MSE SENIOR DESIGN CAPSTONE PROJECT PROPOSAL

PROCESSING METALLIC GLASS FOR ADDITIVELY MANUFACTURED ALUMINUM METAL MATRIX COMPOSITES

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1 Introduction

1.1 Problem Statement

Metal matrix composites have become increasingly popular in the aerospace, automotive, and sport industries due to their light weight and enhanced strength. More specifically, metallic glass as a reinforcement phase has become increasingly popular because of its potential to provide better properties compared to conventional ceramic reinforcements. If a reliable process of making metal matrix composites with metallic glass reinforcements can be created, the aerospace and automotive industries will benefit through weight reduction and better fuel consumption of their vehicles. More specifically, the aerospace and automotive industry are heavily involved in light-weighting their vehicles through two methods: using high performance materials such as composites, and optimizing structures using computational engineering approaches and additive manufacturing [1]. For this project, both additive manufacturing and composites will be utilized by developing a new processing route to create metal matrix composites reinforced with metallic glass through powder metallurgy and additive friction stir deposition.

1.2 Goals and Objectives

The goal of this project is to successfully process and characterize a metal matrix composite reinforced with metallic glass using additive friction stir deposition. More specifically, the objectives of this project are threefold:

1. Devise a ball milling regime that will create homogeneous amorphous metal powder and reduce the glassy metal to an appropriate size.
2. Homogeneously mix the powder into a metal matrix composite specimen as a reinforcement phase through additive friction stir deposition.
3. Optimize mechanical properties of the metal matrix composite by adjusting volume percent of the reinforcement phase and rpms of the additive friction stir deposition machine.

1.3 Deliverables

The primary deliverable will be a detailed report of the findings and an identification of future research directions. Because this project is a preliminary research endeavor, additional research directions will be important for forthcoming teams working with metallic glass. In summary, this project will include the following deliverables:

- A detailed process to make amorphous metal through available powder metallurgy equipment at Virginia Tech
- Additive friction stir deposition processed metal matrix composites reinforced with metallic glass particulates
- A detailed report including characterization of the composite’s properties and recommendations for further research directions

2 Motivations and Contributions to Society

The primary motivation is to directly contribute to the advancement of materials science by establishing a reliable process to produce additively fabricated metallic glass reinforced metal matrix composites. Through distillation of current research into a reliable plan, we hope to foster interest, growth, and support of the applications for metallic glass and additive manufacturing.

2.1 Social Impact

Through the increased use of additive manufacturing, the possibility of part production domestically grows by reducing international sourcing and transport of products. This could have effects on the geopolitical stage as major production hubs, such as China or India, find competition from interior production. The process of design sharing, as opposed to part importation, becomes more feasible in the metals and composites industry. The widespread use of additive manufacturing, not only for metallic glass composites, could lead to greater restrictions and management of information sharing and sales [2].

2.2 Environmental Impact

Additive manufacturing reduces both waste and energy requirements, but still accommodates parts with complex geometries. Through the avenue of additive manufacturing of high strength metal parts on site, the energy used, and greenhouse gases released during transport from production could greatly lower the environmental toll of current industry practices [3].

Additionally, the amount of land taken up by high volume production lines will be reduced as producers shift to space-efficient automated additive manufacturing methods. This shift will result in the reduction of deforestation as well as pollutant production. However, as additive manufacturing technology for polymers has increased in popularity, the concerns surrounding the release of volatile organic compounds (VOCs) have heightened, bringing about various studies into the potential hazards surrounding this production method [4]. As our specific production avenue increases in visibility, alongside metallic additive manufacturing, the dangers pertaining to inhalation of micron level particulates will also become a greater concern and lead to increased regulations for operation and safety [5].

2.3 Economic Impact

Using metallic glass synthesis and additive manufacturing, the time and energy required to create a finished part could be greatly reduced, as manufacturers will no longer have the economic burden of importing vast quantities of raw material or finished parts. Reliance on foreign suppliers will be increasingly lowered, decreasing the amount of capital needed to manage importation and relocation of parts and materials. China, which currently leads the world in part manufacturing for industries such as automobiles, will experience decreased demand causing shifts of pricing in terms of production and shipping [6]. Small scale business models will become more manageable, with more possibilities of innovation and manufacturing pathways. The reinforced metal will have more strengthening mechanisms to allow improvement in areas such as the aerospace, automotive, and sports industry. Ultimately, the growth of this technology will contribute to increased global productivity and has potential to foster decentralized, cost effective production lines [2].

