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RPPR Final Report
as of 15-Mar-2023

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Final Report for Period Beginning 01-Oct-2018 and Ending 15-Aug-2022

Title: Advanced Modeling and Simulation of Thermal Kinetics in Additive Manufacturing

Begin Performance Period: 01-Oct-2018

End Performance Period: 15-Aug-2022

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STEM Degrees: 6

STEM Participants: 12

Major Goals: Major Goals: Additive manufacturing (AM) is inherently a thermal process where layered building is achieved through directed energy input with melting and solidification, energy input for sintering without melting, or binding through a separate agent (e.g., a chemical reaction). In all cases, heat input and its time-dependent propagation through the part plays a critical role. In this project, AM thermal kinetics are studied in two sub-projects. These are: 1) crystal growth modeling in a new powder bed silicon carbide AM process 2) residual stress modeling in wire-arc AM for magnesium alloys.

The tasks for the Powder Bed Silicon Carbide AM were as follows:

- (1). Printer Modification,
- (2). Binder Selection,
- (3). Powder Selection,
- (4). Sintering profile selection,
- (5). 3D printing parameter selection

All the tasks needed some minor modifications as we discovered that the NaOH had a corrosive action on certain metallic parts of the printer. Therefore, a new strategy was developed: instead of using NaOH as the binder solution during 3D printing, the SiC particles were pre-treated with NaOH and dried in air prior to feeding them in the printer. Please see the attached file for the detailed descriptions of the modifications as well as the results.

While most tasks were completed, one task that hasn't been completed and still ongoing is the modeling component for nanowire growth. The experimental work by our group has clearly identified the mechanisms underlying the nanowire growth. One of the Ph.D. students that conducted experiments has also been working on the modeling work and will complete this part of the proposed work by the end of this year. She is currently

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supported by departmental funding as the ARO grant ended in September 2022.

The second project in this proposal is on Wire Arc AM for Magnesium alloys. The objective of the combined numerical-experimental research is to establish the input-output relationships for the magnesium alloys wire arc AM process. The tasks for this project were

- (1). current pulsing,
- (2). torch motion,
- (3). cooling rate,
- (4). In-process metrology,
- (5). finish machining.

The project was challenging due to the difficulties associated with welding magnesium alloys with very little helpful literature. Three main forms of GMAW were tested. DC welding was identified as the only method of layer deposition due to its superior bead deposition, bead shape control, and semi-controllable heat input with our commercial welder. Due to issues with the welding machine, change of PI, and the pandemic-related delays, only two cooling rates could be considered and cryogenic CO₂ cooling method could not be tried before the end of the grant. In addition, in-process metrology has also been deferred. Other than this, all the other tasks have been completed.

However, this project is the dissertation topic of Mr. Christoph Kossack. He has been hired as a teaching assistant professor in our department. He will continue to work on many of the unfinished tasks in the original proposal.

By June 30, 2023, two Ph.D. students supported by the grant will be graduating. One more Ph.D. student also supported by the grant will be graduating by the end of the year. All three of them have contributed significantly to the success of the project.

Accomplishments: Please see the uploaded pdf file.

Training Opportunities:

Several undergrad and grad students received training and participated in professional development activities. Here are some examples:

1. Farjana Sultana, Sujithra Chandrasekaran, Madeline Greenier, and Damien Beasock received training on the use of a Scanning Electron Microscope.
2. Christoph Kossack attended courses offered by COMSOL to learn about using COMSOL technologies to model GMAW and predict residual stresses and distortion.
3. Sean Perry, Farjana Sultana, and Sujithra Chandrasekaran also received training on the use of the 3D printer: 3D Projet460 Plus that was used in this project.
4. In addition, the Crystal Growth component of the project spawned additional research topics on the experimental side. Our department supported several undergrads that worked on these topics. They received training on using the 3D printer mentioned above, making SiC samples, etc.
5. Farjana Sultana and Sujithra Chandrasekaran (two Ph.D. students) mentored two Senior Design teams with each team consisting of 3 students. The teams were working on projects related to 3D-printed SiC screws and applications to graft fixation devices.

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Results Dissemination: The project led to several peer-reviewed papers, conference papers, and conference presentations. Three doctoral dissertations will be based on the work proposed in this project. The three Ph.D. students will be graduating in 2023. Their dissertations are anticipated to lead to additional peer-reviewed papers and conference presentations.

1. El-Ghannam, Ahmed, Sujithra Chandrasekaran, and Farjana Sultana. "Synthesis and characterization of a novel silica nanowire-reinforced SiC thermal material." *Journal of Solid State Chemistry* 297 (2021): 122055.
2. El-Ghannam, Ahmed, Sujithra Chandrasekaran, and Farjana Sultana. "Mechanism of epitaxial growth of silica nanowires reinforcing agent in porous SiC scaffold." *Procedia Manufacturing* 53 (2021): 535-560.
3. El-Ghannam, Ahmed, Sujithra Chandrasekaran, "Enabling 3D printing of SiC using a powder bed binder jet printer" technical poster presentation for "The Carolinas Section Spring event" conducted by "The American Ceramic Society" on May 10, 2022.
4. El-Ghannam, Ahmed, Sujithra Chandrasekaran, "Novel SiC-Mineral binder Composite for thermal and space applications" technical poster presentation for "The Carolinas Section Spring event" conducted by "The American Ceramic Society" on May 10, 2022.
5. El-Ghannam, Ahmed, Farjana Sultana, "Thermomechanical properties of a novel SiC-Mullite thermal material prepared by liquid phase sintering mechanism" technical poster presentation for "The Carolinas Section Spring event" conducted by "The American Ceramic Society" on May 10, 2022.
5. Dan Theologidy, et.al., "3D printing of a Bioactive fixation device for orthopedic applications" Senior Design Poster Expo conducted by University of North Carolina at Charlotte on Dec 12, 2022.
6. Sujithra Chandrasekaran, Chengying Xu, James A. Monroe, Ahmed El-Ghannam, "Thermo-mechanical properties of SiC-mineral binder composites for space applications". (Paper has been peer-reviewed and accepted. Yet to be published online in Proceedings of the ASME 2022 International Mechanical Engineering Congress and Exposition - IMECE2022).
7. Christoph Kossack, "Examination of Gas Metal Arc Welding as a potential method for additively manufacturing parts out of magnesium alloys", Accepted for presentation at AEROMAT 2023, March 14-16, 2023, Fort Worth, Texas.
8. Ahmed El-Ghannam, Tony Schmitz and Damian Beasock, "A novel Approach to Facilitate Densification and Strength of 3D Printed SiC Articles", 42nd International Conference and Exposition on Advanced Ceramics and Composites (ICACC 2018) 'Symposium 15: Additive Manufacturing and 3D Printing Technologies: Emerging Additive Manufacturing Technologies'. January 21-26, 2018, Daytona Beach, Florida, USA

Honors and Awards: Nothing to Report

Protocol Activity Status:

Technology Transfer: Ahmed El-Ghannam and Tony Schmitz; Composite carbide compositions and methods of making the same, U.S. Patent application number 62447641, based on Intl. Application No. PCT/US2018/014234 Accepted and allowed for issuance, February (2022).

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Person Months Worked: 3.00

Project Contribution:

National Academy Member: N

Funding Support:

Participant Type: Co PD/PI

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Person Months Worked: 3.00

Project Contribution:

National Academy Member: N

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National Academy Member: N

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Project Contribution:
National Academy Member: N

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Project Contribution:
National Academy Member: N

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National Academy Member: N

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Project Contribution:
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Funding Support:

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Project Contribution:
National Academy Member: N

Funding Support:

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Project Contribution:
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Funding Support:

Participant Type: Graduate Student (research assistant)
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Person Months Worked: 1.00
Project Contribution:
National Academy Member: N

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Article Title: Effect of processing parameters on the microstructure and mechanical behavior of a silicon carbide-silica composite

Authors: Damian Beasock, T. Michael Stokes, Ahmed El -Ghannam, Tony Schmitz

Keywords: Additive manufacturing, silicon carbide, powder bed, sodium hydroxide

Abstract: Silicon carbide (SiC) is an inert material with excellent thermomechanical properties for many optoelectronic, structural, and ballistic applications. However, manufacturing SiC components is challenging due to the high temperatures (> 2000 deg C) and pressures (1000-2000 atm) required for traditional sintering processes. The objective of the present research study is to enable the additive manufacture of SiC components using a new powder bed, sodium hydroxide (NaOH) binder-jetting process. The innovation is the secondary crystal growth (SiO₂) that serves to bridge and close the gaps between the SiC particles via a chemical attack (oxidation) of their surfaces by the NaOH solution. To evaluate the process, SiC powder was mixed with a NaOH-water solution at room temperature and shaped into 10 mm diameter by 15 mm long cylinders in a mold with compaction by hand pressure only.

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Article Title: Synthesis and characterization of a novel silica nanowire-reinforced SiC thermal material

Authors: Ahmed El-Ghannam, Sujithra Chandrasekaran, Farjana Sultana

Keywords: SiC Thermal material, Thermal shock resistance, Silica gel, Silica nanowires, Dimensional stability, Quartz to cristobalite

Abstract: When forming a 3D SiC object by thermal oxidation of compacted particles, the resultant solid is often found to have limited strength. A major reason for this lack of structural integrity is believed to be due to poor oxygen diffusion and this, in turn, limits the thickness of the silica bonding layer at the interface between SiC particles. In the present study it is demonstrated that SiC particles treated with NaOH will create a silica gel layer that enhances the bonding during thermal oxidation. The surface modified particles were compacted into discs at an applied pressure of 0.47 MPa and then heated at temperatures ranging from 500 to 1000 °C. FTIR confirmed the presence of the characteristic peaks for Si-O and Si-O-Si on the surface of the discs characteristic of the formation of a silica gel layer that bonded the particles at room temperature. SEM-EDX analysis revealed the growth of silica nanowires that bridged the walls of the pores between SiC particles during heat treatment

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Paper Title: LOW COST PLATFORM FOR HYBRID MANUFACTURING OF LIGHT METALS

Authors: Justin West, Emma Betters, Tony Schmitz

Acknowledged Federal Support: Y

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Partners

,

I certify that the information in the report is complete and accurate:

Signature: Harish Cherukuri

Signature Date: 2/10/23 8:23PM

Advanced Modeling & Simulation of Thermal Kinetics in Additive Manufacturing

Grant ID: W911NF1810482

Part 1: Crystal Growth Modeling in a New Powder Bed Silicon Carbide AM process

Abstract

Processing 3D SiC objects by thermal oxidation is cumbersome due to poor oxygen diffusion in SiC and the limited thickness of the silica layer at the interface between particles. In the present study we treated SiC particles with NaOH to create a silica gel layer. The surface modified particles were pressed manually at 0.47 MPa into discs and subjected to thermal treatment in the temperature range 500-1000 °C. FTIR confirmed the presence of the characteristic peaks for Si-O and Si-O-Si on the surface of the discs confirming the formation of a silica gel layer that bonded the particles at room temperature. SEM-EDX analysis demonstrated the growth of silica nanowires that bridged the walls of the pores during heat treatment. XRD showed the phase transformation of the silica gel into quartz and then cristobalite solid solution at higher temperature. In conjunction with the increased growth of the silica nano wires, the strength, toughness, and percent weight gain increased significantly without any measurable change in the dimensions of the discs. FTIR analysis of the condensed volatile species released from SiC discs during heating revealed the involvement of SiO gas in the mechanism of nanowires growth. Preliminary thermal shock resistance tests showed stability of the mechanical properties after water quenching of samples treated at 900-1000 °C. The new mechanism of SiC densification by silica nanowires growth while maintaining dimensional stability opens the door for new SiC manufacturing technology.

Initial studies were conducted to understand the effect of surface modifications of SiC on the microstructure and properties. Samples for these studies were done using powered metallurgy method since the 3D printer modification was still under testing. The goal was to activate the surface of SiC by various concentrations of alkali solution to oxidize the surface and study the effect of thermal treatment parameters on microstructure, density and mechanical properties of surface modified SiC.

Objectives

Task 1: Printer modification

Task 1 in the proposal states that “The current UNC Charlotte ZCorp 510 powder bed printer is configured to print using a calcium sulfate hemihydrate powder and ZCorp proprietary humectant-water solution binder (250 mm × 250 mm print area). The binder delivery system (i.e., HP11 inkjet print head) is compatible with the NaOH binder planned for this research, so a direct replacement of the liquid binder is planned. SiC powder diameters from 80 nm to 100 μm will be tested to verify that uniform layers can be rolled without agglomeration and significant abrasion of the current stainless-steel roller. If roller wear is an issue, the team will repair the roller using low phosphorous electroless nickel plating.” Moreover, the planned Solution for this task in the proposal was: “Uniform SiC powder layers can be rolled and printed for arbitrary geometries. Roller abrasion occurs at a low enough rate that experiments can be completed without repeated roller repair.”

Findings: The NaOH binder solution damaged the printheads during 3D printing. Trials to solve the problem by using a diluted solution of NaOH were not successful. Several trials included

changing the printer head were not successful either. This is due to the corrosive action of NaOH on certain metallic parts of the ZCorp510 printer. Therefore, a new strategy was made to overcome this problem.

Proposed Solution: Instead of using NaOH as the binder solution during 3D printing, the SiC particles were pre-treated with NaOH and dried in air prior to feeding them in the printer. The NaOH treatment created a silica gel layer on the surface of the SiC particles. For 3D printing, a commercially available water-based sugar solution was used as a binder instead of the NaOH solution. During printing, the water-based binder hydrated the silica gel layer and enabled bonding the particles together. Uniform SiC powder layers could be rolled and printed for arbitrary geometries. We did not experience roller abrasion problem and the experiments could be completed without roller repair. See Fig. 1 on page 8 for examples of 3D printing of SiC Discs.

Task 2: Binder Selection

Task 2 in the proposal stated that: “The objective of Task 2 is to select the optimal NaOH concentration that facilitates growth of silica at the grain boundaries of SiC and enhances densification and mechanical properties. Experiments will be completed to test the dependence of SiC microstructure, density, linear shrinkage, and mechanical properties on the NaOH concentration. Various concentrations (1, 5, 10, 15 and 20% weight by volume) will be used for 3D printing of cylindrical SiC samples with 40 μm to 100 μm diameter SiC powders. The samples will be dried at 95 $^{\circ}\text{C}$ for 24 hr and subjected to heat treatment following the same protocol described in Section C (heating rate 1 $^{\circ}\text{C}/\text{min}$ to 100 $^{\circ}\text{C}$, 6 hr hold, 1 $^{\circ}\text{C}/\text{min}$ to 350 $^{\circ}\text{C}$, 6 hr hold, 5 $^{\circ}\text{C}/\text{min}$ to 1100 $^{\circ}\text{C}$, 4 hr hold). The density of the samples prepared using the various NaOH concentrations will be measured and compared. Linear shrinkage will be determined from pre- and post-sintering sample length measurements. SEM-EDX will be used to analyze the outer and fracture surfaces of the SiC samples. The SEM images will be analyzed using ImageJ, a public domain Java-based image processing program developed at the National Institutes of Health, to quantify the porosity and grain boundary sealing. Strength and elastic modulus will be determined from stress-strain measurements using the Instron 5582 UTM. The density and mechanical properties will be correlated to the porosity, morphology, and growth of the secondary crystals on the sintered SiC. This will provide fundamental new knowledge for powder bed 3D printing. The NaOH concentration that produces the highest density and strength will be selected for objectives 3 and 4. The planned Solution in the project stated that: “Material properties and microstructure comparable to solid SiC are obtained. Increased understanding of the secondary crystal behavior and its dependence on binder chemistry are established.”

Proposed Solution: As we described in Task #1, NaOH solution was not used as a binder because of its damaging effect on the printer. It was found that the 3D printed SiC has weak mechanical properties. Therefore, to enhance the mechanical properties, a second NaOH immersion treatment of the 3D printed object was implemented followed by thermal treatment. We studied the effect of impregnating 3D printed SiC discs with various concentration of NaOH on the density and mechanical strength. The optimal NaOH concentration that (i) facilitated growth of silica

nanowires within the pores of 3D printed SiC and (ii) promoted the adhesion at the SiC grain boundaries to enhance densification and mechanical properties were studied.

Findings: To improve the mechanical integrity and handling of the 3D printed objects, a second surface treatment with NaOH was applied. The effect of various NaOH concentrations, on the density and mechanical properties of the 3D printed discs was analyzed and correlated to the silica gel layer and silica nanowires growth. The density data in Figure 10 showed that the density of 3D printed samples impregnated with 10% NaOH is significantly higher than that for samples impregnated with 5% NaOH ($P < 0.01$) and with 20% NaOH ($P < 0.002$). The significant increase in the density of the samples treated with 10% NaOH compared to that treated with 5% NaOH is attributed the reduced porosity and pore size due to the growth of silica nano wires within the porous structure. On the other hand, the significant increase in the silica phase after treatment with 20% NaOH is responsible for the observed lower density of the samples due to the inherent low density of the silica phase compared to the SiC phase.

Task 3: Powder selection

Task 3 in the proposal stated that: “The objective of Task 3 is to select the optimal nm/ μ m particle size ratio that maximizes densification and mechanical properties of the SiC samples prepared by 3D printing. Mixtures of {40 μ m to 100 μ m} and {80 nm to 200 nm} SiC particles will be prepared to produce various ratios (0, 5, 10, 15 and 20% nm to μ m). The mixtures will be applied to 3D print SiC samples using the NaOH binder concentration and amount identified in Task 2. The same sintering profile will also be implemented. The density, linear shrinkage, and mechanical properties will be measured and correlated to the morphology, porosity, and grain boundary sealing. The particle size ratio that yields the highest density and mechanical properties will be selected for further analyses in Task 4. The planned solution stated that: “Material properties and microstructure comparable to solid SiC are obtained. Increased understanding of the secondary crystal behavior and its dependence on powder selection are established.”

Findings: The objective of Task 3 was to select the best combination of nano and micro SiC particles that would assist in 3D printing, enhance densification, and mechanical strength. First SiC particles in the size range 10-100 μ m with 40 μ m as an average particle size were used to 3D print SiC discs. The 3 D printed discs had poor structural integrity after sintering at high temperature (1000 C). Hence, the submicron particles were added to fill the interspace between the large particles. Experimental studies were made to evaluate the effect of different ratios of SiC particles 80 nano to 40 microns on the density and mechanical properties. SEM analysis of samples prepared by manual press suggested that using SiC particles 80 nm is not enough to maximize the density. Increasing the percentage of 80 nm particles in a powder bed printer would not allow even spread of layers during 3D printing.

Proposed solution: In order to, enhance the densification without compromising the homogeneity of the printed layers we chose to use 70 wt% of 40 μ m SiC particles with 15 wt% of 2 μ m and 15 wt% of 600 nm SiC particles. The selection of the particle sizes and the corresponding ratios were based on the size of the pores observed by SEM on the discs made of 80nm and 40 μ m particles. Figure 11 demonstrates that the introduction of the nano and sub-micro particles in the above ratio tremendously increased the density by filling the interspace created by the coarser 40 μ m particles.

Task 4: Sintering profile selection

Task 4 in the proposal stated that: “The objective of Task 4 is to select the optimal thermal treatment parameters that facilitate nucleation and growth of secondary crystals at the SiC grain boundaries and enhance densification and mechanical properties.”

Findings: Experiments were completed to test the dependence of SiC sample microstructure and mechanical properties on the thermal treatment parameters. The DSC curves for all samples treated with various concentrations of NaOH showed an endothermic peak at 525.5 °C indicating the nucleation of the silica crystals (Cristobalite). The samples were thermally treated at the nucleation temperature for different time periods and then the temperature was raised to enable crystallization. DSC analysis showed a major exothermic peak due to crystallization in the temperature range from 667 - 831 °C, 702 - 861 °C, and 689 - 938 °C for samples pretreated with 5 %, 10 % and 20 % NaOH. Measurement of area under the peaks showed an increase in the ΔH as the NaOH concentration increased from 5 % to 20 %. The ΔH for samples pretreated with 5%, 10% and 20% where 45.8 J/g, 87.97 J/g and 446.7 J/g, respectively. The increase in ΔH correlates with the significant weight gain observed by TGA due to nanowire formation by oxidation reaction. The most pronounced effect of the nucleation time is the significant increase in mechanical strength as the nucleation time increased from 1-12 hrs. The samples treated for 1 hour, 6 hours and 12 hours had a strength of 1.646 MPa, 2.710 MPa, 6.855 MPa respectively (Fig. 27). The samples treated at 550 °C for 18 or 24 hours showed lower mechanical strength than that of the samples treated for 12 hours at the same temperature. The nucleation and crystal growth under oxygen and air flow were investigated. The influence of oxygen flow on crystallization and morphology of the secondary crystals were analyzed by SEM-EDX (Figures 32, 33 and 34).

Task 5: AM parameter selection

Task 5 in the proposal stated that: “The objective of Task 5 is to determine the optimal 3D printing parameters that provide efficient interaction between the NaOH binder and the SiC particles as determined by the highest density and mechanical properties of the SiC parts. Experiments will be completed to test the dependence of density and mechanical properties on the 3D printing parameters: To facilitate uniform spreading of thin SiC powder layers, the powder will be loosely packed and subjected to the press rolling technique, where a load is applied to the powder by a rolling spreader rod [15].”

Findings: We changed the protocol for 3D printing of SiC and used water-based binder to print Surface modified SiC. We did not vary the pressure during printing due to unavailability of this function option in the machine. Instead, we obtained homogenous spreading of the particles layer-by layer while printing using an in-built Auger that fluidize the fine particles before each printing in 3D Projet 460Plus printer. The thickness of the layer that produced mechanically stable green discs was 120 μm .

Task 1: Printer modification

The SiC particles were pre-treated with NaOH prior to feeding them in the printer, instead of using NaOH as the binder solution. A commercially available aqueous sugar solution was used as a binder. The binder was supplemented by the printer manufacturer.

Successful Approach: The surface modification of the SiC particles by the NaOH successfully enabled 3D printing of SiC objects using the commercially available water-based binder during printing.

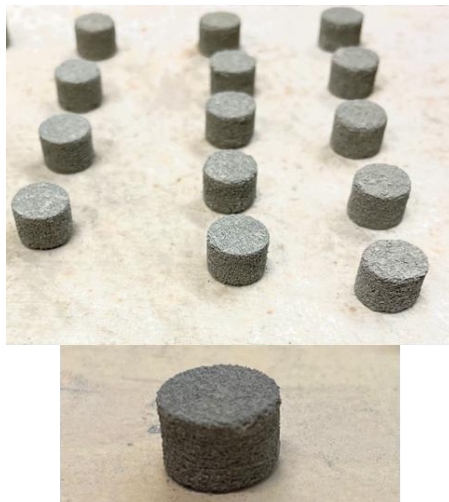


Fig. 1. Digital images of 3D printed discs printed using Projet460Plus. The insets show the magnified image of the printed samples

1.1. Pretreatment of the Silicon Carbide Particles in Sodium Hydroxide

1.1.1. Preparation of NaOH solution

Dry sodium hydroxide pellets obtained from Sigma Aldrich were weighed out on a balance to 200 g increments to make a 20% sodium hydroxide mass by volume solution. Each 200 g increment of sodium hydroxide was put into a 500 mL beaker with a stir bar on a stir plate. 250 mL of nano pure water was added to the beaker and the stir bar was turned on to 40 rpm as the exothermic NaOH dissolution reaction occurred. The contents were poured into a 1 L graduated cylinder and nano-pure water was added until the solution was exactly 1 L. The NaOH final concentration was 20% wt/vol.

1.1.2. Immersion treatment of SiC in the NaOH solution:

Silicon Carbide with an average particle size of 40 μm weighed out into 1 kg increments and placed in a 5-gallon plastic bucket. The 20% sodium hydroxide solution was added to the 5-gallon bucket in a ratio of 1L of 20% sodium hydroxide to each 1 kg of silicon carbide powder. A power drill with a paint mixer attached to its end was used to mix the particles until a homogenous slurry is formed. The bucket was covered with a lid to prevent any evaporation and left for 24 hours.

1.1.3. Washing and drying procedures of the SiC particles:

The NaOH solution was decanted, and SiC particles were washed by adding 1 L of nano pure water per kg of silicon carbide. The particles were resuspended using power driven paint mixer and mixed for 10 min continuously. Then the solution was left to settle for 24 hours. The silicon carbide solution was decanted, and washing was repeated three times to remove excess Na ions. 6 grams of the wet SiC particles were resuspended in fresh 30 mL of nano pure water, and the pH was measured to ensure that the pH lies between 10 and 11. 8 oz of isopropyl alcohol per kg of silicon carbide was added to the wet silicon carbide. This was not stirred and left to dry overnight in the chemical fume hood to facilitate removal of the moisture left over in the wet silicon carbide powder.

The semi-dry particles were taken from the 5-gallon bucket and placed in a large plastic tray to dry in the chemical fume hood which helps increase the surface area exposed to air overnight. After drying, the Silicon Carbide forms a crust like layer, which is hard on the surface and loose inside. To disaggregate the particles, the material was transferred to a closed plastic bottle and milled using alumina beads for 4 hours. After the powder is taken out of the mill, it was sifted to remove the alumina beads and then sifted again using a 200 μm sieve to remove large aggregates that may be present. The powder was taken and put in plastic drying trays and heated to 100 $^{\circ}\text{C}$ for 12 hours in the drying oven. The powder was taken out, allowed to cool to room temperature and re-milled using the same process above. The powder was then used for 3D printing according to the protocol below.

1.2. Testing post processing protocol for strengthening and densification of 3D printed SiC

1.2.1. Effect of crystallization temperature and time on silica nanowires growth

To find the best crystallization temperature to obtain maximum strength and densification, the 3D printed discs were sintered at temperatures such as 800 $^{\circ}\text{C}/2\text{h}$, 900 $^{\circ}\text{C}/2\text{h}$, and 1000 $^{\circ}\text{C}/2\text{h}$. Figure 2 shows the differences in the average compressive strength of the control samples (without secondary treatment with NaOH) heat treated at various temperatures for 2 hours. There was a significant increase in strength between 800 $^{\circ}\text{C}$ and 1000 $^{\circ}\text{C}$. Temperature facilitates the growth of nanowires which grow within the pores and the grain boundaries that densifies the material. Figure 3 shows the SEM micrograph showing effect of temperature in the growth of the nucleation droplets which implies the growth of the nanowires. At 800 $^{\circ}\text{C}/2\text{h}$ the droplet size was measured as $0.58 \pm 0.12 \mu\text{m}$ and at 900 $^{\circ}\text{C}/2\text{h}$ the size of the droplet was doubled to $1.19 \pm 0.18 \mu\text{m}$. At 1000 $^{\circ}\text{C}/2\text{h}$ thin nanowires are grown from these droplets. This proves the mechanism that is involved in strengthening of the discs at 800 $^{\circ}\text{C}$ and 1000 $^{\circ}\text{C}$.

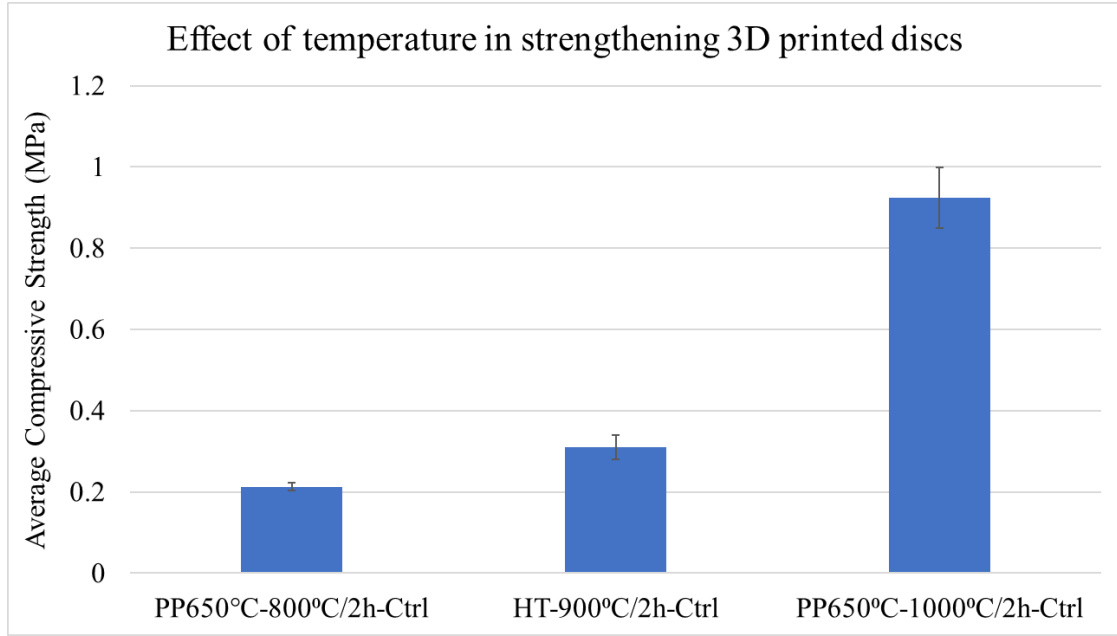


Figure 2: Average compressive strength of 3D printed nano- μ samples heat treated at different temperatures for 2h

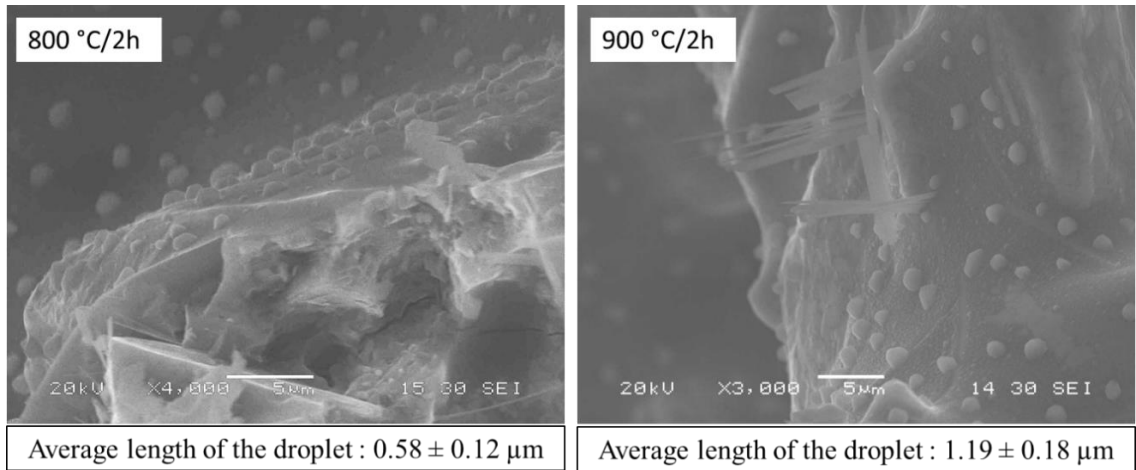


Figure 3: The growth in the size of the silica droplets lengthwise with respect to temperature

Approach to improve the mechanical strength of the 3D printed samples

We immersed the 3D printed discs in NaOH to create additional silica gel layer that contributes to mechanical integrity of the samples upon heat treatment. After 3D printing, SiC discs were immersed in 20% NaOH for 10 min. The samples disintegrated in the NaOH solution and could not keep the physical shape. Therefore, the 3D printed discs were thermally treated prior to impregnation with NaOH. The 3D printed discs were separated into two batches to test two treatment procedures: (i) The first, 3D printed discs were cleaned to remove the loose particles and heat treated at 900 °C for 2h then impregnated in 20% NaOH for 10 min and dried at 100 °C for 24 hrs in the drying oven. (ii) The second, 3D printed discs were heat treated at 650 °C for 5h, cleaned to remove the loose particles and impregnated in 20% NaOH then dried at 100 °C for 24

hrs in the drying oven. The surface chemistry of the dried samples was analyzed by FTIR to determine the formation of silica gel layer. Then the samples were subjected to thermal treatment to enhance the mechanical strength.

