Pacific Northwest
NATIONAL LABORATORY

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Process Intensification for Nanocomposite Aluminum Extrusions

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Executive Summary:

The project evaluated the feasibility of using Shear Assisted Processing and Extrusion (ShAPE) for the fabrication of extrusions with a diameter of 0.5" and higher from an Al-alloy powder feedstock that contains nanoscale second phases for achieving enhanced elevated temperature strength. PNNL has previously extruded 0.2" diameter rods with high strength aluminum alloy powder Al-12.4TM. This project aimed to scale the process up to a diameter of 1.0" at a length of >24" while retaining the excellent mechanical properties achieved for the 0.2" diameter rods. As part of the project, extrusion dies were fabricated with various design features. As an example, scroll features on the tool die face was introduced to enhance powder mixing, and reduce extrusion load. Varying extrusion ratios were used to obtain better microstructural homogeneity. Use of tool design features together with the optimization of process parameters helped in the fabrication of extrusions that were up to 48" long with a diameter of 0.5" using Al-12.4 TM powder feedstock in a two-step process involving cold compaction followed by extrusion. Extrusions with a diameter of 1.0" could also be fabricated, but limitations on the extent of consolidation were experienced as a result of the torque and ram force capacity of the current ShAPE machine.

1. **Project Goals/Objectives:** The primary goal of this project is to demonstrate that Shear Assisted Processing and Extrusion (ShAPE) can be used to fabricate nanostructured aluminum rods, directly from high performance aluminum alloy powder, at a diameter of commercial interest. A secondary goal is to show the potential for process intensification using ShAPE through fewer process steps, and lower extrusion force, compared to conventional powder metallurgy extrusion. The specific objectives are:

   - Fabricate 1.0" diameter nanostructured extrusions in bulk with a length >24", directly from Al-12.4TM aluminum alloy powder without the need for canning, degassing, compaction, and other intermediate steps typical of powder metallurgy extrusion.
   - Achieve a microstructure with an ultrafine grained (UFG <1 μm) aluminum matrix having refined and homogenously distributed nanoscale second phases.
   - Show that the ram force can be significantly reduced compared to conventional powder metallurgy extrusion of the same material.

Background: ShAPE, pioneered at the Pacific Northwest National Laboratory (US Patent 10,189,063), is a new extrusion technology. Maturation of the ShAPE process would create a new cross cutting US manufacturing technology that advances three key the objectives within AMO; namely, nanomaterials processing, process intensification, and materials for extreme and harsh environments. PNNL and SCM Metals Products, Inc. (SCM) are collaborating to mature the ShAPE extrusion process to increase the likelihood of its adoption by industry. PNNL's custom (one-of-a-kind in the world) ShAPE machine will be used to extrude round rods of high strength aluminum alloy feedstock powder provided by SCM. As proof-of-concept, PNNL has previously extruded 0.2" diameter rods with SCM aluminum alloy powder Al-12.4TM. This project aims to scale up the process to a diameter of 1.0" at a length of >24" while retaining the excellent mechanical properties achieved for the 0.2" diameter rods. In prior work on magnesium alloy ZK60, ShAPE has been shown to reduce extrusion force compared to conventional extrusion while work on Al-12.4TM aluminum powder has shown that numerous process steps common to powder metallurgy can be eliminated. The combination of reduced force, smaller equipment, and fewer process steps suggests that significant process intensification may be possible with ShAPE of aluminum alloy powders.

Accomplishments:

- ShAPE extruded 0.5" diameter rod with up to various lengths (48", 42" and 37"). Extrusion was fabricated in a single step through powder compaction and subsequent indirect extrusion steps using Al-12.4TM powder feedstock. Various extrusion ratios were used (40 and 70).
- ShAPE extrusion of 1.0" diameter rod with a length of 9" fabricated from Al-12.4TM powder feedstock with an extrusion ratio of 10.
- Designed and fabricated new tooling and die set for extrusion ratio of 70 at 0.5", 30 at 0.75" and 17 at 1.0".
- Submitted manuscript to peer-reviewed journal: T. Wang et. al., "Microstructural Assessment of a Multiple-Intermetallic-Strengthened Aluminum Alloy Produced from Gas-Atomized Powder by Hot Extrusion and Friction Extrusion," Submitted to Materials, 2020.

