40.0 EVALUATION OF PROCESSING PATH EFFECTS ON MICROSTRUCTURE AND PROPERTIES OF A POWDER-BASED AL-TM ALLOY.

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This project initiated in Fall 2019. The research performed during this project will serve as the basis for a Master's thesis program for Stuart Shirley.

40.1 Project Overview and Industrial Relevance

As the aerospace and automotive industries continue to push the boundaries of fuel efficiency, the demand for lightweight, high-performance materials increases. Titanium and aluminum alloys are the primary metals used to meet the requirements of critical lightweight applications. In the commercial sector, the Boeing 747 is 68% aluminum and 4% titanium; while fighter jets are 50% aluminum and 13% titanium by weight [40.1]. Clearly, titanium is utilized more broadly in critical applications where material cost can be justified. Over the operating lifetime of a fighter jet, material cost is approximately 2%, whereas 50% of the total lifetime jet cost is due to operational costs, which are primarily due to fuel [40.1]. To reduce operational cost, switching titanium parts to less dense aluminum will reduce the mass of the plane and increase fuel efficiency. Savings in fuel due to lightweight materials has become a critical factor in the automotive market as well in order to reduce CO_2 emissions [40.2].

Although titanium meets structural requirements at high temperatures, it is more costly and dense than aluminum [40.1]. Many current aluminum alloys begin to lose mechanical properties above 100 °C [40.1], and thus development of high temperature aluminum alloys could be the solution to cost and weight reduction by replacing titanium components. To this end, aluminum alloys with transition metal alloying elements (Al-TM) have been developed to retain high temperature mechanical properties[40.3]; transition metal elements of interest are discussed below. The retention of ultimate tensile strength (UTS) in Al-TM alloys at elevated temperatures, up to ~350°C [40.4],is due to the reduced solubility of alloying elements, leading to retention of precipitates at elevated temperatures [40.5]. Al-TM is produced as a powder via gas atomization. This process produces a fine grain size and dispersion of second phase particles. While consolidating powder into a bulk form, retaining the resultant microstructure from the powder production process is critical to the high temperature mechanical properties of the alloy. This project will focus on multiple solid-state consolidation processes and the resultant microstructure and mechanical properties.

Solid state processing paths for powder consolidation of interest include canned extrusion, additive friction stir deposition (AFSD), and friction stir extrusion. Each of these three paths will be evaluated for microstructure and mechanical properties. Additional thermomechanical processing via Gleeble® compression testing will be conducted to develop understanding of forging effects on material produced by each of the aforementioned processing pathways.

Typical canned extrusion of powders requires multiple steps to achieve a consolidated bar from powder. Powder is compacted into a can to increase density prior to extrusion, and the can is sealed with a vacuum to isolate the powder from the atmosphere [40.6]. The canned powder is extruded through the die, with the can "jacketing" the extruded shape, and a final step of removing the can from the extruded shape [40.6].

AFSD is a solid-state powder consolidation method that falls within the realm of additive manufacturing. This process is based on a friction stir machine, where powder or solid feedstock are added to the build through a hollow shoulder tool which creates heat through friction and severe plastic deformation to bond powder material to previous layers and build a three dimensional part [40.7].

Friction stir extrusion, related to shear assisted processing and extrusion (ShAPE), is a solid-state method of powder consolidation. In this process, a rotating die is forced into a container of powder, and the material back-extrudes through the center of the die face [40.8]. Friction extrusion offers several advantages, including reduced processing time from powder to rod and improved mechanical processing in comparison to traditional extrusion routes [40.9].

40.2 Previous Work

A primary strengthening mechanism in aluminum alloys is Hall-Petch strengthening, the impediment of dislocation motion by grain boundaries. Strength increases with a reduction in grain size, and the aforementioned rapid solidification that occurs in fine powders is very conducive to forming a fine microstructure [40.10]. Orowan strengthening is the second strengthening mechanism important to high temperature aluminum alloys, is due to dislocations interacting with fine precipitate phases within grains, and is limited by the rate of coarsening of these particles at elevated temperatures [40.10]. Both of these strengthening mechanisms are inhibited by grain growth or precipitate coarsening at elevated temperatures. As temperatures increase, recrystallization and grain growth occur reducing the effect of Hall-Petch strengthening. Additionally, at elevated temperatures diffusion is faster and fine phases within the grain begin to coarsen, reducing the contribution of Orowan strengthening.