1.2.1. FTIR analysis

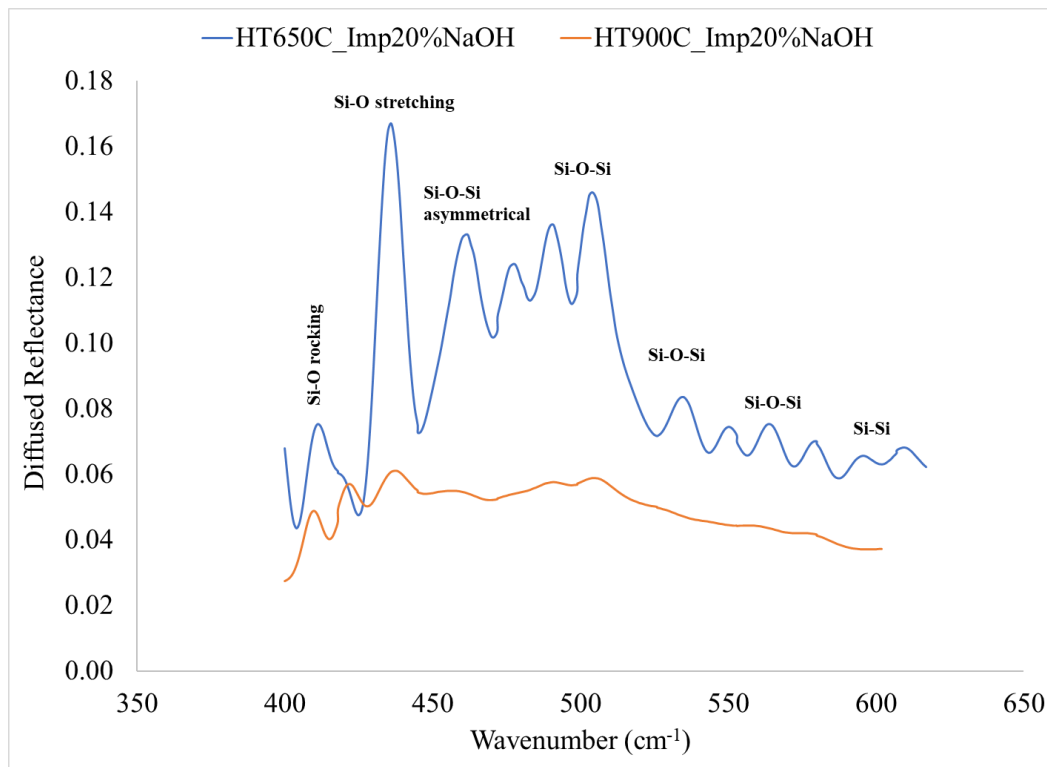


Figure 4: FTIR graph showing the effect of heat treatment temperature prior to impregnation in creating successful Si-O-Si bonds in 3D printed discs using 70:30 nano- μ SiC powders.

The surface chemistry was analyzed by Fourier Transform Infrared Spectroscopy (FTIR; Nicolet 6700 Thermo-Nicolet, Madison, WI) in the diffuse reflectance mode. Potassium Bromide (KBr) was used to collect the background and the spectrum for the sample are then baseline subtracted and smoothened to remove noise. The spectra are then processed and converted to Kubelka-Munk absorbance for the y-axis. Kubelka-Munk (K-M) model is a widely used model in case of diffused reflectance for semi-infinite samples. Figure 4 is the FTIR analysis of samples treated at 650 °C/5h/impregnated in 20% NaOH and 900 °C/2h/impregnated in 20% NaOH. The surface of samples that were heat treated at 650 °C for 5 hours and then impregnated in NaOH showed several high intensity bands for Si-O and Si-O-Si bonds compared to those samples sintered first to 900 °C for 2 h and then impregnated in NaOH.

1.2.2. Mechanical testing

After treatment at 650 °C the silica phases are poorly crystallized cristobalite and quartz solid solutions. These phases can easily be attacked by the NaOH to form abundance of silica gel layer.

On the other hand, the silica phase in samples treated at 900 °C have fully matured into cristobalite which is less reactive than silica phases formed at lower temperature. The formation of high quantity of silica gel layer on the surface of the pores of the 3D printed SiC after NaOH treatment enhanced interfacial bonding between particles and facilitated nanowires growth which in turn improved the mechanical properties (Figure 5).

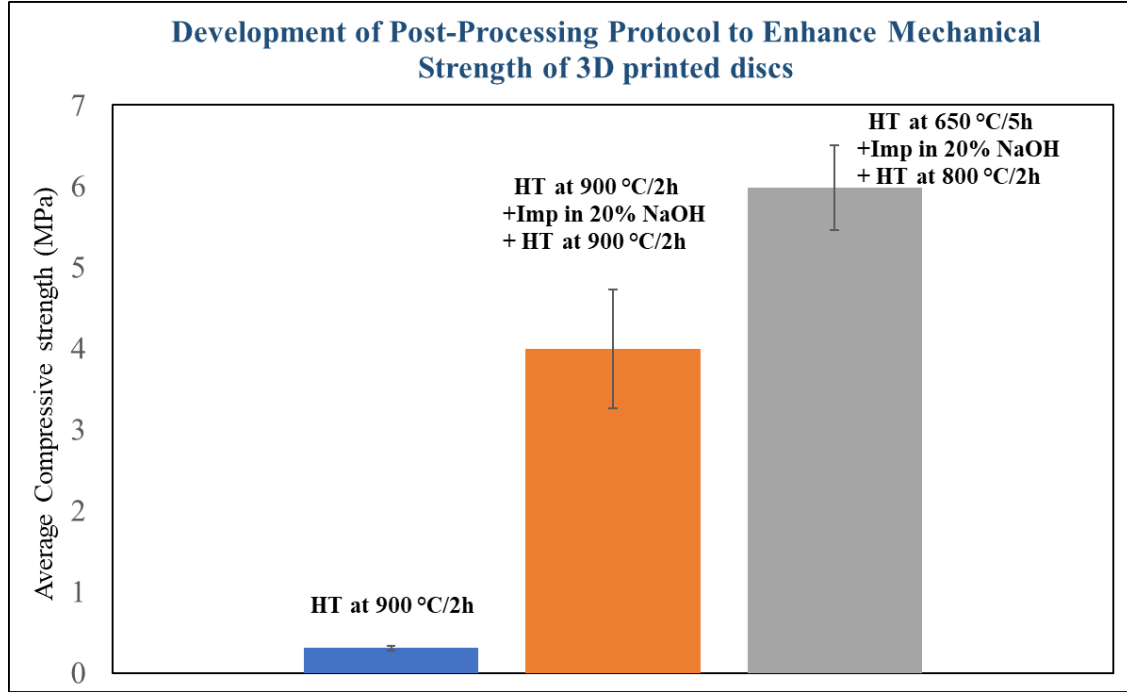


Figure 5: Comparison of the average compressive strength of 3D printed discs with nano- μ powder that are sintered at 900 °C/2h without post processing to the samples that are impregnated with and without the post processing temperature of 650 °C.

To analyze the effect of thermal treatment dwelling time on nanowires growth and mechanical strength, the samples were impregnated in 20% NaOH and heat treated at 1000 C for 2h or 24 h. compressive strength of the samples treated for 24 h (11 MPa) was significantly higher than that (5 MPa) of the samples treated for 2h at 1000C (Fig. 6).

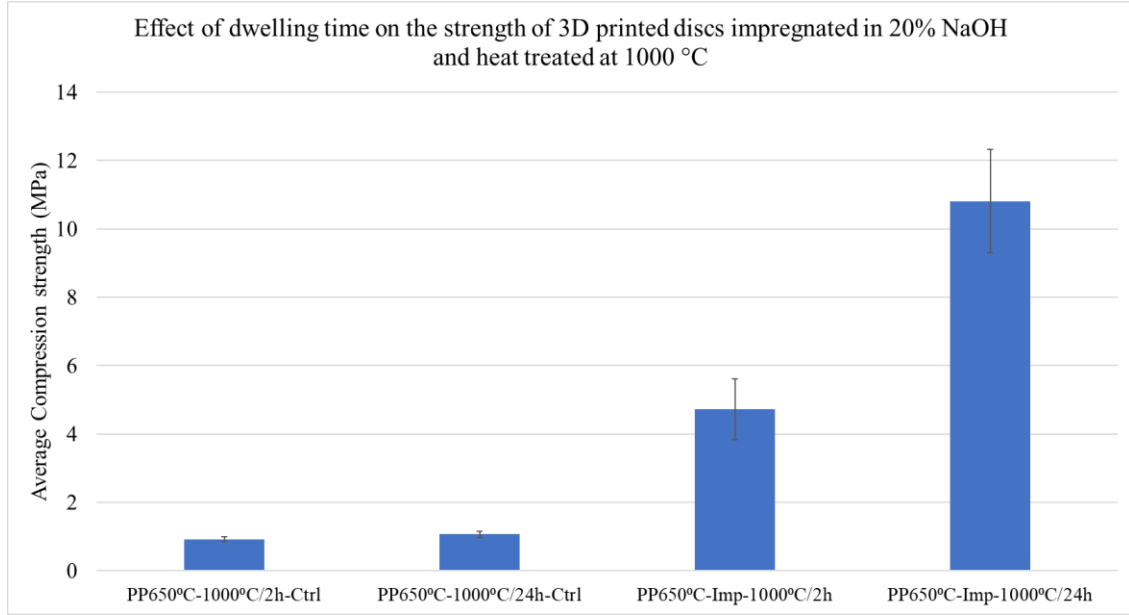


Figure 6: Average compressive strength of 3D printed nano- μ samples heat treated at 1000 °C for 2h and 24h with and without secondary surface activation.

Figure 7 and Figure 9 show the surface morphology of the fractured surface of the 3D printed SiC discs heat treated at 1000 °C for 2h and 24h. From figure 7a and 7c, complete filling of the pores by the nanowires within and around the pores can be observed. Moreover, bridging of the nanowires between the SiC particles and within the pore surface can also be observed from figure 7b and 7d. This can be attributed to the increased strength for the samples treated at 1000 °C/2h in figure 2.

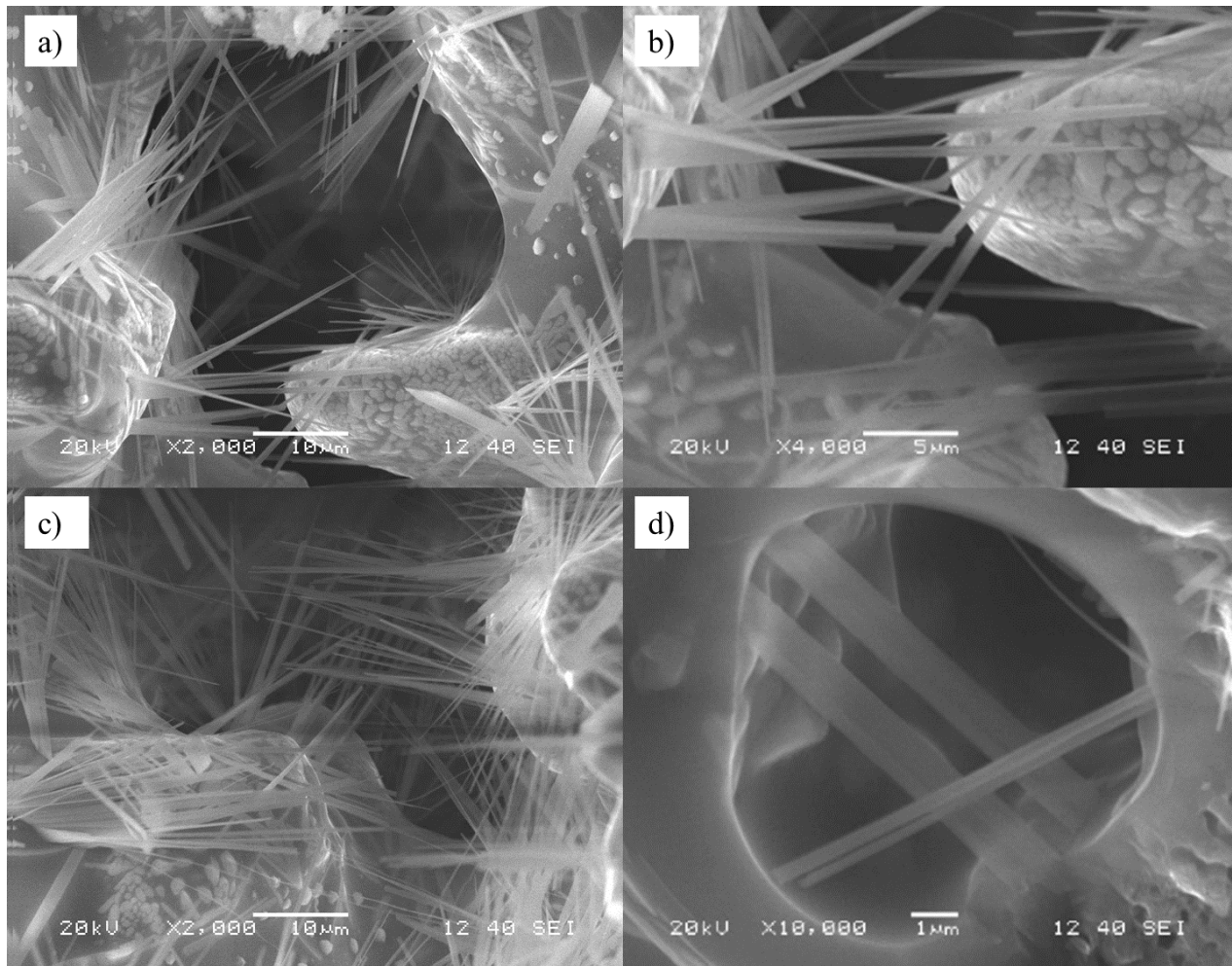


Figure 7: SEM micrographs of the fractured surface showing the growth of nanowires of 3D printed SiC discs impregnated in 20% NaOH and heat treated at 1000 °C for 2h

Figure 9 shows SEM image of the silica nanowires (platelets) growing on the surface of SiC treated at 1000 °C/2h. The surface of the SiC is fully covered with the SiO₂ crystals of different sizes. Measurements of the width of the nanowires grown on the surface of the material was 946nm which was similar to the width of the small crystals next to it that measured 905 nm (figure 8). This indicates that the nano wire growth starts from the crystals on the material surface. Figure 9 shows SEM micrograph of impregnated samples heat treated at 1000 °C/24h. Prolonged heat treatment facilitates enhanced oxygen diffusion that leads to higher formation of silica phases (figure 9a) that flows through the pores during heat treatment and forms thicker silica rock like structures (figure 9d) upon cooling.

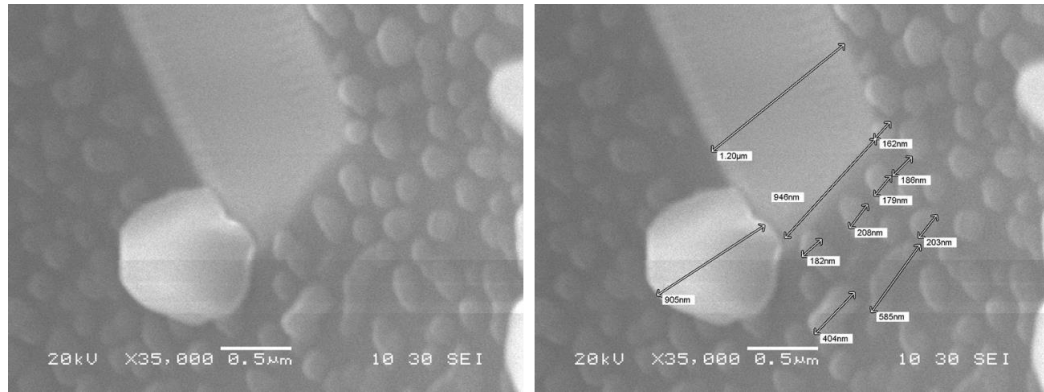


Figure 8: SEM image showing silica nanowires (platelets) grown on the fractured surface of 3D printed SiC impregnated in 20% NaOH and heat treated at 1000 °C for 2h.

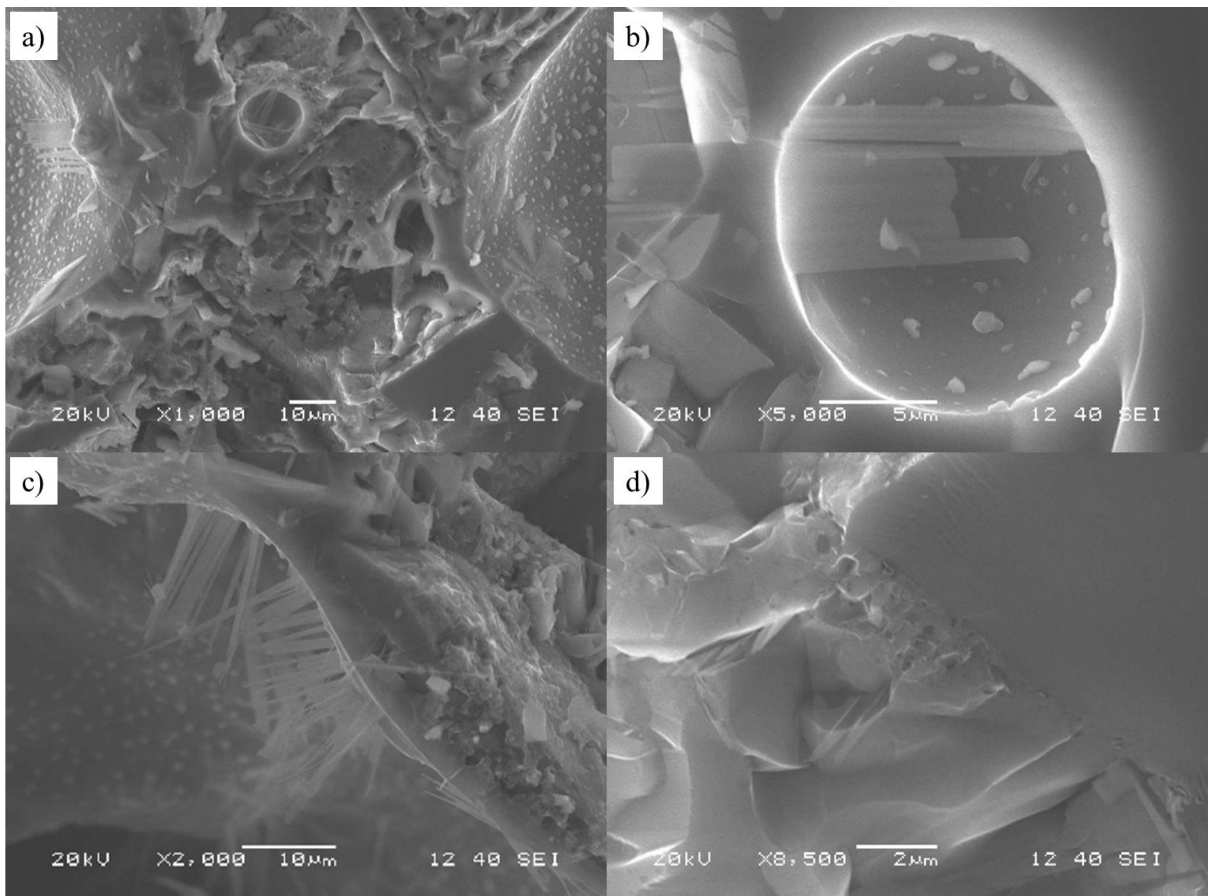


Figure 9: SEM micrographs of the fractured surface showing the growth of nanowires and platelets of 3D printed SiC discs impregnated in 20% NaOH and heat treated at 1000 °C for 24h

Task 2: Binder Selection

To improve the mechanical integrity and handling of the 3D printed objects, a second surface treatment with NaOH was applied. The effect of various NaOH concentrations, on the density and mechanical properties of the 3D printed discs was analyzed and correlated to the silica gel layer and silica nanowires growth. The density data in Figure 10 showed that the density of 3D printed samples impregnated with 10% NaOH is significantly higher than that for samples impregnated with 5% NaOH ($P < 0.01$) and with 20% NaOH ($P < 0.002$). The significant increase in the density of the samples treated with 10% NaOH compared to that treated with 5% NaOH is attributed the reduced porosity and pore size due to the growth of silica nano wires within the porous structure. On the other hand, the significant increase in the silica phase after treatment with 20% NaOH is responsible for the observed lower density of the samples due to the inherent low density of the silica phase compared to the SiC phase.

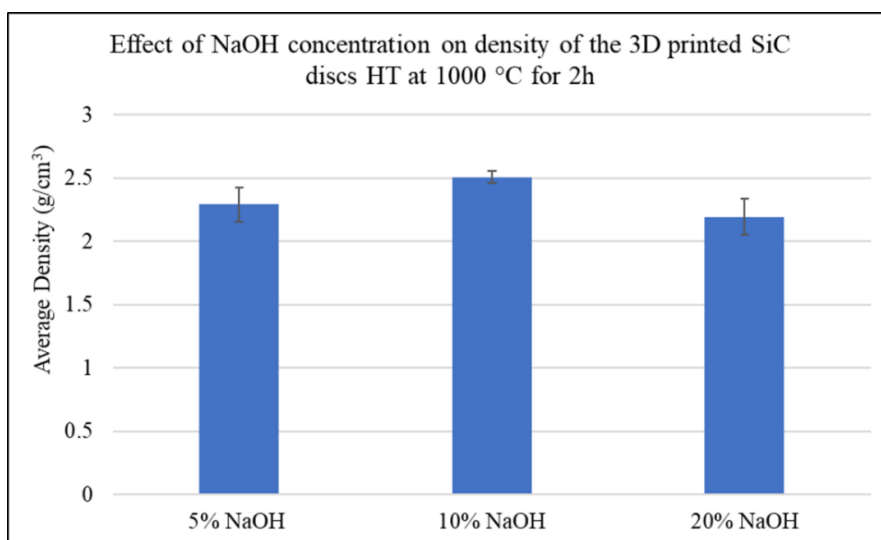


Figure 10: Density data of the 3D printed discs from nano- μ powder, impregnated at varying concentrations of NaOH and heat treated at 1000 °C for 2h.

Task 3: Powder selection

In order to, enhance the densification without compromising the homogeneity of the printed layers we chose to use 70 wt% of 40 μm SiC particles with 15 wt% of 2 μm and 15 wt% of 600 nm SiC particles. The selection of the particle sizes and the corresponding ratios were based on the size of the pores observed by SEM on the discs made of 80nm and 40 μm particles.

The average density of the samples prepared with a 15:1 ratio of 40 μm and <80nm particles treated with 20% NaOH and heat treated at 1000 °C for 2 hours was compared to samples prepared using SiC mixture made of (70 wt. % of NaOH-treated 40 μm powder mixed with 15 wt.% each of 2 μm and 600 nm SiC particles) and impregnated with 20% NaOH then heat treated at 1000 °C for 2 hours. It can be observed from figure 11 that the introduction of the nano and sub-micro particles tremendously increased the density by filling the interspace created by the coarser 40 μm particles.

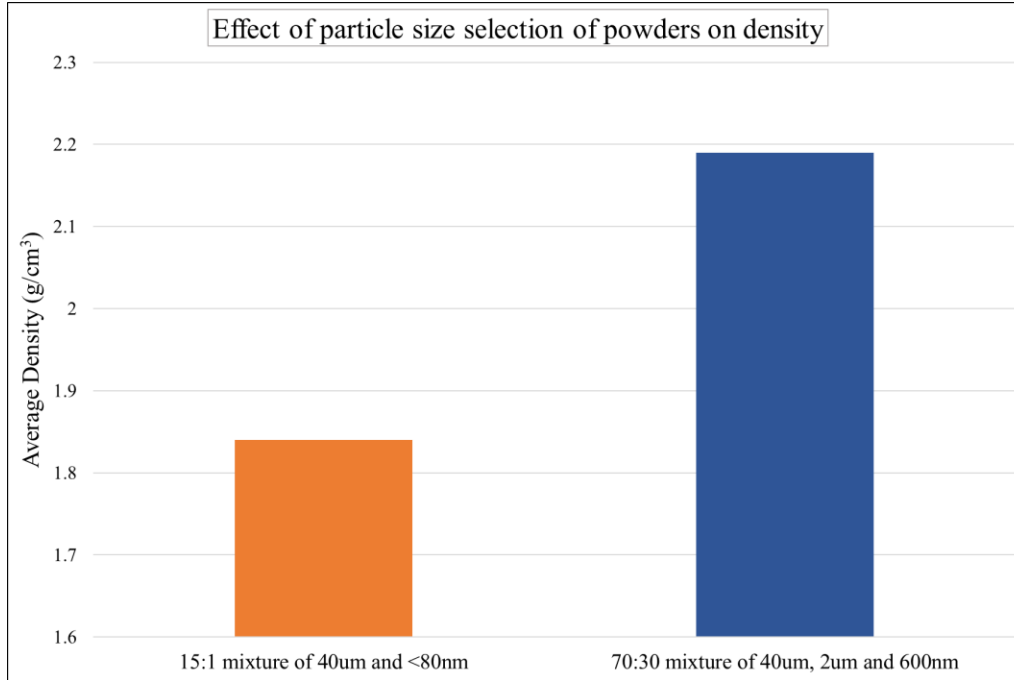


Figure 11: Comparison of average densities of discs printed with 15:1 mixture of 40µm and <80nm powder with 20% NaOH as binder and 70:30 ratio of pre-treated 40µm SiC powder with 30% mixture of 2µm and 600nm powders. Both discs are treated with 20% NaOH and heat treated at 1000 °C/2h.

Figures 12a and 12b are SEM images of SiC discs prepared manually using 40 µm SiC pre-modified with 20% NaOH and (b) using a combination of 70% 40 µm SiC pre-modified with 20% NaOH and 15% each of SiC particles 2µm and 600 nm. The porosity percentage of discs prepared from 40 µm SiC pre-modified with 20% NaOH was 10.54 ± 1.66 % which is significantly higher ($P < 1.48E-9$) than that 5.91 ± 1.29 % of samples prepared from SiC mixture of various particle sizes (Fig.12). The porosity percent was calculated using Image analysis.

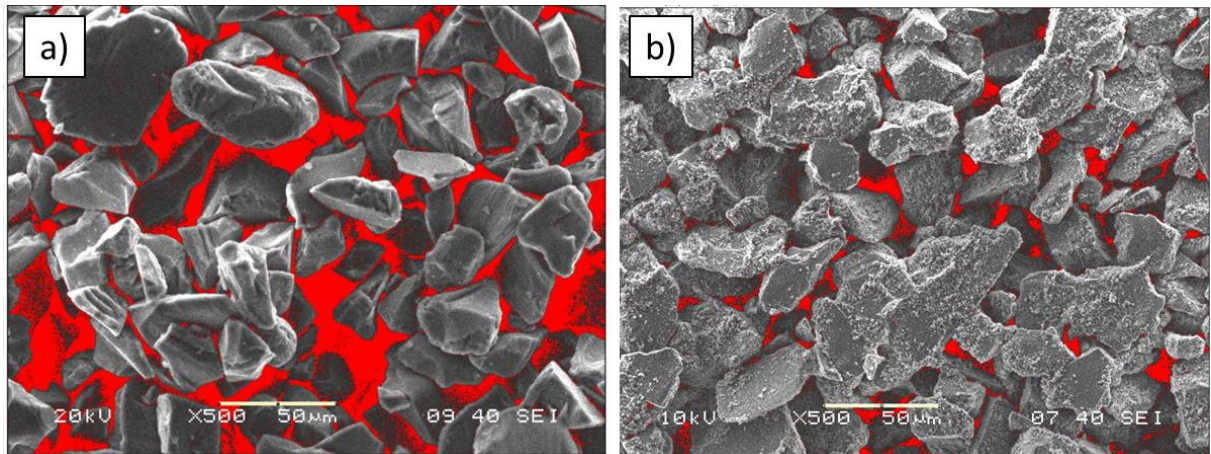


Figure 12: Porosity analysis in ImageJ using SEM micrographs of hand pressed discs at room temperature using a) pre-treated SiC 40µm powders and b) 30% nano-µ SiC powders combined with 70% pre-treated 40µm particles of SiC.

Furthermore, 3D printing of 40 μm SiC particles pretreated with 20% NaOH resulted in poor structural integrity of the printed discs in a ZCorp 510 (Fig. 13a). Therefore, these samples were excluded from further analysis. Figure 13b shows the discs 3D printed using a combination of 70% 40 μm SiC pre-modified with 20% NaOH and 15% each of SiC particles 2 μm and 600 nm in ProJet460Plus. The printed discs showed better structural integrity and a smooth surface finish than the once from ZCorp 510 printer.