Task 1: Extrusion Tooling Integrated with ShAPE Machine

During the course of the project, tooling and fixtures have been designed and fabricated for indirect extrusion of 0.5" and 1.0" diameter rods. Flat face and scrolled tools were designed and fabricated for powder consolidation. The hardware has been installed in the ShAPE machine and checked for fit and function. Tools with the following extrusion ratios have been designed and fabricated as part of the project, e.g. 40:1 and 70: 1. Aggressive scroll pattern has been used on the tool face to promote extensive deformation of the powder feedstock. Representative images of the extrusion tooling having a diameter of 4.2" re captured below (Fig. 1)

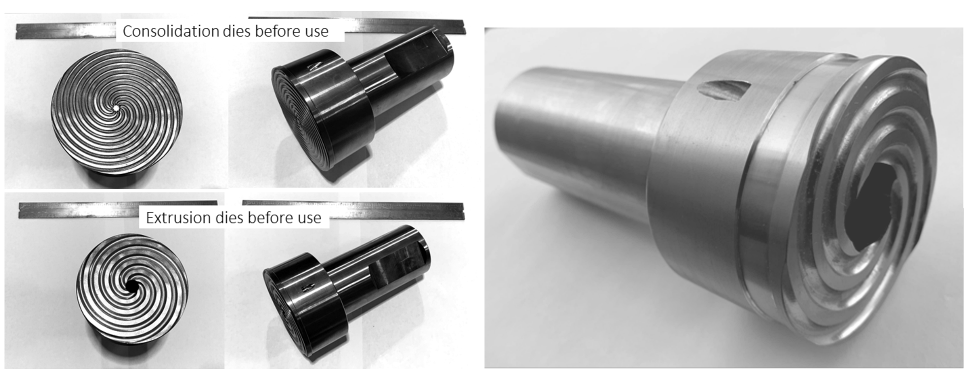


Fig. 1 Extrusion tools designed for the project. Scrolled-face tool for powder consolidation and indirect extrusion.

Task 2: Process Development

Indirect ShAPE extrusion on Al-12.4TM powder feedstock has been carried out at various extrusion ratios. ShAPE processing was carried out in two steps, (i) powder consolidation, and (ii) indirect extrusion. Al-12.4TM powder feedstock was first cold compacted, and subsequently friction consolidated using the consolidation dies. During friction consolidation, the die (without extrusion orifice) is rotated at a certain rpm, while applying a pre-determined axial force, typically at 150 rpm and 60 kN ram force. Simultaneous application of axial force together with frictional heating results in formation of a dense top layer. ShAPE indirect extrusion was carried out in the next step. The extrusion die was rotated and pressed into the consolidated billet at a fixed ram velocity of 4 mm/min. At the start of the indirect extrusion step, a tool speed of 300 rpm was selected to overcome the machine torque limitation. Afterwards, the tool speed was decreased to 80 rpm to maintain a steady process temperature during the actual extrusion event. As an example, Fig. 2a shows the extrusion die assembly in contact with the consolidated powder billet after ShAPE extrusion event, while Fig. 2b captures the ShAPE extrudate coming out through the extrusion die orifice.

(a) (b)

Fig. 2 (a) extrusion die assembly in contact with the consolidated billet, (b) ShAPE extrudate coming out through the die orifice.

Details about the ShAPE process is shown in Fig. 3, which summarizes the machine data output during extrusion of 0.5” diameter rod. Specifically, the plots below show data for extrusion force, spindle power, and die face temperature as a function of extrusion length. The force plot has a shape typical of conventional extrusion with a breakthrough force that drops to a lower steady state force. The steady state extrusion force was just 9,000 lbf which is very low for an extrusion of this size. The steady state spindle powder was noted to be 5 kW, which is quite low in comparison to billet pre-heating and container heating used in conventional hot extrusion processes.

