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COMPONENTS

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Directed Light Fabrication of Rhenium Components

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Abstract

Directed Light Fabrication (DLF) is a direct metal deposition process that fuses powder, delivered by gas into the focal zone of a high powered laser beam to form fully dense near-net shaped components. This is accomplished in one step without the use of molds, dies, forming, pressing, sintering or forging equipment. DLF is performed in a high purity inert environment free from the contaminants associated with conventional processing such as oxide and carbon pickup, lubricants, binding agents, cooling or cleaning agents. Applications using rhenium have historically been limited in part by its workability and cost. This study demonstrates the ability to fuse rhenium metal powder, using a DLF machine, into free standing rods and describes the associated parameter study. Microstructural comparisons between DLF deposited rhenium and commercial rhenium sheet product is performed. This research combined with existing DLF technology demonstrates the feasibility of forming complex rhenium metal shapes directly from powder.

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Conventional Processing

Refractory metals are difficult to fabricate at an acceptable cost using conventional processing methods. Because of its high-strain hardening characteristics, rhenium requires many iterations of forging and annealing to form a part by conventional metal working techniques. Consequently, powder metallurgy techniques and chemical vapor deposition (CVD) technologies are often used. However, these are expensive and time consuming processes requiring multiple processing steps including fabrication of mandrels, patterns and dies. For example, CVD requires the fabrication of a suitable mandrel over which rhenium is chemically deposited at high temperature. The mandrel must then be removed by machining, melting or acid leaching. Due to its expense and high cost of recovery, it is used sparingly in general applications, typically as an alloying addition. It work hardens extremely when cold and may only be worked 10% to 15% before requiring a high temperature anneal to fully recrystallize. The primary factors which have limited the use of these alloys are their cost and difficulty in fabrication using conventional methods. Forming and machining these alloys can be difficult and costly with waste associated with these processing methods. Casting technology is not feasible for the highest melting temperature refractory alloys due to temperature limitations and constraints on crucible and mold materials. Powder metallurgy techniques have been applied to these materials, such as high temperature HIP bonding and compacting. These techniques also have their limitations with respect to cost, speed, achievable geometry, required tooling and waste product.

In the case of rhenium, hot working must be carried out in a hydrogen or vacuum environment. In air the metal readily oxidizes to heptoxide (melting point 297 C), so hot working above 500 C is not possible, further complicating the fabrication process. After extraction, Re powder typically under goes compaction, vacuum pre-sintering at 1200C, H₂ sinter, and hot pressing, to 90% density [1].

Hipping, then strip forming into bar using cold rolling or swaging is limited to 10% reduction, and many high temperature (1300 to 1500 C) anneals are often performed. This extensive value-added processing contributes to the high cost and fairly limited range of commercial shapes.

Directed Light Fabrication

DLF processing has been developed to provide a pure, single-step, waste-free process to fabricate near-net-shaped parts using almost all metals including rhenium. [2-6]

The high value added and the ability degree of to produce complex shapes not otherwise achievable will open a wide range of economically feasible uses for this and other refractory metal components.

DLF is a free-form metal fabrication process. That is, one can form a functional, fully dense, metal component without the use of a die, pattern, or mold, and without the use of metal casting or metal forming equipment (Figure 1). Consequently, it bypasses traditional forming processes and powder metallurgy processing and reduces the processing steps required

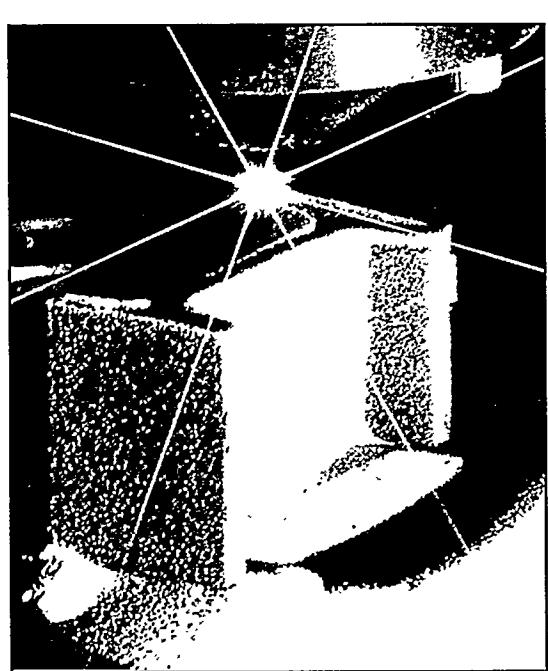


Figure 1. DLF free-form laser deposition of a fully dense hollow I-Beam of 316 stainless steel from a computer solid model.