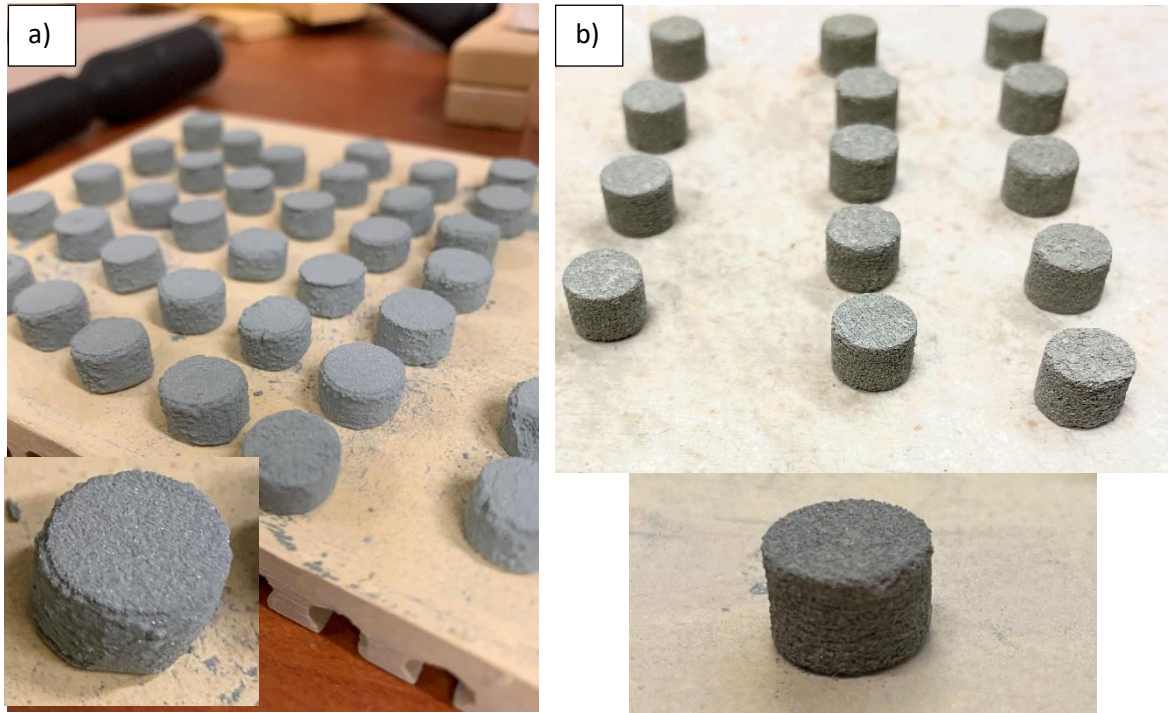


Figure 13: Digital images of 3D printed discs printed on a) Z-Corp510 with 40 μm SiC particles and b) nano-micro combination of particles printed in ProJet460Plus. The insets a) and b) shows the

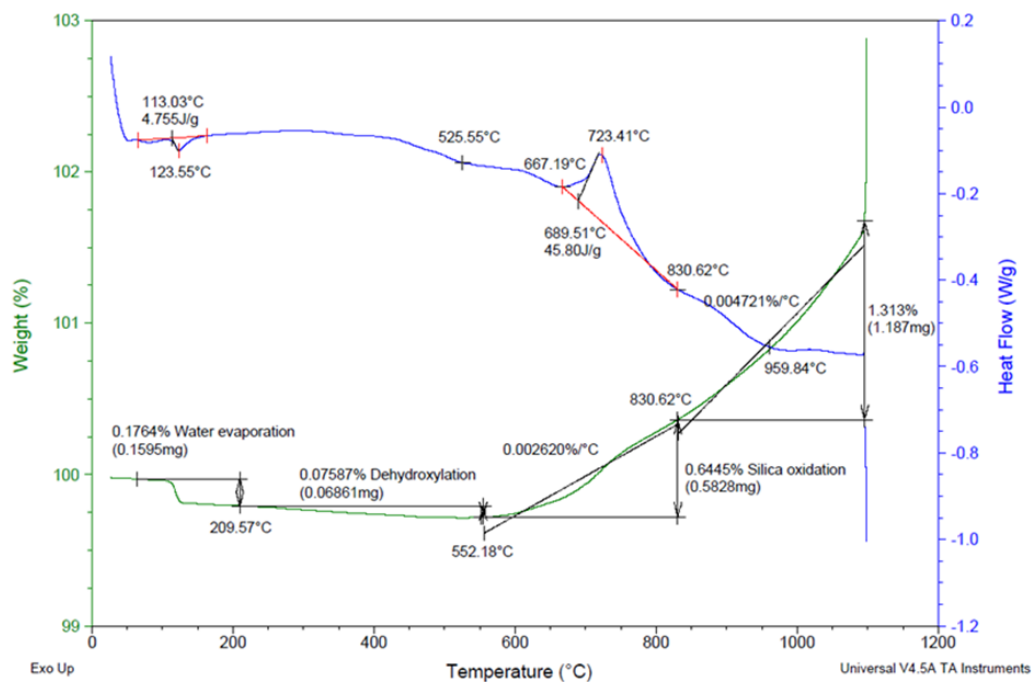
Task 4: Sintering profile selection

Experiments have been completed to test the dependence of SiC sample microstructure and mechanical properties on the thermal treatment parameters: *Temperature, time, rate of heating, and heating environment*

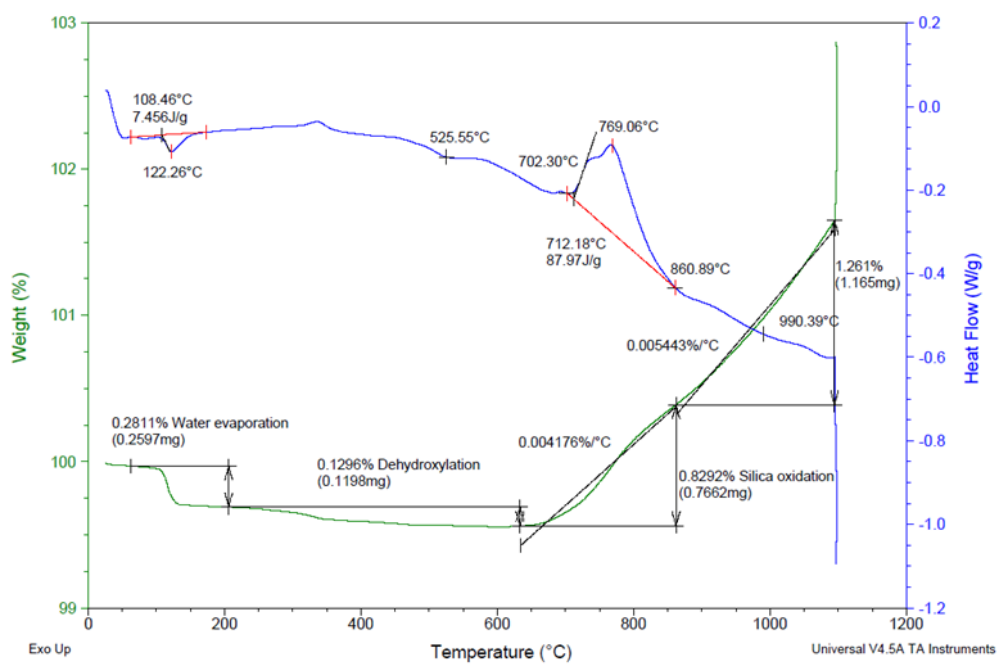
4.1. Differential Scanning Calorimetry (DSC) and Thermogravimetric Analysis (TGA)

Thermogravimetric (TG) and differential scanning calorimetry (DSC) analyses (Fig. 14) were performed on SiC discs (5 mm diameter x 4 mm length) pretreated with 5 %, 10 % and, 20 % NaOH, to determine the nucleation and phase transformation temperatures of the silica phase on the SiC surface. Samples were heated in air environment at a heating rate of 10 $^{\circ}\text{C}/\text{min}$ over the range from room temperature to 1100 $^{\circ}\text{C}$ and maintained at 1100 $^{\circ}\text{C}$ for 30 mins. The samples were cooled to room temperature and analyzed by SEM.

(a)



(b)



(c)

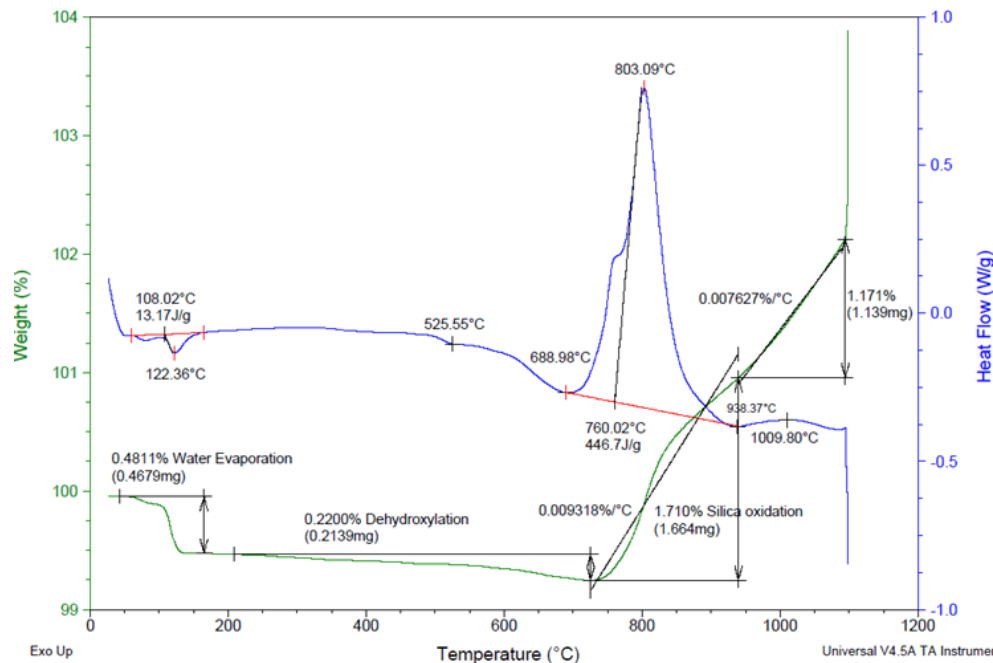


Fig. 14: DSC-TG analysis of 40 μm SiC mixed with (a) 5 %, (b) 10 % and (c) 20 % NaOH

Differential scanning calorimetry shows an endothermic peak at around 122 $^{\circ}\text{C}$ indicating the water desorption in the temperature range 75 – 180 $^{\circ}\text{C}$. In association with the desorption, TG analysis shows 0.1764 %, 0.2811 %, and 0.4811 % weight loss respectively for samples pretreated with 5 %, 10 % and 20 % NaOH. This is similar to previous studies that reported desorption of water from amorphous silica in the temperature range 100 – 200 $^{\circ}\text{C}$ [1, 2]. Water desorption amount increases with the increase in the NaOH concentration due to the increased formation of silica gel layer. This has been confirmed by FTIR analysis that showed a significant increase in the area under the peak for the Si-OH and the Si-O-Si (Fig. 15 and 16).

4.1.1. Evidence of the formation of Silica gel layer after treatment with NaOH:

SiC scaffolds were prepared using four different concentrations of sodium hydroxide (NaOH). SiC powder (Beta 3C, US Research Nanomaterials, Houston, TX), in the size range of 1–40 μm , was mixed with polyethylene glycol (PEG) in a ratio of 60/40. A volume of 60 μl of 5, 10, 15, 20, or 40% (W/V) NaOH was mixed with 1,000 mg of the SiC/PEG powder mixture. The particles (1,000 mg) were loaded into a 10-mm diameter tungsten carbide die, pressed at 50 MPa, and left to dry at room temperature overnight. The scaffolds were subjected to a heat treatment protocol at 350 $^{\circ}\text{C}$ for 24 hr to burn off the sacrificial PEG polymer. The temperature was then raised up to 900 $^{\circ}\text{C}$ for 2 hr. This was done using a controlled rate of heating and cooling of 1 $^{\circ}\text{C}/\text{min}$.

The surface chemistry of the scaffold was analyzed by Fourier transform infrared spectroscopy (FTIR; Nicolet 6700 Thermo-Nicolet, Madison, WI) under diffuse reflectance mode. Spectra was collected after 200 scans at 4 cm^{-1} resolution. The area under the peak for various functional groups was measured using the built in the Omnic software 7.0.

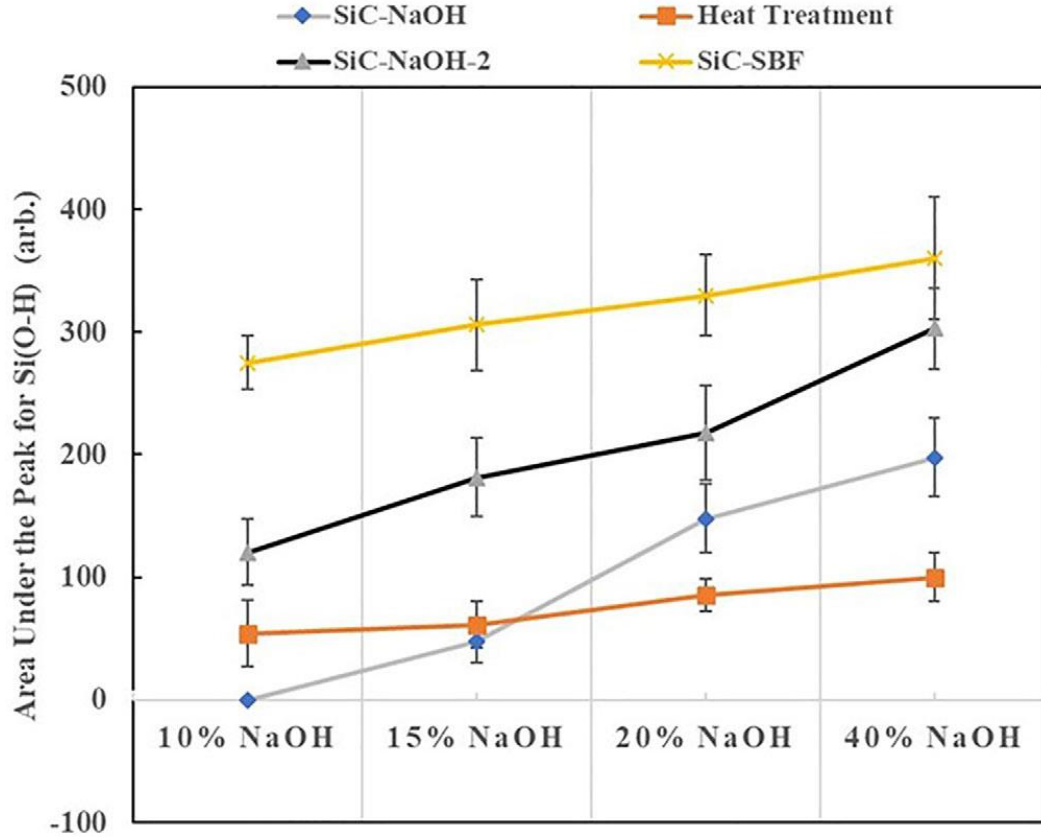


Figure 15: A comparison of the area under the peak for the Si—OH stretching band ($2,800\text{--}3,700\text{ cm}^{-1}$) for SiC samples treated with various NaOH concentrations, heated at $900\text{ }^{\circ}\text{C}/2\text{ hr}$, immersed in NaOH/10 hr, and incubated in SBF for 48 hr. As expected, the area under the peak increased as the NaOH concentration used to treat the surface of SiC increased. The area under the peak decreased after thermal treatment due to evaporation of water, however a significant increase is observed after immersion in NaOH for 10 hr (SiC—NaOH-2) as well as after immersion in SBF.

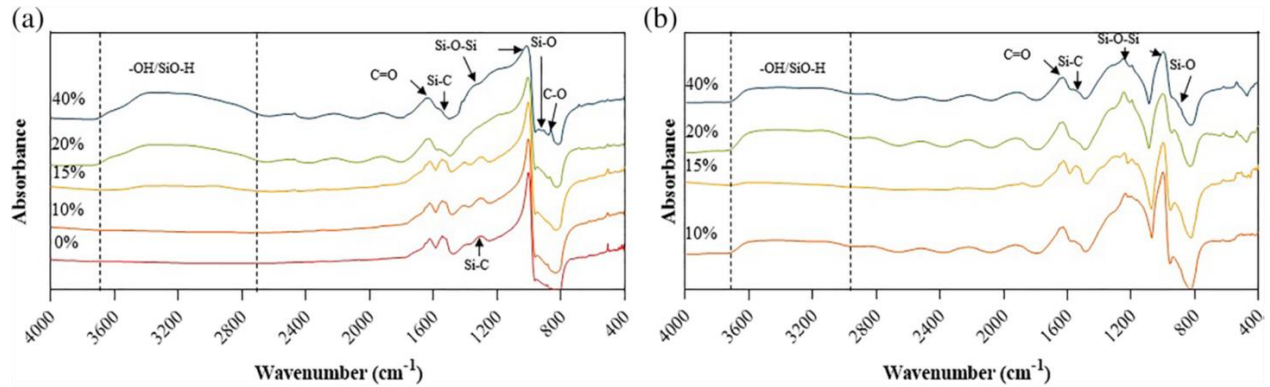
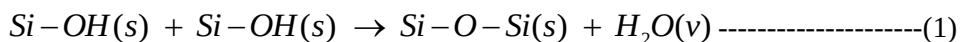


Figure 16: (a) FTIR analysis of the SiC powder prior to and after surface treatment with various concentrations of NaOH. Upon addition of NaOH, a broad OH band appears in the wave number range $2,800\text{--}3,700\text{ cm}^{-1}$ due to hydrated silanol groups present on the material surface. The peaks centered at $1,530$ and $1,300\text{ cm}^{-1}$ show the Si—C stretching vibration. (b) FTIR spectra of the SiC scaffolds subjected to heat treatment at 900°C for 2 hr. The intensity of the Si—C band centered at $1,530\text{ cm}^{-1}$ decreased in all spectra. The bands at 980 and $1,200\text{ cm}^{-1}$ are due to Si—O—Si vibration.

Following desorption, the silica gel layer undergoes condensation of the hydroxyl groups that leads to elimination of water molecules and formation of Si-O-Si siloxane bridges according to the eq (1),



The 0.219 % weight loss in the temperature range 200 – 725 °C is attributed to the dehydroxylation process of silica gel as well as the release of SiO and Si (OH)₄ molecules. Previous studies using FTIR analyses confirmed the release of silicon oxide and silanol groups from the silica gel layer on the surface of SiC [3, 4] (Fig. 17). Dehydroxylation of pure silica in the temperature range from 200 – 700 °C was reported in the literature with associated weight losses depending on the surface area of the material [5, 6].

4.1.2. Evidence for the release of SiO gas from the surface modified SiC:

SiC powder was mixed with 20% NaOH and placed in a beaker. The beaker was covered with Aluminum foil sheet that was placed 1 inch over the powder. The beaker was placed on a hot plate and heated at a rate 50 °C/min up to 500 °C for 25 min then cooled down. The surface of the aluminum foil facing the SiC powder was analyzed by Fourier transform infrared spectroscopy (FTIR; Nicolet 6700 Thermo-Nicolet, Madison, WI) in the diffuse reflectance mode (DRIFT). Spectra were collected after 200 scans at 4 cm⁻¹ resolution. In addition, the outer and fracture surface chemistry of the SiC discs were analyzed after the mechanical test.

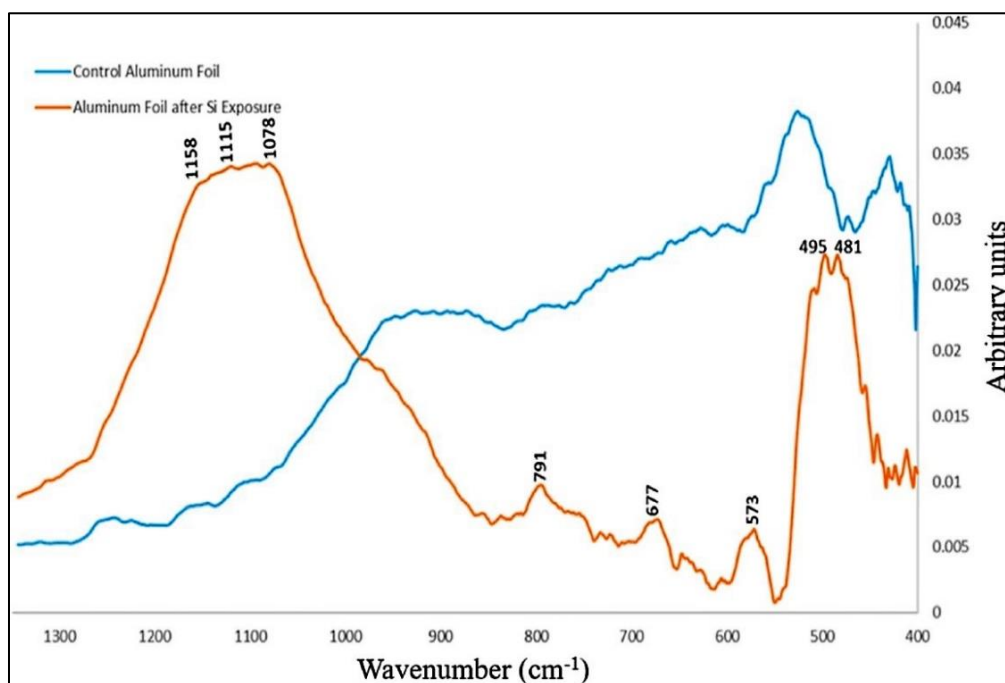


Figure 17: FTIR analysis of the aluminum foil placed at a distance for the NaOH-treated SiC powder heated at 500 °C. The bands in the wave number range 980–1230 cm⁻¹ is for the Si–O–Si stretching, the shoulder at 950 cm⁻¹ is due to the Si–OH vibration, the bands in the wavenumber range 450–550 cm⁻¹ are the Si–O rocking, at 791cm⁻¹ due to the Si–O bending.

The DSC curves for all samples treated with various concentrations of NaOH showed an endothermic peak at 525.5 °C indicating the nucleation of the silica crystals. Previous studies showed that the T_g of sodium silicate glass that has 1:2 Na:Si ratio was 440 °C [7]. In our study, the ratio of Na:Si is much lower than 1:2 and hence the nucleation temperature is expected to be high (figure 18).

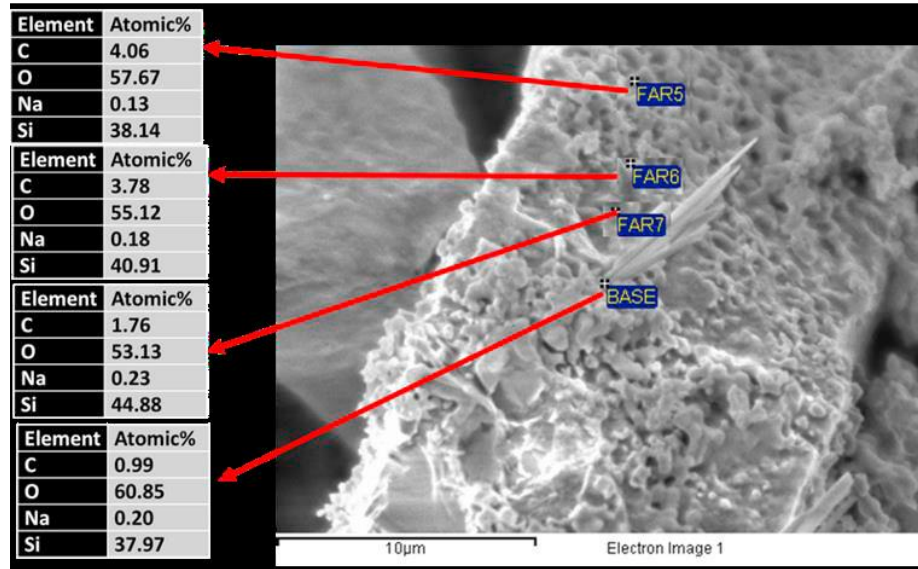
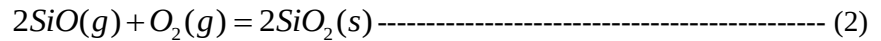


Fig. 18: SEM fracture surface analysis of the SiC disc showing the EDX elemental composition at various distances from the base of a silica nanowire.

DSC analysis showed a major exothermic peak due to crystallization in the temperature range from 667 - 831 °C, 702 - 861 °C, and 689 - 938 °C for samples pretreated with 5 %, 10 % and 20 % NaOH. Measurement of area under the peaks showed an increase in the ΔH as the NaOH concentration increased from 5 % to 20 %. The ΔH for samples pretreated with 5%, 10% and 20% where 45.8 J/g, 87.97 J/g and 446.7 J/g, respectively. The increase in ΔH correlates with the significant weight gain observed by TGA due to nanowire formation by oxidation reaction according to equation (2)



Indeed, SEM analysis confirmed the DSC analysis and showed significant increase in the nanowire density for samples pretreated with 20% NaOH compared to 5 % and 10 % NaOH samples heated at 1100 °C (Fig. 19).

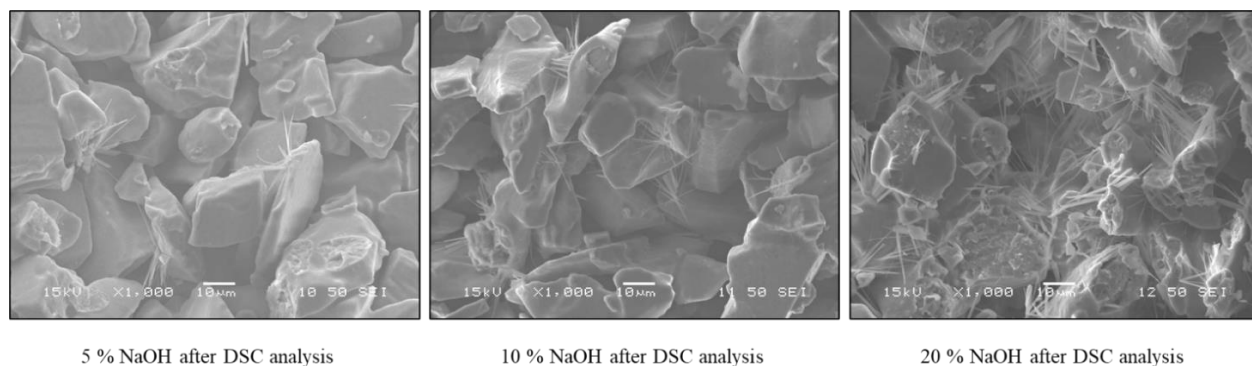


Fig. 19: SEM analysis of DSC-TGA samples pretreated with 5 %, 10 % and, 20 % NaOH

Quantitative image analysis was done using SEM images for studying the influence of NaOH concentration on nanowire growth and number density. Nanowire number density of the fracture surface was analyzed by using ImageJ software showed statistically significant higher density of nanowires in the samples pretreated with 20% NaOH compared to other samples (Fig. 20). For the quantitative analysis of number of nanowires per surface area and nanowire diameter, broken nanowire that does not contacted to any surface were excluded, only nanowire that is connected to the surface were considered for diameter and nanowire number density measurements. The nanowire number density ($1/\text{mm}^2$) as well as the width of the nanowires increased according to the concentration of the NaOH used to pretreat SiC in the order 20 % > 10 % > 5 % (Fig. 20 & 21).

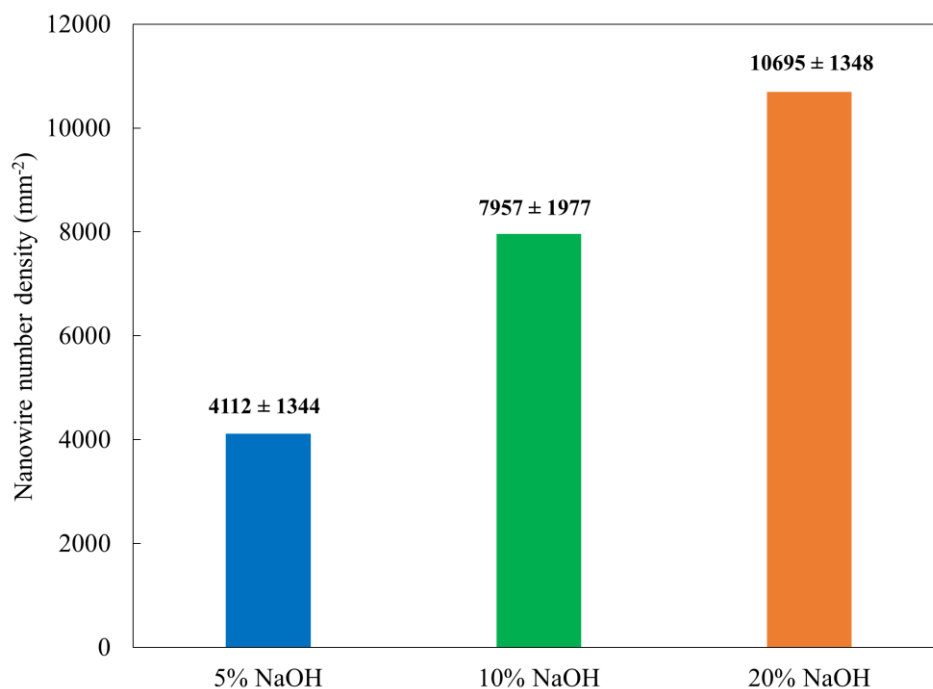


Fig. 20: Nanowires average number density (mm^{-2}) in samples pretreated with 5 %, 10 % and, 20 % NaOH

The increased number of silica nanowire crystals in the 20% NaOH is attributed to high number of crystal nuclei formation in the sample (Fig. 21). The nucleation process is enhanced by the presence of Na ions as well as the hydroxyl groups present in the silica gel layer.