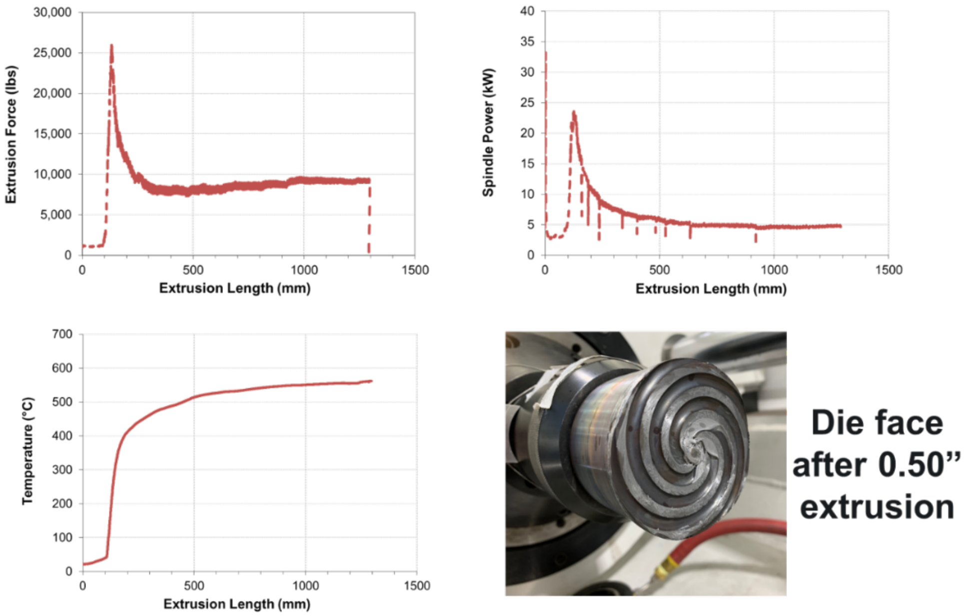


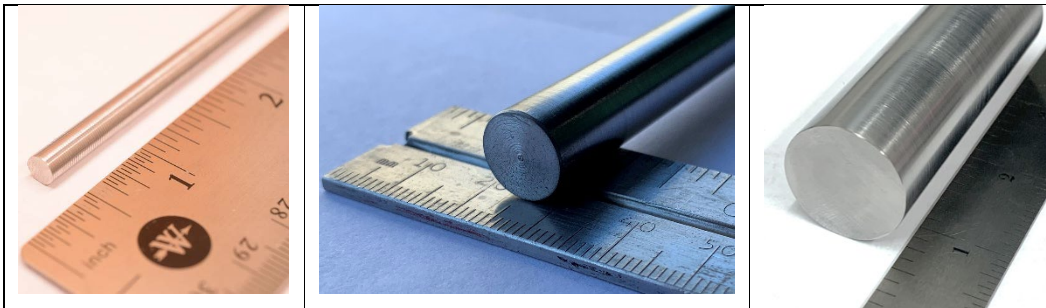
Fig. 3 ShAPE process data output recorded during extrusion of 0.5” diameter rod.

Through our ShAPE process, we have been able to successfully consolidate and extrude rods of various diameters using Al-12.4TM powder feedstock. Successful extrusion of a 9” long rod with a diameter of 1.0” could be achieved through ShAPE processing trial, which is shown in Fig. 4. To begin with, Al-12.4TM powder was first densified into a billet with solid top face through multiple steps using a scrolled consolidation tool. Subsequently, a die with an inner orifice diameter of 1.0” and extrusion ratio of 10 was used for indirect ShAPE processing. The final extrudate contained within the extrusion die is shown in Fig. 4a. The actual extrudate after extracting from the die, is shown in Fig. 4b. Fig. 5 shows another example of a 0.5” diameter extruded rod with length > 40”. A close-up representation of our ShAPE extrudates with the surface machined for aesthetics (e.g. 0.5” dia and 1.0” dia rods), that we were able to fabricate is shown in Fig. 6a, 6b and 6c. Although a 1” diameter was achieved, the centerline of the extrusion was not fully densified and contained significant porosity since a large enough extrusion ratio could not be achieved due to limitations on the ShAPE machine ram force and torque capacity.

(a) (b)

Fig. 4 (a) 1.0” dia extrudate contained within the extrusion die, (b) actual 1” dia. extrudate

Fig. 5 ShAPE extrudate with 0.5” diameter.



(a)	(b)	(c)

Fig. 6 Close up view of ShAPE Extrudates; (a), (b) 0.5” dia (a, b); 1.0” dia (c).

