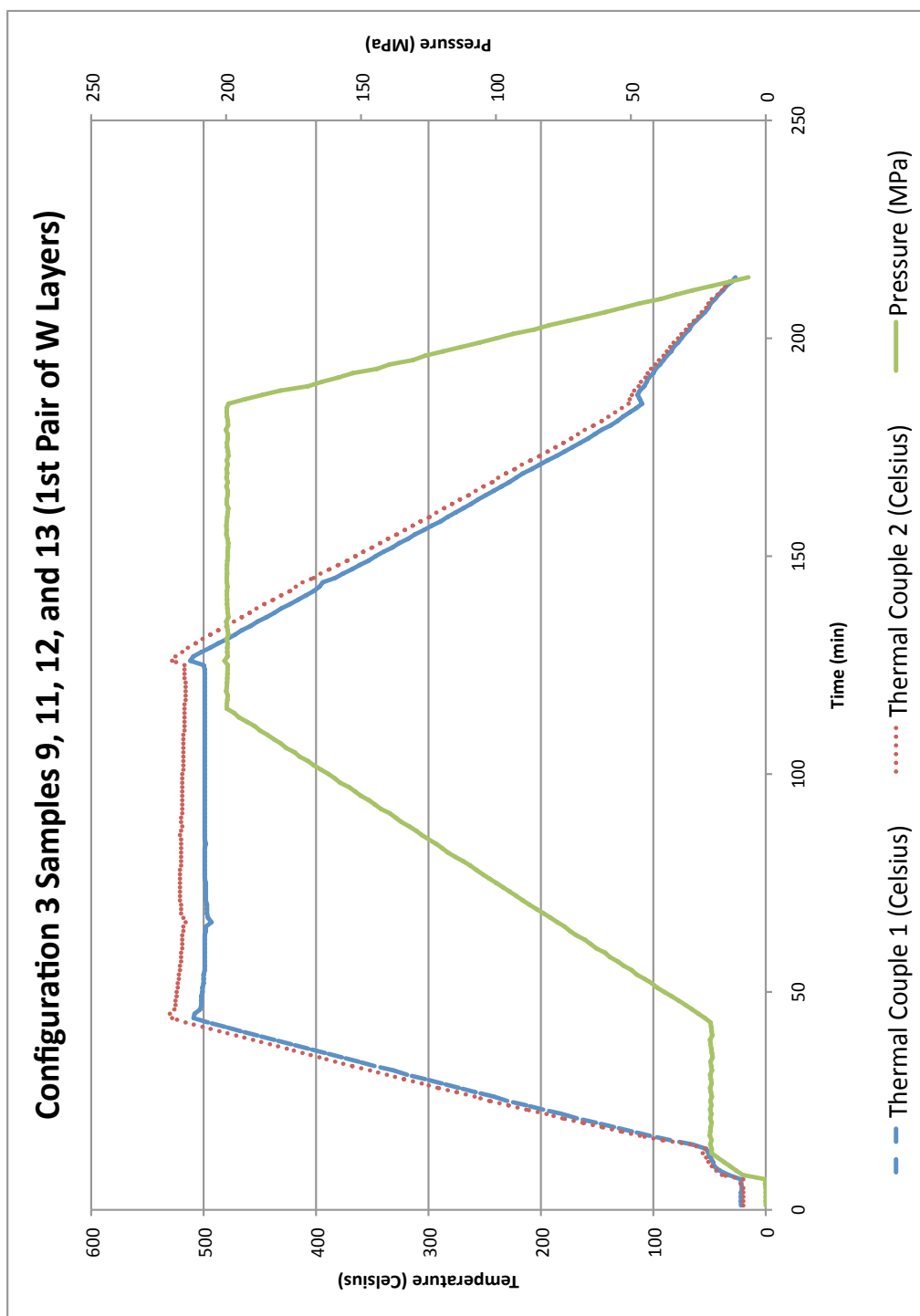


Diagram 30 – HIP 11: HIP process applied to Samples 9, 11, 12 and 13, Configuration 3 to secure the first pair of W fiber layers.