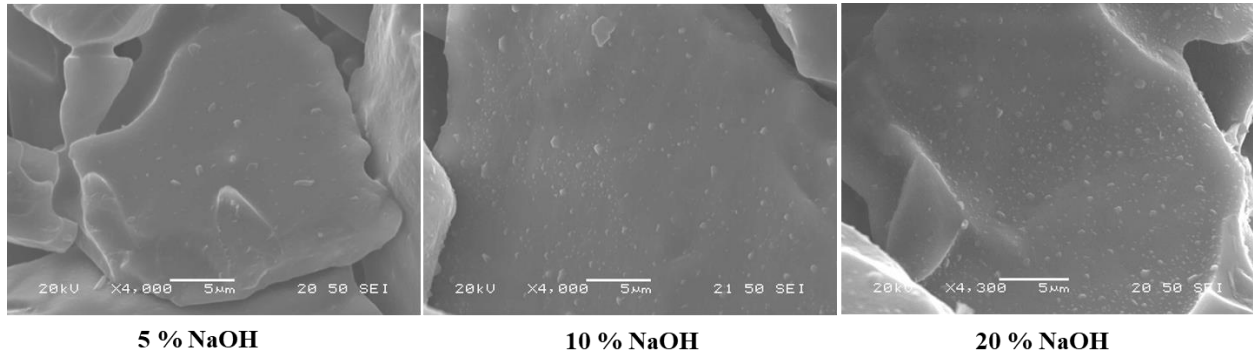


Fig. 21: SEM analysis of samples pretreated with 5 %, 10 % and, 20 % NaOH sintered at 725 °C/15 mins

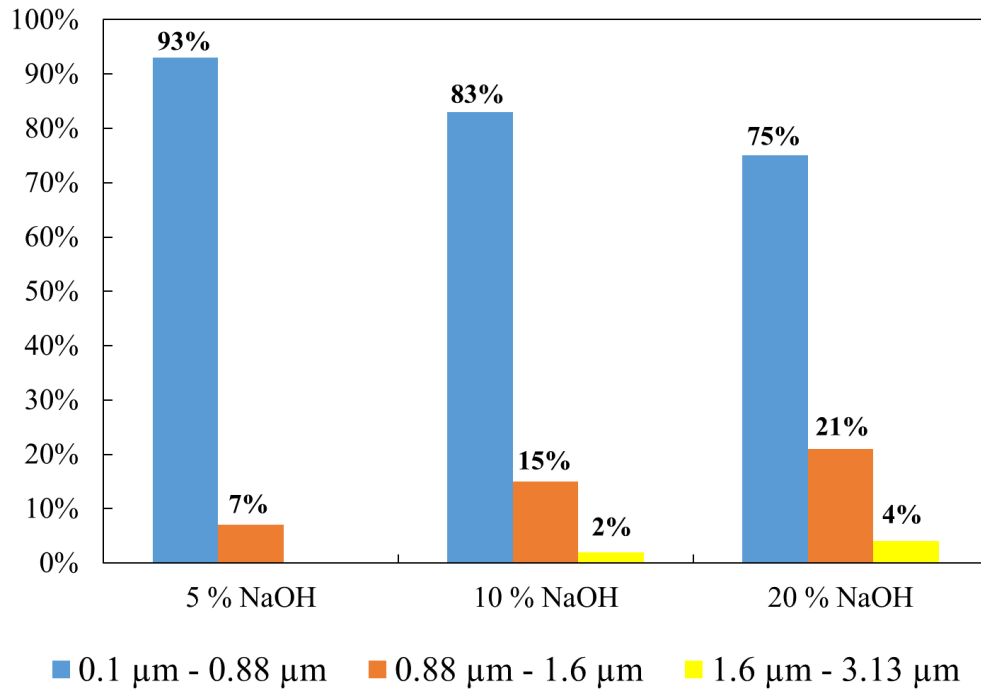


Fig. 22: distribution percentage of the diameter of nanowires present in porous SiC discs pretreated with 5 %, 10 % and 20 % NaOH

Figure 22. shows that the width of the nanowires increased according to the concentration of the NaOH used to pretreat SiC in the order 20 % > 10 % > 5 %. The increase in the nanowire's diameter could be attributed to the increase amount in SiO vapor released from the silica gel layer and participate in the growth of nanowires. The sample treated with 20% or 10% showed fusion of multiple nanowires to make wide plates near the surface of the material. As the wide plates grow some of them were very symmetric and many others would separate into individual bands. In

addition to the fused nanowires, a population of nanowires are remained without fusion. In conjunction with the differences in the nanowires dimensions and populations TG analysis showed significant increase in the weight gain. The rate of weight gain for all samples take place in two steps. The initial weight gain step took place in the temperature range 552-831 °C, 636-861 °C and 726-940 °C for sample pretreated with 5%, 10% and 20% NaOH, respectively. The rate of weight gain in the first step increased with the increase in the concentration of NaOH in the order of 20.8 ug/min, 34.1 ug/min, and 77.76 ug/min for 5%, 10% and 20% NaOH, respectively. The second weight gain step took place in the temperature range 831-1100 °C, 861-1100 °C and 940-1100 °C for sample pretreated with 5%, 10% and 20% NaOH, respectively. Similar trend in the rate of weight gain was also observed in the second step weight gain. The rate of weight gain in the second step increased with the increase in the concentration of NaOH in the order of 44.13 ug/min, 48.75 ug/min, and 73.2 ug/min for 5%, 10% and 20% NaOH, respectively. For the 20% NaOH constant rate in the two steps weight gain observed due to presence of thick silica gel that is deprived of oxygen. For the 5 % and 10% the weight gain increased in the second step compared to the first step due to the passive oxidation. The initial high rate of weight gains is due to the oxygen diffusion into the oxygen deficient silica layer. As more oxygen diffusion takes place concentration gradient decrease and the rate of diffusion decreases.

4.2. Silica nanowire growth mechanism and growth modeling

Fig. 23 shows the silica nanowire growth mechanism of nanowire formed inside the pore of SiC discs (5 mm diameter x 4 mm length) pretreated with 20 % NaOH. Silica gel coated samples were heated at 525 °C/ 15 mins and SEM analysis showed the formation of droplets or nuclei. At the early stage of droplet formation, structure is amorphous silica rich in hydroxyl (OH^-) ions on the surface. As the heat treatment continues, up to 700 °C these droplets go through dehydroxylation reaction and form dangling bonds. Silica droplets become active due to presence of these dangling Si-OH bonds as well as oxygen deficiency in the subsurface of the silica layer. Motivated by the oxygen concentration gradient the droplets serve as active sites for oxygen diffusion and silica nanowire growth.

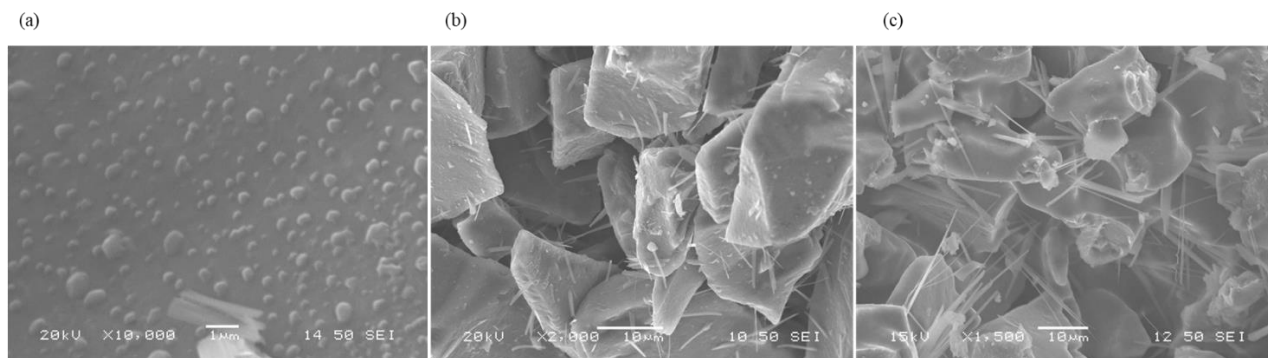


Fig. 23: SEM images of (a) silica nuclei on the surface of SiC particles formed at 525 °C, (b) small silica nanowires formation at 800 °C, (c) nanowires grew into bigger nanowires at 1100 °C

SEM image in Fig. 24 shows the growth of the nanowire along the grain boundaries and also the formation of silica crystals. This implies that the presence of defects on the SiC surface makes a more active droplet that serve as a high energy site for nanowire formation.

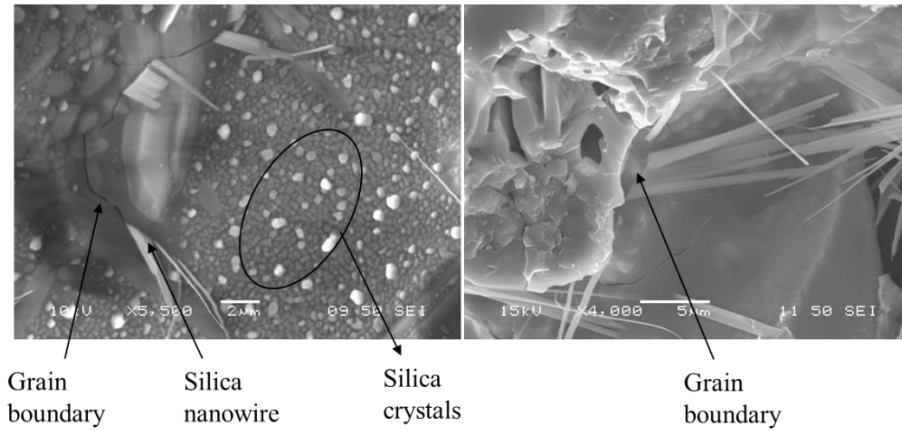
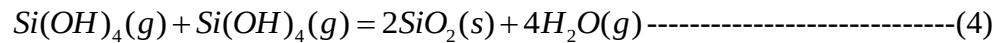


Fig. 24: Plenty of nucleation droplets and surface defects on the left and the Silica nanowire formation along the defect on the right

The above analyses suggest that the accumulation of SiO and $\text{Si}(\text{OH})_4$ vapor inside the pores of the SiC discs enhances the formation of SiO_2 molecules inside the pores of SiC discs according to the equations (1) and (4).



The SiO_2 molecules preferentially deposits on the active silica droplets to form the Si-O-Si long chains responsible for nanowire growth. As the nanowire starts growing the outer most surface layer on the growing wire is the active site because the Si-O moiety is in contact with air. The side Si-O-Si moiety of the nanowire are already bonded with maximum nearest neighbor so they have less motivation to react with vapor molecules inside the pore.

As such, the growth of the silica nanowire takes place through a vapor-solid (VS) growth mechanism. A schematic of this growth mechanism is shown in fig. 25 where the main growth process is the deposition of SiO_2 molecules.

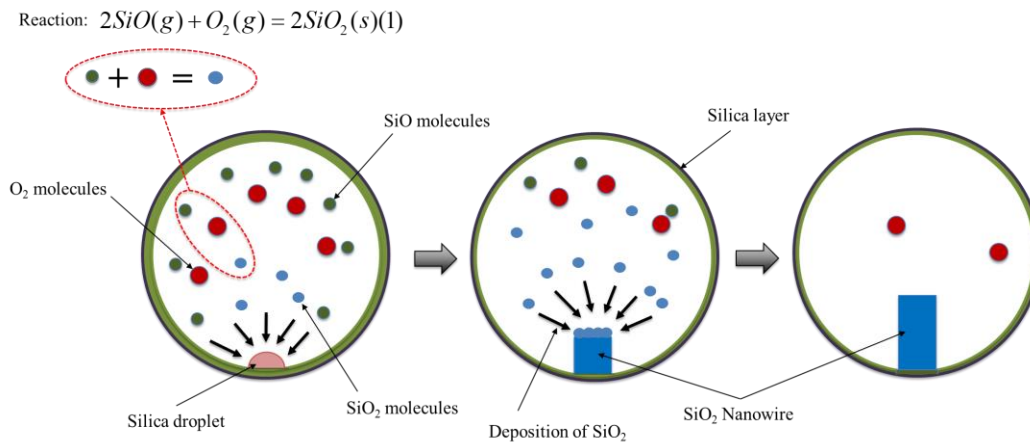


Fig. 25: Scheme of the Silica (SiO_2) nanowire formation by VS mechanism inside the pore of SiC discs

In this model, nanowire diameter, D is same as silica droplet size and assumed to be remained same whereas nanowire length is changing due to deposition of SiO_2 molecules. We focus our attention on the first kinetic reaction equation (1) happening inside the pore of the SiC disc.

Reaction rate is proportional to a power of the concentrations of reactants according to the law of mass action [8]. Here the reaction is second order in terms of SiO and first order in terms of O_2 . System of ordinary differential equations (ODEs) is shown below, which governs the change of concentration over time for the reactants i.e., SiO and O_2 and product SiO_2 .

$$\frac{1}{2} \frac{dC_{\text{SiO}}}{dt} = -KC_{\text{SiO}}^2 C_{\text{O}_2} \quad (3)$$

$$\frac{dC_{\text{O}_2}}{dt} = -KC_{\text{SiO}}^2 C_{\text{O}_2} \quad (4)$$

$$\frac{1}{2} \frac{dC_{\text{SiO}_2}}{dt} = KC_{\text{SiO}}^2 C_{\text{O}_2} \quad (5)$$

The reaction rates are highly dependent on temperature and can be shown using an Arrhenius term of the form

$$K = A \exp\left(-\frac{E_a}{RT}\right) \quad (6)$$

Here A is a pre-exponential constant, E_a is the activation energy of the reaction, T is the sintering temperature and $R = 8.314 \text{ J.mol}^{-1}\text{K}^{-1}$ is the universal gas constant.

The deposition rate of the SiO_2 molecules depends on the concentration of SiO_2 molecules and temperature according to Hertz-Knudsen equation [9],

$$Z = C_{\text{SiO}_2} \sqrt{\frac{k_b T}{2\pi m}} \quad (7)$$

Here C_{SiO_2} is the SiO_2 molecules concentration obtained by solving ODEs (3, 4, 5), k_b is the Boltzmann constant, and m is the mass of SiO_2 molecule.

Silica nanowire vertical growth rate can be measured as changing its volume during the introduction of the SiO_2 molecules on the top surface of the nanowire.

$$\frac{dL}{dt} = Z\Omega \quad (8)$$

Forward Euler method was used for solving the system of ODEs (3, 4, 5). Fig. 26 shows that the nanowire length is increasing according to the NaOH concentration in the order $20\% > 10\% > 5\%$. Therefore, the current growth model exhibits the similar trend found through the experimental data for the influence of NaOH concentration on silica nanowire growth.

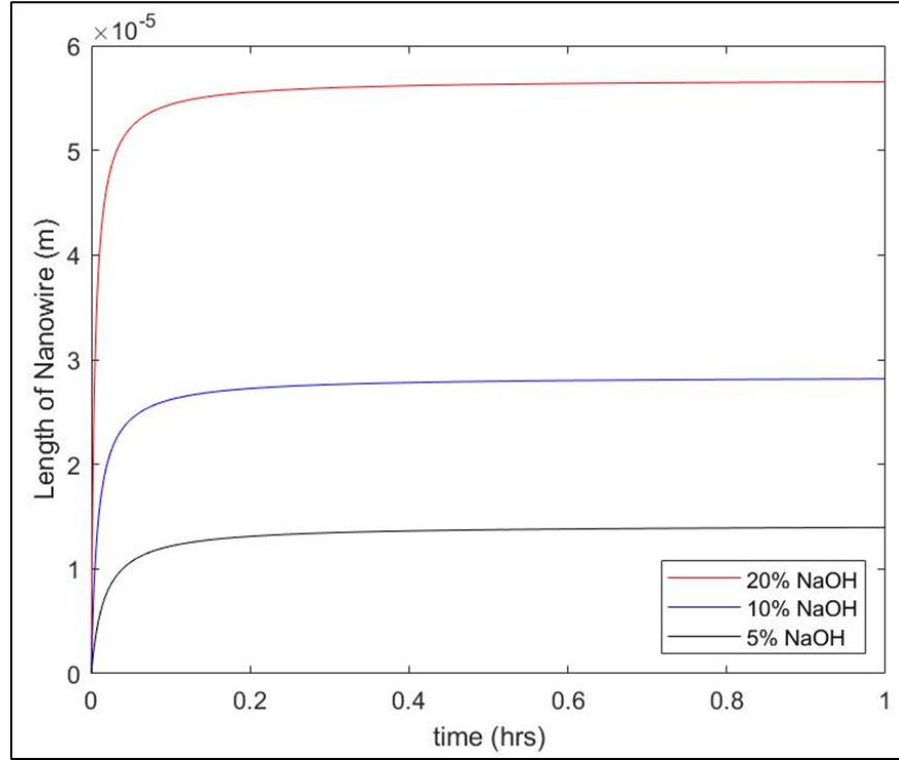


Fig. 26: Variation of nanowire lengths with respect to time for different NaOH concentrations

The nucleation time that produces the highest density and sealing of grain boundaries will be selected. The *dwelling time at the crystal growth temperature* will then be varied to determine the optimal time for crystal growth.

4.3. Effect of nucleation time on the crystal growth

3D printed samples were heat treated in two step procedures for nucleation and crystal growth. After various thermal treatment protocols, the density, linear shrinkage, porosity, grain boundary sealing, and mechanical properties were measured.

The 3D SiC discs (10 mm dia x 7 mm) were printed on a ProJet 460 Plus printer and subjected to heat treatment at 650°C/ 5h to enhance mechanical integrity. Air pressure was used to remove loose particles attached to the discs. To test the effect of nucleation time on the density and mechanical strength, the 3D printed SiC discs were impregnated with 10% NaOH/10 min then thermally treated in two steps; 550°C/1h, 6h, 12, 18 or 24 h followed by a second heat treatment at 900°C/4h.

The most pronounced effect of the nucleation time is the significant increase in mechanical strength as the nucleation time increased from 1-12 hrs. The samples treated for 1 hour, 6 hours and 12 hours had a strength of 1.646 MPa, 2.710 MPa, 6.855 MPa respectively (Fig. 27). The samples treated at 550 C for 18 or 24 hours showed lower mechanical strength than that of the samples treated for 12 hours at the same temperature. The **decrease in mechanical** strength is due to the inhibition of the growth of the nanowires. Indeed, fracture surface analysis of samples

showed extensive growth of nanowires within the pores and microcracks after treatment at 550 °C for 12h and 900 °C for 4h (Fig. 28). In conjunction with the growth of nanowires the density and strength were significantly increased. When the nucleation time was further increased to 24h, minimal growth of nanowires was observed most probably due to increased oxygen diffusion in the silica layer prior to heat treatment at 900 °C. The growth of silica nano wires is motivated by the oxygen concentration gradient. Therefore, the extensive oxygen diffusion during 24 hours treatment at 550 °C has the potential to reduce the concentration gradient and the driving force for the growth of nanowires.

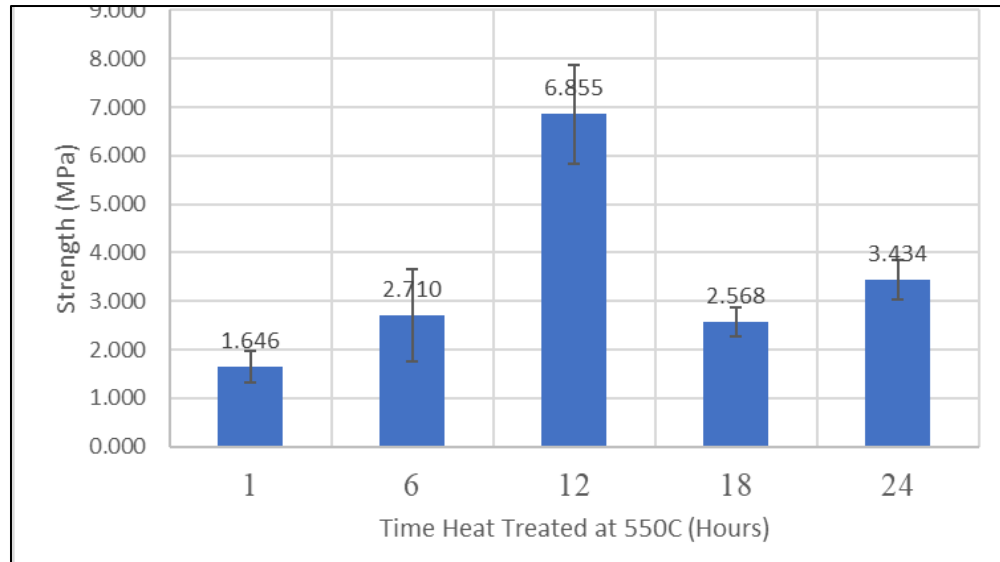


Figure 27: Mechanical Strength of 3D printed SiC samples treated at 550°C/1h, 6h, 12 hours, and 24h followed by 900C/4h after impregnation in 10% sodium hydroxide concentration.

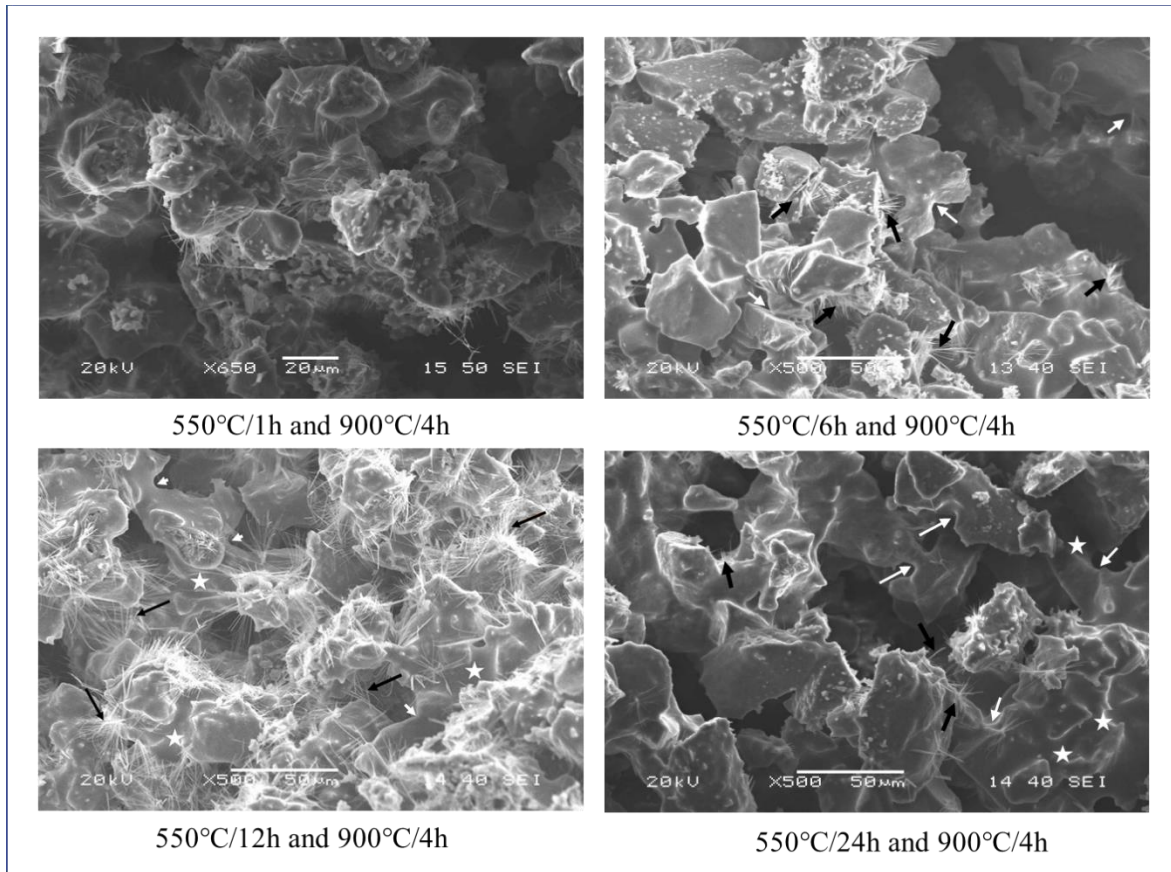


Figure 28: SEM morphology of fractured surface of 3D printed discs treated at different nucleation times then at 900 °C/4h. The black arrows point to the nanowires growing on the surface of the pores.

Due to the significant increase in the amount of wires grown in samples heat treated at 550°C for 12 hours, more analysis was done on samples subjected to one step heat treatment at 550°C for 12, 6 and 1 hour in order to see the impact of nucleation time without sintering at 900°C.

Quantitative analysis of the nano wires density was performed using SEM and image analysis program. The average number of nanowires per $100 \mu\text{m}^2$ was 29, 40, and 112 for the samples treated at 550 °C for 1, 6, and 12 hours prior to heat treatment at 900 °C/4hrs (Fig. 29). Occasional fusion of the nanowires was observed for the samples treated at 500 °C 12 hrs + 900 °C/4hrs. Fusion of nanowires can be attributed to the high density of nuclei. Therefore, the high mechanical strength of the 3D printed SiC samples after treatment at 550 °C/12 hrs+900 °C/4h can be attributed to the significant increase in the density of nano wires per unit surface area of pore walls.

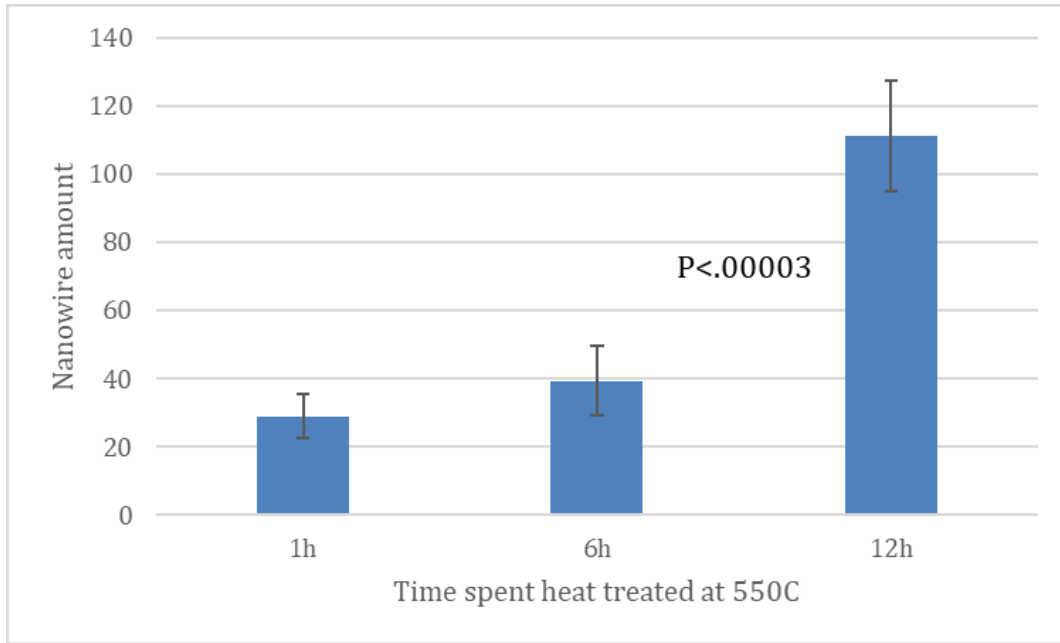


Figure 29: Quantitative analysis of nanowire amount in samples heat treat for different time periods at 550°C. It was seen that the average number of nanowires in a given area of 100 μm^2 was 29 for the sample treated for 1 hour, 40 for the sample treated for 6 hours, and 112 for the sample treated for 12 hours.

The samples impregnated with 10% NaOH and heat treated at 550°C different time periods prior to heat treatment at 900 °C/4h showed comparable density (figure 30). The density of these composites are controlled by many factors: (i) the formation of the silica gel layer, (ii) The oxygen diffusion during the growth of nano wires and (iii) the initial porosity characteristics of the 3D printed samples. The silica phase (2.6 g/cc) has lower density than the SiC (3.2 g/cc). Therefore, formation of silica would reduce the density of the samples. On the other hand, as the samples oxidize during silica nano wires growth, the density is increased due to the weight gain following oxygen diffusion. The growth of nano wires also reduces the porosity of the material. The complex contribution of each factor on the density of the material have resulted in comparable density among samples. However, the mechanical strength showed a significant difference as discussed above.

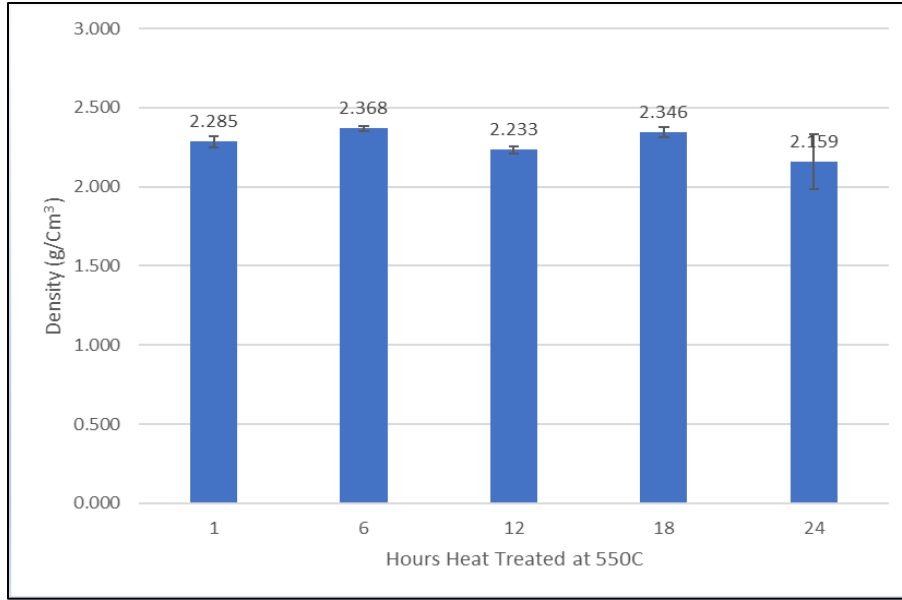


Figure 30: Density of 3D printed SiC samples treated at 550C/1h, 6h, 12 hours, 18h, and 24h followed by 900°C/4h after impregnation in 10% sodium hydroxide concentration

4.4. Dimensional stability after post processing of the 3D printed samples

After heat treatment of the samples, the face of the sample that was not in contact with the alumina plate was found to have small amounts of deformation around the edges of the top face. This shrinkage was found to increase with the concentration of sodium hydroxide (figure 31). Therefore, we tested the effect of NaOH concentration on the dimensional stability.

The samples impregnated with 1% sodium hydroxide and heat treated for 1 hour at 550°C showed deformation of 0.67% (n=3). The 10% samples heat treated at 550°C/1h showed a deformation of 35.81% (n=3). At 550°C/6h, the deformation increased for the 1% sodium hydroxide concentration to 13.64% (n=3). This increase in deformation is a statistically significant change with a p value of 0.013 compared to that of the deformation that occurred at 500°C/1h. The sample impregnated with 10% sodium hydroxide showed a statistically insignificant drop to 29.06%. (n=3).

