

Additive Manufacture of Refractory Metals for Aerospace Applications

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High temperature refractory metals are required for a number of high temperature propulsion applications. Refractory metals are expensive, difficult to manufacture with high buy-to-fly ratios, and few vendors. Additive manufacture (AM) is used to produce C103, Molybdenum (Mo), and Tungsten (W) reaction chamber and thrust stand-off as well as Iridium ultra-fine lattice catalysts for integration into 1 N green propulsion thrusters. Refractory AM is in development and like traditional AM alloys requires substantial post-processing to include powder heat treatment, surface finish enhancement, inspection, and machining before placed in service. The combination of limited feedstock sources, high temperature processing, oxygen sensitivity, fracture prone nature, and need for elevated temperature mechanical testing limit the number of qualified facilities capable of post-processing AM refractory materials, which add to cost and schedule constraints. However, properly implemented refractory metal AM can overcome existing manufacture limitations by greatly increasing design flexibility, new material options, reduced price, decreased lead-time, and leverage the ever growing AM commercial supply base.

Nomenclature

<i>AM</i>	=	additive manufacture
<i>DED</i>	=	directed energy deposition
<i>EB-PBF</i>	=	electron beam powder bed fusion
<i>EDM</i>	=	electro discharge machining
<i>HfC</i>	=	hafnium carbide
<i>HIP</i>	=	hot isostatic press
<i>ICME</i>	=	integrated computational materials engineering
<i>L-PBF</i>	=	laser powder bed fusion
<i>LP-DED</i>	=	laser powder directed energy deposition
<i>Mo</i>	=	molybdenum
<i>MSFC</i>	=	Marshall Space Flight Center
<i>Nb</i>	=	niobium
<i>NTP</i>	=	nuclear thermal propulsion
<i>PSD</i>	=	particle size distribution
<i>RCS</i>	=	reaction control system
<i>Re</i>	=	rhodium
<i>RHEA</i>	=	refractory high entropy alloys
<i>Sa</i>	=	areal average surface roughness
<i>SEM</i>	=	scanning electron microscopy
<i>t</i>	=	layer thickness
<i>Ta</i>	=	tantalum
<i>T_m</i>	=	melt temperature
<i>UTS</i>	=	ultimate tensile strength
<i>W</i>	=	tungsten
<i>WT</i>	=	wall thickness
<i>Z</i>	=	build direction
<i>ZrC</i>	=	zirconium carbide
<i>ρ_{rel}</i>	=	relative density
<i>μ-CT</i>	=	x-ray micro-focus computer tomography
<i>μm</i>	=	micrometer
<i>%RD</i>	=	percent relative density
<i>%wt</i>	=	weight percent

Introduction

Refractory metals and alloys are used for service in extreme high temperature environments. In aerospace examples of such applications include reaction control system (RCS) thrusters, nuclear thermal propulsion (NTP) fuel clad, hypersonic wing leading edges, hypergolic and green propulsion chambers and catalyst, power conversion systems, and electric propulsion. Refractory metals are desirable due to a high melt temperature (T_m) and the ability to retain strength and hardness at elevated operating temperatures. In power and propulsion refractory metal parts tend to be primarily thin-walled geometries such as converging-diverging nozzles as shown in Figure 1.

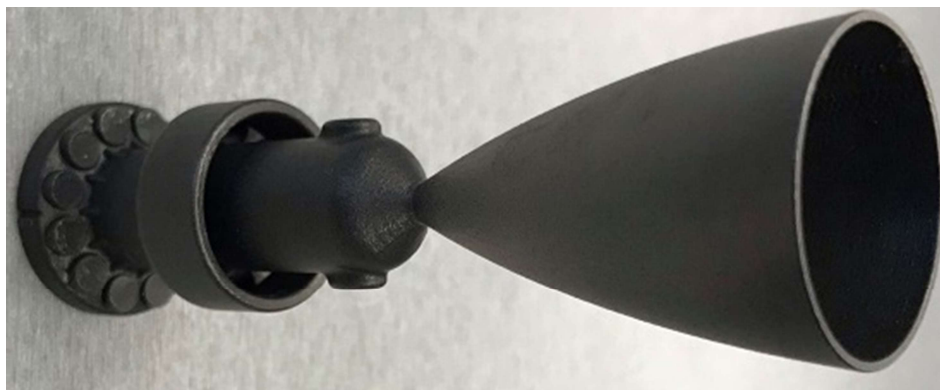


Fig 1. Demonstration 5 N thruster made from Inconel 718 L-PBF AM.