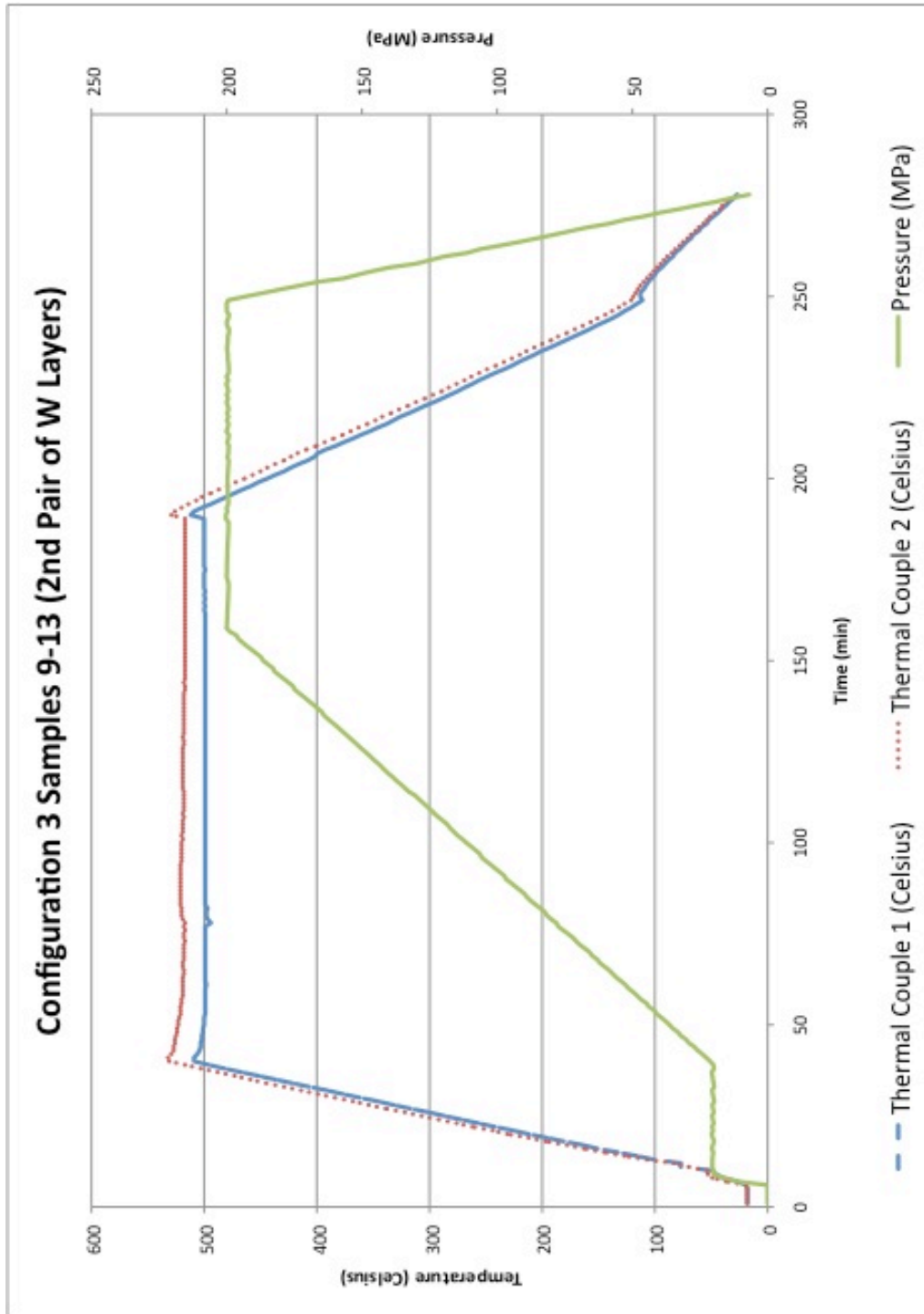


Diagram 31 – HIP 12: HIP process applied to Samples 9-13, Configuration 3 to secure the final pair of W fiber layers. The vacuum seal on Sample 12 failed.

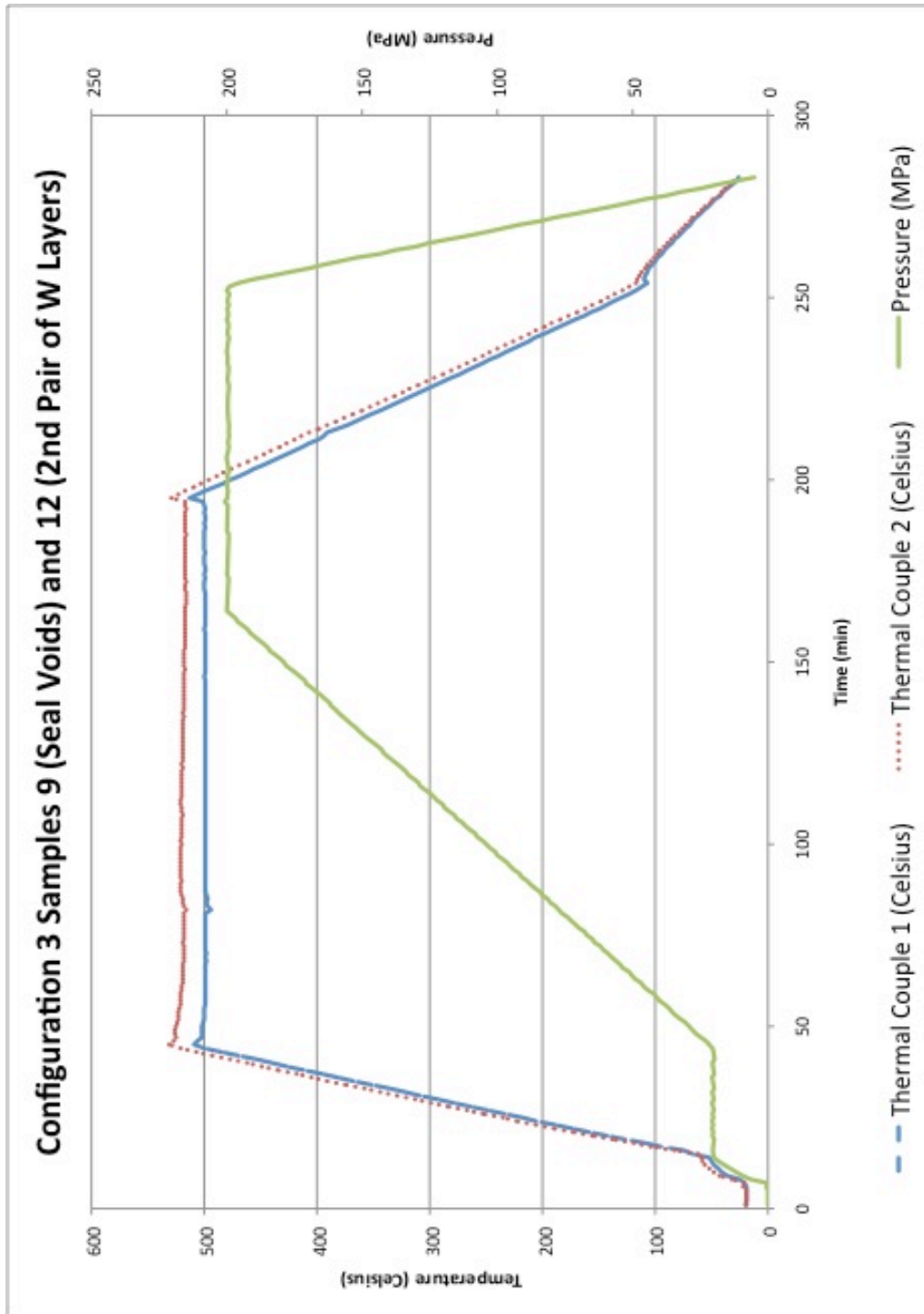


Diagram 32 – HIP 13: HIP process applied to Samples 9 and 12, Configuration 3. Sample 12 had the final two layers of W fibers applied. Sample 9 was HIP treated again to see if the interface gaps between the aluminum tubes that remained after the first successful HIP cycle could be closed in a densified sample.

Appendix D – Finite Element Modeling

In this analysis we are trying to model the mechanical behavior of a metal matrix composite made up of an aluminum matrix with tungsten fibers. Such a material was tested under tension on November 2, 2009. The goal of the test was to get an estimate of the strength of our composite to gauge how close to obtaining the target strength of 690 MPa. As it turns out this first successful tensile specimen measured an ultimate tensile strength of 648 MPa, and there is some speculation as to how the composite obtained such a high strength. Additionally due to the processing of the specimen the aluminum matrix became annealed, thus losing strength. It is thought that aging the composite after processing back to the original temper would increase the strength of the composite further. The following finite element study was done to shed some light why the high strength observed in the tensile test as well as give us some idea what effect aging will have on the composite's strength.