3 Literature Review

This section includes a synthesis of three major subjects that pertain to this project: metallic glass, metal matrix composites, and additive friction stir deposition. To be coherent, the materials science of metallic glass will be discussed first as it is the first benchmark of this project.

3.1 Materials Science of Metallic Glass

3.1.1 Overview

Glass is defined as being an amorphous solid lacking any long-range order. Typically, metals contain long range order in the form of crystal structures such as face centered cubic. This widely accepted characteristic of metals was challenged 60 years ago at California Tech after the first metallic glass was formed during an experiment involving the gun quenching of a gold-silicon binary alloy [1]. Since then, amorphous metal has been determined to have unique properties including high hardness and strength with measured yield strengths reaching about 2 GPa, high elastic limits of about 2%, high corrosion resistance, low coefficient of friction, and good soft magnetic properties [7] [8].

Although metallic glass has these properties, their capabilities are limited by their thermal instability. Until recently, only thin ribbons of metallic glass could be made due to the high cooling rates needed. But more research has led to the development of bulk metallic glasses (BMG), which are characterized as having one dimension equal to or greater than one centimeter [7]. To further understand how to form BMGs, the thermodynamics and kinetics of glass formation must be discussed.

3.1.2 Thermodynamics and Kinetics of Glass Formation

Table 1 of the Appendix shows important terms that will be used in the following section.

The composition of the alloys greatly determines their glass forming ability. Alloys close to deep eutectics of quaternary systems have shown the most success in forming BMGs because they are less likely to form intermetallics [9]. To further optimize an alloy's glass forming ability, the reduced glass transition temperature needs to be maximized. When the reduced glass temperature is increased, the critical cooling rate decreases as shown in Fig. 1.

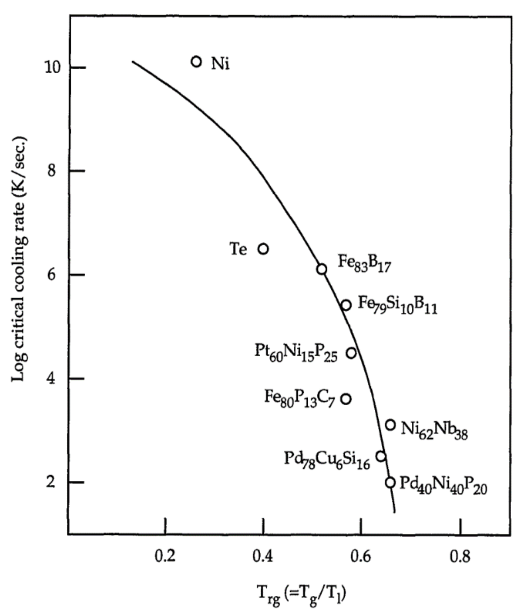

Fig. 1: Calculated cooling rates for glass formation of several alloys plotted against reduced glass transition temperature [7]

Using these parameters, BMG forming alloys such as $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10}Be_{22.5}$ were optimized to obtain a near maximum T_{rg} of 0.67 and a resulting critical cooling rate of 1 K/s [7]. Because of its incredibly small critical cooling rate, this alloy can be amorphized from the liquid state by simply quenching it in water.

Although this proves that BMGs can be formed without the need for complex rapid solidification techniques, problems still arise in the need for further processing. This project requires metallic glass in the form of powders, not bulk materials. Luckily, it has been determined that metallic glasses can be formed through mechanical alloying.

3.1.3 Mechanical Alloying of Metallic Glass

Mechanical alloying involves repeatedly cold welding and fracturing a metal powder mixture in a high energy ball mill. When specific powder compositions are mechanically alloyed for a specific amount of time, amorphous metals can be produced. This is ideal for this project because the goal is to reinforce aluminum with glassy particles. Thus, powder metallurgy is the most efficient process for this project as the metallic glass will already be in particulate form when created. This technique is also beneficial due to the low operating temperatures which reduces the effects of oxygen on the metallic powder. And because the operating temperatures are low, the process the alloys take to become glass differs greatly from the liquid processing routes.

The amorphization process is complex and is still not certain; however, Fig. 2 and Table 2 give a possible description of the amorphization process for a Zr-Al-Cu-Ni alloy.