Traditional refractory metal manufacture is expensive due to the high cost of the feedstock, difficulty to achieve near net shape forming and machining, heat treatment, joining, and inspection. Since refractory metals tend to be primarily thin-walled designs such as converging-diverging geometries or clad, part production routinely results in 95-98% of the stock machined away, constituting a 20:1 to 50:1 buy-to-fly ratio. Assuming a 20:1 buy-to-fly ratio, the cost of a part in feedstock alone is 5% and 95% in machining waste, not including machining time or machining waste disposal, which is proportional to machining mass. In addition, refractory metals are relatively difficult to machine, resulting in a limited number of machine shops with the requisite equipment and experience to meet exacting aerospace specifications. Ultimately, a machined refractory metal part can cost several thousand dollars but the waste can be several tens of thousands of dollars.

NASA Marshall Space Flight Center (MSFC) has been developing additive manufacture (AM) for propulsion systems in order to produce complex and optimized components from an array of materials with numerous advantages over traditional manufacturing. AM refractory investigations have found that L-PBF AM of refractory metals and alloys offer significant cost and schedule savings, as well as engineering advantages. A previous development effort of laser powder bed fusion (L-PBF) AM of C103 (Nb-10Hf-1Ti) found that the cost to generate the same part via AM as machined was significantly reduced even when taking into account powder feedstock (33% more expensive than wrought feedstock), print time, heat treatment, final machining, and waste disposal [1, 2]. Unlike machining, AM allows for the desired near-net shape to be produced with minimal machining. AM waste is typically 5-10% of the printed part mass resulting from over-sized powder, support structures, and sacrificial geometric features at interfaces to be machined away to meet surface finish requirements. Consequently, the AM buy-to-fly ratio is approximately 1.1:1 [1, 2]. The vast majority of AM powder is reused for additional builds as long as PSD, morphology, and chemistry remain within feedstock specifications as with any other AM powder feedstock that is recycled. Therefore, like a machined part, an AM part will also cost several thousand dollars; however, unlike traditional manufacture, the waste cost constitutes a few hundred dollars not tens of thousands of dollars. The difference in feedstock costs alone to arrive at an equivalent part constitute an order of magnitude cost reduction by using AM [1, 2]. The cost savings, reproducibility, schedule control, and in certain cases properties of AM parts have significant aerospace industry implications for broad implementation of an alloy that may have been previously avoided due to the aforementioned constraints. In addition, the number of commercially available AM vendors that are sufficiently qualified to print refractory metal parts increases annually. C103, tungsten (W), Molybdenum (Mo), Tantalum (Ta), and Rhenium (Re) are examples of refractory metals that are already utilized or in development using L-PBF, electron beam PBF (EB-PBF), electron beam wire DED (EW-DED), and laser powder DED (LP-DED) AM. However, there is wide variability in the maturity of these refractory metal options, with highly weldable C103 experiencing rapid development in comparison with fracture prone W and Mo. Examples of small refractory L-PBF AM parts produced by NASA are shown in Figure 2.

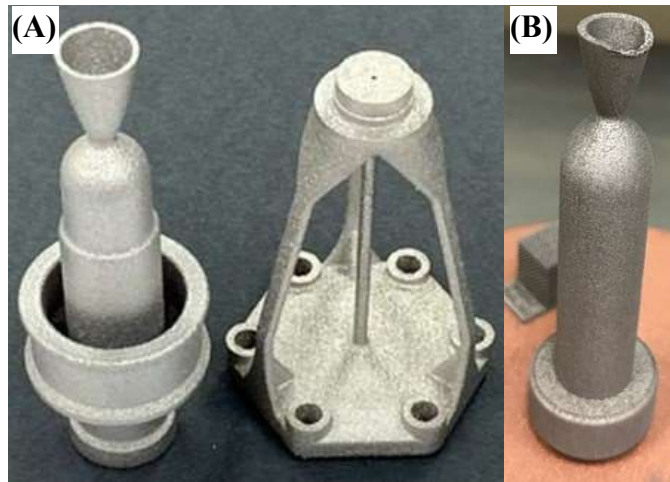


Fig 2. AM C103 1 N reaction chamber and thrust stand-off (A) and AM tungsten chamber (B).