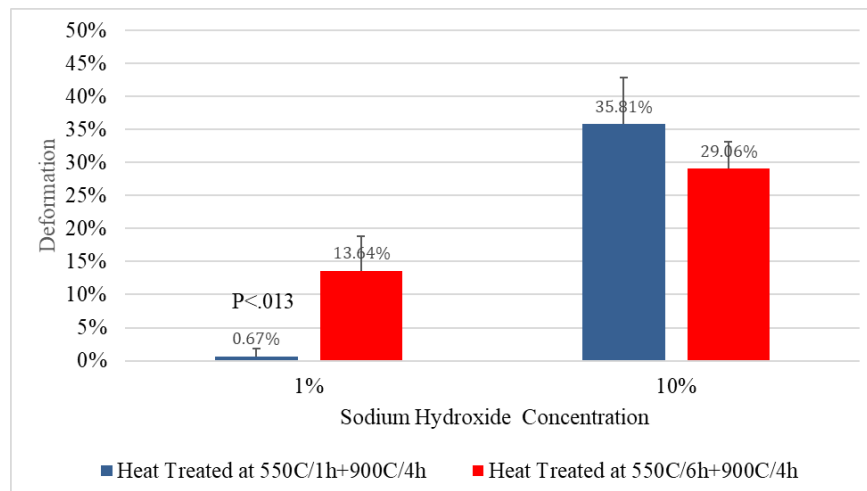


Figure 31: Deformation percentage for 3D printed SiC samples treated at 550°C/1h and 550°C/6 hours followed by 900°C/4h after impregnation in 1% and 10% sodium hydroxide concentrations

4.5. Effect of oxygen environment during sintering and strengthening of the 3D printed SiC discs

The lowest crystallization temperature of 800 °C was chosen in order to understand the effect of oxygen in the crystallization and growth of nanowires. The abundance of oxygen at high temperatures may lead to extensive oxidation and poor mechanical integrity. Hence the study was carried out at 800 °C for both air and oxygen environment. The 3D printed SiC discs were sintered at 800 °C in air and in oxygen environment to understand the effect of oxygen in the growth of nanowires. Figure 32a shows formation of large nucleation droplets at a temperature of at 800 °C for 2h without oxygen. In Figure 32b due to the presence of oxygen environment, the growth of thin nanowires of an approximate average size of 200 nm were observed. The formation of SiO₂ nanowires requires diffusion and deposition of oxygen onto oxygen deprived SiO nucleation droplets. Hence the introduction of oxygen environment promotes the growth off nanowires even at temperatures as low as 800 °C. The EDX on figure 33 proves that the droplets formed at 800 °C in oxygen environment has a Si:O ratio of 1: 2.4 which shows that adequate availability of oxygen to form SiO₂ nanowires. Figure 34 shows SEM images of the SiO₂ droplets after heat treatment at 800 °C in air. Upon heat treatment, the water molecules evaporate from the silica gel and the material matures into SiO₂ droplets.

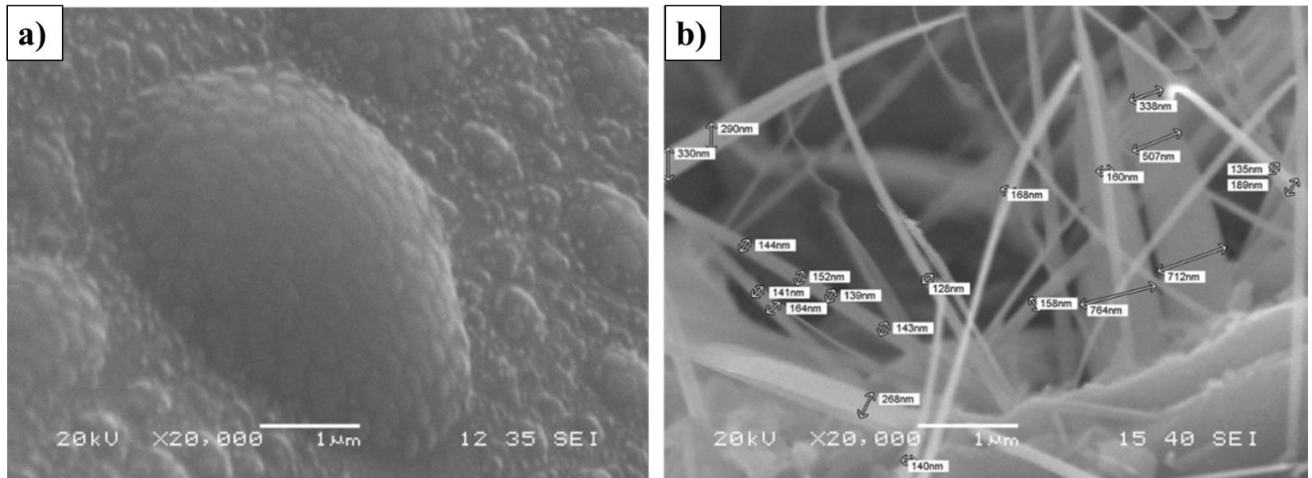


Figure 32: SEM Micrographs of 3D printed discs heat treated at 800 °C for 2h a) in air and b) in oxygen environment

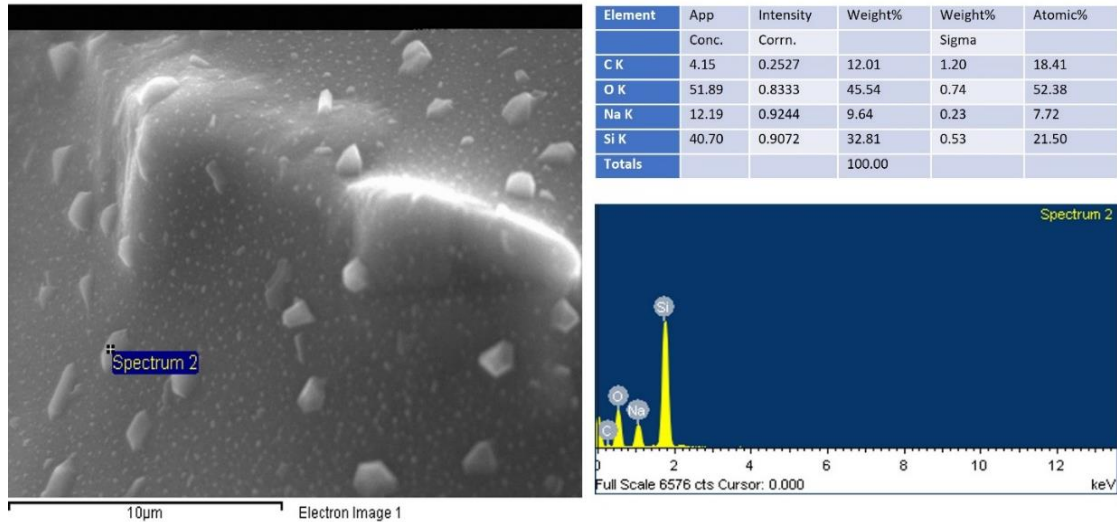


Figure 33: EDX analysis of the nucleation droplets on the 3D printed nano- μ sample heat treated in oxygen atmosphere after impregnation in 20% NaOH and heat treated at 800 °C for 2h.

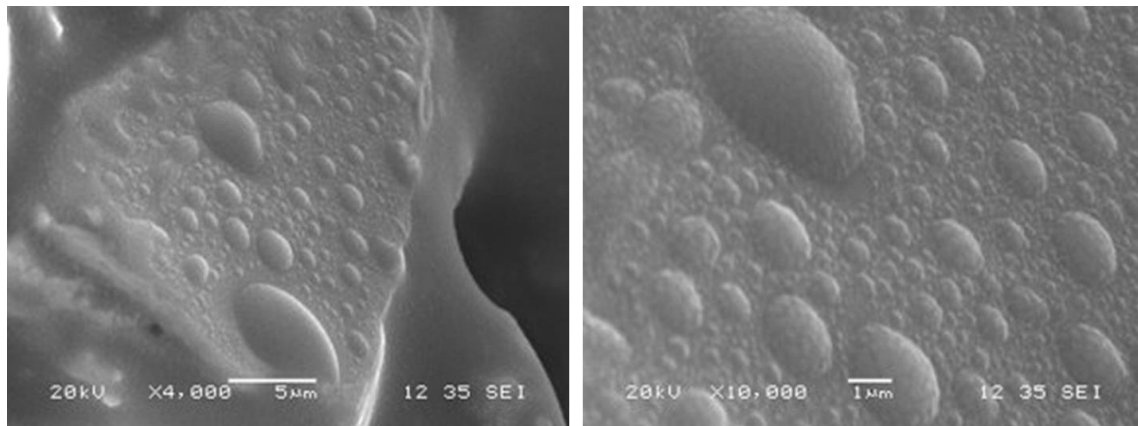


Figure 34: SEM micrographs of the SiO₂ droplets after heat treatment at 800 °C in air.

Conclusion Increased understanding of the secondary crystal behavior and its dependence on sintering profile were established.

Task 5: AM parameter selection

The hypothesis is that spreading the SiC powder into defect-free layers and providing efficient mixing with the NaOH binder during jetting will optimize the homogeneity and growth of the secondary crystals and lead to enhanced densification. The rationale is that uniformity of the green part microstructure is necessary to ensure the elimination of defects during sintering. The high surface area of fine ceramic particles causes increased cohesive strength for unpacked powder and decreased flowability. SiC powder were spread into defect-free layers and the conditions for homogenous spreading of the particles layer-by layer while printing were identified. 3D Projet460Plus printer contains an in-built Auger that fluidize the fine particles before each printing to ensure the homogeneity of the spread layers.

Post-processing of the green parts included two major steps:

- Thermal treat the green parts at 650 °C to provide initial mechanical stability (figure 5)
- The discs were then subjected to NaOH impregnation treatment that facilitates bonding between particles and increase the silica gel layer precursor for silica nanowire formation. (figure 4)

Efficient surface interaction between the NaOH binder and the homogenous SiC layer is dependent on the NaOH flow rate and quantity during the binder jetting. Efficient NaOH mixing with the SiC powder is essential for homogenous growth of secondary crystals to seal the gaps between the SiC particles.

Part 2: Residual Stress Modeling in Wire-Arc AM for Magnesium Alloys

Additive Manufacturing of Magnesium Alloys via GMAW

Experimental Setup/Results and Multi-physics Modelling

Abstract

Gas Metal Arc Welding (GMAW) is implemented as a method of additively manufacturing magnesium structures with high strength-to-weight ratios. This process was examined experimentally, via the construction of a CNC welding machine, and modelled using COMSOL Multi-Physics. Multiple welding parameters were tested to determine the optimal settings for depositing AZ61a magnesium wire on a AZ31b substrate. Welding methods including DC, DC Pulse, and Synergic Pulse were analyzed with both high and low energy inputs. Finding functional welding parameters proved difficult and time consuming as there exist no dedicated magnesium welders with preprogrammed material presets. As a result, material presets for aluminum had to be used, making the tuning of weld parameters challenging due to a high difference between machine set parameters and the actual outputs of the welder. Welding data, in the form of actual welding current, voltage, and wire feed rate, were collected and used in the analysis of weld bead shape, substrate penetration, and layer fusion. The focus then shifted on how to best assemble these beads into multi-row/multi-layer (MRML) blocks. Numerous overlap percentages were tested to minimize internal voids as much as possible. Different bead stagger patterns were investigated to determine how to best deposit these layers on top of one another. Using the found parameters and deposition patterns, MRML magnesium blocks were created on our CNC welding machine. These blocks were then cut into tensile test specimens using wire EDM and tested in our Instron 5582. Tensile test data was obtained on two different bead print directions: i.) normal to the tensile test pull force and ii.) in-line with the tensile test pull force. While the yield strength of the material was not affected by the print direction, it did impact the fracture stress limit. SEM analysis of the fracture surface was performed as well as optical microscope analysis of the grain structure of the printed material. The SEM analysis not only was used to determine the ductility of the material but was also a useful tool in locating different types of weld imperfections at the fracture surface which resulted in the fracture of the samples. Finally, a MRML COMSOL simulation of the material deposition method was created. The Goldak double ellipsoid heat transfer model was used to determine its ability to simulate the heat input during the welding process of a single bead on a substrate. The model was then expanded to utilize an element activation method to simulate the heat transfer as well as the deposition process. The final 3D model simulates the complete thermal history of a three-layer block of material being built using GMAW.

Experimental Setup

GMAW CNC - Design and Testing

Very little data exists on the behavior of magnesium alloys when used in the gas metal arc welding process. Initially, these experiments were to take place on a 5 axis Mori-Seiki CNC machine at UNC Charlotte. However, before installing this welding system on the Mori-Seiki, a prototype of a 3-axis CNC welding machine was built to first determine potential spatter, fumes, and burn through, all of which could severely damage the Mori-Seiki. The welding CNC that was built is depicted in figure 1. The welding takes place inside an enclosure constructed out of 6061 aluminum. The largest magnesium substrate installed to date is a 305 mm x 305 mm x 13 mm plate, which was used for individual bead test welds. Larger plates, however, can be installed as well. The maximum potential printed part height is currently 100 mm, but this can be quickly increased to up to 300 mm by replacing the three vertical supporting structures and the linear guide of the z-axis. The enclosure is raised off the ground to aid in heat removal during welding. An array of fans is installed underneath the back end of the enclosure which blows ambient air across the outside bottom of the enclosure. A large cooling fan was installed outside the weld enclosure to aid in the interpass cooldown

process to decrease downtime between welds. Two gas hoses are running into the enclosure at both ends, which can be used to flood the area with UHP argon gas from a separate tank with a dual flow regulator. The suspension of the workpiece in a 100% argon environment does greatly improve the final quality of the bead. The edges of the enclosure are sealed with fire resistant material to ensure no escape of the argon gas through any existing cracks. However, this would require far too much gas due to the open nature of this enclosure and the pre-weld treatment steps that must be taken prior to each bead deposition. Ergo, the bulk of the research was performed using only the shielding gas from the weld torch to protect the weld pool from contaminants.

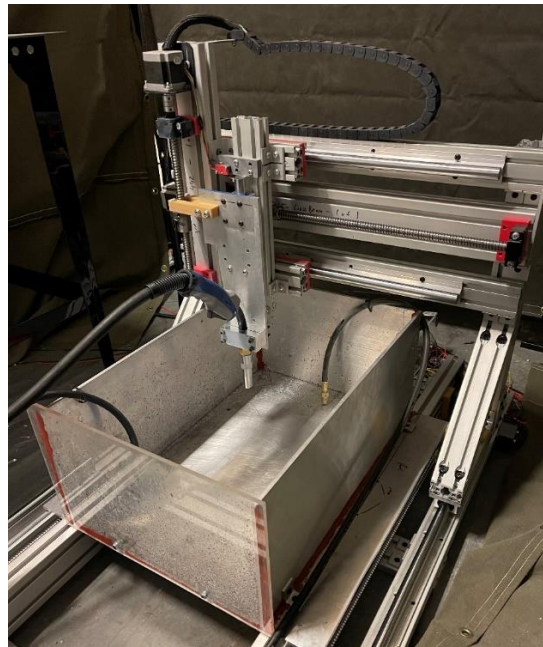


Figure 1 GMAW CNC

The welding substrate is clamped to the bottom of the aluminum enclosure via two large aluminum cross-beams and four bolts, as seen in figure 2. Grounding of the substrate is achieved by either clamping the welding ground cable to the clamps or by attaching it to a grounding bolt underneath the enclosure.



Figure 2 Clamping and grounding

The welding machine used for these experiments is the Welbee P400 from Daihen OTC, depicted in figure 3. Originally, these experiments were performed with the Millermatic Alumapro, which proved ineffective given its optimization for aluminum welding only. The P400 allows for more flexibility when trying to determine custom weld parameters and provides the option of uploading custom wave forms for pulse MIG welding. The welding torch trigger wires were fed through a relay-trigger connected to the Mach3 controller of the CNC, enabling torch activation via CNC M-code in the welding program. The torch itself is fastened to the z-axis with an aluminum clamping system we designed and manufactured. An emergency stop was also installed to prevent excessive damage in the case of welder misfire, burn through, or CNC code error.

It was initially assumed that magnesium and aluminum would have similar welding characteristics. Calibration of the machine and initial tests were performed using 5056 aluminum wire on the Millermatic. Aluminum is much cheaper than magnesium, so the process of building large sample blocks and then machining them into tensile test specimens was to be perfected on aluminum first. This was accomplished with relative ease. Figure 3 shows the results of these initial tests.



Figure 3 Deposited aluminum block tests

Experimental Research Task 1: Current pulsing and other MIG methods

Challenges in magnesium GMAW

When the switch from aluminum to magnesium was made, the original assumption of the similarities between the two in welding was quickly proven incorrect. Magnesium is much more sensitive to even the slightest changes in input parameters. When the heat input is too low, the bead will not penetrate the substrate nor fuse with beads layered on top it. When magnesium is melted it has a very low viscosity, so when the welding heat input is just a little bit too high, the melted wire will flow across the substrate instead of sticking to it. Furthermore, due to the very light weight of magnesium (magnesium to aluminum density ratio = 0.63), the weld droplets do not separate from the wire and fall onto the substrate in a consistent manner, but rather stick to the weld wire and form large individual drops. This leads to only two, equally detrimental, potential outcomes. The first possibility is that the large drop eventually separates from the weld wire, causing a sudden large change in the distance between weld wire and substrate, which results in sudden drops/spikes in welding current and voltage. This can lead to a misfire and to an intermittent bead profile. Figure 4 displays a plot that shows these fluctuations in the welding data. The top bead in figure 5 shows the resulting shape when this occurs. This behavior can't be predicted and if this occurs mid-process during the manufacturing of a component on a CNC, then the resulting part would be unusable.

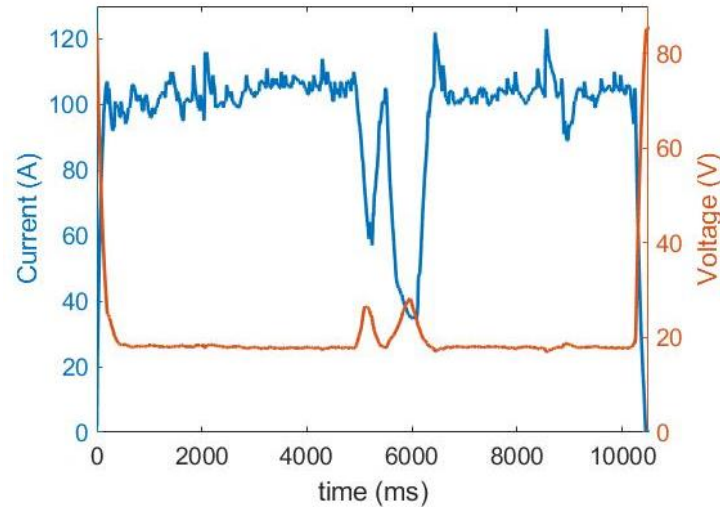


Figure 4 Current and voltage fluctuations resulting in intermittent bead profile



Figure 5 Magnesium bead shapes with different input parameters

The second possibility is that the weld bead continues to grow but does not drop at all, but rather sticks to the contact tip. When this happens, the accumulated heat will melt the contact tip, and if this is not noticed quickly enough to abort the process, can lead to the weld tip exploding, spraying copper puddles all over the substrate. An image of an extreme case of such a melted tip is shown at the top of figure 6. The sensitivity to changes in welding parameters is further visible by the other two beads in figure 5. The middle bead shows the material behavior as the heat input is slightly increased. Adherence to the plate is slightly improved, so is the issue of intermittence. The material, however, begins to flow away from the main bead structure. The bottom bead shows the material when the heat input is slightly increased further. The bead no longer has any discernable shape and the material is flowing all over the substrate. The room for error in having the correct settings is extremely small.



Figure 6 Melted contact tips from Mg GMAW

Magnesium, unlike aluminum, also has the ability to ignite and burn. Even though in theory the heat input must be extremely high for this to occur, it is still a potential danger, and has in fact happened in one occurrence during our experiments. During a high energy input test, the crater fill operation at the end of the weld punched through the magnesium substrate, causing it to catch fire and burn for 10 to 15 seconds. This happened during a hand-welding test and the material flowed through the weld table holes onto the floor. Figure 7 shows the resulting damage to the material. Had this occurred on the Mori-Seiki the damage could have been severe. A good way to hedge against such potential dangers is by always using an additional aluminum baseplate underneath the magnesium substrate.



Figure 7 Burn through of magnesium substrate

Special care must be taken to prep the substrate correctly prior to welding. A file is used to remove any material from the edges of the plate. This is done to ensure that the substrate lays flat on the welding support surface so that proper heat transfer between support and substrate can occur. Next the part is sanded down multiple times with 80 grit sandpaper and 300 grit sandpaper. The remaining oxide powder is then washed off with acetone. Next, a stainless steel brush is used to prepare the area chosen for welding to remove any last surface impurities. Due to the large amount of weld soot during magnesium welding, the substrate is brushed down with the stainless brush between every weld and the soot is removed.

Three main forms of GMAW were tested: DC, DC Pulse, and Synergic Pulse. DC welding is a continuous input of energy at a pre-set current and voltage input. The voltage and current may oscillate on their own throughout the process to maintain a relatively continuous heat input to the workpiece. In DC pulse, the input current oscillates up and down following a pulse wave function resulting in an average input current/voltage for the duration of the weld. One of the biggest benefits of this process is a higher deposition rate, which is one of the key reasons GMAW is considered for this project. This process also reduces spatter and improves overall weld quality. Lastly, synergic pulse welding can stabilize the welding arc, which is of great importance when considering the bead-detachment problem discussed in the previous section. In this process, the welding current is set, and the machine calculates the optimal welding voltage, $V_{\text{calculated}}$.

for that current. The calculated voltage can then be fine tuned by either increasing or decreasing it. All tests discussed in this paper were performed with 1.2 mm diameter AZ61a welding wire and AZ31b substrates. The 100% argon shielding gas flow rate was set to 25 cfh.

Synergic Pulse Welding

After numerous tests using different parameters for synergic pulse welding, a semi-successful set of parameters was found using high energy input. Initial tests were performed by hand to speed up the tuning process. The weld was split up into three separate stages: Initial Condition, Main Condition, and Crater Fill condition. The Initial Condition set the welding current to 170 A and the welding voltage to $V_{\text{calculated}} + 50$. This was done so that the initial heat would better penetrate the substrate. After 1.5 seconds, the main condition kicks in, which was set at 170 A $V_{\text{calculated}} + 0$. When the weld is complete, the crater fill condition runs for 1.5 seconds at a current of 140 A and a $V_{\text{calculated}} - 40$. The crater fill option rounds out the end of the bead to avoid a long sloping end to the bead, thus the heat input for this stage is greatly reduced. Figure 8 displays the actual welding condition for this process sampled by the P400 at 100 hz. The mean power output during the main condition was 5213 W with a standard deviation of 67.6 W. Even though this process was initially considered successful, it was later determined that this spray like method from the pulse welding did not penetrate the substrate enough. Most of the heat went into the melting of the wire itself, resulting in globular weld beads that did not fuse to one another when stacked on top of each other. Figure 9 shows a single (top) and two-layer (bottom) bead when these parameters are implemented on the CNC machine. Figure 10 shows a side view of the two-layer bead.

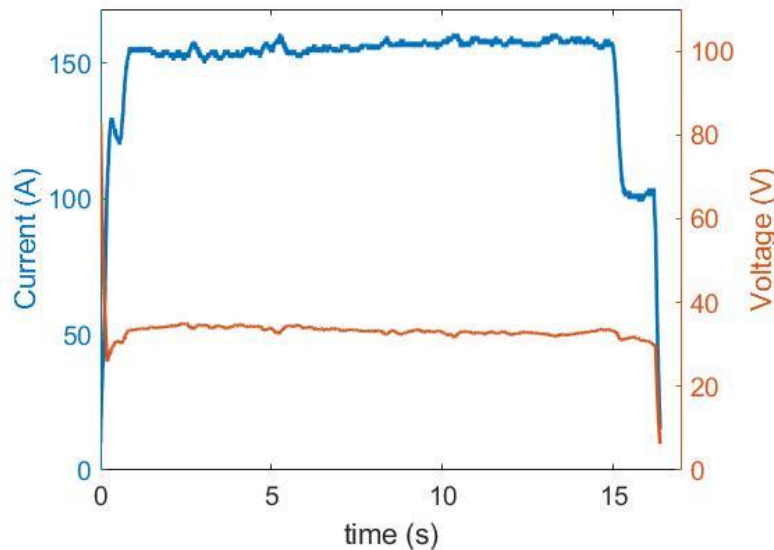


Figure 8 High energy synergic pulse welding condition



Figure 9 Single and double layer bead of magnesium using synergic pulse welding



Figure 10 Side view of double layer bead of magnesium using synergic pulse welding

DC MIG Welding

Standard DC MIG welding at low energy input was implemented for the magnesium welding process. At high energy input, this process resulted in a very high amount of spatter. These tests were performed by hand at first and then on the CNC. Figure 11 displays a single bead when deposited the low energy input process is used. The surface of the bead is much smoother than with high energy synergic pulse. However, there is a lack of substrate penetration on the bottom layer, resulting in a cause of concern about future layer fusion. This, however, was later corrected by further parameter tuning. Figure 12 displays the welding condition for the duration of the weld.



Figure 11 Low energy input DC weld of magnesium

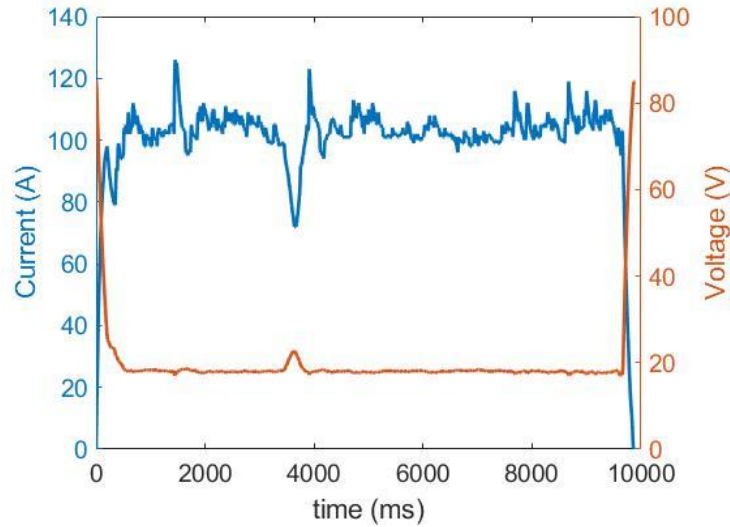


Figure 12 Welding condition for low energy input DC weld of magnesium

As can be seen in figure 12, a small current/voltage drop/spike is observed indicating the presence of a potential minor gap in the bead. The majority of the weld, however, is stable. The power output for this setup has a mean of 1864 W, much lower than that for pulse welding. These parameters were further improved, with the final setting shown in the conclusion section.

Equipment challenges

Typically, establishing the correct welding parameters is achieved by starting with a pre-existing set of parameters based on wire material, wire thickness, and substrate thickness. The desired bead is then obtained by tuning the input parameters, voltage and amperage. For example, if the bead first appears to have too much of a caterpillar shape, the user can slightly increase the voltage to flatten the bead. While doing this the amperage should remain the same. The first problem that was discovered was that the inverter's actual output was drastically different than the set parameters on the machine by the user. Clamp meters were then used to confirm that these readings were not just errors in the inverters measurement system. These discrepancies between set voltage/current and actual output voltage/current are substantial. Figure 13 displays the output voltage and output current of the inverter in DC pulse mode, showing a mean voltage of 31 V and a mean current of 193 A during deposition. The set input parameters for this weld were 15 V and 250 A. This discrepancy pattern between input vs output parameters was observed in all welding methods, as can be see in Table 1. Pulse welding showed the most drastic differences between input and outputs when comparing the different welding methods. The output power was consistently over double the set power and could not be lowered. This made the pulse deposition method not usable for our experiments.

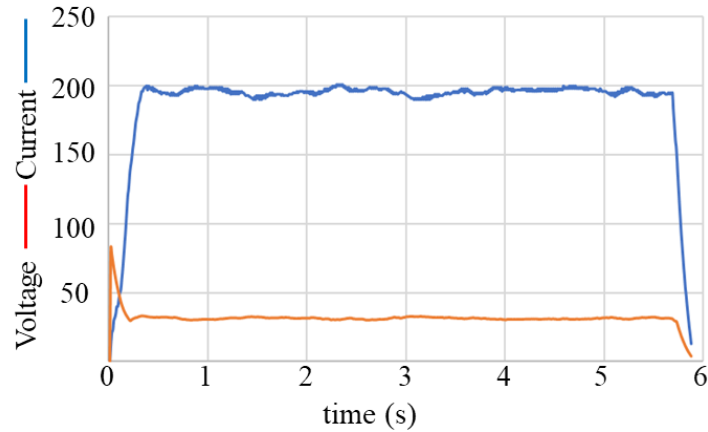


Figure 13 DC Pulse measured output voltage and current when set at 15 V and 250 A

Table 1 Input vs Output Parameters

DC						
Test	A_set	A_actual	ΔA	V_set	V_actual	ΔV
1	200	150	50	12	14	2
2	200	135	65	15	17.7	2.7
3	250	168	82	15	18.6	3.6
Pulse						
Test	A_set	A_actual	ΔA	V_set	V_actual	ΔV
1	250	193	57	15	31	16
2	250	175	75	10	30.5	20.5
Synergic						
Test	A_set	A_actual	ΔA	V_set	V_actual	ΔV
1	180	134	46	0	21.3	-
2	180	142	38	-100	13.5	-
3	250	175	75	-100	20	-
4	100	122	-22	-100	16.7	-

Aluminum wire was subsequently tested to determine if this difference also appears when using the set wire material. The aluminum output parameters matched the input parameters, so it was clear that the issue was with the magnesium. An attempt was made to map the input parameters for magnesium to desired output parameters, but this proved impossible due to the impact that changing one parameter has on the output of the other. Examples of this can be seen in table 1 in both the DC and Pulse weld data. In DC, with the set current being held constant at 200 A, changing the set voltage from 12 to 15 not only increases the output voltage but also causes the output current to drop. Changing the set current also has a direct impact on the output voltage. This phenomenon cannot be removed using a standard industrial welder with wire material presets. Performing a normal tune of the parameters by adjusting them one at a time was therefore not possible and made finding optimal welding parameters extremely challenging and time consuming. This problem also made it increasingly challenging to tune the required welding parameters as multi-layer blocks were manufactured. DC welding was used for the remainder of the project as the only method of layer deposition due to its superior bead deposition, bead shape control and semi-controllable heat input with our commercial welder.

Fusion in multi-row/multi-layer blocks

Another obstacle to overcome was finding the parameters that would result in the optimum temperature of the material being deposited. It is possible to maintain good control of the shape that is to be printed with the magnesium. This, however, requires a reduction in the heat input during the deposition process and results in beads of a caterpillar like shape. This causes not only the bead to not fully penetrate the substrate, but also causes large voids in the material as layers are being added on top of each other. Figure 14 shows how these beads appear inside a welded block.

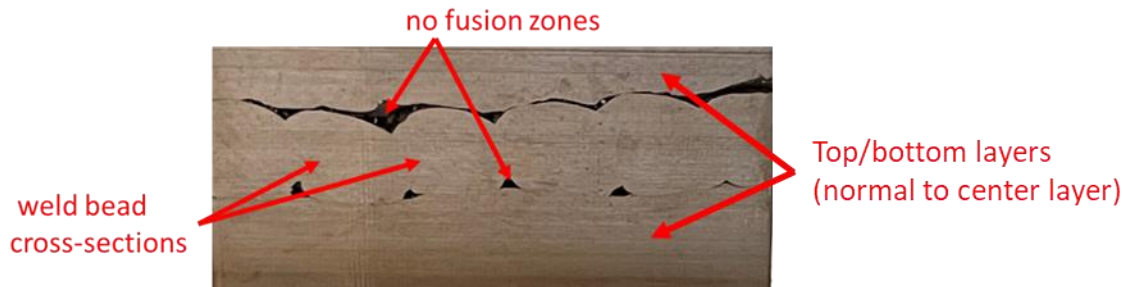


Figure 14 Low heat input layer cross-section

An increase in heat input is required for the layers to be fused on top of one another. There is a very fine line between not enough heat and too much heat when welding magnesium. Not enough heat and issues like those in figure 14 occur. On the other hand, if the heat is even the slightest bit too much, the deposited material will lose its shape and flow uncontrollably over the substrate surface. Figure 15 shows how even the slightest change in heat input can cause a good bead (top two beads) to lose its shape when the welding temperature is raised by increasing the voltage current by just 0.2 V. If the temperature is increased any more, the bead will have no distinguishable shape at all.