The tensile specimen tested in the lab was a metal matrix composite made up of an aluminum alloy 6061-T6 matrix and tungsten fibers. The shape of the specimen was cylindrical with tungsten fibers, 200 μm in diameter, in the axial and hoop directions. The specimen behaved in a brittle fashion showing a linear increase in stress with strain before suddenly breaking within the gauge section at 648 MPa. No secondary increase in the stress-strain curve was observed after fracture. The inner and outer diameter of the gauge section was 7.5 and 9.2 mm respectively. Approximately 140 tungsten rods were arranged in the axial direction with two tungsten wires wrapped tightly within the specimen in the hoop direction. For this

particular specimen 20% of the cross section was tungsten with the remaining being aluminum alloy 6061-T6. It has been measured on other samples that the process used to create the composite anneals the aluminum alloy 6061-T6. For this analysis we are assuming this annealing is similar to aluminum alloy 6061-O.

For the finite element method (FEM) analysis three different meshes were created. Two were with solid Type 1 elements and the third being made up of tetrahedral Type 15 elements. Of the solid Type 1 models one was course with 1,280 elements and the other fine with 15,500 elements. The tetrahedral model was made up of 3,180 elements. With these meshes it is felt that a conclusion can be reached to determine if the results are converging. Due to prior experience with LS-DYNA and the need to minimize computational time Type 1, elements were used instead of higher order elements. It is thought higher order elements will converge faster at the cost of more computational time. Each mesh had other attributes changed to analyze tensile strength and changes to material in the case of the course model. The meshes can be seen in Figures 91-93 for the course solid Type 1 model, the fine solid Type 1 model, and the tetrahedral Type 15 model.

Several differences from the actual specimen are being introduced into the models to reduce computational time. It is assumed that in the mechanical test that the tungsten wire wrapped in the hoop direction did not significantly contribute to the axial strength, thus the tungsten wire is being left out of the model. Additionally the scale of the FEM model is significantly different, as we will only have eight tungsten rods in our models that are on the scale of meters in dimensions. The cross section

percentage of tungsten to aluminum is preserved however. The inner and outer diameter of the cylinder in the FEM models in 4 and 7.5 meters respectively. The tungsten rods, 0.5 meter in diameter are equally spaced along the centerline of the thickness of the cross section.

Each of the three meshes underwent a tensile analysis. After seeing the results were converging the course model was used to see how using aluminum alloy 6061-O instead of aluminum alloy 6061-T6 changed the tensile behavior.

Each model implemented the *TIED_SURFACE_TO_SURFACE keyword to attach the tungsten rods to the aluminum matrix. Such a keyword is thought to be appropriate since under SEM observation the actual fracture surface of the tested specimen showed minimized pullout of the tungsten fibers, suggesting our process created a very strong interface between the components.

To see at what strength the models failed at *PRESCRIBED_MOTION_SET was used create a plane of nodes that would move at a fixed rate. This is the most idealistic way to model a strain controlled tensile test. All models were run explicitly.

The material models implemented in this FEM analysis provided the greatest challenge and education. The original plan to use the *JOHNSON_COOK keyword had unexpected instability issues. They were not related to the loading but the material model itself and unfortunately lead to the pursueute of a different material model. The material model used instead was *PLASTIC_KINEMATIC with no hardening, thus a perfectly plastic model. Although this sort of model further

decreases the possibility of matching the experimental data, this model was stable and lead to interesting results. The values used for the material models are presented in Table 11 below and were obtained from matweb.com with the exception of the tungsten rods, which were independently tested.

Table 11 – Material Properties for the FEM analysis.

	Density (kg/cm)	Elastic Modulus (GPa)	Poisson's Ratio	Yield Strength (MPa)	Strain to Failure
Al 6061-T6	2704	68.9	0.3	276	0.12
W	18360	400	0.27	2400	0.006
Al 6061-O	2704	68.9	0.3	55.2	0.25

Each of the models varied greatly in time to compute. The course models were each approximately seven minutes to run with the tetrahedral models coming in at 27 minutes. The fine models took four hours or more to complete.

For tensile analyses the fracture pattern is considered to be convergent with the course model. First all the tungsten rods snap, and in some cases in multiple locations. Then the aluminum fractures from the inside diameter to the outside. This behavior is very similar to cup and cone failure commonly observed with ductile fracture and is seen in the fracture surface of the experimental specimen, sample 5 of Configuration 1. Figures 94-98 show the progression of failure in the fine model. Models that had tungsten rods fracture in multiple places ended up with a non-planar fracture surface similar to what was actually tested.

The tensile strength of the models is presented in Table 12 below. Overall the results of each model with aluminum alloy 6061-T6 match the measured experimental strength. This is unexpected, as one would have expected for the aluminum's strength to have decreased due to the annealing in the HIP cycle, and would have hoped the T6 temper would instead increase the strength. The aluminum alloy 6061-O, a material expected to be close to the strength of the real specimen had a strength less than 648 MPa.

Table 12 – Fracture strength of FEM models.

	Fracture Strength (MPa)	Experimental Specimen (MPa)
Course Al 6061-T6	643	648
Fine	638	
Tetrahedral	644	
Course Al 6061-O	516	

From the data it can be said that the course model converges for axial strength. However due to the use of a perfect plastic material model, the strength values obtained are less than what can be experimentally measured. The aluminum alloy 6061-O model should come close to the experimental results but is instead over 100 MPa less. This shows how important it is to have accurate material models to predict or recreate actual material behavior. That said, it is seen that an aluminum matrix in the T6 condition significantly increases the tensile strength. Additionally as long as a firm interface is kept between the tungsten fibers and the aluminum matrix the high strength we desire can be obtained as these FEM models show what can be done if fiber pull out is ignored or minimized.

Considering the assumptions of the material models and geometry the results are surprisingly close to the experimental results. It is not hard to imagine that with a plastic hardening or a Johnson-Cook model the experimental results can be matched. Thus it is thought that the mechanical strength observed in our specimen is due simply to the materials acting as two springs against the applied load. It does not appear that any other microscope interaction between the components is aiding in the mechanical strength. A plot of stress vs time can be seen for several elements across the plane that fractures in the course model in Diagram 33. Notice that unlike our experimental results the aluminum matrix recovered the load after the tungsten fractures. It is thought that in our experiment that when the tungsten rods fractured, thus transferring an extreme overload to the aluminum matrix the matrix, simply fractured instantly instead of continuing to carry the load. A Johnson-Cook material model may show this behavior.

In conclusion several FEM models were created to simulate actual tensile testing of an in-development metal matrix composite (sample 5 of Configuration 1). The models were intended to gain insight to the contributing factors to the tensile strength of the composite. Course models with solid Type 1 elements were seen to be accurate, and the need for a better material model such as Johnson-Cook was highlighted. However with a perfectly plastic material model, it was seen that the mechanical strength of the composite comes primarily from the individual component's material properties and not from microscopic interaction between the components. It was also seen that aging the aluminum back to the T6 condition can

significantly increase the tensile strength of the composite as long as the strong interfacial pressure between the components remains intact.