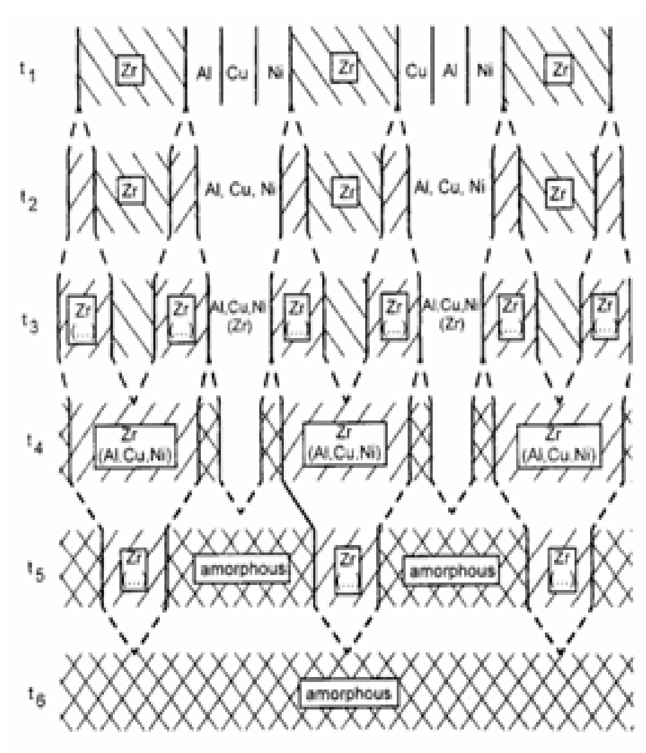

Fig. 2: Amorphization Process of Zr-Al-Cu-Ni Alloy During Mechanical Alloying [10]

Note that each section of Fig 2. refers to the microstructure of a single metal particle.

Table 2: Description of Amorphization of a Mechanically Alloyed Zr-Al-Cu-Ni Alloy

Time	Microstructure Description
t_1	Powder particles form a layered structure of all the constituents through continuously cold welding and fracturing.
t_2	Solid solutions of Al, Cu, and Ni form due to the negative heat of mixing and combined pipe diffusion along the cores of dislocations created during the impact of the milling balls [10].
t_3	The atoms of the smaller elements begin to diffuse into the larger zirconium atoms through bulk diffusion [10].
t_4	Mixing of the elements leads to a largely supersaturated solid solution as intermediate reaction products [10].
t_5	An amorphous phase begins to form starting at the grain boundaries and interfaces. This is caused by supersaturated layers being heavily deformed during a collision event which leads to regions along interfaces having excess chemical energy with respect to the formation of an amorphous phase [10].
t_6	The amorphous phase spreads until the powder becomes fully amorphous

The amorphization process discussed in Table 2 is a time dependent process, with total milling energy being the primary factor. Increasing the total milling energy can be done by either increasing the ball to powder ratio or by increasing the rpms of the ball mill. Another interesting characteristic of the amorphization process, shown in Fig. 3, shows that increasing the total energy of milling leads to powders of different compositions having a gradually more similar time to amorphization.

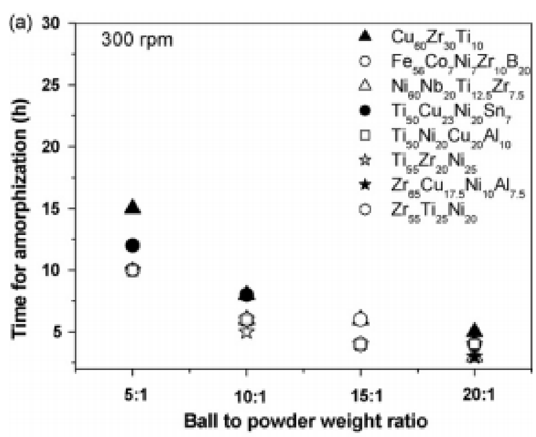

Fig. 3: Time required for amorphization for several powder compositions at 10:1 ball to powder ratio for different rpms [11].

This common minimum time to amorphization may be due to the structure of amorphous phases being like each other for the BMG forming compositions. The minimum time can also be used during the project as a baseline to check the glass formability of the composition chosen.

The last behavior of importance is the existence of an energy window. Fig. 4 shows the energy window of a Zr-based BMG forming alloy.