Figure 15 Too hot and too cold image

This is also an issue when starting to add multiple lines together as the required overlap will cause the weld material to flow uncontrollably when re-melted. Figure 16 shows how the manufactured block will continue to lose its shape the more material is added to it. This makes predicting the required row spacing to achieve the desired overlap impossible and can lead to intermittency issues during the weld. As can be seen in figure 16, the single beads on top have a perfect consistent shape. Unfortunately, as soon as a second bead is added to the original single bead with a 30 percent overlap, the outside of the weld flows onto the substrate. The more beads are added in this manner, the further the layer loses its shape.



Figure 16 Side flow of overlapping beads

The interpass temperature for welding with magnesium also plays a key role in dealing with the fluidity problem of melted magnesium. Tests were performed at different cooldown temperatures between weld beads. It was found that the temperature of the substrate must return to close to ambient temperature before additional beads could be deposited. Without allowing this cooldown, the temperature buildup in the substrate and deposited beads gets so high that the material will spatter and flow without any control. To lower the interpass time, two cooling methods were applied. An array of fans installed underneath the welding enclosure removed the heat that was transferred from the substrate into the machine's enclosure. An additional fan was installed above the enclosure to speed up the cooldown between beads. The interpass time for 80 mm beads was 3 minutes while the interpass time for 175 mm beads was 6 minutes. This process could be vastly improved with advanced rapid cooling methods and was originally planned as part of this project. Due to faculty changes and project reassignments, this research report only reflects 2 out of the 3 years that were originally allocated to this project. With additional time, advanced cooling techniques could be studied as planned.

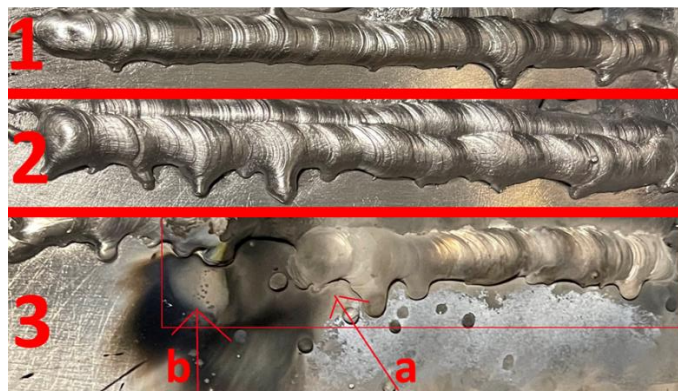


Figure 17 Overlap flow problems and intermittence

In addition to these side-flows of the deposited magnesium material, other factors can cause sudden stops in the weld. The most common issues are oxidation on the weld wire, oxidation deposits on the substrate, and slipping of the wire-feeder wheels due to bends in the weld hose. The third bead in figure 17 failed halfway through the weld due to the interference of the side-flow of the previous pass and due to a stop in the feeding of the wire. At location (a) the wire stopped feeding but arcing continued. The torch was at location (b) when the emergency shutoff button on the CNC was pushed. If this is not detected quickly enough the wire will continue to heat up and stick to the nozzle, which can cause it to melt and pop.

The wire must be cleaned manually inch-by-inch before it can be used in this process. This is done by spooling it onto an empty spool while cleaning it with 600 grit sandpaper. Once the desired length of wire has been sanded, it gets respooled onto the original spool while whipping down the wire with denatured alcohol to finish the cleaning process. Similar cleaning procedures are also applied to the substrate, which first gets sanded down, then gets cleaned with a wire brush, and then finally gets wiped down with acetone. Since all this is done manually it is a very time-consuming endeavor. This process could be automated but that would require the design and build of a separate wire-prep machine.

The soft nature of magnesium wire also makes feeding it through the weld hose very difficult. Keeping the hose as straight as possible at all times reduces the chance of the material to stop feeding. The addition of Weld-Aid Lube-Matic cleaning and lubrication pads at the inlet of the wire feeder helped in further cleaning the wire to ensure the liner does not get clogged with dust and the lubrication reduced the wire feeding problem. During the manufacturing process, the deposited beads and substrate need to be cleaned with a stainless-steel brush after the deposition of each weld to remove the soot that accumulates around the weld pool (black soot is visible in Figure 17). The dust is then vacuumed out of the enclosure before the next bead is added.

Warping of the substrate is also of concern. The substrate is fastened in the CNC with two aluminum bars acting as clamps. During deposition, the thermal stresses inside the substrate cause it to deform so much that the force it puts on the aluminum clamps causes them to bend. This is shown in Figure 18. The left side of the image shows the new substrate clamped in the enclosure and the right image shows the deflection in the clamps at the end of the manufacturing process. When the clamps are removed, the substrate continues to deform, with an end to end lateral offset of up to 2 cm. This will be shown in detail in the following sections. Of course, this problem can be easily resolved by simply using a thicker substrate. This would significantly increase the cost of this research due to the relatively high cost of AZ31b magnesium plates. It was therefore decided that thinner plates of 10 cm thickness would be continued to be used for these experiments.

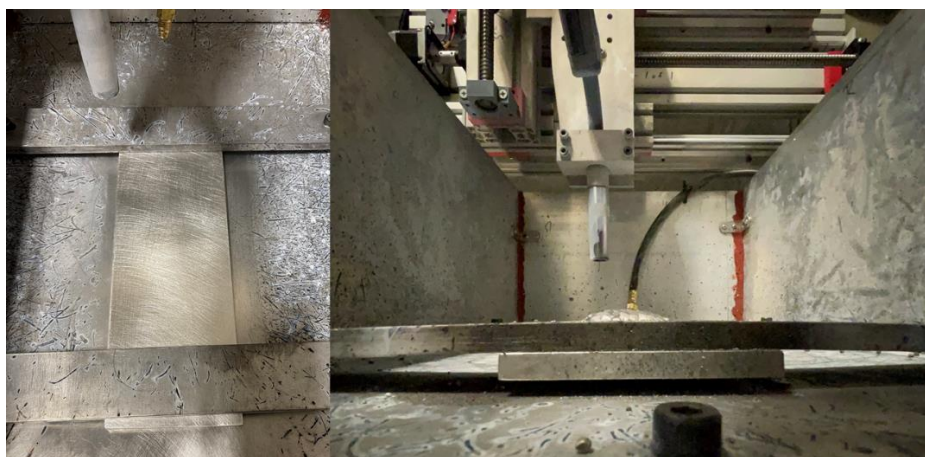


Figure 18 Clamping and warping of the substrate inside the CNC

The shielding gas for the magnesium welding process is also of the highest importance. For these experiments, 100% argon gas was used. The welder itself produced a gas flow rate of 35 cfm. The enclosure itself could also be flooded with argon through two hoses inside the enclosure, one of which can be seen in figure 18. Flooding the work area with argon prior to welding did result in a much better-quality weld. However, due to the required cleaning process between the deposition of each beads and the open-box nature of the enclosure, the quantity of argon gas required to perform these tests was excessive. It was therefore decided to rely solely on the contamination protection provided by the shielding gas from the

weld torch for these tests, even though this would result in a less than perfect final product. The addition of a continuously argon flooded closed off enclosure would result in far superior results. The design and build of such a machine is planned for future work. Additionally, the design and build of a magnesium dedicated inverter for this additive manufacturing process is also part of future research plans. With a magnesium dedicated machine these parts could be printed with high efficiency and near net shape, but this process would have taken close to a year to perfect. As such, it was decided to move forward with in-hand components to obtain initial data on the process.

Conclusion

Extensive testing has shown that DC welding as apposed to pulse welding is a better method for the deposition process when using a commercial welder. This is due to pre-existing material presets that make the control of energy output of the welder difficult when using non-standard welding wires such as magnesium. Pulse welding of magnesium would require the design and build of a custom magnesium-dedicated inverter. Successful deposition parameters for DC welding were found, the bead profile of which was shown in figure 15. The deposition for these beads featured the following parameters, setup and power output:

- i. Set current: 200 A (resulting in measured current of 130 A)
- ii. Set voltage: 15.5 V (resulting in measure voltage of 18.6 V)
- iii. Wire feed rate: 13.6 m/min
- iv. Nozzle to workpiece offset: 12 mm
- v. Torch travel speed: 750 mm/min

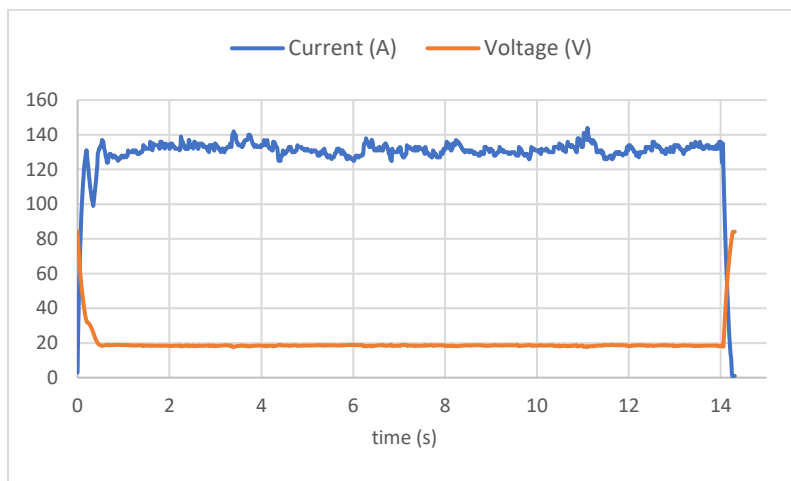


Figure 19 DC pulse inverter output (set: 200A/15.5V)

Experimental Research Tasks 2 and 3: Torch Motion and Cooling Rate

Internal voids are one of the biggest concerns when creating multilayer blocks using GMAW. This became obvious already when doing the initial test with aluminum wire. The torch path when layering the beads on top of one another is crucial in obtaining parts with minimal internal defects like voids.

Torch Motion: Bead and layer orientation

Numerous approaches to determine the best orientation and stagger for building layer-upon-layer blocks were tested. It was determined right at the offset of these tests that depositing the beads directly on top of one another was not going to lead to good results as this will immediately lead to the material slipping off the sides of the pre-deposited bead. The resulting edge has the appearance of the top of a loaf of bread and

this phenomenon is therefore referred to as loafing. In order to ensure subsequent layers remain on top of previously deposited material, there must be a staggered offset when starting a new layer. The amount of this offset depends on the width of the beads. An offset of 30 percent of the bead-width was found to be sufficient to avoid loafing of the material.

The first multilayer block of AZ61a magnesium that was manufactured can be seen in the following figures. This block consists of 65 individual beads that were deposited with a machine set amperage of 220 A and a voltage of 15V. The actual output was measured at 150 A and 18 V. The beads were spaced 5.5 mm apart on each layer resulting in an overlap of around 50%. As can be seen in the pictures, this overlap caused significant flow on the sides of the beads, but a rough net block shape was still retained. Between each bead deposition the workpiece was brushed and cleaned. The block itself was 175 mm in length, 80 mm in width, and 25 mm in height at its base. The lengths and widths were reduced for each of the subsequent layers, of which there were 6 in total. It is important to note that at the start of the fifth layer the first bead failed halfway through the deposition process. This bead was fixed by running a second bead from the other side into the end of the failed bead. Figure 20 shows multiple angles of the final block that was created. The bottom right image in figure 20 shows the warp that occurred in the substrate, with a end-to-end deflection of 22 mm.



Figure 20 AZ61a manufactured block

The deposited material was then machined into a block, as can be seen in figure 21. Upon inspection it was found that minor voids were occurring in select areas across the sides of the block. Numerous additional settings were tested following this experiment to reduce these voids, including welding parameter changes, layer orientation, overlap percentage changes, and clamping methods.

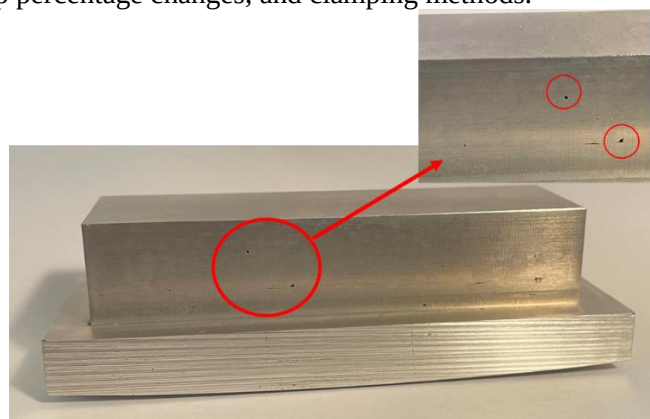


Figure 21 Machined AZ61a block with minor voids

It was determined that the most likely cause of these voids were pockets in the deposited layer caused by wire material that was flowing off the bead during overlapping depositions. An example of such pockets is shown in figure 22. As can be seen, even though the previously deposited layers appear to be nicely fused together, they are still most likely suffering of internal voids due to such pockets not being entirely removed when the subsequent bead is being deposited on top of them. Resolving this issue was the biggest challenge moving forward.

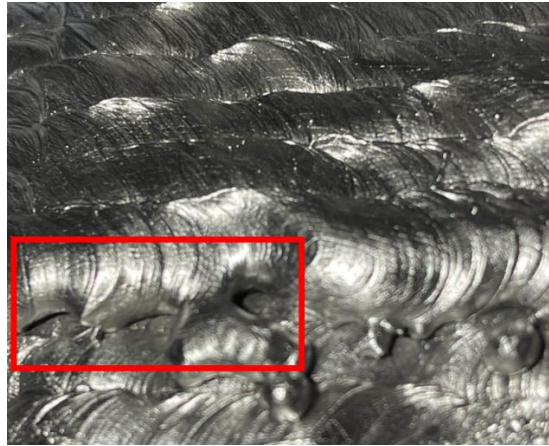


Figure 22 Pockets leading to voids in deposited material

Instead of welding each layer using different percentages of overlap, it was decided to space deposited beads apart from one another, creating a channel of empty space between them, and then depositing an additional bead into the empty channel. The first test using this method was done by utilizing a zero percent overlap. This can be seen in the left picture of figure 23. The next layer would be deposited into the channel between the beads. The problems with excessive flow caused by overlapping the beads was immediately reduced, resulting in layers that kept their shape much better than before. The downside of this approach is that naturally the final part will have a pyramid like shape, as every subsequent layer will be deposited with a shift of half the width of the bead. The first small test result of this staggering approach can be seen in the right-side picture of figure 23.



Figure 23 Zero overlap layer with no space in channel

Following the success of this method, multiple layer bead spacing approaches were tested, increasing the space between beads up to 80% of the bead width. It was determined that a bead gap of 4 mm resulted in the final best shape of the deposited block.

Due to the pyramid like structure of the final part, the first large block that was manufactured was done so using short beads that were deposited across the width of the substrate instead of long beads deposited

across the length of the substrate. This was done to ensure that there would be more usable material for machining tensile test specimens. This also meant that the heat input per bead was less than half than what it would be if long beads were deposited. Given the lower final temperature of the beads, side flow caused by overheating was also reduced. This, however, also meant that the deposition print direction would be normal to the direction later to be applied during the tensile test. This meant that any cracks/voids due to pockets would most likely cause the material to fail quicker given their orientation.

Material deposition for overhanging geometries has yet to be investigated. It is feared that when performing such magnesium welding operations on a machine such as the Mori, considerable damage to the machine can occur given the spatter and soot associated with this method. Given that this machine has a z-axis that is constrained in vertical motion, it is also not possible to install an enclosure on the worktable as any off-axis rotation of it would cause the welding torch in the spindle to collide with the enclosure. The custom CNC built by us could be modified by raising the supporting structure of the z-axis and installing a fourth axis to tilt the build plate. This would greatly reduce the monetary risks involved rather than trying such a deposition process on the Mori Seiki. Adding the fourth axis is easily achievable, but would require a little time to design and build. This was part of the original plan but due to the loss of one year of research has not been accomplished yet. This, however, will still be completed and the findings submitted.

Cooling Rate

As mentioned, due to the lack of time there were only 2 cooling rates investigated as last October: i. natural cooling and ii. Forced air convective cooling via a fan. The primary benefit of increasing the cooling rate is the reduction in time of the manufacturing process, since the increase in cooling greatly reduces the interpass temperature between beads, as previously stated. In order to reach the final goal of building layered blocks large enough so that tensile test specimens could be machined out of them, detailed tests on cooling rates, including the addition of the cryogenic CO₂ cooling method, had to be postponed. These tests can and will still be performed. Some choices had to be made to get as much work done as possible in the reduced 2 year, so unfortunately some tasks had to be deferred to a later date -process metrology also falls into this category of work to be done.

Experimental Research Task 5: Finish machining

Tensile test manufacture

The first large block using the new stagger method was created and can be seen in figure 24. As can be seen in the final shape of this block, the layers retained a decent shape and excessive flow was almost entirely controlled. This block consists of 137 individual 80 mm long beads. After each bead was deposited, the substrate and deposit were thoroughly brushed and wiped clean manually. The block was then cut in a wire EDM to create tensile test specimens, as can be seen in figure 25.



Figure 24 Short bead block sample for tensile test specimens



Figure 25 Wire EDM AZ61a tensile test specimens

The tensile test specimens were created using the ASTM E8/E8M-13a specification for subsize specimens and had a total length of 100 mm, a gauge length of 25 mm and a 6 mm x 6 mm testing cross section. Only the top 2 layers of samples, layer A and layer B in figure 25, were usable for the tensile tests as layer X included some material from the substrate. Voids in these specimens were also still present. While some of the sample surfaces were free of voids, others did show the presence of voids. These voids impacted, of course, the tensile test results, as will be seen later. These voids were primarily visible in the B layer specimens, but it is assumed they were also present internally in the A layer. Figure 26 shows the surface quality of these specimens. The top are the A layer specimens and the bottom are the B layer specimens.



Figure 26 Tensile test specimens – short beads

The same staggered pattern was then used to print more magnesium blocks, but this time in the long direction. This block is seen in figure 27. Due to the required stagger shape, the final number of tensile test specimens that could be extracted out of the block was much lower than that of the previous block. There were 5 usable samples that could be extracted out of this block. The presence of voids was still clearly present, as can be seen in figure 28. Some of the samples clearly still showed the presence of the issue with pockets between the beads. The machined samples from this block followed the same ASTM standards as before but had a considerably longer length of their grip section. As will be shown in the next section, due to the soft nature of the material the samples were prone to slipping in the Instron clamps. This additional length gave us more surface area for the clamps to hold on to the specimens.



Figure 27 Long bead block sample for tensile tests



Figure 28 Tensile test specimens – long beads

In summary, there were two different print directions being tested with these samples. Samples from the magnesium block in figure 24 were the result of short beads that are printed in the direction normal to that of the applied force in the Instron (*short-bead-normal* - SBN). The other samples, depicted in figure 28, were the result of long beads that were printed in the direction in-line with the pull force applied by the Instron (*long-bead-inline* - LBI). Both sets of samples were printed with the same heat input, using the same channel deposition approach, and the same staggered layering.

Tensile test results

Tensile tests were performed in the Instron 5582. The short-bead specimens displayed in figure 26 were tested first. Initial tests failed due to clamping issues. As can be seen in figure 29, the material was so soft that the Instron clamps could not hold on to the samples because they deformed the material. This problem was mostly corrected when subsequent test specimens had a larger portion of the grip length inserted into the clamps. Testing the samples with the longer grip length did not suffer from this problem as frequently, but unfortunately multiple data sets obtained from the Instron were not usable due to the clear presence of slipping of the sample. Future tests will be performed with a custom clamp insert and a custom test specimen grip sections that will act as anchors and rely solely on gripping force. The results for the specimens that were pulled to fracture are presented in the following section.



Figure 29 Specimen slip and deformation during tensile test

Figure 30 displays the combined test results for all samples that were tested from the SBN test group. As can be seen, some of these samples suffered from minor oscillations in the elastic region but still yielded usable results. A mean modulus of elasticity of 30 GPa was found for these samples. The yield strength for the material was determined to be in the range of 110 MPa to 130 MPa. Determination of the exact tensile strength proved problematic due to fracture of the samples occurring at different stress magnitudes. This is due to the crack propagation that takes place inside these sample during the strain-hardening phase of the tensile test. Voids in these samples can be in the form of different size cracks, and their size and location will ultimately determine when the specimen fails. Given the location of where these samples ultimately failed, it was clear that there must have been one small, but long void created between two beads during the deposition of the top layers of the magnesium block. This can be seen in figure 31, showing how all the samples from this layer failed at the same off-center location. This theory was confirmed when analyzing the fracture surface with the SEM. Even with this shortcoming, the material behavior during these tests remained consistent for all samples up to the points of fracture. The highest measured stress at the point of fracture was 174 MPa for the SBN group.

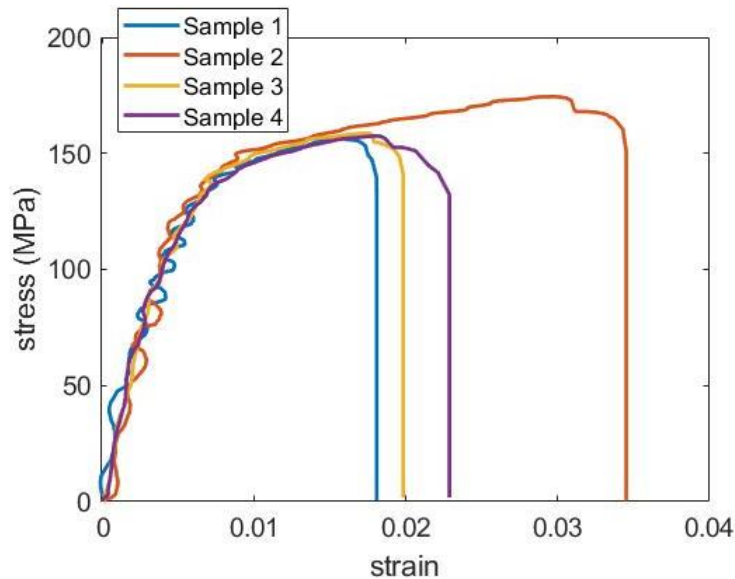


Figure 30 Stress-strain curves for SBN samples

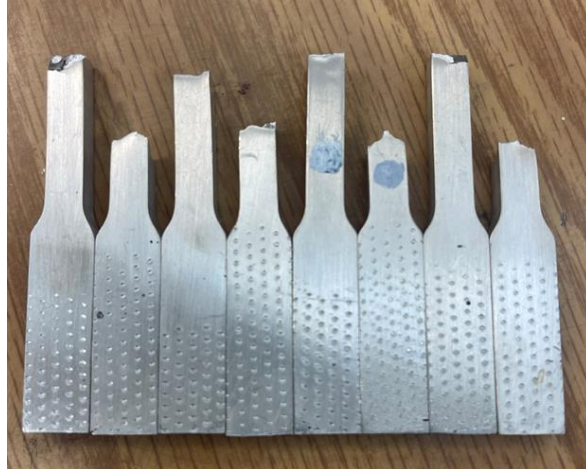


Figure 31 Examples of fractured SBN samples

Figure 32 shows the results when the LBI test data is compared to the SBN results. Just like in the FDM of plastics, the print direction has a great impact on the final strength of the material. As stated, the LBI samples also had minor voids, but now these cracks are in-line with the applied force, allowing the material to remain intact at higher forces. The elastic region and yield strength of these two sets of data are almost the same, but the final fracture point of LBI samples far exceeds that of the SBN samples, measuring as high as 217 MPa. It is important to note that unfortunately many of the LBI samples had a void at the radius of the tensile test specimen. Since these voids were present at this stress-riser, those samples failed at that point just before reaching the yield strength, rendering the results unusable.

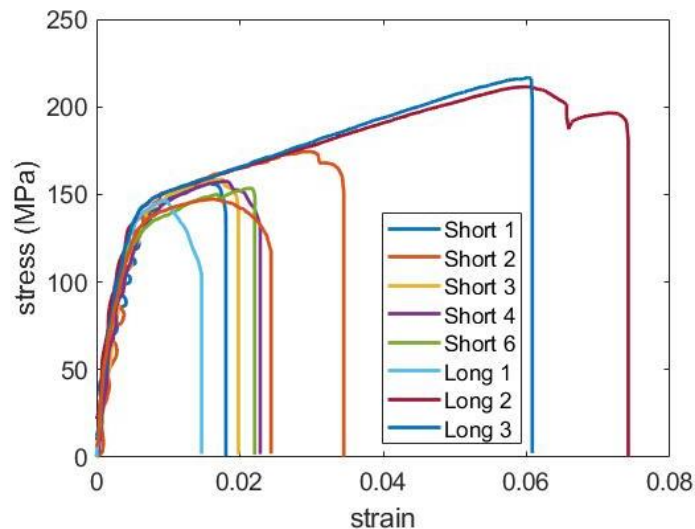


Figure 32 Combined SBN/LBI stress-strain results

SEM analysis of fracture surface

SEM analysis was performed of the fracture surface. This was not only used to determine the material behavior, but also to inspect the material for imperfections left behind from the deposition process. Figure 33 shows two images of fully fused sections after fracture, with the cup like structures of the surfaces demonstrating the behavior of ductile material.

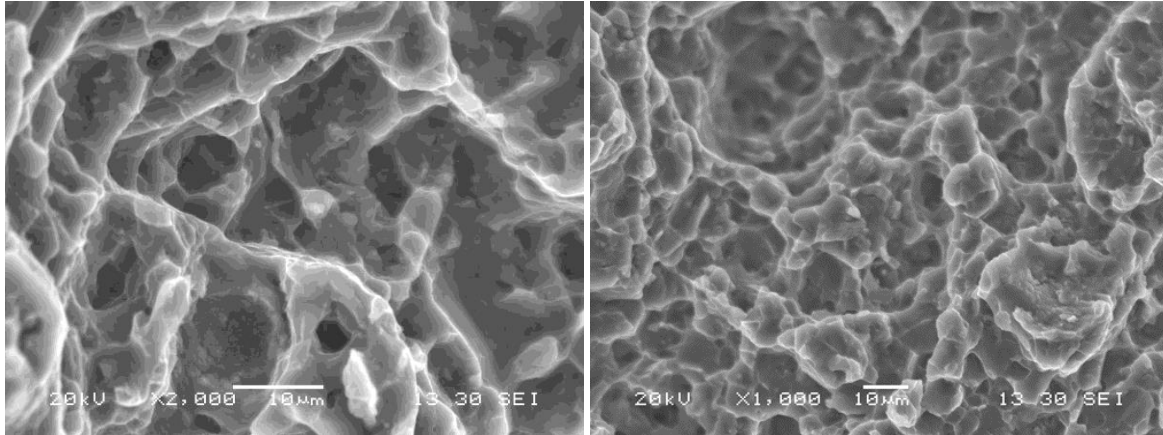


Figure 33 SEM of fused fracture surface

The imperfections found during the SEM investigation were considerably more interesting. One of the biggest problems in welding magnesium is the presence of spatter, which is very difficult to control when depositing magnesium at an accelerated rate and at higher temperatures. As mentioned before, due to the low viscosity of the magnesium alloy, it has the tendency to flow over previously deposited material before it solidifies, instead of fusing together with it. One of the dangers in this is that it could potentially trap soot and/or spatter inside the built piece. Figure 34 shows what appears to be a trapped spatter ball inside the structure. This is the equivalent of having a void in the material as it is not fused to any of the surrounding material, making the structure weaker. The surrounding surface also shows the lack of fusion in this area, indicating that this entire region was caused by the side-flow of magnesium during deposition.

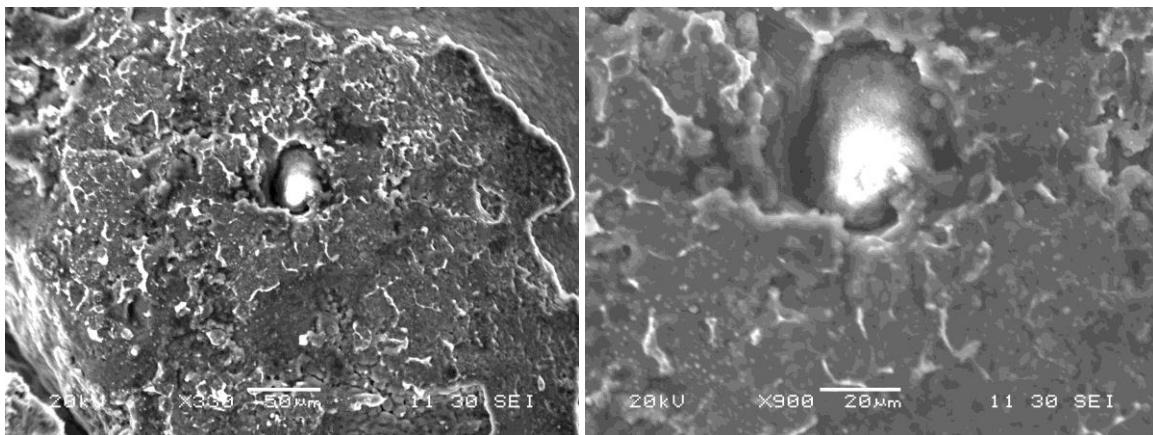


Figure 34 Trapped spatter inside the welded material sample

The presence of smaller cracks and hollow voids was also discovered, as can be seen in Figure 35. The left side of the image shows a small crack in the surface while the right side of the figure shows a void going into the material. All these imperfections could be removed, but as mentioned before, this would require a custom-built inverter that can be specifically tuned for the deposition of this material. Commercially available machines can do a decent job with single, or perhaps even double stacked, beads, but for multi-layer deposition, a custom machine would be required.