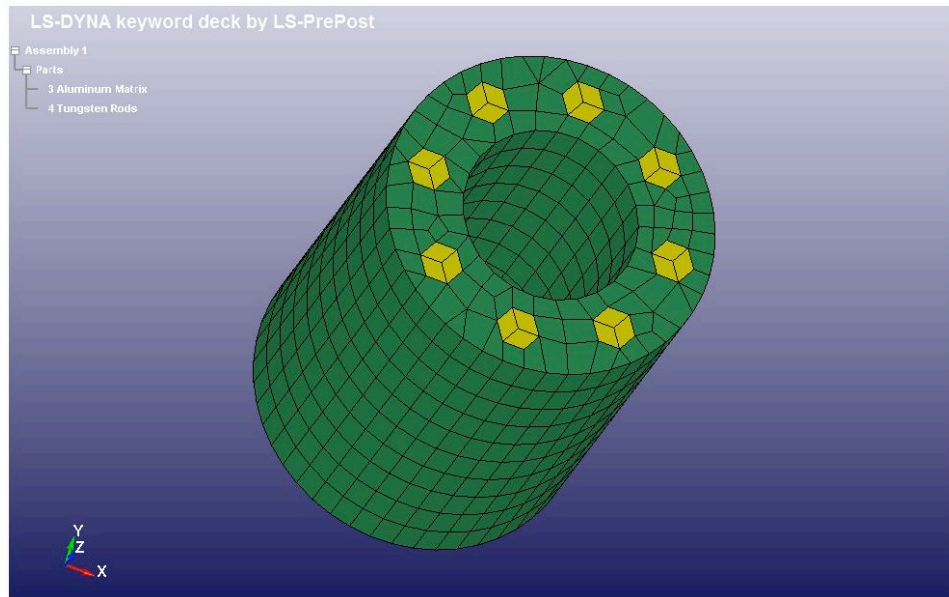


Figure 91 - Course solid type 1 model.

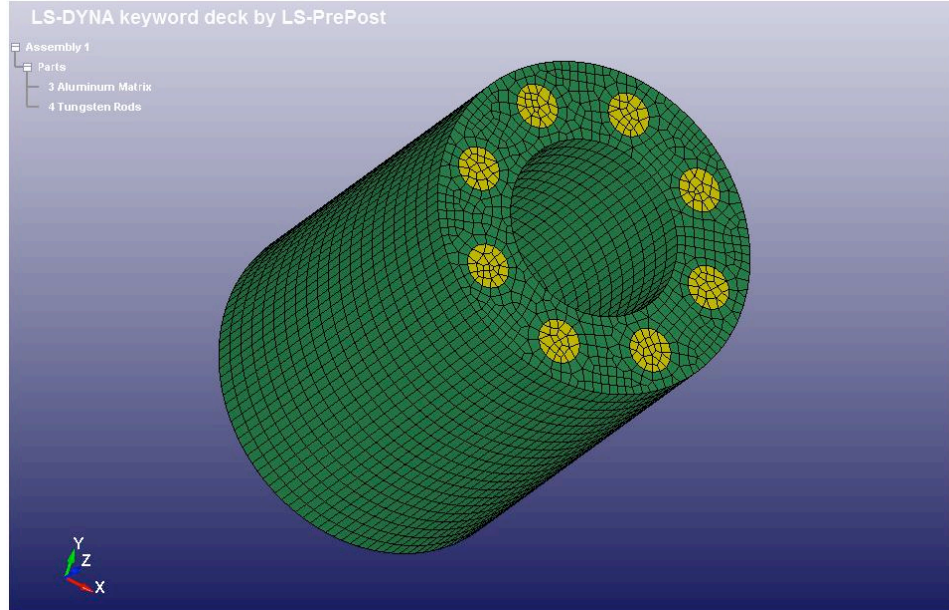


Figure 92 - Fine solid type 1 model.

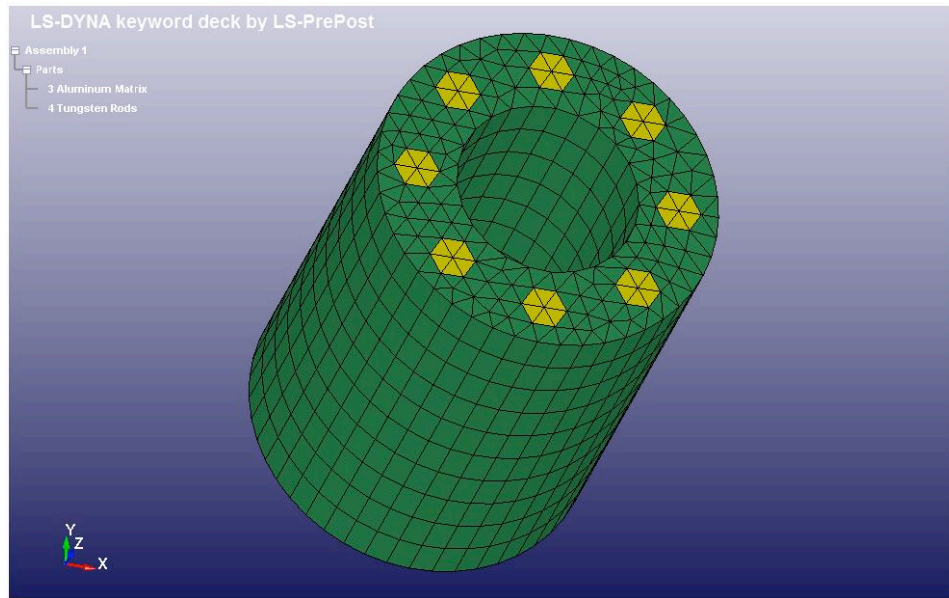


Figure 93 - Tetrahedral type 15 model.