One of the primary limitations of refractory AM is the limited number of refractory feedstock vendors that offer powder or wire that meet AM specifications, as well as the limited number of alloy formulations of engineering utility for AM. Refractory components have been traditionally been produced via powder metallurgy, plasma deposition, electro-deposition, with final geometry obtained with electro discharge machining (EDM). Often angular powder for each element was mixed at the requisite weight percent (wt%), then consolidated through hot isostatic press (HIP) or deposited, forged, then heat treated to arrive at the desired homogenized microstructure. Although mixed powder can be used for AM lab-scale feasibility studies, it is not well suited for industrial production applications where the desired elemental distribution, PSD, and morphology require consistency. Mixed powders can also segregate during sieving due to the differences in mass of the elements and powder particle size, requiring an additional mixing step before each reuse. Due to the high melting temperature of refractory metals, inert gas atomization (the most common form of AM powder production) cannot easily process most of these metals. Most refractory metal powders are produced through plasma spheroidization of angular powder, plasma wire atomization, rotating electrode process, and plasma or induction atomization of ingot. Additional feedstock methods are in development but are not commercially available at this time. Commercially available elemental AM grade powder options include W, Mo, Ta, and Nb; while available alloy powders include C103, Ta3W, T5W, Ta13W, with FS85 (Nb-28Ta-10W-1Zr) in development.

Another major difficulty of refractory metal AM is the inherent fracture prone nature of many of these materials during melt and solidification. Due to a combination of the high thermal gradient that results in high residual stress across the part, and low ductility result in micro-cracking. Low ductility is a well-known attribute of refractory metals and many refractory alloys of interest were developed to increase ductility to enable traditional manufacture. For example, Re is added to W and Mo (such as W-25wt%Re, Mo-44wt%Re, Mo-47.5wt%Re) in order to provide sufficient ductility to allow for post near-net shape machining. Re is advantageous due to a high T_m , increased ductility, and enables the alloy to retain mechanical properties at elevated temperature as Re does not suffer from grain growth as drastically as the other refractory metals. Unfortunately, Re is extremely expensive, powder feedstock is mixed (non-alloyed), powder is angular, and the number of commercial suppliers are extremely limited. For powder-based AM methods, pre-alloyed powder with spherical morphology and near fully dense particle density is desired, which has become AM industry standard. For these reasons, contemporary AM methods and materials require development in order to meet the need of high temperature power and propulsion applications.

Refractory metal components can also suffer from changes in mechanical properties during operation at elevated temperature as grain growth occurs with continued exposure to operating conditions. Materials that are strengthened by fine grain microstructure generally become ineffective at elevated temperatures as the grain growth occurs over cumulative operational service. In addition, there is potential for solid solution strengthened alloys to suffer from elemental diffusion that can result in non-uniform elemental distribution that impacts material chemistry (e.g. micro-segregation). To address the limitations of the existing refractory metal and alloy inventory efforts are underway to develop new refractory metal formulations designed and optimized specifically for AM processes. These AM optimized refractory metal formulations are in development to address improved printability, sustained properties at elevated temperature, cost, and availability in order to meet increased demand primarily by the aerospace, defense, medical, and energy communities.

Methodology

The objective of this paper is to conduct a technology gap review of the current state of refractory metal AM and to provide an example of develop a maturation path to meet needs. Efforts emphasize refractory metal formulation optimization for AM, evaluate potential feedstock production options, AM parameter development, heat treatment options, mechanical and microstructural characterization, inspection, component level test. Three phases of refractory metal development process were identified and are in currently being utilized.

The first phase is to use AM with existing refractory metal powder feedstock such as W, Mo, Ta, and C103. High T_m materials combined with low ductility are inherently fracture prone and micro-cracking is often observed due to high thermal gradient from melt to solidification resulting thermally induced residual stress. The impact on heat treatment and surface enhancement/coatings are evaluated to determine if the micro-cracks can be overcome.