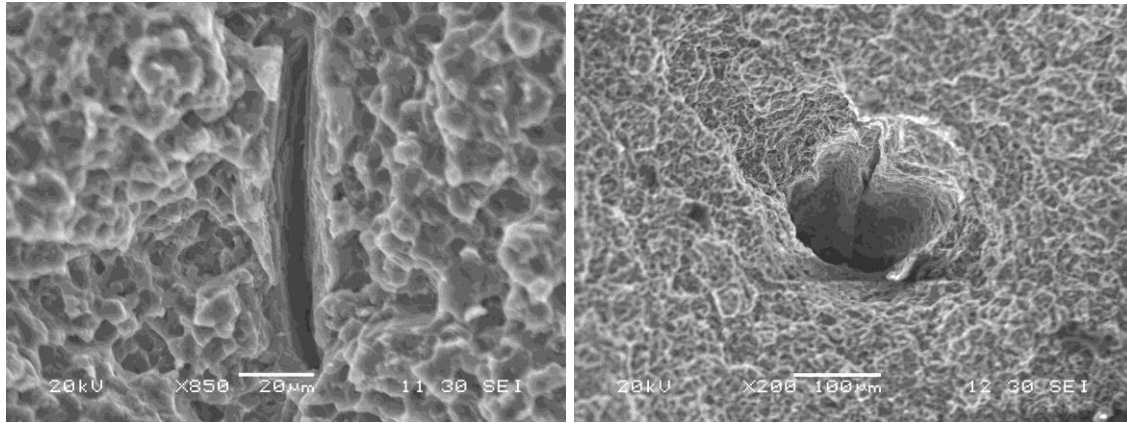


Figure 35 Crack and void inside the the welded material samples

Optical microscope analysis of grain boundaries

Optical microscope analysis was performed to examine the grain structure for the printed magnesium. Simple polishing of the material surface was insufficient, so an etching agent had to be introduced. It was found that a Nital solution was required to achieve the desired results. The material was very sensitive to this etching agent, so etching had to be done by submerging the sample surface for no more than 10 seconds during the etching process. The following figures show the results of this analysis. Figure 37 shows that the grains are roughly 10 μm in diameter. Due to the small weld wire, the pre-weld microstructure can't be determined. However, Rajakumar et al did a similar analysis of weld joints when joining two AZ61a plates. In this analysis they found that the AZ61a base material had a diameter of about 38 μm . Our MRML samples show significant grain refinement, as the grain size is reduced by close to a factor of 4. This is most likely due to the relatively rapid cooling process. Since only one bead at a time is deposited, the bead loses its heat fast due to conduction to the surrounding material and due to the forced convection by the externally mounted fan. The presence of β -phase precipitates is prominently present in our images as well.

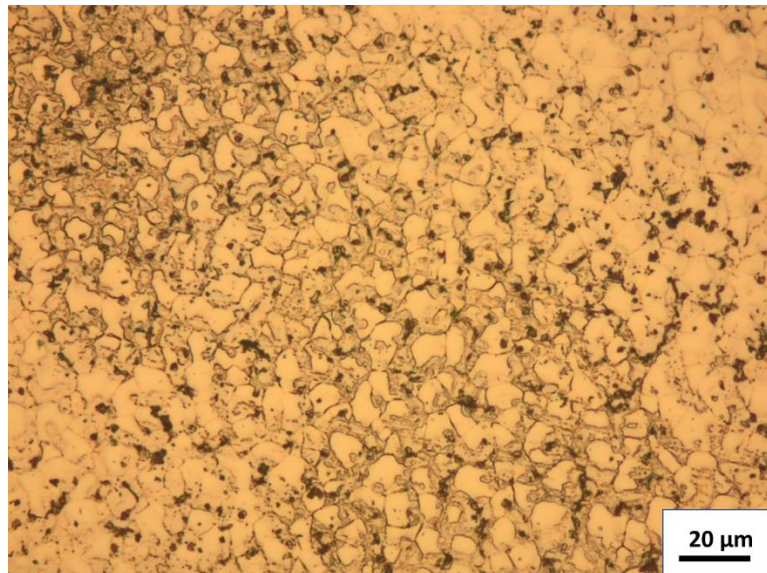


Figure 36 Optical micrograph of welded material – 50x

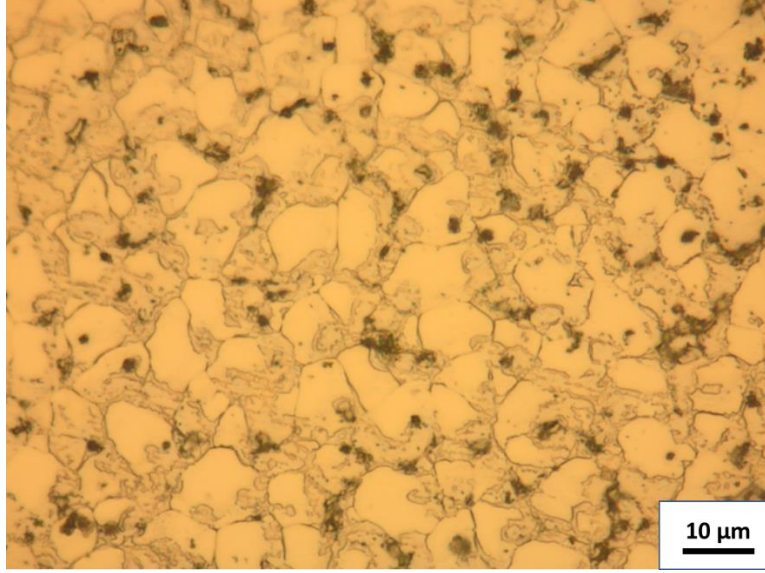


Figure 37 Optical micrograph of welded material – 100x

Conclusion

Material properties and data on microstructure of welded multilayer magnesium blocks were investigated. Additional tests are currently being performed utilizing a different magnesium alloy, AZ91, and a different welding wire diameter, 1.6 mm. The final results will be submitted upon completion of the work.

Multiphysics GMAW Simulations

Modelling of the GMAW process was another project objective. Instead of interweaving the simulation research with the experimental research findings, the simulation work is presented in the following section.

Initial heat source modeling

Modelling of the welding process is an inherently complicated process as it involves multiple physics aspects, multiple heat transfer aspects, as well as a unique material deposition method. It is in general not something that lends itself to three-dimensional modelling. Furthermore, the deposition process and cyclical thermal loads that are applied to old and new layers/elements make this modelling process an even greater challenge. The material deposition in MIG welding is not via a continuous stream of material being deposited onto a substrate, such as in direct energy deposition (DED). Instead, the filler material drops at random time steps from the tip of the weld wire onto the substrate. The substrate is heated up with the welding arc so that the weld droplets can fuse with it. COMSOL multi-physics is used to setup a coupled model that can calculate the heat transfer and track the thermal history of the part so that the residual stresses in the part can be determined. The information in this section reflects the steps that were taken in year 1 to model the heat source for this simulation.

The moving heat source for this simulation is based on the Goldak double-ellipsoid model, the governing equations of which are shown in equations 1 and 2.

$$f_1 = \frac{6\sqrt{3}f_f Q}{abc_f \pi \sqrt{\pi}} \exp\left(\frac{(z-z_{focus})^2}{c_f^2} + \frac{(x-x_{focus})^2}{a^2} + \frac{(y-y_{focus})^2}{b^2}\right) \quad \text{Eqn. 1}$$

$$f_2 = \frac{6\sqrt{3}f_r Q}{abc_r \pi \sqrt{\pi}} \exp\left(\frac{(z-z_{focus})^2}{c_r^2} + \frac{(x-x_{focus})^2}{a^2} + \frac{(y-y_{focus})^2}{b^2}\right) \quad \text{Eqn. 2}$$

Q represents the power input of the heat source, which is determined by welding current times voltage. The model is split up into two halves, one in front of the ellipsoid center and one behind it. f_f and f_r represent the fractional factors of the deposited heat. The constraint $f_f + f_r = 2$ must be maintained. The ellipsoid gets its shape from the constants a , b , c_r , and c_f . x_{focus} , y_{focus} , and z_{focus} represent the center of the double ellipsoid as it moves along the weld path. The weld path in our simulations is along the z direction. Therefore, x_{focus} and y_{focus} remain constant at the surface of the plate, while z_{focus} moves at the feed speed of the CNC torch, which is 500 mm/min. The power input for our simulation was based on the welding conditions observed in our low energy input experiment, and was set to 1900 W. For our simulation, given the observed welding behavior during our experimental work, the overall width of the horizontal ellipsoid is set to 8 mm ($a = 4$ mm) while the penetration depth is set to 4 mm as well ($b = 4$ mm). Figure 38 displays the heat source shape and input when simulated in COMSOL.

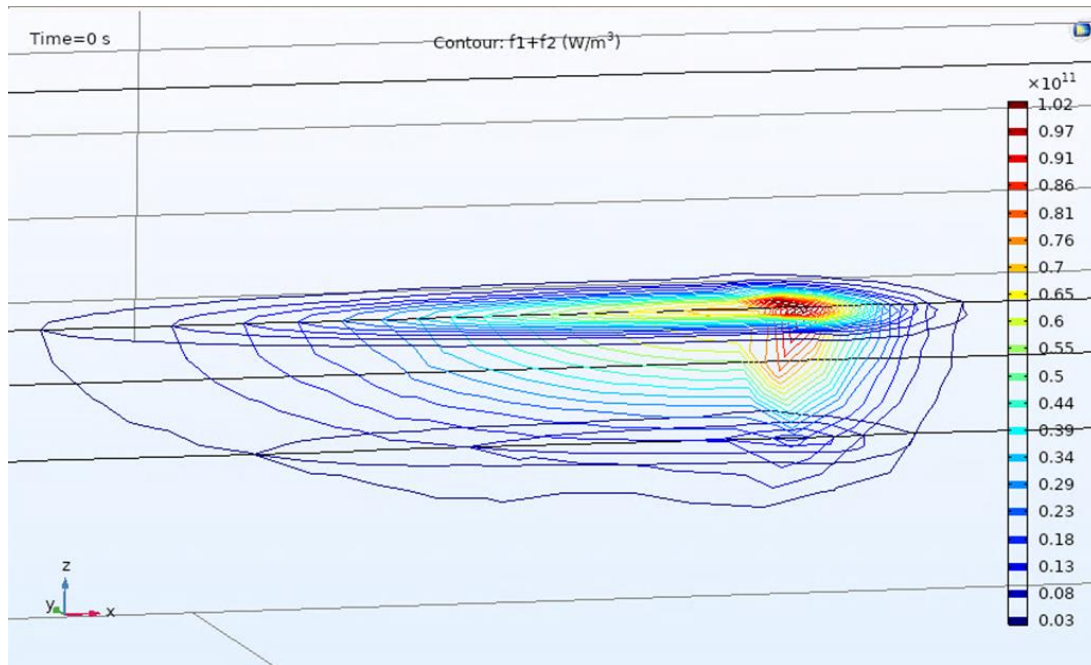


Figure 38 Goldak double ellipsoid heat source in COMSOL

A simple model tracking the heat input during the deposition of one bead of weld wire onto a substrate was created. The substrate and weld bead were given dimensions identical to those used in our experiments. The substrate dimensions, in imperial units, are 12 in x 12 in x ¼ in. The weld bead has a length of 170 mm, a width of 8 mm and a height of 4 mm. A symmetry plane in heat transfer and solid mechanics is used to reduce the number of elements. The model was meshed with 5 different zones, as shown in figure 39. Each of the zones implement a free tetrahedral mesh. Zone 1 and Zone 2 include the weld bead fusion, and the heat affected zone of the substrate. Both have a maximum element size of 1.5 mm to fully capture the implemented heat source. These zones are blended into the surrounding zones meshes. Zones 3 and 4 are the areas surrounding the heat affected zones and have a maximum element size of 8 mm. Zone 5, which is also the zone for the system constraints, has a maximum element size of 3 cm.

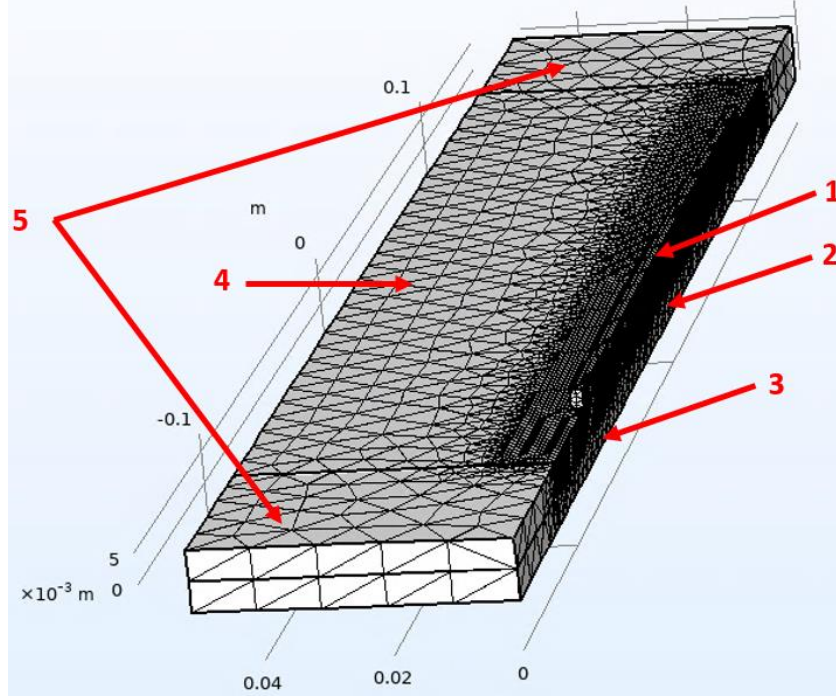


Figure 39 Substrate and weld bead computational mesh setup

Heat Transfer model setup

The time dependent model solves for heat transfer in the system using equations 3 and 4.

$$\rho C_p \frac{\partial T}{\partial t} + \rho C_p \mathbf{u} \cdot \nabla T + \nabla \cdot \mathbf{q} = Q + Q_{ted} \quad \text{Eqn. 3}$$

$$\mathbf{q} = -k \nabla T \quad \text{Eqn. 4}$$

where ρ is the density, C_p is the specific heat capacity, T is temperature, Q is the heat source, Q_{ted} is thermoelastic damping, and k is the thermal conduction coefficient. The entire part is assigned an initial temperature of 293.15 K. The sides and top surface, with exception of zone 5, are set to both surface-to-ambient radiation and convective heat transfer.

Solid Mechanics model

This model is coupled to the heat transfer problem. The bead material, Zone 1, is activated step by step as a function of the z_{focus} of the heat source model using the activation function $z_{focus} > centroid(z)$. The bottom of the substrate has a prescribed displacement of $v > 0$, as it could not warp through the enclosure baseplate. The rotation of the bottom face is also fixed about the x and z axis. Lastly, the top of Zone 5 has a fixed constraint as it is held in place by the aluminum clamps. The COMSOL equations solved in the solid mechanics module are shown below in equations 5 through 9.

$$0 = \nabla \cdot (FS)^T + \mathbf{F}v, \quad F = \nabla \mathbf{u} \quad \text{Eqn. 5}$$

$$s = s_{ad} + J_i F_{inel}^l (\mathbf{C} : \epsilon_{el}) F_{inel}^T, \quad \epsilon_{el} = \frac{1}{2} (F_{el}^T F_{el} - I), \quad F_{el} = F F_{inel}^T \quad \text{Eqn. 6}$$

$$\epsilon = \frac{1}{2} [(\nabla \mathbf{u})^T + \nabla \mathbf{u} + (\nabla \mathbf{u})^T \nabla \mathbf{u}] \quad \text{Eqn. 7}$$

$$\mathbf{C} = \mathbf{C}(E, v) \quad \text{Eqn. 8}$$

Initial Model Results

The initial program simulated 20 seconds of welding, capturing the entire path of the heat source along the bead. Figures 40 and 41 depict the heat source half way through the weld, at $t = 10$ s, in the xy-plane and zy-plane, respectively.

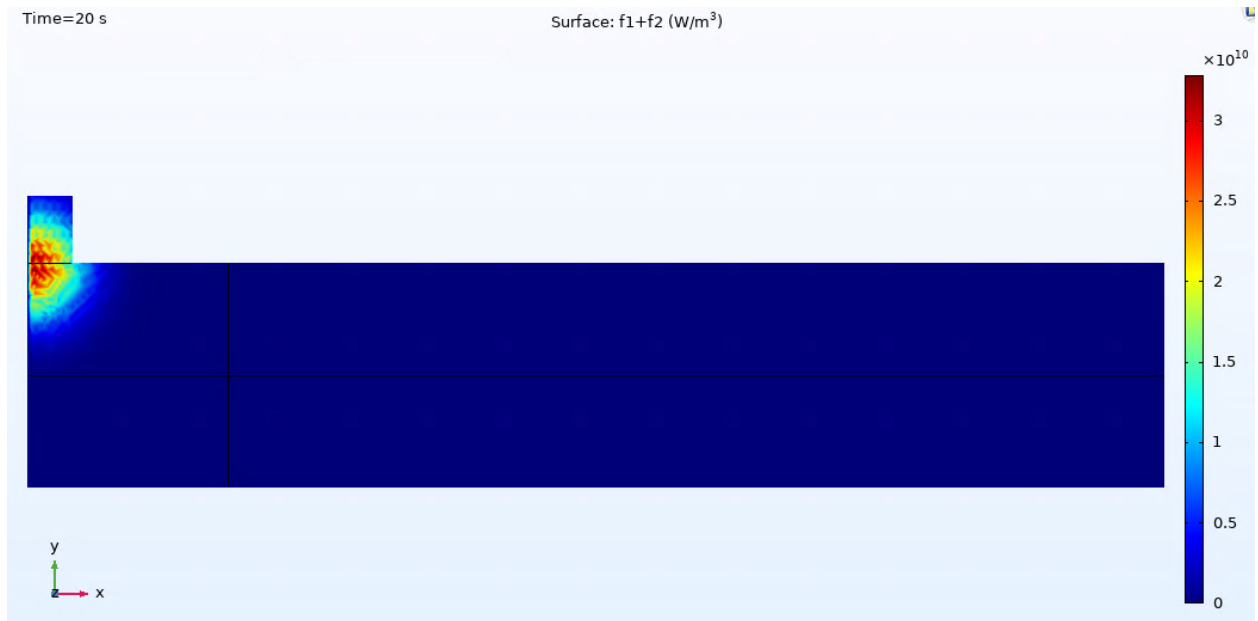


Figure 40 Goldak heat source at $t = 10$ s in xy-plane

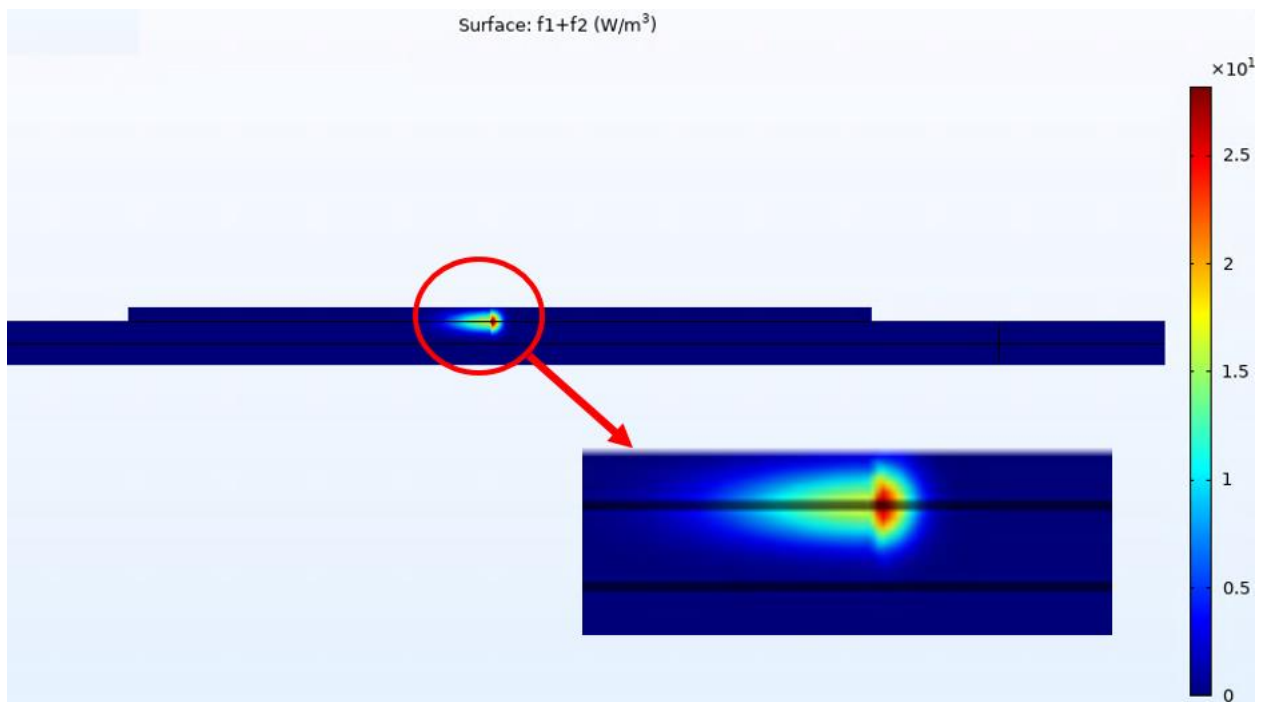


Figure 41 Goldak heat source at $t = 10$ s in zy-plane

It is important to note that in subsequent simulations the heat source will only be applied to the substrate instead of the weld bead itself, as the weld droplets will fall onto the plate after being melted off the weld wire. An attempt was made to simulate this by setting the initial condition to all newly activated elements in the weld bead block to 1,100 K, but this caused numerical instabilities and the solver could not compute the results. A solution to this problem could be adding an additional heat source function that would be only active for a range of newly activated elements, which would rapidly increase their temperature to the estimated weld droplet temperature before removing the heat source again. This heat source would move along with z_{focus} , but instead of being one point in space it would be a $\pm$ range around z_{focus} .

Figure 42 and Figure 43 show the temperature of the bead and substrate at $t = 20$ seconds in the xy and zy planes, respectively. These images confirm that this low energy input has a relatively small penetration depth of only a few millimeters. The temperature at the edges of the plate is between 300 K and 400 K, which is also in line with experimental observations.

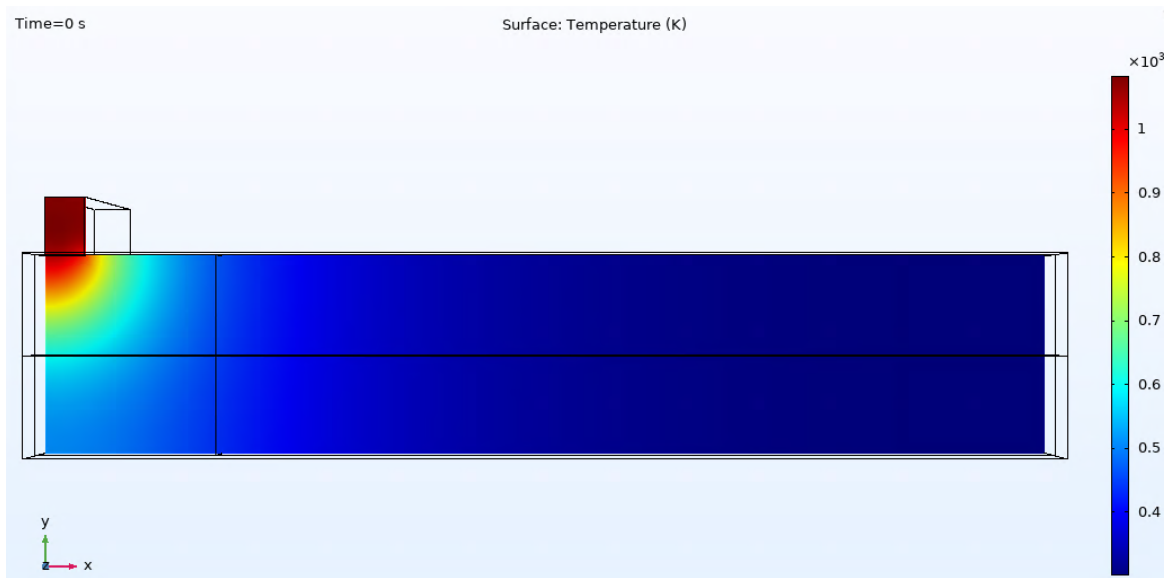


Figure 42 Structure temperature in xy-plane at $t = 20$ s

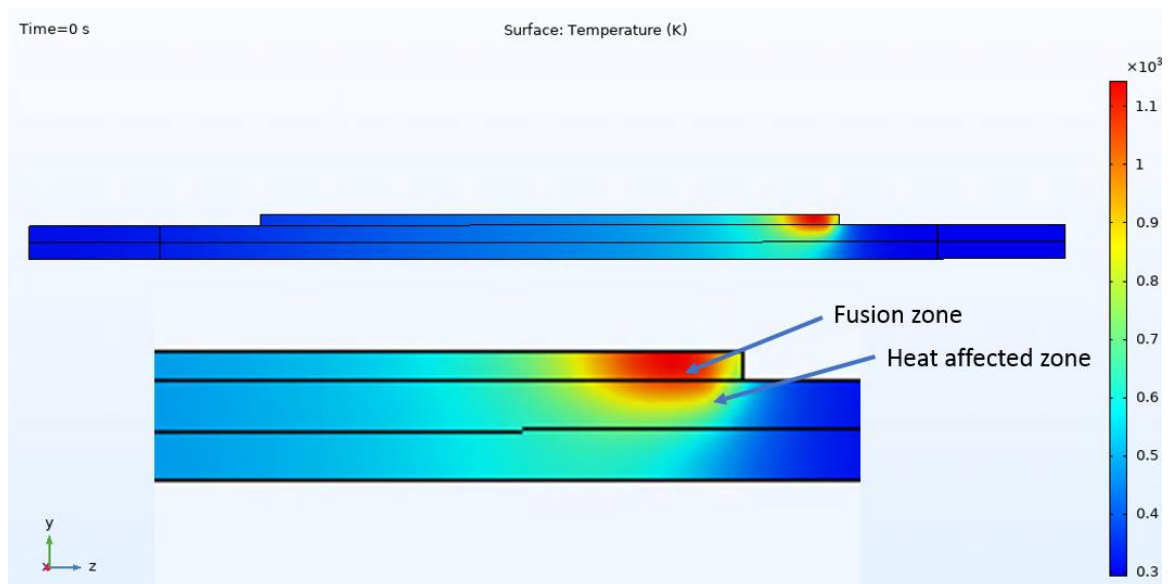


Figure 43 Structure temperature in zy-plane at $t = 20$ s

Displacement of the plate was also computed and is presented in figure 44. Displacement in the y direction was of the highest magnitude and is therefore displayed here. This displacement is at the time of the end of the weld while the sample is still clamped down and has yet to cool. A spring back analysis will be completed to determine the amount of warping caused by this single bead worth of heat input. The von mises stress at $t = 20$ seconds is depicted in figure 45. The highest amount of stress is located at the bottom of the plate underneath the heat source. At this time, however, a large section of the fusion and heat affected zones are still in a molten state. A cool-down period needs to be implemented to determine any residual stresses. After these initial test simulations a more advanced and elaborate model was created.

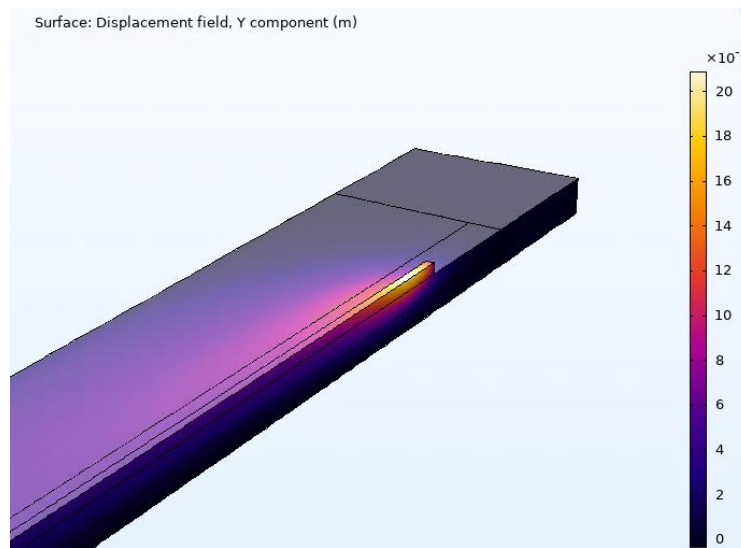


Figure 44 y-displacement at $t = 20$ s

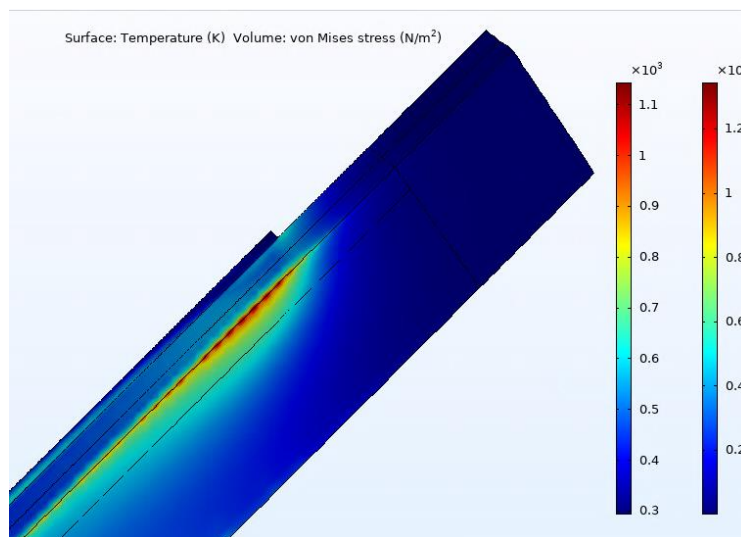


Figure 45 von Mises stress at $t = 20$ s

Multibead GMAW Deposition and Thermal Simulation

Model Geometry

COMSOL Multiphysics was used to develop a model for the additive welding process. The primary goal was to develop a model that would get as close as possible to simulate the deposition process and subsequent heat transfer as it would occur in real life. The FEA model includes a 20 cm x 10 cm x 1 cm substrate onto which 9 beads are deposited. The geometry for the simulation can be seen in figure 46.