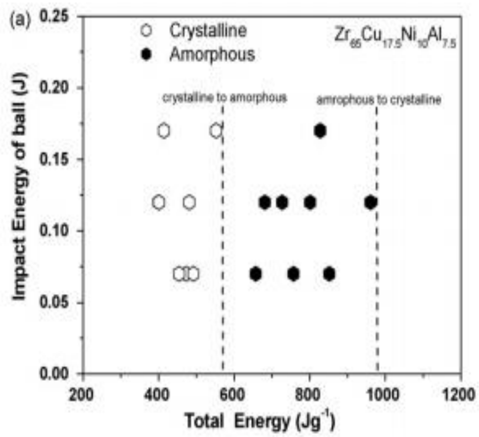

Fig. 4: Impact Energy of Ball Versus the Total Energy of Milling with Energy Window Circumscribed [11]

If the parameters of the milling process lead to a total energy of milling outside of the energy window, a crystalline powder will be the result. Therefore, during the project careful consideration of total energy of milling will be done. This consideration will be discussed more in-depth in the methodology section.

Next, the interactions of the metallic glass powder and a crystalline metal matrix will be discussed.

3.2 Metal Matrix Composites Reinforced with Glassy Particulates

3.2.1 Overview

Metal matrix composites (MMC), especially aluminum based metal matrix composites, are becoming increasingly more popular in the aerospace, automotive, sports, and electronics industries due to their light weight and enhanced mechanical properties [8]. Metal matrices can be reinforced with particles or fibers, but only particulate reinforced MMCs will be discussed because this project will only deal with particulate reinforced MMCs. Usually, micron-sized ceramic powders are used, but the interfaces between the powders and the metal matrix are not ideal due to the differences in thermal expansion coefficients [8]. Recently, it has been postulated that metallic glass powders could be an alternative to ceramics and would have better interfaces with the matrix because both the reinforcement and matrix constituents possess metallic bonding and similar thermal properties [8].

3.2.2 Properties of MMCs

Because particle reinforced MMCs have a homogenous distribution of metallic glass powders, the properties of the MMCs will be isotropic. This is ideal because most engineering materials used in the target industries are isotropic. The most important property of the MMCs is their enhanced strength. An example of this increase in strength can be seen when creating a metal composite consisting of aluminum 2024 and an iron-based metallic glass. When a composite containing 60% by volume metallic glass was made, the strength was increased from 97 MPa to 403 MPa while still retaining a fracture deformation strain of 12% [12]. This strengthening phenomenon is a result of several strengthening mechanisms listed below:

- Hall-Petch effect
    - Glass particles act as grain boundary pinners which in turn reduces the grain size of the composite. Reducing the grain size leads to an increase in strength [12].
- Orowan strengthening

  - Particles act as barriers to dislocation motion and deformation which increases the strength [12].
- Enhanced dislocation density
  - High density of dislocations may result from the processing of MMCs due to the thermal mismatch of the glassy particles and metal matrix. Increases in dislocation density causes an increase in strength of the composites [12].

Lastly, because of the uniformly dispersed reinforcement phase, MMCs can be processed using common processing techniques such as hot rolling, hot forging, hot extrusion, and machining [13]. Unconventional processing techniques can also be used such as friction stir processes. Friction stir processes include severely plastically deforming the surface to strengthen the base metal. Hidetoshi Fuji et al. proved that MMCs reinforced with metallic glass particles could be created through friction stir processing by making grooves in the base metal and filling those groves with the glass powders [14]. Once the high-speed rotating cylinder passed over the grooves, the particles homogeneously dispersed into the metal matrix. This process is like additive friction stir deposition, which is the process this project will be implementing. Additive friction stir deposition (AFSD) uses the severe plastic deformation mechanisms of conventional stir processes but adds material in layers which leads to three-dimensional parts. Further details of this process are discussed in the next section.

3.3 Additive Friction Stir Deposition

AFSD is a non-beam additive process that involves feeding material through a rapidly rotating shoulder onto a metal substrate [15]. Once the feed material contacts the substrate, it is heated, softened, and bonded to the substrate through plastic deformation at the interface. This plastic deformation process occurs while the shoulder is moved transversely to make layers of feed material, and by adding subsequent layers on top of each other, three dimensional parts are made [15]. These three-dimensional parts have several beneficial properties that distinguish this additive process from other beam-based ones such as having better microstructures, being faster, having more build capability, being able to use a wide range of materials, and having no melting occur. These capabilities are listed in more detail in Table 4 of the Appendix.

All these characteristics are a good indication that a strong metal matrix composite reinforced with metallic glass particles can be produced. AFSD has already been shown to produce metal matrix composites reinforced by ceramic particles. The main limiting factor for metallic glass particle MMCs is the operating temperature of the process. If the temperatures become too high, the glass particles may recrystallize and lose their attractive properties. To avoid this, an experimentation plan has been devised, with the details of this plan discussed further in the following section.