The second development phase utilizes additions of dispersoids (generally ceramic nano-powder). Dispersoids act as heterogeneous nucleation sites in the melt pool during solidification and induce formation of refined equiaxed grains, decreasing residual stresses and associated cracking [2]. The AM refined grain structure is retained at elevated temperature due to grain boundary pinning of the metal matrix by nano-scale ceramic particulates, known as dispersoid strengthening [3, 4]. Dispersoids must have chemical affinity for the metal matrix and must be thermodynamically stable above metal solidification temperature, which limits options to carbide-based ceramics when applied to refractory metals [4]. Oxide-based ceramic dispersoids are heavily utilized in lower temperature Al-base and Ni-base alloys, but will experience melting due to the high temperature of the melt pool in L-PBF and DED AM of refractory metals [4, 5]. For example, T_m for W is 3410 °C, Mo is 2610 °C, and C103 is 2349 °C. Carbide dispersoid candidates with the appropriately high T_m include HfC at 3900 °C and ZrC at 3420 °C. Although shown to be advantageous in stabilization of Mo in EB-PBF HfC is much more expensive than ZrC and HfC has a high thermal neutron absorption cross section, which is undesirable in nuclear applications. For this reason, ZrC is often considered the preferred dispersoid for refractory AM purposes. Dispersion strengthened refractory alloys have been shown to improve printability and mechanical properties at elevated temperature [3, 4]. One previous study of W-0.5wt%ZrC via L-PBF AM that showed a significant decrease in micro-cracking of up to 88.7%, improved printability, and improved retention of mechanical properties after exposure to elevated temperatures [4]. Clearly, dispersoid additions in AM can improve printability and mechanical properties but still suffer from constraints related to the parent metal characteristics.

The third phase relates to a new family of thermodynamically stable refractory alloys designed specifically for the AM process. Cost and AM optimized solid-solution alloy development takes the base element of interest and determines which alloying elements can be used to reduce or eliminate micro-cracking, while providing sufficient strength for post-process machining. Although this is a relatively new field, feasibility studies using mixed powder for L-PBF have been conducted to produce W-5Nb and W-7Ni-3Fe solid solution alloys. Results show improved printability over elemental W, while using cost effective secondary elemental additions and show promise for additional development [5]. Refractory high entropy alloys (RHEA) offer potential for improved mechanical properties at elevated service temperatures and are under development by the AM community. Design of RHEAs is a computationally intensive process due to the very large number of possible alloy compositions. Therefore, an Integrated Computational Materials Engineering (ICME) approach integrating melt-solidification mechanics simulations and machine learning is necessary. These simulations are intended to predict potential solid solution RHEA candidates optimized for a specific AM process, while avoiding intermetallic phases, unfavorable ductile-to-brittle transition temperatures, micro-cracking, etc. [6]. The alloys must be weldable (printable) and not necessarily needing properties to survive traditional manufacture, which means that significant reductions in Re content (single digit wt%) or eliminated entirely to reduce cost is highly desirable. This approach is in the nascent stage but is expected to become the dominant method in new alloy development across all AM materials and methods. An example of W L-PBF AM is now discussed to address the previously mentioned constraints that exist throughout the entire logistical supply chain, from powder availability to part inspection.

Powder Characterization and Optimization

An example development effort is discussed for L-PBF AM of W. W powder was obtained from EOS North America for use in the MSFC EOS M100 L-PBF AM platform. The W powder was found to have angular powder morphology, near-full dense particles, with particle size distribution (PSD) of from 36 to 15 μm with a d_{90} of 25.9 μm and d_{50} of 16 μm measured using a Retsch Camsizer XT. The W powder was used to print a series of W specimens, which served well for the intended application [2]. However, in efforts to improve flowability and increase the part density the powder underwent spheroidization using the MSFC Tekna Tek15 radio frequency plasma spheroidizer as shown in Figure 3.

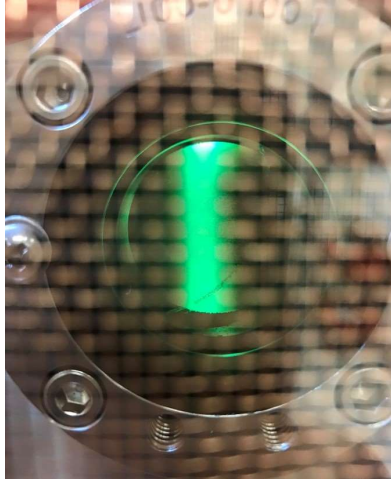


Fig 3. MSFC Tekna Tek15 plasma spheroidization of W powder.

The advantages of the spheroidization process is that it can process any high temperature material and a limited number process parameter variables such as process gas composition and powder flow rate. A comparison of the as-received W powder and RF spheroidized powder is shown in Figure 4. The powder morphology is improved considerably, measured using a Malvern Morphologi G3 optical analyzer at MSFC and found to have a median circularity of 0.97, which is considered an indicator of highly spherical powder.