One method to address thermal limits of aluminum is to alloy with several transition metals that have low diffusion coefficients including; Fe, Cr, Ti, V, Nb, and Mo [40.10]. The use of transition metals with low diffusion coefficients in an aluminum matrix is critical to creating a high temperature aluminum alloy. Since these elements diffuse slower at elevated temperatures, coarsening of particles on grain boundaries or within the grain occurs at reduced rates. Stabilized phases continue to contribute to strengthening by pinning grain boundaries thus reducing precipitate growth and inhibit dislocation movement by Orowan strengthening [40.10].

Powder aluminum alloys are produced through gas atomization, this allows for a great flexibility in composition and range of powder size from approximately 10 to 1000 μm [40.11,40.12]. Gas atomization of the liquid melt forms very small powder particles, and these particles undergo very fast cooling rates in comparison to casting; thus the term rapid solidification (RS) [40.12]. Al-TM alloys produced through rapid solidification have been reported with grain size of 4-5μm [40.9], with further refinement achieved in consolidation [40.9,13].

Via the gas atomization process, it is also possible to produce alloys with alloying element concentrations greater than the solid state equilibrium [40.10]. These supersaturated solid solutions increase the fraction of second phase formed on the dissolution of the saturated phase [40.10], thus increasing the Orowan strengthening contribution. Limited solubility at elevated temperature prevents coarsening of second phases. In high temperature aluminum alloys similar to Al-TM, the fine second phase particles are typically metastable and formed during rapid solidification (RS) [40.10]. These metastable strengthening phases have been shown to transform to more stable equilibrium phases in the temperature range 400-500°C [40.10,40.14,40.15]: thus, consolidation of powder should be performed at low enough temperatures to retain these phases.

Retaining the fine particles formed during RS is essential to the performance of these alloys at high temperature. Consolidation of these powders to a bulk form must be conducted via a solid-state process at relatively low temperatures to retain the second phase particles. Previous research on aluminum alloys with transition metals has been conducted primarily on melt-spun ribbons [40.15,40.16], and RS powders consolidated through extrusion [40.17]. This work seeks to further investigate Al-TM alloy microstructures produced by RS powder consolidated through extrusion and several solid-state friction-based processes, as mentioned previously.

40.3 Recent Progress

40.3.1 Thermal Stability Testing

Several authors report transformation of the primary strengthening phases for aluminum alloyed with transition metals between 400-500°C determined by differential scanning calorimetry, with a corresponding drop in mechanical properties [40.10,40.14,40.15]. Quasicrystal phases are the primary strengthening phases present in these alloys and have been shown to transform around 450°C into more stable phases [40.10]. Thermal stability tests were conducted on extrusions produced from the Al-12.4TM alloy as outlined in

Table 40.1 with hardness data and microstructural changes to be reported in the next period. This thermal stability testing is to compare the Al-12.4TM alloy with that of previously published work and inform later thermomechanical processing and alloy evaluation.

40.3.2 Forging Microstructure Characterization

Several impeller forgings were produced from Al-TM extrusions with a 6.25:1 extrusion ratio, as shown in **Figure 40.1**. Extrusion ratio is the cross section of the container divided by the cross section of the extruded part [40.18]. The forging was sectioned and macro-etched to reveal the flow lines of the forging shown in **Figure 40.3**. Samples were etched in 9% NaOH solution at 65°C for 5 minutes then wiped with a cotton ball soaked in 3% nitric acid to de-smut the surface. The resultant macro-etch shows good fill of the die cavity and flow of material. Of most interest, there is an etching response indicated by the arrows in **Figure 40.3** that appears to be shear banding or a remnant microstructural inhomogeneity from the extrusion process. Shear bands are the result of a local plastic instability during deformation, however an increase in strain rate sensitivity reduces the onset of strain localization [40.19]. **Equation 40.1** gives the tendency for a material to form localized shear bands[40.19], as α increases shear banding is more prevalent.

$$\alpha = \frac{\gamma - 1}{m} \qquad (40.1)$$

With increasing strain rate sensitivity, m, shear banding would be less likely to form. Alloys similar to Al-TM consolidated through extrusion have been shown to exhibit a strain softening behavior at elevated temperature and strain rate, however they also exhibit an increasing strain rate sensitivity as processing temperature increases [40.14]. As the material is strain softening, the γ strain hardening coefficient would decrease. Thus, in Al-TM alloys, tendency to form shear bands would be low as γ is negative and m is increasing. .