4 Proposed Methodology

Our experimentation process will consist of two primary phases: forming the metallic glass powder and forming the metal matrix composite for characterization. These two phases are discussed in the following two sections. Furthermore, Fig. 5 of the Appendix shows a graphic depicting the proposed experimental methods.

4.1 Metallic Glass Formation

Before the glass particles can be made, a specific composition needs to be selected. A zirconium-based composition has been selected as the first choice. Specifically, a composition of $Zr_{51}Cu_{20.7}Ni_{12}Al_{16.3}$, also known as Zr51, will be used. This composition is based off of the work done by Shen et al. where they developed the alloy by using three binary eutectic compositions [16]. Eutectic compositions of the Zr-Cu, Zr-Ni, and Zr-Al systems were combined in equal parts to develop this new alloy. This metallic glass system has proven to be more thermally stable than other Zr-based glasses due to the new approach used. Zr51 was measured to have a crystallization temperature of 503°C, and was heated to 400°C for 60 minutes without any crystallization [16]. Because of its high thermal stability, Zr51 will be able to withstand the elevated temperatures of the additive friction stir deposition process.

The only risk using this alloy is it has only been produced using liquid processing routes. To mitigate this risk, we will have a secondary alloy composition of $Zr_{65}Cu_{17.5}Ni_{10}Al_{7.5}$, or Zr65. The benefits to using Zr65 as a second choice is it has proven to form amorphous powders through mechanical alloying, and the elemental powders will already be purchased [11]. However, Zr65 has a crystallization temperature of 431°C which would require changing the parameters of the AFSD process to lower the operating temperature.

Using the chosen powder compositions, a milling regime will be used to make amorphous powder. A high energy ball mill with a ball to powder weight ratio of 15:1 and an rpm of 300 will be used to form the amorphous powders. According to Bhatt et al., using these milling parameters results in a time to amorphization of approximately 5 hours [11]. Stainless steel vials and balls will be used to process the powders, as well as toluene. Toluene will be used to restrict oxidization and to prevent excessive welding [11].

4.1.1 Proof of Concept for Amorphous Powders

A small first batch of powders will be created as a proof of concept. This small batch will be placed into the x-ray diffractometer to prove their amorphous microstructure. Time permitting, this small batch can also be placed into a differential scanning calorimeter to measure its glass transition temperature and crystallization temperatures. Once this proof of concept is produced, larger batches can be made, and the next phase of the project can begin.

4.2 Metal Matrix Composite Processing and Characterization

For the next phase, the amorphous powders will be mixed into a metal matrix through additive friction stir deposition. Aluminum will be used as the matrix material because it has been proven to make reliable parts through the AFSD process.

4.2.1 Proof of Concept for Metal Matrix Composite

A simple and fast way of taking the aluminum matrix and adding the metallic glass powders is to drill holes into the aluminum feed material and then deposit the metallic glass powders into the holes. This process will be used to create a proof of concept MMC. Once this proof of concept MMC is made, its microstructure can be observed in the SEM to validate that the metallic glass dispersed into the aluminum matrix and did not crystallize during the additive process.

4.2.2 Characterization and Optimization of Mechanical Properties

Once the proof of concept is made, a more robust method of creating the MMC can be done. The chosen method of making this composite is to mix aluminum alloy powder with the metallic glass through planetary ball mixing and then consolidate the powders through both cold isostatic pressing (CIP) and sintering. This process will be used to make a feed rod with better dispersed reinforcement particles. This feed material can then be used to fabricate larger parts for microstructure and mechanical analysis.

If there is enough time, a central composite experimental design will be conducted using the rpms of the machine and the volume percent of metallic glass as the factors. A response surface can then be generated for both the strength and ductility of the material. Using these two response surfaces, optimal compositions and processing conditions can be determined depending on the desired strength and ductility of the composite.

5 Project Management Plan

5.1 Scope

The scope of this project is limited to the development of a suitable production method for metallic glass synthesis. This involves obtaining raw material, developing a successful ball milling process, characterizing the amorphous powder, developing a successful additive process, and characterizing the resulting MMC. From this process, deliverables including a detailed report on the findings and methods for producing both amorphous powders and a metal matrix composite will be provided.

5.2 Proposed Schedule

Fig. 6 in the Appendix illustrates a proposed schedule for this project. This schedule is subject to change depending on the rate at which experimentation yields results, or if unknown interruptions occur such as a resurgence of the COVID-19 virus.