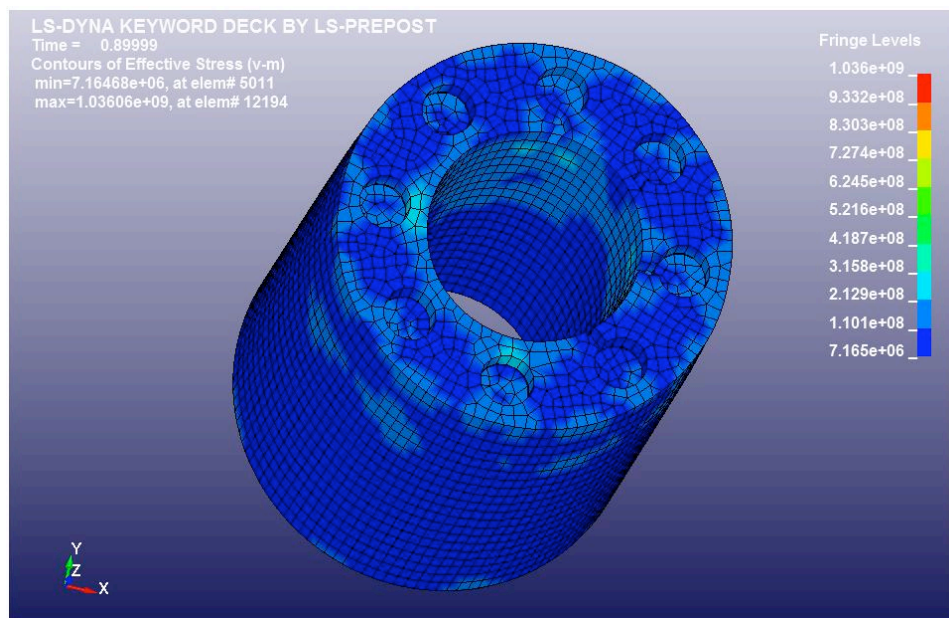


Figure 94 - Fine model with initial tungsten rod fracture. Note failed elements are deleted in model.

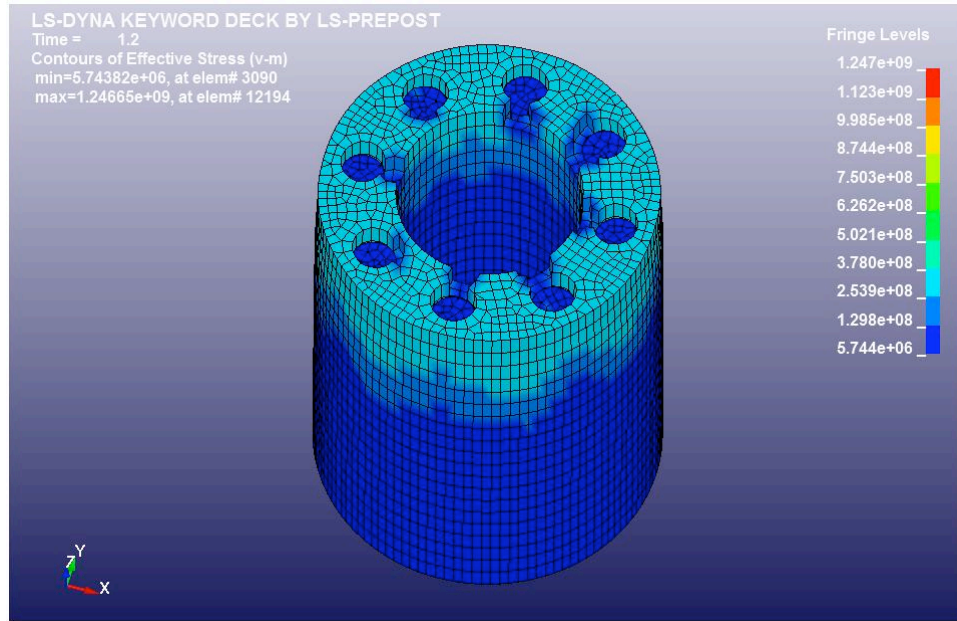


Figure 95 - Fine model with initial aluminum matrix failure. Also notice the slight necking of the aluminum matrix. Note failed elements are deleted in model.

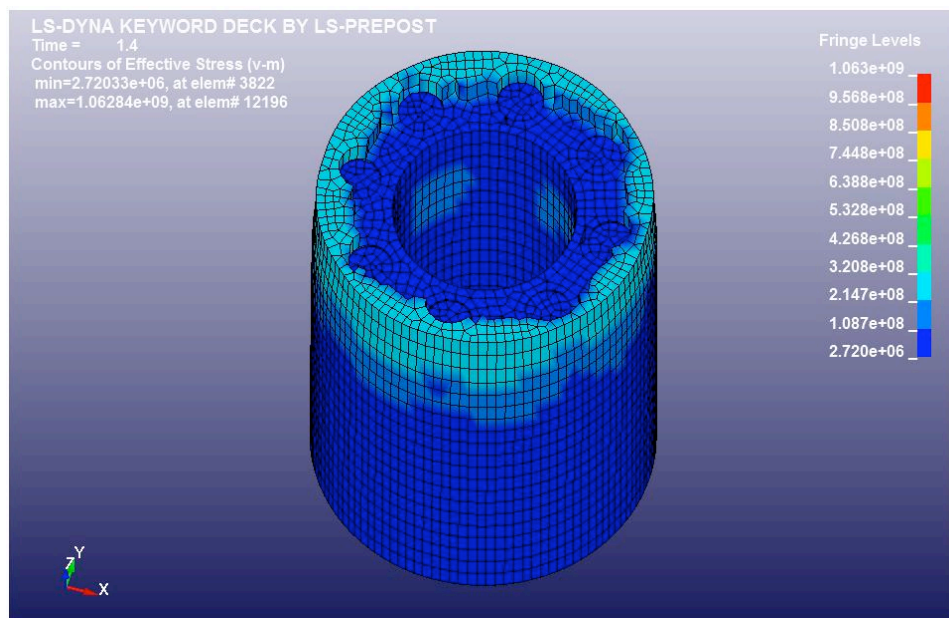


Figure 96 - Fine model with continued necking and failure of the aluminum matrix. Note failed elements are deleted in model.

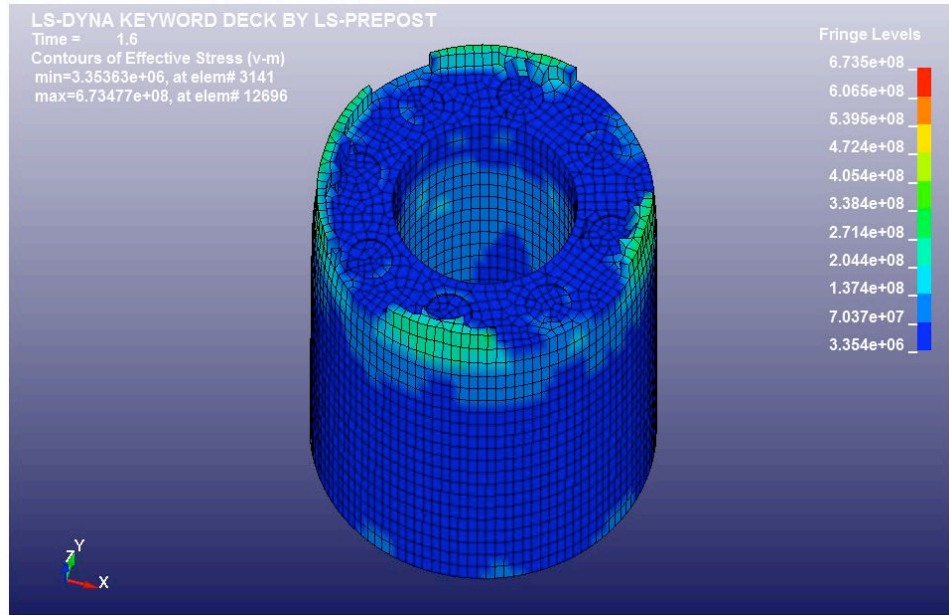


Figure 97 - Fine model with the outermost fibers of the aluminum matrix breaking last. Note failed elements are deleted in model.