Task 3: Microstructural Characterization

Microstructural characterization involved optical microscopy, hardness measurement, and SEM imaging at the area of interest along the length of the ShAPE extrudates. As an example, the 37” rod that was fabricated at an extrusion ratio of 40, was cross sectioned near the beginning and end of the extrusion to understand the extent of consolidation and flow of material into the orifice. The flow pattern along the centerline and extent of mixing has been noted to be substantially different at the beginning of the extrusion where the temperature is lower (400-450C) compared to the end of the extrusion where the temperature is higher (550C). A representative image of the extrusion cross-section along with the remnant of the starting powder feedstock is shown Fig. 7. High magnification SEM images from the edge and centerline of the extrudate confirm that much less refinement of the aluminum matrix and second phases occurs along the centerline as compared to the edges (where fully processed material enters the extrusion orifice). Insufficient refinement could be the reason behind the formation of striated flow marks along the extrudate centerline. Based on the microstructural observations, a higher extrusion ratio, and conically tapered orifice, would be beneficial to provide more extensive mixing along the centerline, together with ShAPE processing at the lowest temperature possible. This can be accomplished by using lower rpm but will most likely require a cooled powder container and scrolled die to keep temperature <450C.

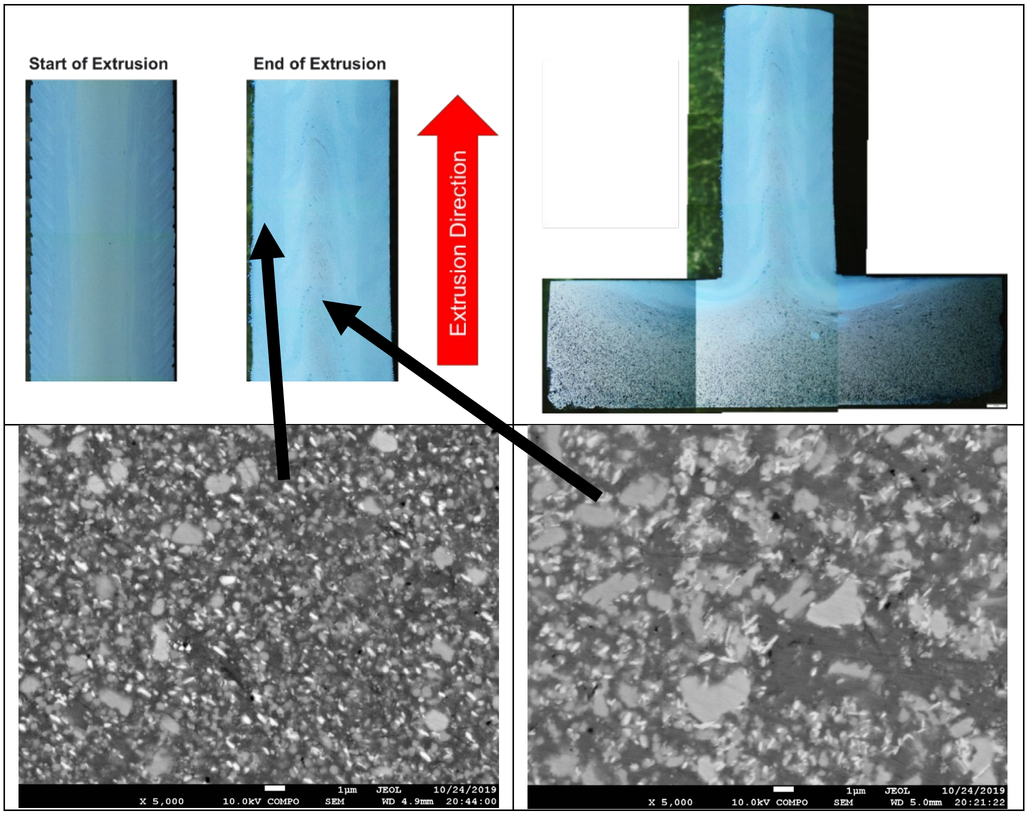


Fig. 7 Cross-sectional images of 0.5" dia ShAPE extrudate showing less refinement of second phases along the centerline compared to the periphery.

Microhardness measurements were carried out at multiple locations along the length of the extrusion and across the diameter. Hardness values at the start of the extrusion were noted to be higher and it reduces gradually at the end of the extrusion. Further, a depression in the hardness values along the centerline has been observed, which gets more severe towards the lower end of the extrusion. Therefore, we believe the presence of increased mixing and lower temperature (i.e. more load transfer between the aluminum matrix and second phases particles), which is the typical ShAPE condition at the start of the extrusion leads to higher hardness. Based on the microscopy, temperature, and hardness data, we hypothesize that a higher extrusion ratio combined with processing at lower temperature would be effective at eliminating hardness variation along the length, and across the diameter, of the extrusion.