5.3 Budget Costs and Procurement

This project will use the $500 given by the Virginia Tech Materials Science and Engineering Department to purchase materials that are not already available in Dr. Yu's labs. This may include bulk metal powder of zirconium, copper, aluminum, and nickel. More expenses may be necessary for the purchase of robust milling media such as tungsten carbide milling balls. More information on possible suppliers and costs of the raw materials of this project is shown in Table 4 of the Appendix.

5.4 Team and Personnel

The primary research team will consist of four undergraduate students: Ariel Lee, David Sanches, Rob Palisin, and Will Blankenship. This research team will be advised by Professor Hang Yu and Joey Griffiths, a graduate student. Outside professional resources for guidance and suggestions will be accepted from Professor Kathy Lu, Professor Carlos Suchicital, Professor Alex Aning, and other personnel from the Materials Science and Engineering department at Virginia Tech. Lastly, the academic advisor to this team will be Professor Thomas Staley.

5.5 Communications and Documentation

It is recognized by this senior design team that communication is necessary for the success of this project. Constant interpersonal communication will be made possible using the messaging app Slack, which will be utilized as both the primary means of organization and recording of interactions. Documents and resources will be shared and maintained in Google Drive, allowing for asynchronous work and information sharing. Quality evaluations will be done as a group, with the primary team member reviewing their own work alongside their teammates. Meetings with the supervisor will be meticulously recorded to allow for reference access and updates to be shared with those who cannot attend. A universally accessible calendar, with predetermined free times for meeting scheduling will be created and updated as changes to personal schedules are made. Lastly, a Gantt chart will be created to better schedule and plan the division of labor between team members, with adjustments to be made in the future as conditions change.

5.6 Facilities and Equipment

Facilities and equipment will be supplied primarily by the Materials Science and Engineering department under the umbrella of the College of Engineering of Virginia Tech. The primary location for this project will be the teaching labs in Randolph. Trainings will be required to work with the necessary lab equipment such as the ball mills, the x-ray diffractometer, and the differential scanning calorimeter. The additive friction stir deposition machine will only be operated by trained graduate students or Virginia Tech faculty and will not require training.

5.7 Risks

The risks involved with this experiment include the following:

- Handling of metal powders
- Working with X-rays
- Working with flammable materials (Toluene)
- Working with high energy ball milling equipment

To mitigate these risks, the proper training will be completed for using equipment that emits radiation, as well as training for ball milling equipment. First, the chemical health and safety training required by all students using the teaching labs will be taken. Further trainings such as powder handling and processing training will be prepared to properly work with the metal powders. The flammable liquids safety training provided by VTEHS will be taken in order to handle toluene. The safety data sheets for each hazardous material used will also be on hand and studied by each team member prior to experimentation. Proper personal protective equipment will be enforced such as long pants, gloves, and protective laboratory eyewear for all users.

5.8 Legal Procedures

Currently, no legal action needs to be taken. To prevent any legal actions, each undergraduate member will undergo all the necessary trainings, and any proprietary work that comes from this project will be handled by Dr. Yu.

5.9 Constraints and Challenges

The main constraints concerning this project is successfully producing amorphous powders through mechanical metallurgy. This has been mitigated by selecting two compositions with one being a novel composition with increased temperature stability, and the other being a backup composition that has proven to be successfully mechanically processed into amorphous powder. Other challenges include unknown obstacles that need to be accounted for, such as a resurgence of the Covid-19 virus. To account for this, the schedule for the project has most of the progress being in the first semester so that the second semester can be used to either optimize the metal matrix composite or to analyze all the data obtained and put together a presentation and report. Limited access to equipment and machines will be a challenge if the fall semester of 2020 were to be online. One potential solution to overcome this situation is to code and run computer simulations on models that this team creates.

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7 Appendix

Table 1: Important Thermodynamic Terms Relating to Metallic Glass Formation

Thermodynamic Term	Definition
Critical cooling rate (T_c)	The cooling rate needed to obtain a metallic glass from the liquid state [7].
Glass transition temperature (T_g)	The temperature at which a liquid glass becomes solid. Is also defined as being the temperature at which the viscosity of the glass equals 10^{13} poise [7].
Freezing temperature (T_m)	The temperature at which the liquid freezes to the equilibrium crystalline phases [7].
Reduced glass transition temperature (T_{rg})	A crucial factor for glass formation and is equal to the glass transition temperature divided by the freezing temperature [7].