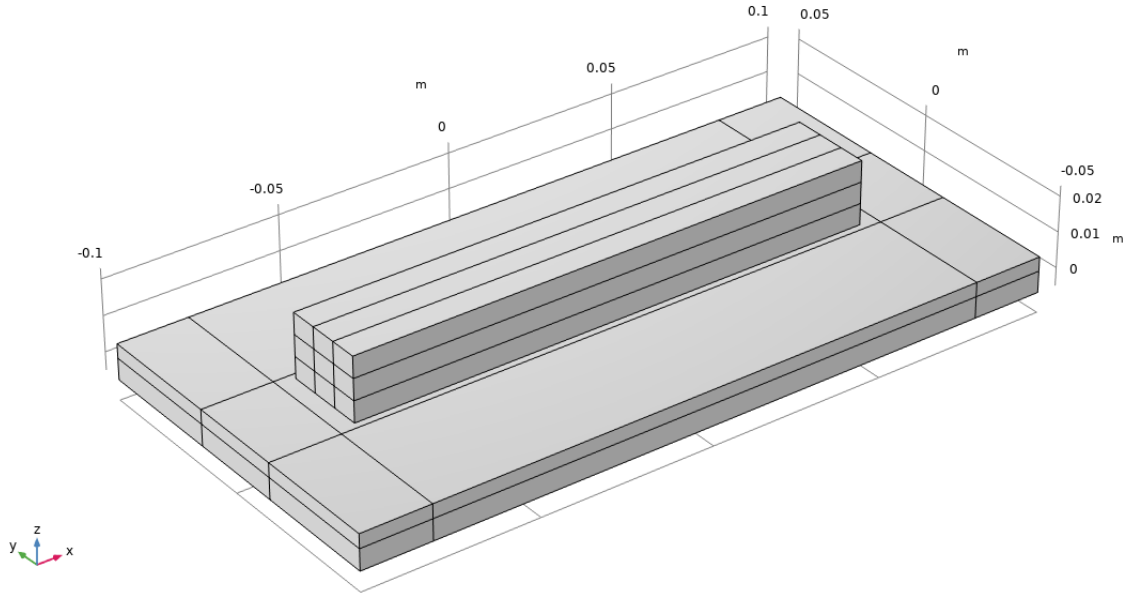


Figure 46 Model Geometry for additive welding operation

The individual beads have a width of 8 mm, a height of 6 mm, and a length of 150 mm. They are deposited with the torch moving in the positive x-direction. Figure 47 show the order in which the beads are deposited. Beads 1, 6 and 7 are deposited at $y = -8$ mm, beads 2, 5 and 8 are deposited at $y = 0$ mm, and beads 3, 4 and 9 are deposited at $y = 8$ mm.

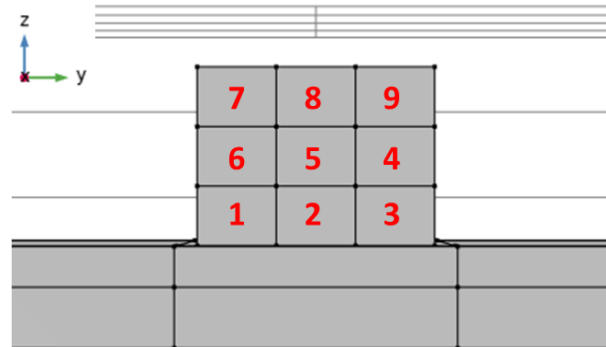


Figure 47 Order of bead deposition

Heat input and element activation setup

The exact same heat input model is used for the heat source in this simulation as in the initial models: the Goldak double-ellipsoid model. The governing equations for the front and the rear halves are represented by equations 1 and 2. The power input is represented by Q and has a magnitude of 2700 W for the simulations. This value was taken from the measured power output of the welder during the deposition

process. As stated in the previous report, the model is split up into two halves, one in front of the ellipsoid center and one behind it. f_f and f_r represent the fractional factors of the deposited heat. The constraint $f_f + f_r = 2$ must be maintained. The ellipsoid gets its shape from the constants a , b , c_r , and c_f . The constant a represents the maximum half-width of the heat source, 4mm, and the constant b represents the maximum penetration depth of the heat source, 4 mm. The length of the front section is represented by c_f , 4 mm, and the length of the rear section is represented by c_r , 16 mm. x_{focus} , y_{focus} , and z_{focus} represent the center of the double ellipsoid as it moves along the weld path. The weld path in this simulation is along the x direction.

The velocity of the heat source, x_{focus} , is 0.0133 m/s, or 800 mm/min, as was input into the welding CNC. The model was set up so that different weld conditions could be simulated by having to change as few parameters as possible. The parameter *bead_time* is calculated by dividing the length of the bead by the velocity of the heat source. The *interpass_time*, which is the parameter that sets the time between bead deposition, can be set to the desired duration. Using these two variables the parameters for the welding start and end times of the beads are automatically calculated. The start and end times of the beads are used in the piecewise functions that compute x_{focus} and y_{focus} . While the y_{focus} piecewise function only switched between the constants that represent the three possible center y-locations of the beads (-8mm, 0 mm, 8 mm), the x_{focus} piecewise function was a simple linear position function of time. For example, the x_{focus} function for bead 2 was $x_{focus2} = -0.075[m] + vel*(t-t_{2_start})$.

The Goldak functions f_1 and f_2 were constrained both in time and space to lower computational costs. The values of f_1 and f_2 were only calculated for each bead during the actual deposition process. Additionally, the computational region for the heat input was also restricted to nodes with coordinates xyz that were within the double ellipsoid volume during the current time step. The Goldak heat input was also restricted to the substrate, or to the previously deposited bead onto which the new bead is being deposited on. This was done to simulate only the heat input caused by the welding arc. The heating/melting of the bead material itself will be discussed later. Lastly, in order to make the simulation results smoother, the heat input was ramped up and ramped down at the beginning and end of each bead. This was done with the addition of a smoothed step function with a transition zone of 100 ms.

Unlike in other additive manufacturing process simulations, such as powder bed fusion simulations for example, depositing material in a welding simulation is not simply a matter of converting already present material in one form into another form (powder into solid). Welding involves the deposition of new material. What was not there before, must suddenly appear, and do so in a physical state representing reality. Several steps were taken to simulate this process as realistically as possible. A bead activation function, *bead_function*, was created to activate the elements as they would appear during deposition. This was done by activating the elements as a function of x_{focus} position, time, and domain. The first few terms of this function are shown in equation 9, with activation of the remaining 7 beads following the same logic.

$$bead_{function} = (X_{focus} > X) * (dom == 13) + (X_{focus_2} > X) * (t > t_{2_start}) * (dom == 16) + \dots \quad \text{Eqn. 9}$$

This function, along with the COMSOL constraint *solid.wasactive*, was used in the Solid Mechanics Element Activation setting. Only domains representing the individual beads were selected for this function. Elements that are not active are simulated as being air, with the proper properties assigned to them. This had to be done because all elements that constitute the final geometry of the part had to be included in the domain and mesh of the model at every time step. Thus, even though the elements are not active, heat transfer is being calculated for them. This made the process more challenging and will be shown in the following section on heat transfer. As soon as the elements are activated, the element properties switch from air to that of the metal being deposited. This, as will be seen, can be challenging as it causes some instabilities in temperature results. The substrate material always remained active.

What remained was the temperature of the newly activated elements. These elements cannot be activated at a temperature that does not represent the temperature of a melted weld droplet. Instead, they should be at a temperature slightly above the melting temperature of the material. The assumption is being made that when the weld wire material is being melted and the droplet is of sufficient size it detaches from the weld wire and falls into the weld pool. The temperature of this droplet is assumed to be slightly above the melting temperature of the material when it hits the weld pool. It is not possible in COMSOL to set an initial temperature condition to newly activated elements during a simulation, which would have been the easiest solution to this problem. Instead, a different function had to be implemented.

The total energy input required to melt each bead is calculated based on the deposited material's mass, specific heat, and temperature change. This energy requirement is then removed from the heat source total that is available to the Goldak heat input. This artificial heat source for melting the beads is applied to a small section of the bead ahead of the center of the Goldak heat source. As such, it is applied only to inactive elements that have the property of air. This general heat source is applied through an if-statement in the COMSOL code represented in equation 10, where Q_{bead} is a general internal heat source to achieve the desired final temperature of the bead material for the moment it gets activated. As can be seen, the heat source only gets to applied to the inactive elements 3 mm ahead of the center of the heat source and keeps the temperature of these elements at 1100 K.

$$if((X < (x_{focus} + 3[mm])) * (x_{focus} < X) * (T < 1100[K]) * (t < (t_{end} - 0.2[s]), Q_{bead}, 0)) \quad \text{Eqn. 10}$$

Figures 48 and 49 illustrate this process as a slice plot through the center of bead 1 during its deposition.

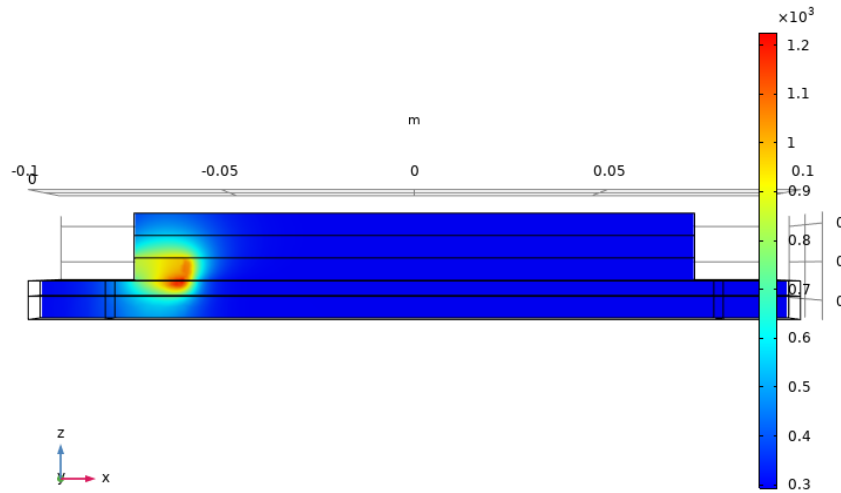


Figure 48 Slice plot of temperature at center of bead 1 during deposition – whole model

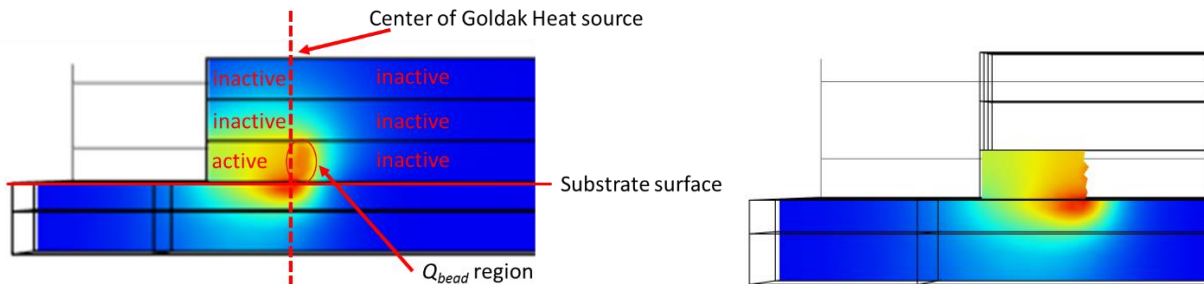


Figure 49 Zoomed in region demonstrating bead heating and element activation

As can be seen in these figures, the bead heat source does not get applied to any of the already active elements. Furthermore, due to the inactive elements having the properties of air, there is virtually no impact that this heat has on the temperature of the active material below it. In the initial 2D simulation tests to determine if this was a valid approach, this artificial heat source was even applied to all inactive elements for the entire duration of bead deposition. It was shown that there was virtually no heat transfer from the preheated inactive elements of air to the solid layers below. This can be seen in figure 50, which shows the temperature distribution of the model towards the end of the bead deposition.

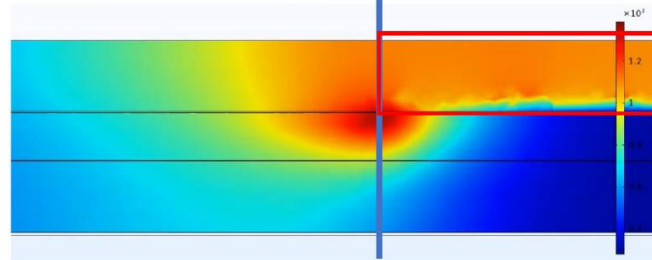


Figure 50 Full bead heating applied during deposition

Heat Transfer setup and mesh

The material properties of the elements were defined based on the active/inactive state of the material. This was done using a series of if statements in the heat transfer setup. For example, to define the material's density, the statement `if(solid.isactive, mat1.def.rho(T), mat2.def.rho_gas(T))` is used. This switches the thermal and material properties of the elements between solid and air depending on the state of the element. It is important to note that this direct switch was possible for all properties without causing any numerical instabilities with the exception of thermal conductivity, k . The thermal conductivity for the solid is in the range of 150 to 180 $\text{Wm}^{-1}\text{K}^{-1}$, while the range for air is between 0.025 to 0.07 $\text{Wm}^{-1}\text{K}^{-1}$. Both are a function of temperature. When using the values for air directly, instabilities in the computation of the temperatures occurs, as can be seen in figure 51. In COMSOL, one can assign measuring probes to domains. A temperature probe for maximum and minimum node temperatures was assigned to each of the domains, which is what is shown in these figures. It is worth noting that these spikes are not the general trend of the temperature distribution and represent only a single node within each of the domains. It was still decided to attempt to reduce these random spikes, especially for the probes measuring the minimum temperatures.

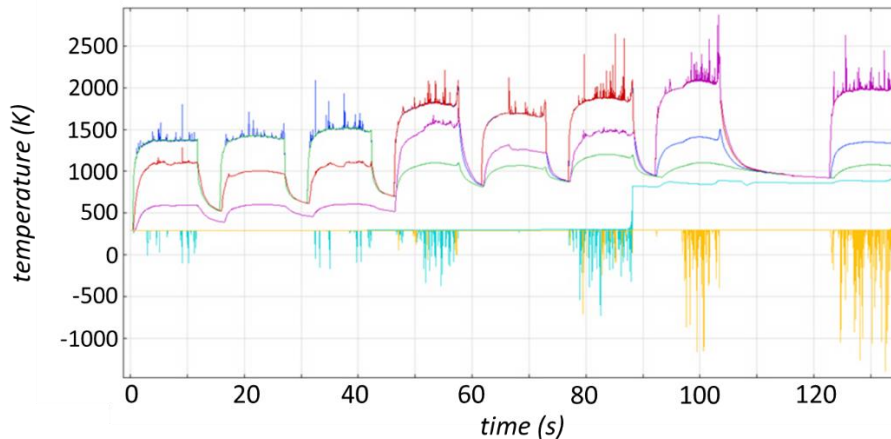


Figure 51 Instabilities in temperature calculations caused by large jumps in thermal conductivity

Minimum bead temperatures reached negative spikes as low as -1400 K, making this model unrealistic. This instability was removed by determining and setting a constant thermal conductivity variable for air of 0.5 $\text{Wm}^{-1}\text{K}^{-1}$. This ensured smooth computation results while still maintaining 3 orders of magnitude

separation between the k for the solid and that of air. As will be shown later, this low thermal conductivity coefficient did not impact the conductive heat transfer through the inactive elements.

For the substrate, two boundary conditions were used. All surfaces that were exposed to the ambient air had a radiation heat transfer and a convection heat transfer boundary condition assigned to them, using a material emissivity of $\varepsilon = 0.1$ for radiation, and a coefficient of $h = 80 \text{ Wm}^{-2}\text{K}^{-1}$ for the convection. This convection coefficient was used to simulate the forced air being applied to the material during the weld interpass windows. The conditions for the individual beads was more complicated. It is not possible to apply boundary conditions in COMSOL to internal boundaries, so other steps had to be taken to simulate heat transfer to the environment as beads were deposited during the simulation. For this, each bead interface had to be assigned a unique heat transfer condition based on time and element activation of itself and the surrounding beads. Figure 52 will be used as an example of how these boundary conditions were applied to the fourth bead.

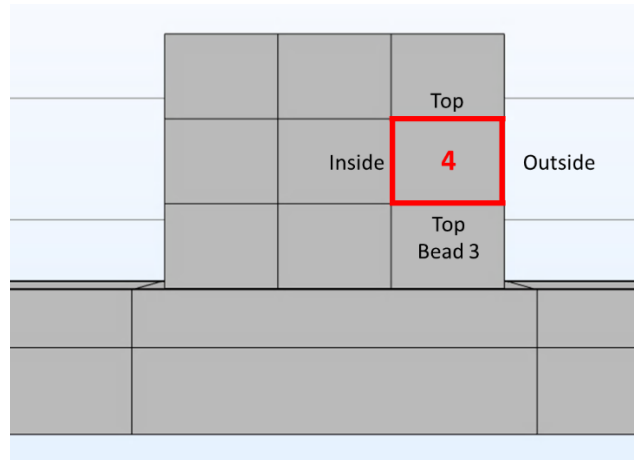


Figure 52 Boundary conditions applied to bead 4

Activation of the outside boundary conditions for bead 4 only required that the elements themselves were active. Both surface-to-ambient (radiation) heat transfer and convective heat transfer were applied to that side of the bead using the requirement *solid.isactive* in COMSOL. The outside boundary will have these boundary conditions for the remainder of the simulation since the outside boundary will always remain exposed to ambient conditions. The inside and top boundaries needed additional requirements. For example, the inside boundary would also have surface-to-ambient and convection applied to it, but only if i.) the solid is active and ii.) the material deposited for bead 5 in the center does not exist yet. This was done by making the application of the boundary conditions dependent on the location of the heat sources for beads 4 and 5. An example of how this was applied to the radiation heat transfer equation for the inside is shown here: $\text{if}((x_{\text{focus5}} \geq X) * (X \leq x_{\text{focus4}}) * (t < t_{5_end}), 0.1, 0)$. The same logic applies to the top boundary of bead 4, but that boundary is dependent of the material deposition of bead 9 as well. The boundary heat transfer for the bottom of bead 4 is included in the top boundary conditions for bead 3 and follows the same logic as that for the top of bead 4.

Heat Transfer simulation Results

The method by which the simulation was set up captured the element deposition and heat transfer of the process. To illustrate the results, the following figures show the heat transfer and element activation during the deposition of several beads.

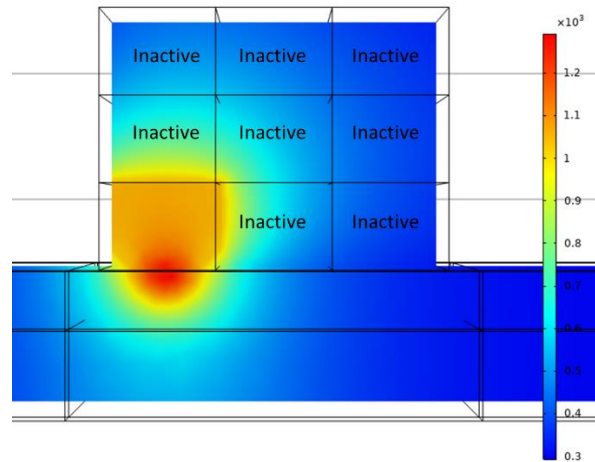


Figure 53 ZY-plane cross-section view of temperature distribution in bead 1

Figure 53 shows the temperature distribution about halfway through the deposition of the first bead of the block. The shape of the fusion and heat affected zones can be clearly seen. The temperatures of the newly deposited bead are slightly below that of the heat input supplied by the Goldak heat source, as expected. The heat transfer downward into active elements of the substrate and to the ambient side can be seen as well. At this point in the simulation, only the substrate and portions of the first bead have active elements. The fact that the heat distribution appears symmetric with the COMSOL boundary conditions on the left and the custom inside and top bead boundary conditions to the right and top of the bead, indicates that the specially created conditions work as intended. There is of course a very small amount of conduction being calculated across the inactive beads since the thermal conductivity coefficient could not be set to zero, but the impact this has on the temperature profile was shown to be negligible. The temperatures during this operation show the expected values during deposition for this low heat input GMAW process.

Figures 54 and 55 show the evolution of the material deposition and depict the state of the process when it has reached bead 7 of the top layer.

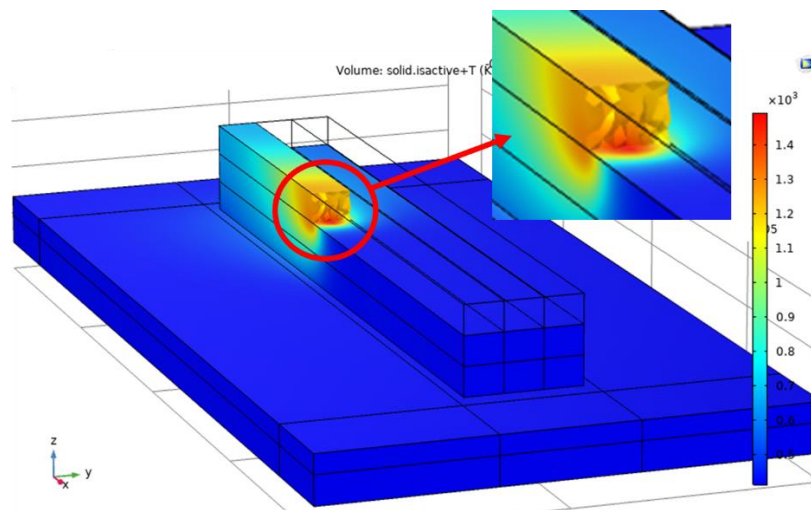


Figure 54 Bead 7 profile and temperature distribution during deposition

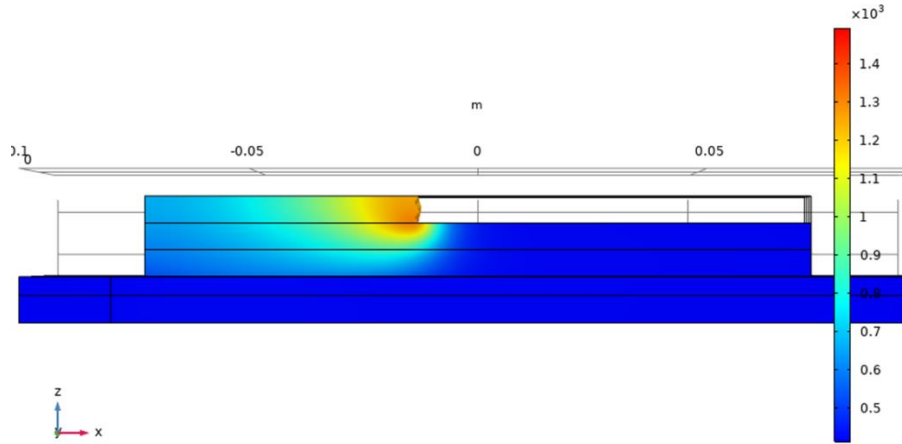


Figure 55 Element Activation and temperature profile of bead 7

These two figures show how the deposition and temperature distribution continue to resemble the expected results for this process. It is important to note that the interpass time for this simulation was set to only 60 seconds to reduce the duration of the simulation. For real world applications, the interpass time must be at least three to four times that value to avoid overheating of the material. Sufficient time must be given so that the input heat has time to leave the material. If interpass times are too short, the material will melt too rapidly and potentially ignite. The reader will have observed that temperatures up to 1000 K are present in figure 55 at points all the way down to the bottom bead layer, which would not be the case if proper interpass durations were applied. These results do present an additional benefit of this simulation. The simulation can now be used to determine the best interpass duration that would allow the material to return to the desired starting temperature for continued deposition. This would eliminate the need for excessive experimental testing and save time and money spend on material.

As stated above, the computational time for this model is considerable. The results shown in this report are from a model that took 16 hours to run. This is in large part due to slow server issues, but also due to the very fine mesh required for this process. Figure 56 shows the mesh that was used in this simulation.

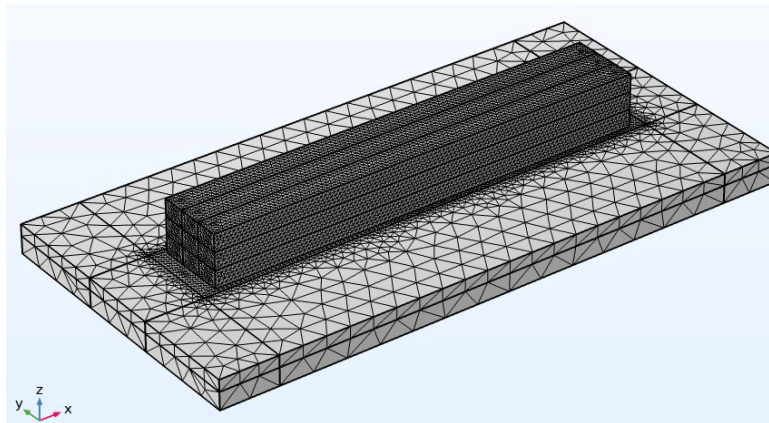


Figure 56 Simulation mesh

The Goldak heat source has a very small volume and it was important to capture as much of the heat source as possible. This meant that the mesh for the beads and for the part of the substrate that deposition was on had to be very small. A maximum element size of 1.5 mm was used for the Goldak input domains. The remaining substrate could be meshed with significantly larger elements.

As in the previous simulation, probes were used again to measure the maximum and minimum node temperatures in all the domains of the model. Figure 57 shows these results for the simulations. In this plot, bead 5 was omitted to show the rate of the temperature decrease more clearly during the interpass windows. As can be seen, even with a 130 second cool down window, the maximum temperature in the beads is still 400 K and even the minimum temperatures are still above 300 K. It takes close to four minutes until the bead temperatures are back to the ambient temperature of 293.15 K. This was also the case during the experimental part of the project, where cool down took several minutes depending on interpass duration between previously deposited beads. Bead 4 in the simulation showed a particularly large jump in maximum temperature due to it being deposited directly on top of the previous bead, which has not had enough time to cool down.

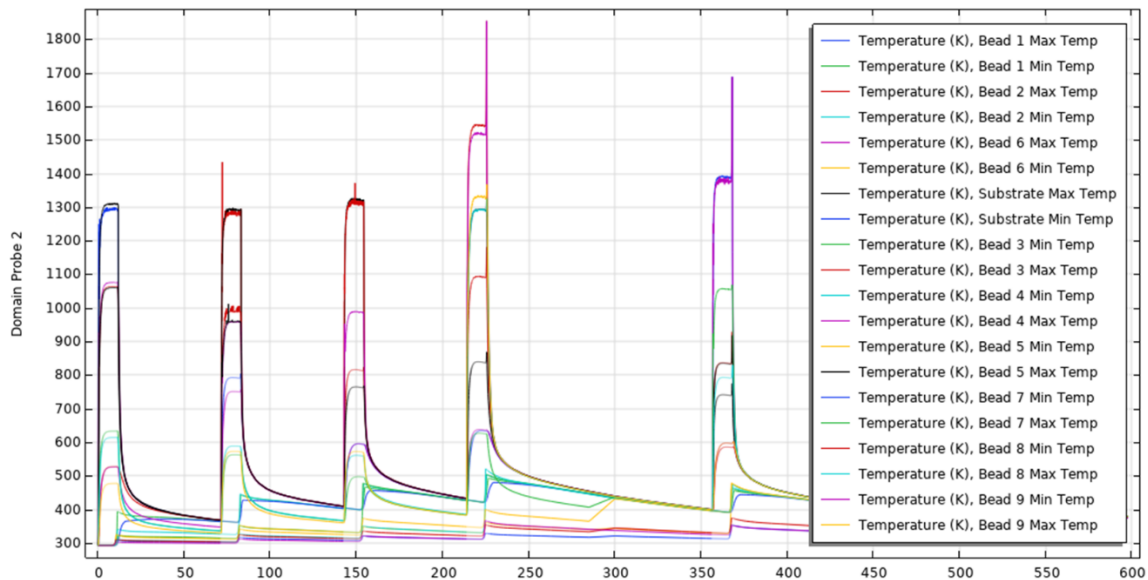


Figure 57 Maximum and minimum temperature probes for each time step of the simulation

This setup did remove the big drops in minimum temperature of the previous simulation, which dropped as low as 1400 K. However, now there are still spikes at the end of each bead deposition phase. This is because as the heat source reaches the end of the bead, the boundary condition there does not allow the heat to be removed at a rate fast enough, causing a sudden build up of energy in the inactive elements. A better view of this is depicted in figure 58 where it can be clearly seen how the temperatures goes up at an increasing rate toward the end of the bead. This could be removed by adding a time dependent heat flux at the back of the welded block that allows the excess heat to be removed more rapidly as the Goldak heat source approaches the end of the bead. One could also edit the ramp down rate of the Goldak heat source and/or the bead heating source. However, since only a few inactive elements were affected by this phenomenon, there has been no attempt made as of now to try and remove these spikes.

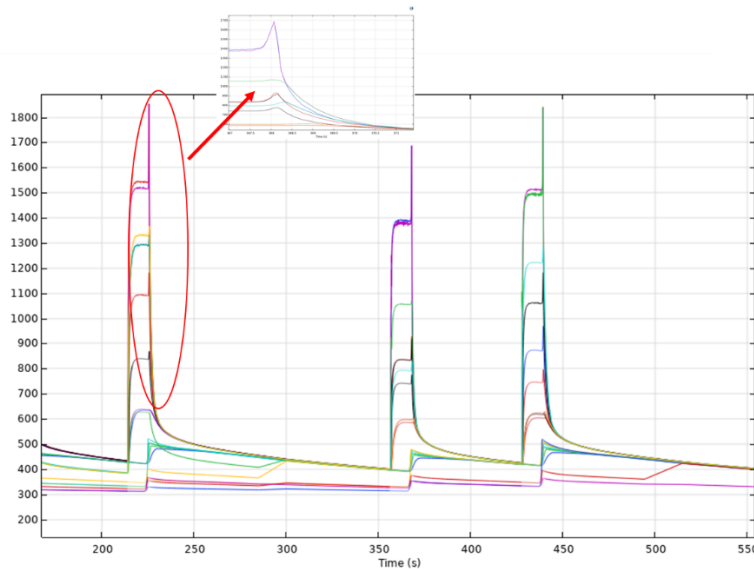


Figure 58 Maximum temperature spikes at the end of bead deposition

Heat transfer and deposition simulation conclusion

The material deposition and heat transfer has been successfully modeled. Minor temperature spikes remain in a few elements, but these can be readily removed in either post processing or through the addition of an artificial heat sink at the end of the welded block. These solutions will now be used in a structural mechanics simulation that uses the simulated heat transfer results and the COMSOL Thermal Expansion coupling module in order to determine the residual stresses in the part. Even though the substrate in the physical experiment was clamped on both ends during the manufacturing process, the simulation will fix only one side of the substrate. As shown in the experimental section, the warping of the substrate is substantial during this process. This simulated clamping condition would allow the substrate to deform unrestricted over time due to material addition and heat input. Another possibility is to apply the clamping condition as is, run a structural mechanics simulation, and then use the results of that simulation as inputs to a third simulation: a spring back simulation in which one of the clamps is incrementally removed. This work will be completed as part of my PhD dissertation, and will be submitted to the Army Research Office as soon as the models have been completed.

Final Remarks

As stated in previous sections, some research tasks could not be completed because of lost time due to a multitude of reasons, including faculty changes, project assignment changes, supply chain / order delays, covid-impacts, and more. However, these tasks are currently still being worked on and will be completed within the year. The idea of using GMAW for AM of magnesium shows great promise but requires the design/build of components dedicated to working with this material. The fact that reasonable results were attainable at all with only commercially available welders with hard presets for other materials already shows that this is doable. If all these equipment obstacles were removed, the final results would be of great value.