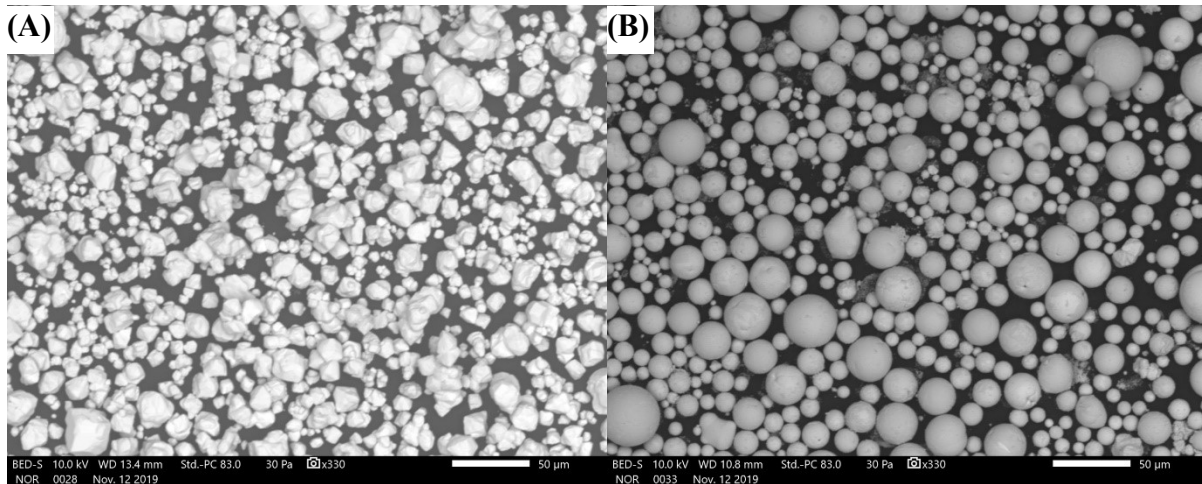


Fig 4. As-received angular W powder (A) and RF plasma spheroidized W powder (B).

Subsequent L-PBF AM trials were also conducted using W and Mo powder obtained from Tekna that had spherical morphology, near-full density, with a PSD from 45 to 15 μm with a d_{10} of 18 μm . Flowability and uniform powder distribution across the build plate was improved when using spherical powder, as expected.

L-PBF AM Parameter Development of W and Mo

W specimens were produced on the NASA MSFC EOS M100 L-PBF AM platform. Metallographic, heat treatment, mechanical property, surface finish, and gas leak test specimens were produced using either 304 stainless steel or CU110 commercially pure copper build plates. Mo parameter development was halted soon after initiation due to Covid-19 pandemic protocols enacted at MSFC. Therefore, EOS North America was contracted to conduct Mo parameter development for the EOS M100 and was still in work at time of publication. An example of W metallographic and mechanical test specimens produced by MSFC are shown in Figure 5. The angular W powder, spheroidized W powder, and commercially available spherical W powder was used for development prints. Improved powder flowability did indeed result in a more uniform powder distribution across the build plate when using spherical powder and improved printability; however, it does not necessarily equate to improved microstructure.

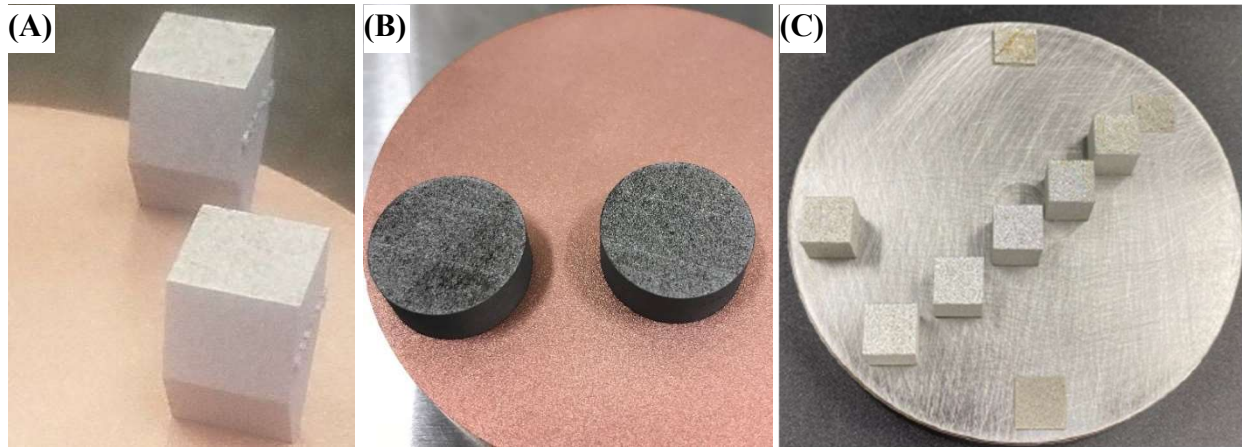


Fig 5. L-PBF AM W metallographic (A), mechanical test (B), and Mo metallographic specimens (C).