Tkach Metal Forming Consultants graciously provided modeling of the production process for the Al-TM impeller forging this can be seen in **Figure 40.2**. The figure shows the predicted effective strain and temperature at the end of the forging operation. These results informed the areas of interest outlined in black boxes in **Figure 40.3**. **Figure 40.4Figure 40.5** are optical metallographic images of microstructures from two of the aforementioned areas, with the light and dark regions believed to be the powder particles consisting of relatively equiaxed grains. Further characterization with scanning electron microscopy (SEM) and electron backscattered diffraction (EBSD) is necessary to confirm the grain size and morphology of the forged regions of interest. The thermal model of the forging indicates that adiabatic heating is occurring during the forging process which could affect the previous discussion on shear banding. Further literature review and microstructural analysis will be conducted to determine if adiabatic heating could be causing shear banding in the forgings.

40.3.3 Tooling development

Additive Friction Stir Deposition (AFSD) is a process pathway for the consolidation of Al-TM powder that has been of interest and scope in this project. Production of metallurgical AFSD samples will be conducted by Virginia Tech and University of Alabama. The machines at these universities require a solid feedstock to build a part and are not equipped to directly produce samples from powder. University of Alabama has produced samples from green compacted powders in the past and agreed to attempting production of Al-TM builds using this method.

An additional route to prepare feedstock is to pack the powder into a tube and use this as a feedstock in the AFSD. In this method powder will be loaded into a capped tube then compressed to approximately 60% density before capping the tube. Two dimensions of tube will be produced, one at a 0.375" in cross-section for direct loading in the AFSD machine and a second tube of 0.75" in cross section. The larger cross-section will be rolled down to 0.375" in square cross section for use with AFSD, this route will lead to a larger portion of the feedstock consisting of Al-TM powder precursor. Following this route of material preparation for AFSD requires a clamp and plunger fixture to be manufactured to aid in the production of these "packed" tubes. A preliminary design for this fixture can be seen in **Figure 40.6**. There are three primary components, the clamp/case, a strip plate, and the plunger. The clamp will hold and support the tube during powder compaction and align it with the plunger. The plunger will transfer the load to the powder inside the tube, and the strip plate prevents the tube from staying on the plunger as it is pulled out of the tube after compaction.

40.4 Plans for Next Reporting Period

Below are outlined several interest areas of this project to be discussed in the spring.

- Powder preforms for AFSD at Virginia Tech and University of Alabama
- Thermomechanical testing: Greeble compression testing, and strain rate testing of ShAPE and AFSD material
- Forging EBSD microstructure comparison with predictive modeling

40.5 Acknowledgements

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40.6 References

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40.7 Figures and Tables

Table 40.1:Thermal stability tests on extruded Al-TM

TEMP FOR 1HR (°C)	350	400	450	500	550
	X	X	X	X	X

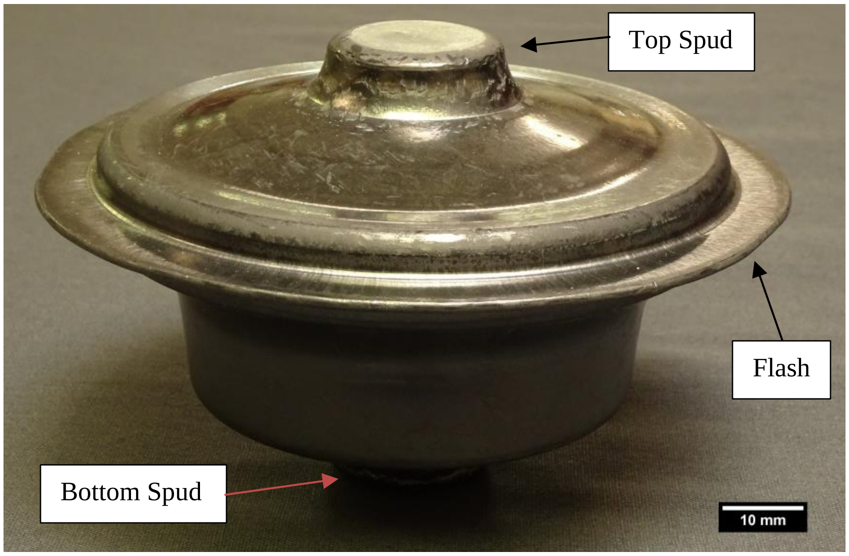

Figure 40.1:As received closed die forging of an impeller produced from an Al-TM extrusion. Produced in a single strike of the mechanical forge press, microstructural response to the experienced strain rate is of interest and will be characterized in future work. Labeled regions for reference in microstructures presented in Figure 40.4 and Figure 40.5.