Table 3: Distinguishing Capabilities of AFSD and their Corresponding Descriptions [15]

Capability	Description
Solid-state nature	The highest temperature observed during the AFSD process is normally 60% to 90% of the feed material. Thus, issues seen in beam-based techniques such as porosity, residual stresses, and hot cracking are not present.
Characteristic microstructure	Beam-based techniques lead to microstructures consisting of columnar grains due to the epitaxial solidification. AFSD, however, results in refined equiaxed grains that are formed during dynamic recrystallization. Because of the equiaxed grains, AFSD processed materials have isotropic properties compared to the beam-based materials' anisotropic properties. Therefore, AFSD materials are more suited for load bearing applications.
Diverse feed material options	AFSD uses rods of metal that can be cast or wrought. Because of its access to cheap processing methods for the feed material, AFSD is more convenient and economical than beam-based approaches. Beam-based processes require powder with specific compositions, shapes, and sizes which raises the cost of operation.
Wide range of applications	AFSD has been successful in creating parts made from various alloys including aluminum alloys, magnesium alloys, titanium alloys, and nickel-base superalloys. Metal matrix composites have also been fabricated by consolidating pre-mixed powders.
Speed	AFSD build rates are comparable to other large-scale additive manufacturing process such as wire

	arc additive manufacturing. More importantly, the build rates of AFSD are much higher than beam-based processes.
Scalability	Because AFSD is not limited by the need for a vacuum chamber or powder bed, large-scale production can occur. Parts with dimensions of 1.5 meters have successfully been produced.