Heat Treatments

W specimens underwent a series of heat treatments at MSFC to include stress relief (SR), hot isostatic press (HIP), and recrystallization. SR was conducted from 1100-1200 °C in vacuum of 10^{-4} Torr for 1 hour. HIP was conducted at 1700-1800 °C from 172-193 MPa for 1-4 hours [7]. Recrystallization occurred from 1250-1350 °C in vacuum of 10^{-4} Torr for 1 hour. It must be mentioned that heat treatment of refractory metals requires furnaces to hold a high degree of cleanliness, treated in vacuum or ultra-high purity argon, often wrapped in a foil with high oxygen affinity such as Ta. The resulting structure-property relations for AM W specimens are now discussed.

Microstructural Characterization

As-built and heat treated W specimens had Relative density (ρ_{rel}) determined using Archimedes' method or helium pycnometry and density ranged from 93-95 %RD and Mo was found to be 97.9 %RD. These value are substantially lower than standard L-PBF AM density cutoff threshold of 99.5 %RD, but is not unexpected considering the inherent micro-cracks that form during the AM process. The microstructure was evaluated using optical microscopy as shown in Figure 6 for both W and Mo.

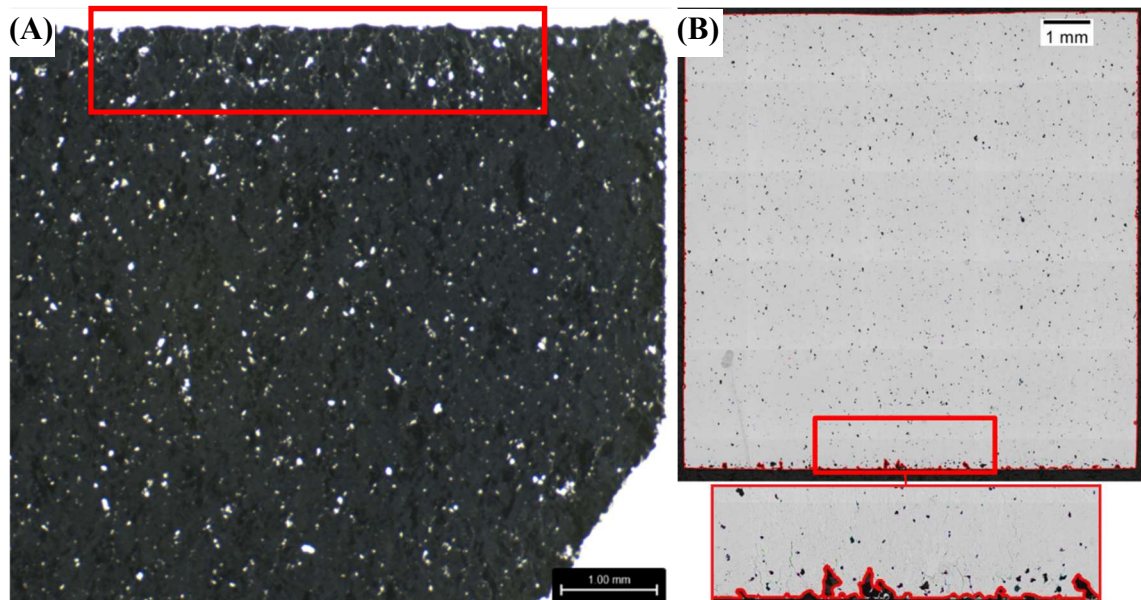


Fig 6. (A) Scanning electron micrograph of L-PBF as-built W [4] and (B) optical micrograph of as-built Mo. Red boxes indicate areas of substantial surface connected micro-cracks.

Mechanical Testing

Meso-scale tensile specimens in the as-built and heat treated conditions underwent tensile testing at ambient temperature with load applied parallel to the build direction (Z). The median ultimate tensile strength (UTS) is shown in table 1:

Table 1: Tensile data of L-PBF AM W at 20 °C.

Condition	UTS _m (MPa)
As-Built	157.65 ± 17.9
Stress-Relieved	164.34 ± 29.7
Recrystallized	177.30 ± 4
HIP	In work

For comparison, the UTS of wrought W at 20 °C is 349.26 MPa, which means the L-PBF AM W is 50.7 % the strength of wrought. This result is not surprising when we consider ρ_{rel} ranged from 93-95 %RD and the micro-cracks inherent in the microstructure act as stress concentration sites to an already fracture prone material undergoing tensile loading at well below the ductile to brittle transition temperature. Future tasks include tensile testing as a function of elevated temperature since when W AM components are in service they will not be operating in a temperature range that would make them brittle.