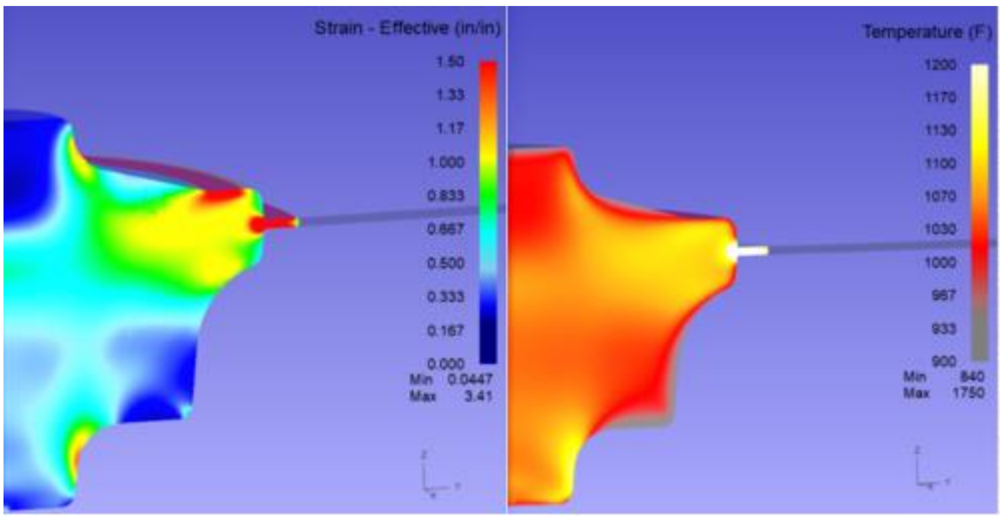

Figure 40.2: Deform™ finite element model of effective strain (left) and temperature (right) experienced in Al-TM at end of forging cycle. Modeling courtesy of Tkach Metal Forming Consultants.

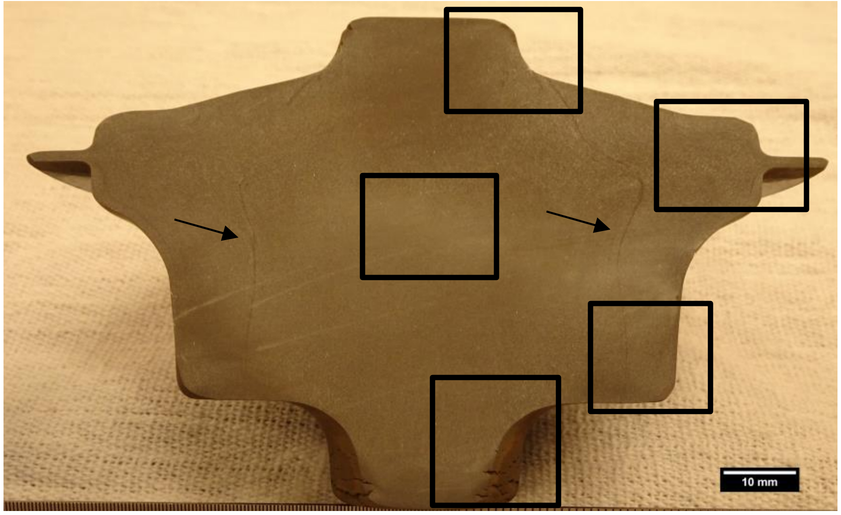

Figure 40.3: Cross section and macro-etch of forging shown previously. Etched in 9% NaOH solution at 65°C for 5 minutes then wiped with a cotton ball soaked in 3% nitric acid to de-smut the surface. Boxed areas indicate regions of interest due to expected thermal and strain gradients predicted by finite element modeling. Arrows indicate heavily etched flow lines at the ~1/4 radius position of the forging, further characterization is needed to determine if this is a microstructural affect or difference in chemical composition.