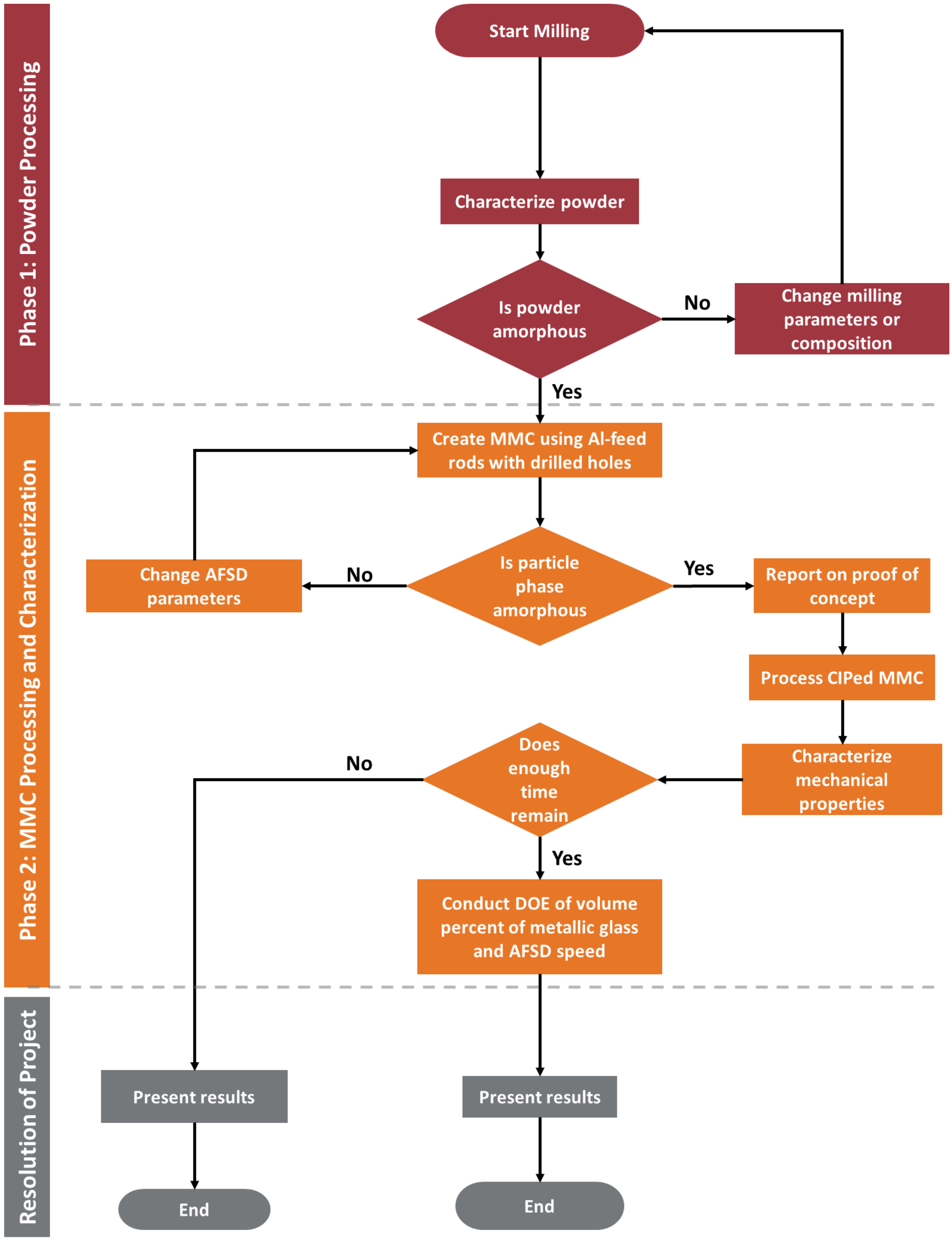

Fig. 5: Proposed Experimentation Plan

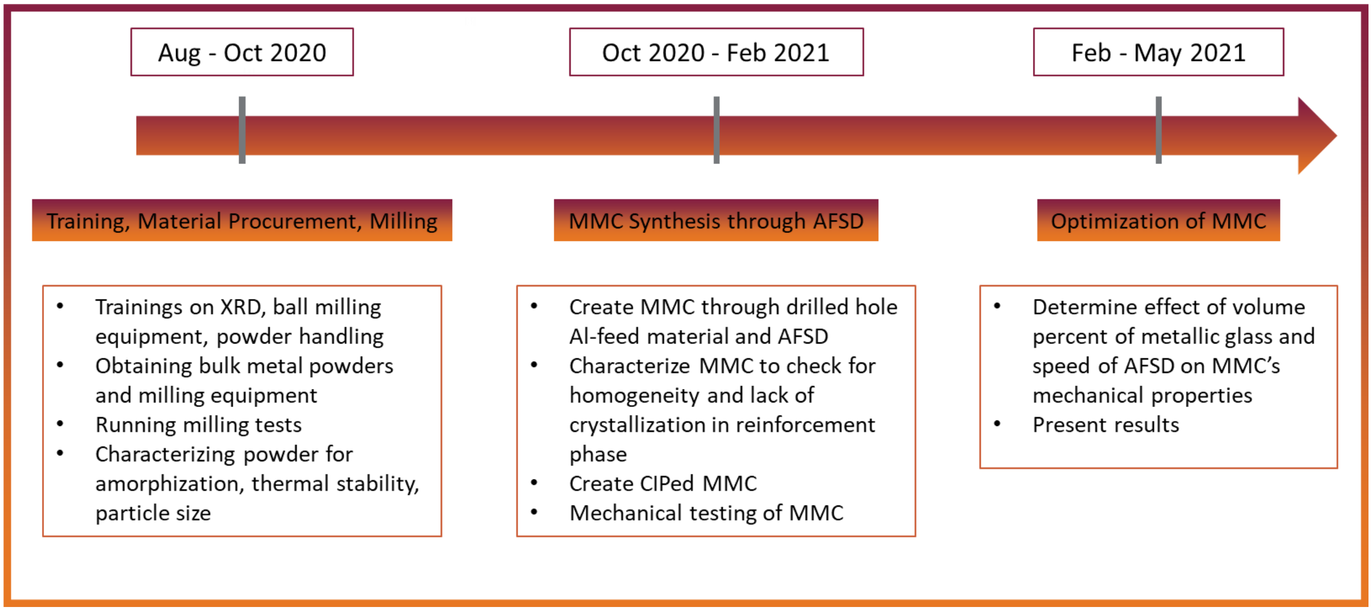

Fig. 6: Proposed Project Schedule

Table 5: Suppliers and Cost Estimates for Raw Materials [17, 18]

Material	Possible Supplier	Particle Size (if Powder)	Purity (if Powder)	Estimated Cost
Zirconium powder	ProChem Inc.	80 mesh	99.9%	$115 for 100 grams
Copper powder	ProChem Inc.	100 mesh	99%	$70 for 500 grams
Aluminum powder	ProChem Inc.	100 mesh	99.9%	$70 for 500 grams
Nickel powder	ProChem Inc.	100 mesh	99.5%	$35 for 250 grams
Tungsten carbide milling balls	MSE Supplies	N/A	N/A	$159 for 1000 balls