Surface Finish

As-built areal surface roughness (S_a) as a function of geometric angle was measured on a number of specimens using a Keyence VR-3200 wide area 3D scanner using a magnification of 80x in super-fine mode. Surface finish was found to be typical for L-PBF AM, taking into account the layer thickness, laser focus diameter, and powder PSD. Table 2 shows the resulting AM W surface finish values measured as a function of angle from Z.

Table 2: Surface Finish of As-Built L-PBF AM W.

Condition	S_a (μm)								
	0° (vertical)		15°		30°		45°		90° (horiz)
	Blade-Facing	Back	Up-Facing	Down-Facing	Up-Facing	Down-Facing	Up-Facing	Down-Facing	Up-Facing
W As-built	5.5	6.3	5.4	7.3	6.1	8.9	9	15.7	-

Additional surface finish improvement is readily achievable using commercially available surface modification methods such as shot peen, chemical etch, slurry hone, electro-polish, etc. Future tasks will focus on thoroughly evaluating the applicability of these surface finish modification methods to W, Mo, C103, and other refractory metal options as they become implemented. Such surface enhancement methods will be critical to optimize AM surfaces not only to improve fatigue life of refractory AM components but will also impact protective coating adherence, which are commonly used to protect oxygen sensitive refractory metals at elevated temperature. Coating examples include molybdenum-silicide or R512E (Si-20wt%Fe-20wt%Cr), which are commercially available; however, the deposition process will likely require optimization based on the AM part surface condition [8], [9]. In addition to adherence the performance of the coating to protect the refractory metal substrate will also require characterization [10], [11].

Leak Testing and Coatings

Due to the known issue of micro-cracks that are volumetrically distributed throughout the W AM material it is important to characterize the impact on not only the mechanical properties but also gas permeability since many of the applications used in propulsion will require some degree of gas retention. Leak test specimens were printed from L-PBF AM W with 0.5 mm, 0.76 mm, and 1.0 mm wall thickness (WT) as shown in Figure 7. Four specimens for each wall thickness were produced in order to undergo heat treatment schedules previously discussed prior to testing. Specimens were tested with as-build surface finish and no additional post-processing beyond powder removal and heat treatment was applied.



Fig 7. As-built L-PBF AM W leak test specimens with 0.5 mm wall thickness.

Leak specimens were attached to flexible plastic tube and the joint between the specimen and the tube sealed with epoxy. The other end of the tube was attached to a regulator and gaseous nitrogen supply. The specimen was immersed in water and the nitrogen pressure was gradually increased incrementally from 68.5 kPa (10 psig), 137.9 kPa (20 psig), 206.8 kPa (30 psig), and 275.8 kPa (40 psig) to observe gas leakage through the walls. All AM W specimens in as-built and heat treated conditions experienced leakage at a range of relatively low internal gas pressures. The 0.5 mm WT specimens experienced a slight leak at 10 psig, as observed by the formation of a single bubble that slowly grew over time and experiences bubbles free flowing through the walls at 20 psig. The 0.76 mm WT specimens experienced a slight leak at 20 psig and free flowing leakage at 30 psig. Finally, the 1.0 mm WT specimens experienced a slight leak at 30 psig and free flowing leakage at 40 psig. An example of the leak testing qualitatively showing gaseous nitrogen leak through the leak specimen wall as a function of pressure is shown in Figure 8. Specimens in the HIP condition showed the same leak result indicating that the process was ineffective at closing micro-cracks, which was not unexpected considering the max HIP furnace temperature of 1700-1800 °C is low compared to the desired HIP temperature of 70% of T_m or approximately 2400 °C. One potential option to prevent gas leakage is to increase wall thickness but this has obvious drawbacks such as increased weight and does not address the underlying issue of micro-cracking. Obviously, gas permeable materials are not desirable for most propulsion applications, except perhaps transpiration cooling. For this reason, ongoing efforts are underway to determine the influence of coatings to mitigate gas permeability of AM W and Mo parts. These coatings are simply a near-term option until more advanced AM methodologies (dispersoids and new alloys) become widely available, which will have been optimized to minimize if not eliminate unnecessary micro-cracking or excessive porosity.