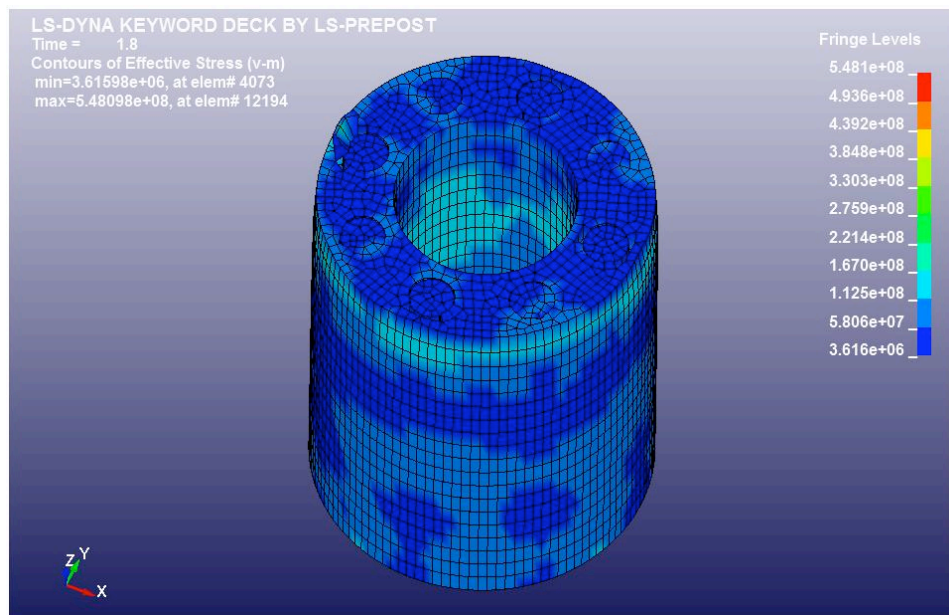


Figure 98 - Fine model when fully fractured. Note failed elements are deleted in model.

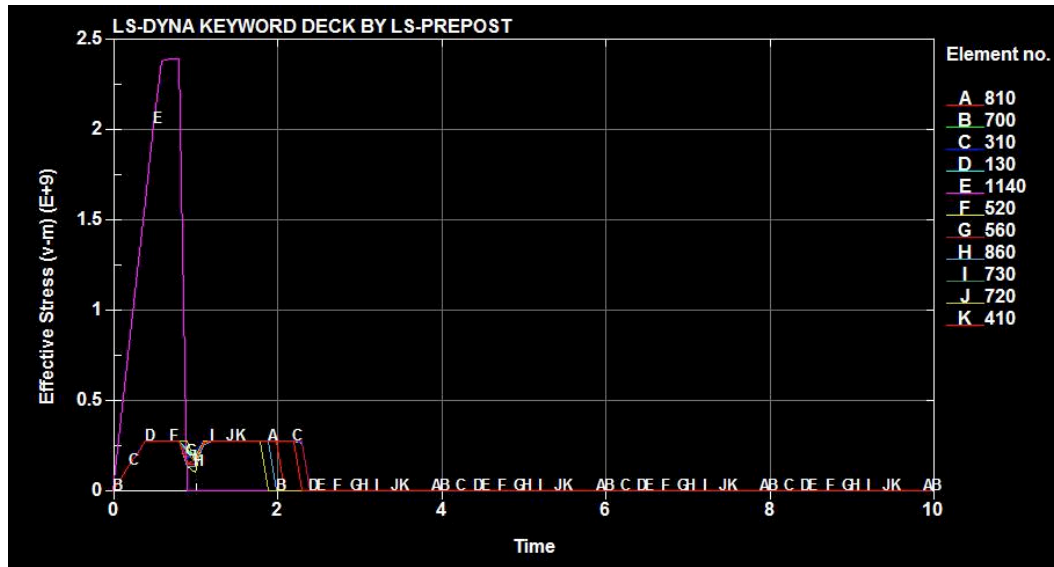


Diagram 33 - Stress vs Time from the course tensile model for several elements along the fracture plane.

Appendix E –Compression Calculation

To show that after the HIP treatment that the components of the composite have residual plastic stresses due to thermal expansion the following analysis is offered. A single W rod will be analyzed surround an Al 6061-T6 cylinder whose outer radius would be the addition of the radius of the W rod and half of the smallest spacing between the W fiber Configurations that was experimented. This model ignores the fact that more material exists outside of the aluminum cylinder, and neglects the end effects of the cylinder-rod geometry. Thus, it is assumed that the W fibers do not interact with each other as far as thermal stresses are concerned and that the stresses induced in the region of each W fiber in the overall composite can be approximated with a cylinder of aluminum.

Thermal expansion must be considered first to set up the analysis. Following Equation 16 the initial expansion of the W fiber is used to determine initial inner diameter of the aluminum tube before cooling.

$$\delta_W^{Thermal} = \alpha_W \Delta T * R_W \quad (16)$$

$$R_{Al-initial} = R_W + \delta_W^{Thermal} \quad (17)$$

Where R is the related radius, alpha is the related coefficient of thermal expansion, and delta-T is the change in temperature. Using the result from Equation 17 and Equation 18 for aluminum the radius of the aluminum cylinder is found after cooling as if the W rod was not present in Equation 19. The radial difference, delta, between what the components want to shrink to is then computed in Equation 20.

$$\delta_{Al}^{Thermal} = -\alpha_{Al} \Delta T * R_{Al-initial} \quad (18)$$

$$R_{Al} = R_{Al-initial} + \delta_{Al}^{Thermal} \quad (19)$$

$$\Delta = R_W - R_{Al} \quad (20)$$

With delta computed and geometry of the components known as if they were not interacting the following equation can be used to find the interfacial pressure, P between the components when they are fitted together [23].

$$P = \frac{2\Delta}{2 \left[\frac{R_{Al}}{E_{Al}} \left(\frac{b^2 + R_{Al}^2}{b^2 - R_{Al}^2} + \nu_{Al} \right) + \frac{R_W}{E_W} (1 - \nu_W) \right]} \quad (21)$$