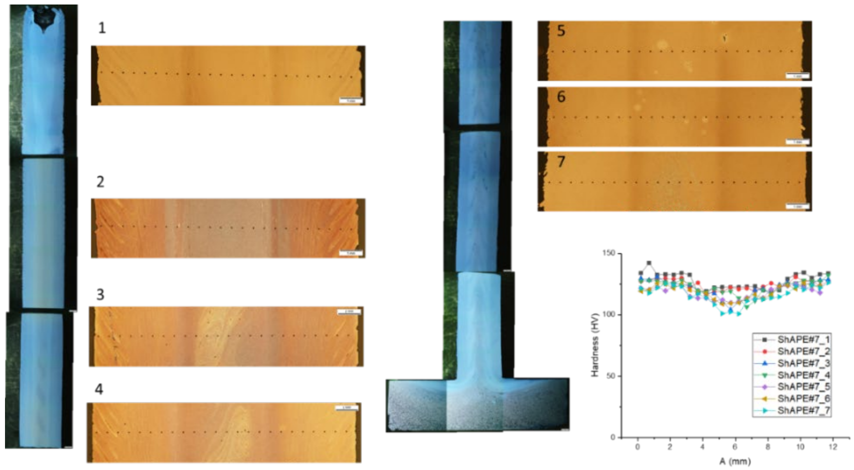
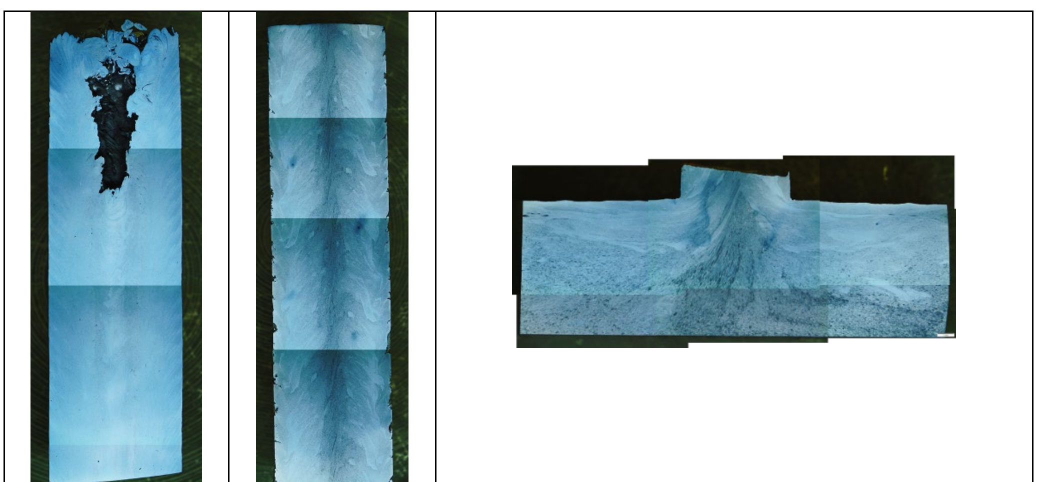


Fig. 8 Vickers hardness of the 37” long extrudate, extrusion ratio = 40

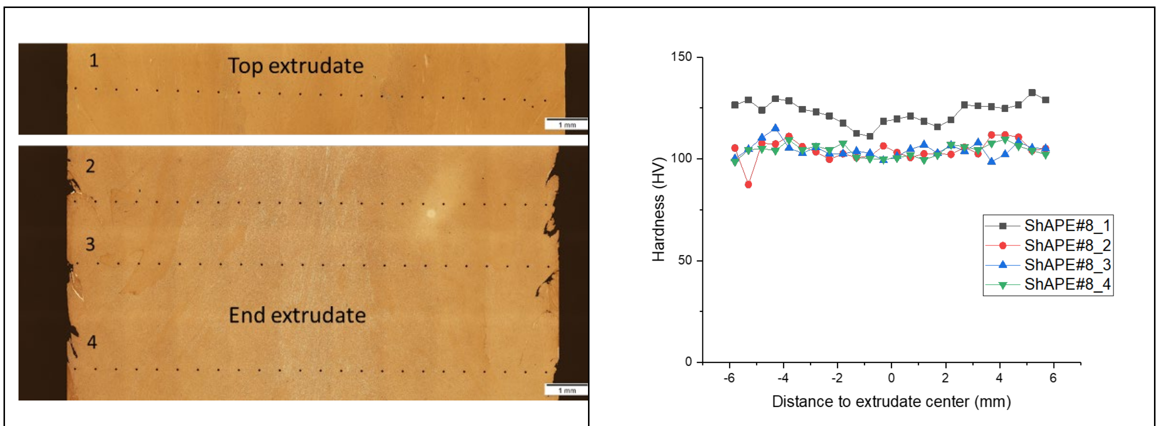
We were able to extrude another 0.5” dia rod with a length of 42” at an extrusion ratio = 70. The extruded rod was cross sectioned near the beginning and at the end of the extrusion to understand the nature of powder densification and subsequent flow of material into the extrusion orifice. Fig. 9a-9c, show optical micrographs of the longitudinal cross-section at different positions along the length of the 0.5” extrudate. Fig. 9a, shows the cross-section at the beginning of the extrudate. A big hollow is noticed at the start measuring about 0.5” long, which subsequently gets filled up as more material is extruded. Fig. 9b shows the cross-section of the extrudate at the very end of the process, which appears to be completely consolidated. Cross-section of the processed billet is shown in Fig. 9c, which appears to be well consolidated near the top as well. Only at the very bottom, presence of porosity is noted. Based on limited optical microscopy analysis, it appears that the powder material is going through a swirl motion during ShAPE extrusion. Increase in the extrusion ratio definitely led to more swirl-like features.