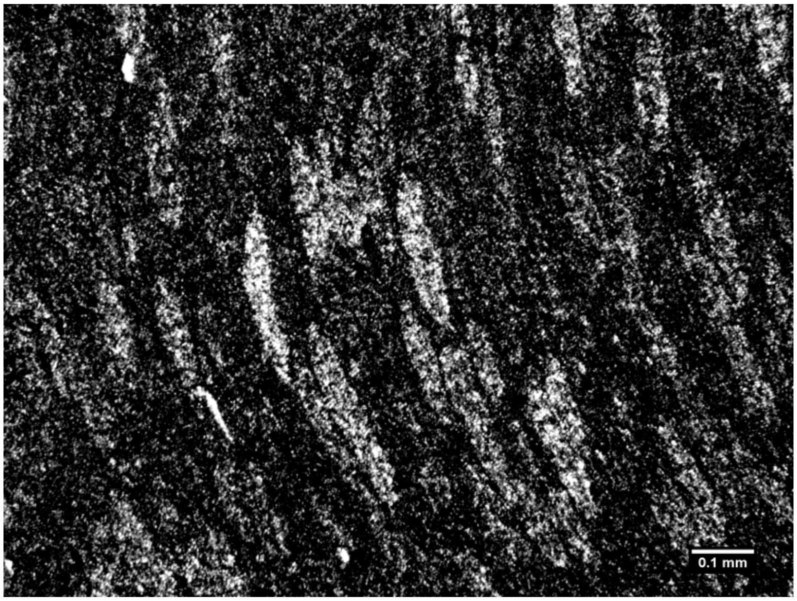

Figure 40.4: Optical metallographic image of the forging microstructure near the flash, etched in Keller's reagent. The microstructure shows the resultant flow and deformation of powder particles with what appears to be an equiaxed microstructure within powder particles. Further characterization with EBSD needed to confirm the equiaxed grain morphology within powder particles.

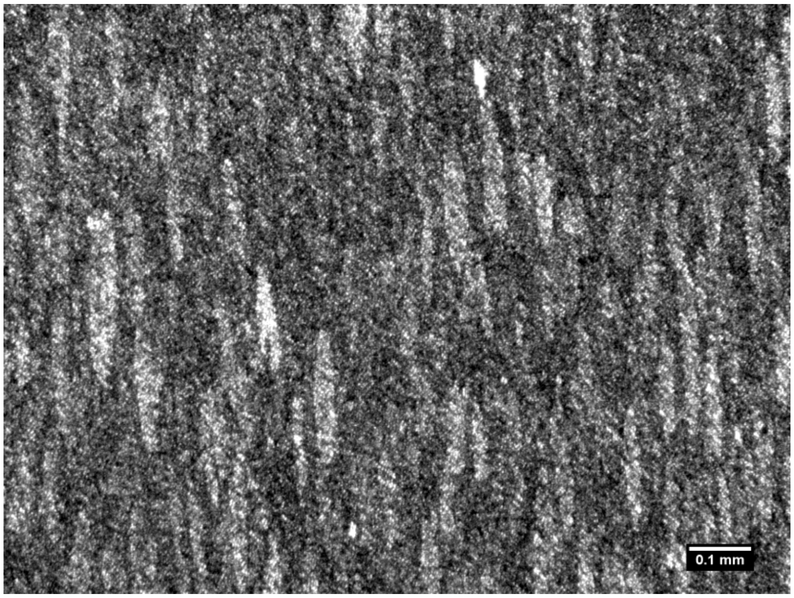

Figure 40.5: Optical metallographic image of the forging microstructure near the top spud, etched in Keller's reagent. This microstructure is taken in the region of the top spud where “dead metal” is expected to have microstructure similar to the extruded material. Shown is a microstructure of elongated powder particles from extrusion with what appears to be an equiaxed microstructure within powder particles. Further characterization with EBSD needed to confirm the equiaxed grain morphology within powder particles.

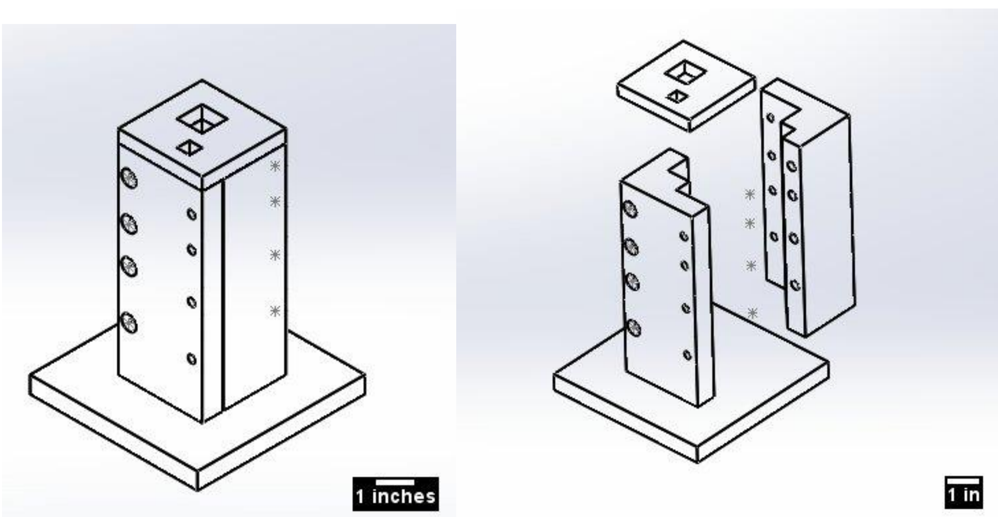

Figure 40.6: Clamp design for AFSD preforms, a) assembled clamp b) exploded view of the clamp for loading material.