Where b is the outer radius of the aluminum cylinder, E is the related elastic modulus, and tau is the related poisson's ratio. When the geometrical values chosen from the experimental Configurations found in section 3.1 and material values obtained from matweb.com as follows are inserted into Equations 16-21 it can be shown that plasticity will be induced into the aluminum cylinder.

$$\begin{aligned}
E_{Al} &= 68.9 \text{ GPa} \\
E_W &= 400 \text{ GPa} \\
\nu_{Al} &= 0.3 \\
\nu_W &= 0.27 \\
\alpha_{Al} &= 23.6 * 10^{-6} / C \\
\alpha_W &= 4.4 * 10^{-6} / C \\
\Delta T &= (500 - 22) C = 478 C \\
R_W &= 0.0001 m \\
b &= (0.0001 + 0.00006) m = 0.00016 m \\
Y_{Al} &= 276 \text{ MPa} \\
Y_W &= 2,400 \text{ MPa} \\
Y &= \frac{HV}{3}
\end{aligned}$$

The resulting pressure stress at the interface of the W rod and aluminum cylinder would be 252 GPa. If the results of annealed the aluminum matrix for Configuration 2 are averaged from Table 4 the yield stress of the aluminum is 154 MPa. The resulting interfacial pressure stress is less than the elastic limit for tungsten, 2,400 MPa, but close to or exceeding the yield stress of the aluminum matrix, 154 to 276 MPa. If the outer radius of the aluminum cylinder is expanded to infinity it is seen that the interfacial pressure stress approaches a limit of 492 MPa. Thus it can be concluded that due to thermal contraction of the components in this composite a plastic zone is induced around the W fibers in the aluminum matrix.

With the previous analysis determining that plasticity could be induced into the aluminum matrix the following analysis will estimate the radius of the plastic zone surrounding each fiber. A perfect plasticity model is used for the aluminum matrix with the yield strength of the aluminum being taken as 276 as annealing of the

aluminum due to the HIP treatment will now be neglected. The maximum stress that the Al 6061-T6 can support, 310 MPa is loaded on the interior surface of the aluminum cylinder. Plane strain will be enforced with the end effects of the geometry ignored. The aluminum matrix will be assumed to be initially isotropic and a Tresca Yield surface used to determine plasticity.

The boundary conditions of an infinitely long W rod in an aluminum cylinder are as follows:

$$\sigma_{rr} \big|_{r=R_{Al}} = -P \quad (22)$$

$$\sigma_{rr} \big|_{r=b} = 0 \quad (23)$$

The use of variable r defines the radial distance of interest around the concentric center of the geometry. With the use of Airy stress functions the general equations for radial and tangential stress are derived with variable constants A and B.

$$\phi = P \left(A * r^2 + B * R_{Al}^2 \ln r \right) \quad (24)$$

$$\sigma_{rr} = \frac{1}{r} \frac{\partial \phi}{\partial r} = P \left(2A + \frac{B * R_{Al}^2}{r^2} \right) \quad (25)$$

$$\sigma_{\theta\theta} = \frac{\partial^2 \phi}{\partial r^2} = P \left(2A - \frac{B * R_{Al}^2}{r^2} \right) \quad (26)$$

Using the boundary conditions, Equations 22 and 23, the constants A and B can be found from Equations 25 and 26.

$$B = \frac{-b^2}{b^2 - R_{Al}^2} \quad (27)$$

$$A = \frac{R_{Al}^2}{2(b^2 - R_{Al}^2)} \quad (28)$$

Using Equations 25-27 the general equations of stress are:

$$\sigma_{rr} = \frac{P * R_{Al}^2}{b^2 - R_{Al}^2} \left(1 - \frac{b^2}{r^2} \right) \quad (29)$$

$$\sigma_{\theta\theta} = \frac{P * R_{Al}^2}{b^2 - R_{Al}^2} \left(1 + \frac{b^2}{r^2} \right) \quad (30)$$

The Tresca Yield surface is defined in Equation 31 as follows:

$$\tau_{\max} = \frac{Y}{2} = \frac{\sigma_{\theta\theta} - \sigma_{rr}}{2} \quad (31)$$

$$Y = \sigma_{\theta\theta} - \sigma_{rr}$$

The initial yield pressure is found by combining Equations 29-31.

$$P_Y = \left(\frac{b^2 - R_{Al}^2}{2b^2} \right) Y \quad (32)$$

The equations for the elastic zone, or where r is greater than c , the plastic boundary, but less than b , the outer diameter of the aluminum cylinder, are found using Equations 29, 30, and 32. The pressure is set to the initial pressure.

$$\sigma_{rr}^E = \frac{c^2 Y}{2b^2} \left(1 - \frac{b^2}{r^2} \right) \quad (33)$$

$$\sigma_{\theta\theta}^E = \frac{c^2 Y}{2b^2} \left(1 + \frac{b^2}{r^2} \right) \quad (34)$$

To find the equations of stress within the plastic region of the cylinder the equation of equilibrium must be used with a replacement of variables from Equation 31.

$$\begin{aligned} \frac{\partial \sigma_{rr}}{\partial r} + \frac{\sigma_{rr} - \sigma_{\theta\theta}}{r} &= 0 \\ \frac{\partial \sigma_{rr}}{\partial r} - \frac{Y}{r} &= 0 \end{aligned} \quad (35)$$

Integrate Equation 35 and solve for the resulting constant, C using Equation 33 since radial stress is continuous with r equal to c.

$$\sigma_{rr}^P = Y \ln r + C \quad (36)$$

$$C = \frac{c^2 Y}{2b^2} \left(1 - \frac{b^2}{c^2} \right) - Y \ln c \quad (37)$$

Equation 36 can be rewritten with the result of Equation 37.

$$\sigma_{rr}^P = \frac{Y}{2} \left(\frac{c^2}{b^2} - 1 \right) - Y \ln \left(\frac{c}{r} \right) \quad (38)$$

To find the plastic boundary, c the radius is set to the inner radius of the aluminum cylinder in Equation 38, and Equation 22 is set equal to Equation 38.

$$P = Y \ln \left(\frac{c}{R_{Al}} \right) - \frac{Y}{2} \left(\frac{c^2}{b^2} - 1 \right) \quad (39)$$

Solving for c numerically with the applied pressure P of 310 MPa on the aluminum cylinder with the same dimensions as defined previously it is found that the whole cylinder would be in the plastic domain. Taking the outer radius of the aluminum cylinder to infinity results in the plastic boundary approaching a limit of a radius at 184.8 μm . By assuming the W rod does not deform the thickness of the plastic region around each W fiber would be 84.8 μm .