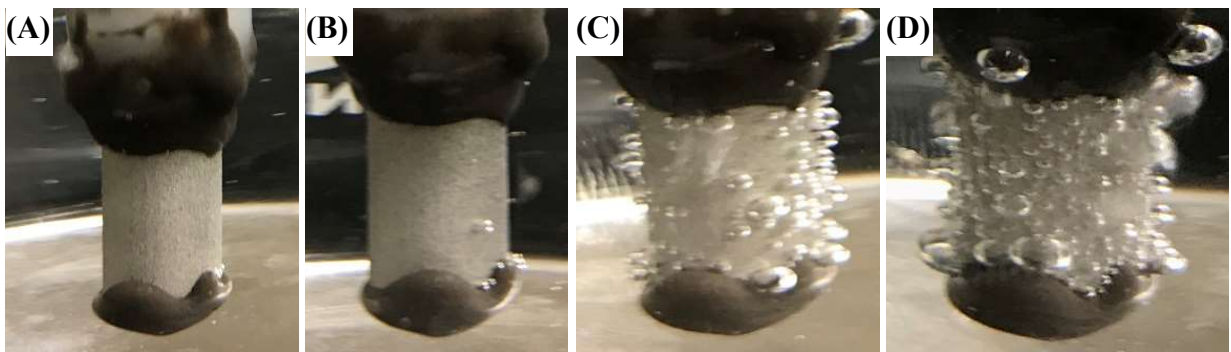


Fig 8. As-built W 0.5 mm WT specimen leak testing at 10 psig (A), 20 psig (B), 30 psig (C), and 40 psig (D).

Potential coating methods include electro-forming, vacuum plasma spray, and LP-DED to add a thin layer (0.1-0.5 mm) coating of the same material as the AM substrate or a compatible material. Coatings used in refractory metals are typically applied to protect oxygen sensitive materials during elevated service, but in this circumstance may also help to address gas permeability for low pressure applications where AM parts with micro-cracks may still be considered functional. Coating adherence will also be characterized as a function of surface finish from as-built to polished using a variety of surface enhancement techniques previously discussed.

Non-Destructive Evaluation Limitations

Non-destructive evaluation methods such as x-ray computer tomography (CT) and x-ray micro-focus computer tomography (μ -CT) are relied upon heavily for inspection of complex AM components. The high spatial resolution provided by μ -CT is able to discern fine geometric features, particularly for parts printed from relatively low effective atomic number elements that have low radio-opacity, meaning that a sufficient x-ray flux can penetrate into the part and to the detector to yield a sufficiently high signal-to-noise ratio that allow for images to be resolved with adequate resolution [12], [13]. Unfortunately, x-ray CT and μ -CT are unsuitable for inspection of refractory metal parts since refractory metals have high atomic numbers, which are have high radio-opacity resulting in high scatter, low penetration depth, and poor signal-to-noise ratio, which result in unresolvable images let alone defect detection [2]. Neutron CT is a potential option in theory but has traditionally required access to national user facilities, which is not practical in a production environment. However, commercial neutron CT vendors are now a potential option, although part activation and cool-down may have significant implications on part schedule and handling. Destructive methods such as automated sectioning and imaging produced a CT-like model but not suited for production. Ultrasonic inspection is generally difficult to apply to AM due to the inherently rough surface finish of AM parts and would require surface enhancement to allow for appropriate application. Alternative non-destructive evaluation methods are required for pragmatic utilization on AM refractory metals and alloys.

Conclusion

Refractory metal AM been demonstrated as viable with W, Mo, Ta, C103, as well as several refractory alloys in development. Recommendations for future work include surface finish enhancement evaluations, coating process and material evaluations, elevated temperature mechanical testing, component hot fire testing, and continued development of dispersoid strengthened refractory metals and RHEAs to offer a greater array of formulations to meet the increasing demand for complex components operating at extreme temperature environments.

Acknowledgements

The authors would like to thank Marvin Barnes, Daniel Cavender, Paul Gradl, Zachary Jones, Megan Le Corre, Christopher McKinney, Matthew Medders, John Peugeot, Sara Rengifo, Jhonathan Rosales, Pat Salvail, Jeffrey Sowards, Jamelle Williams, and Ryan Wilkerson of NASA MSFC; Keith Peterson of NASA Ames Research Center, Ankit Saharan and Mahemaa Rajasekaran of EOS North America; and Francisco Medina and Edel Arrieta of the University of Texas at El Paso. This project was funded under NASA Center Innovation Fund and Technology Excellence fund.

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