(a) (b) (c)

Fig. 9 Longitudinal cross-section of 0.5” diameter extrusion, (a) at the start of the ShAPE process, (b) at the end of the ShAPE process, (c) processed billet; extrusion ratio = 70.

Hardness measurements at various locations on the longitudinal cross-section of the 0.5” diameter rod was carried out to understand the effect of ShAPE processing on the mechanical behavior of the fabricated extrudate. Fig. 10a shows the locations of the four lines on the longitudinal cross-section of the 0.5” diameter extrudate, along which hardness scans were carried out. The corresponding hardness map is shown in Fig. 10b. Hardness values at the top of the extrudate is noticed to be higher than what is noted at the end of the extrudate. Such a variation in the measured hardness values is possibly associated with the underlying material microstructure. The process temperature varies along the length of the extrudate during ShAPE as well, with considerably lower temperature being recorded at the start of the process in comparison to a significantly higher temperature noted at the end of the extrudate. A lower process temperature would result in a finer Al grain structure, and related higher hardness. On the other hand, a higher process temperature would lead to recrystallization and grain coarsening, which would eventually result in a lower measured hardness values, as observed in the end section of the fabricated extrudate. Based on the current observation, we feel that active cooling at the orifice of the extrusion die would be critical in order to generate a microstructure that would show uniform mechanical properties. Additionally, the hardness plot shown in Fig. 10b displays no depression in the hardness values at the center of the extrudate at the end section, which is a desirable feature. A depression at the center of the extrudate (seen with lower extrusion ratio e.g. 40:1) is associated with less or no ShAPE processing of the feedstock powder. An increase in the extrusion ratio to 70:1 appears to solve this particular problem of a lightly processed centerline.



(a) (b)

Fig. 10. Hardness measurement on 0.5” diameter extrudate, (a) locations of hardness scans on the longitudinal cross-section, (b) hardness plot. Extrusion ratio = 70.

Conclusion:

Through this project, we were able to ShAPE extrude 0.5” diameter rod of various lengths (48”, 42” and 37”). Extrusions were fabricated in two steps through cold powder compaction and subsequent indirect extrusion using Al-12.4TM powder feedstock. Various extrusion ratios were used (40 and 70). For ShAPE extrusion of 1.0” diameter rod, a 9” long extrusion could be fabricated from Al-12.4TM powder feedstock with an extrusion ratio of 10. The process energetics were found to be very low. As an example, the steady state extrusion force was noted to be 9,000 lbf for a 0.5” diameter extrusion, which is very low for an extrusion of this size. The steady state spindle powder was noted to be 5 kW, which is quite low in comparison to billet pre-heating and container heating used in conventional hot extrusion processes. Microstructural characterization of the extruded rods indicated presence of heterogeneity along the center of extrudates in comparison the extrudate edge. However, increase in extrusion ratio helped in the refinement of powder feedstock and achieve a more uniform microstructure. Based on the microstructural observations, a higher extrusion ratio, and conically tapered orifice, would be beneficial to provide more extensive mixing along the centerline, together with ShAPE processing at the lowest temperature possible. This can be accomplished by using lower rpm but will most likely require a cooled powder container and scrolled die to keep temperature <450 °C.

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