These results of the plastic zone around each W fiber are just a estimation. In actuality the aluminum matrix is much softer due to being annealed by the HIP treatment, and the aluminum matrix is not initially isotropic due to being heavily plastically deformed during the HIP treatment by flowing into the W structure. These two phenomena have conflicting results on the material properties of the aluminum matrix with the annealing reducing the mechanical strength and the plastic flow increasing the mechanical strength. Additionally the requirement to have the elastic deformation of the W fiber is not enforced to remain in contact with the inner radius of the aluminum cylinder. A full plastic analysis accounting for the history of the aluminum tubes and deformation of the W fiber is necessary to accurately predict the size of the plastic zone around each W fiber due to thermal contraction.

Appendix F – SEM Analysis of Sample 9

After four HIP treatments that exposed the first pair of W fiber layers to approximately 9 hours of pressurized conditions at 500 Celsius a SEM was used to determine that no detectable diffusion of the components occurred. Without diffusion of W into the Al 6061 as observed in sample 6 of Configuration 1 it is concluded that no intermetallic compound was created. Further evidence of the lack of diffusion is the sharp boundary between the components as seen in Figure 99. Had an intermetallic compound been created the boundary between the W fiber and aluminum matrix would be indistinct. The detection of aluminum approximately 4 um deep into the W rod is of the same order of magnitude commonly associated with an SEM's spatial resolution of 1 um [24]. Thus the observed penetration of aluminum atoms could be due to noise within the SEM. However this amount of diffusion between the components is considered acceptable for our application. Figures 100 and 101 show the intensity of the elements detected as the line was scanned as seen in Figure 99. Notice the sharp boundary between elements supported the conclusion that little to no diffusion occurred.

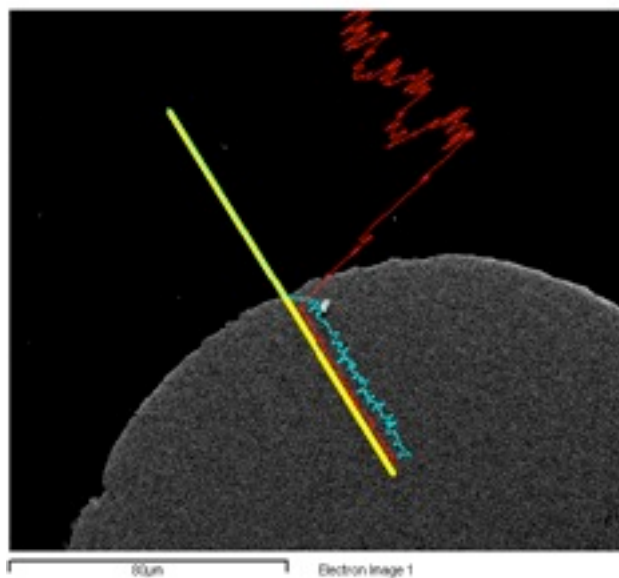


Figure 99 – SEM line scan of the cross section of a W fiber exposed to all four HIP treatments. Notice the sharp boundary between the W fiber seen in gray and the aluminum matrix seen in black.

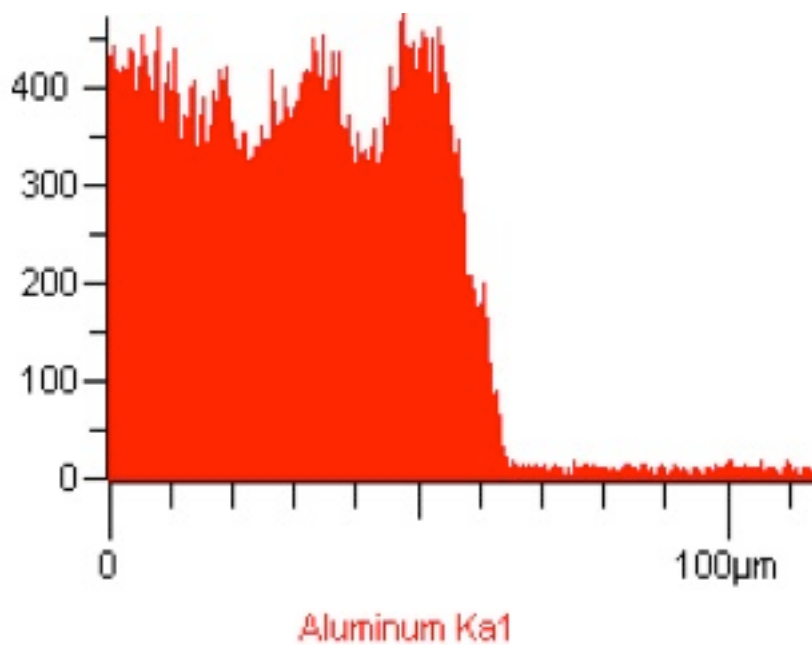


Figure 100 – SEM line scan from Figure 99 of the aluminum. At a distance from the beginning of the scan larger than 60 um the peaks are thought to be noise.

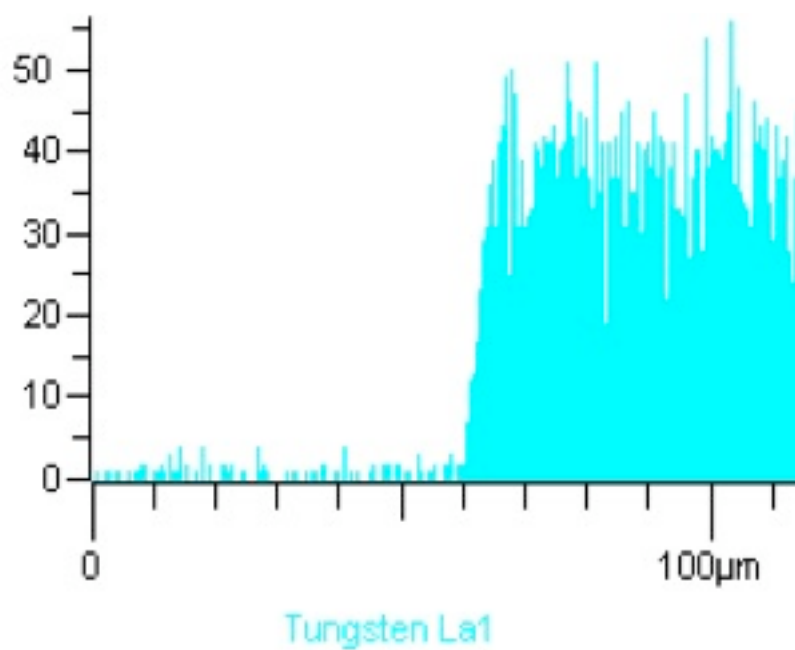


Figure 101 - SEM line scan from Figure 99 of the tungsten. At a distance from the beginning of the scan smaller than 60 um the peaks are thought to be noise.

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