to achieve a finished product. Unfused material is not contaminated or altered by the process allowing the powder to be reclaimed, making the process waste-free. The process is environmentally contained

in a high purity inert argon atmosphere and no solvents or lubricants, such as those used in forming processes are used, improving the purity of the final product over conventional processing. Complex components are possible, reducing or eliminating welding or assembly steps, minimizing weight in the structure, and minimizing the cost of raw material. In Figure 2, we have compared conventional chemical vapor deposition and forming by powder metallurgy processing and DLF processing of rhenium.

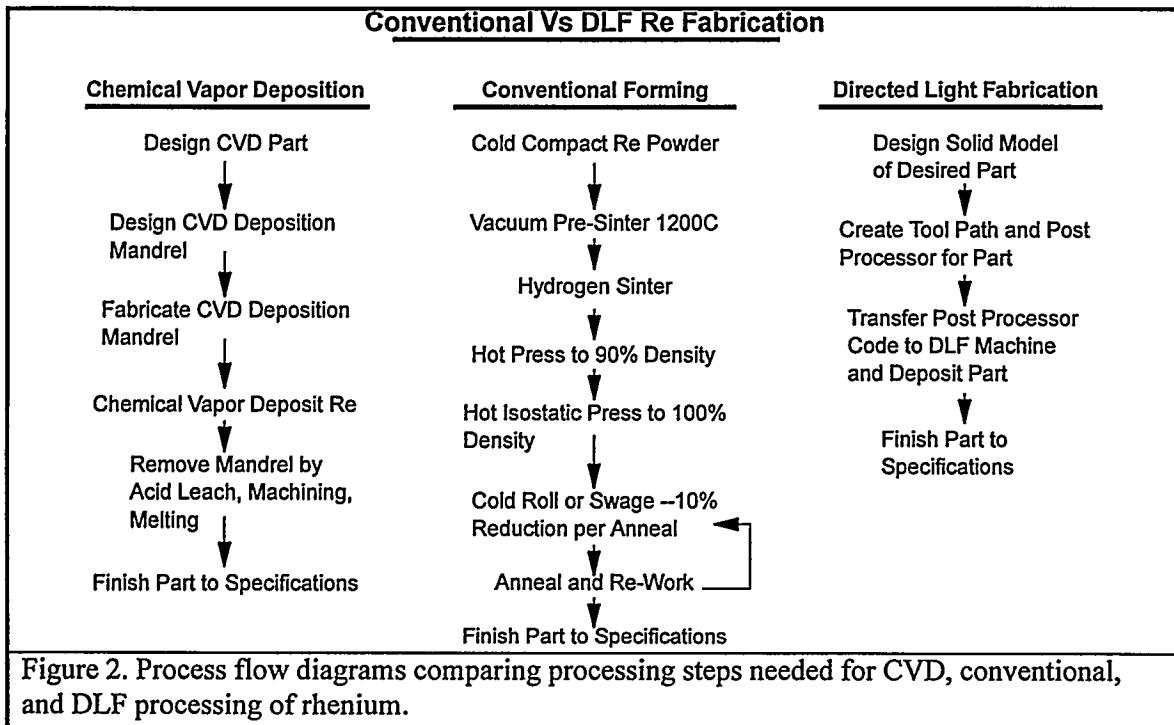


Figure 2. Process flow diagrams comparing processing steps needed for CVD, conventional, and DLF processing of rhenium.

The DLF process is accomplished by creating a solid model of a desired component on a computer, producing a tool path from the solid model, and then using the tool path to guide a laser metal deposition system to form successive thin cross-sectional layers defined by the part boundaries until enough layers are fused to form the component. Figure 3 schematically shows the process with a 3-

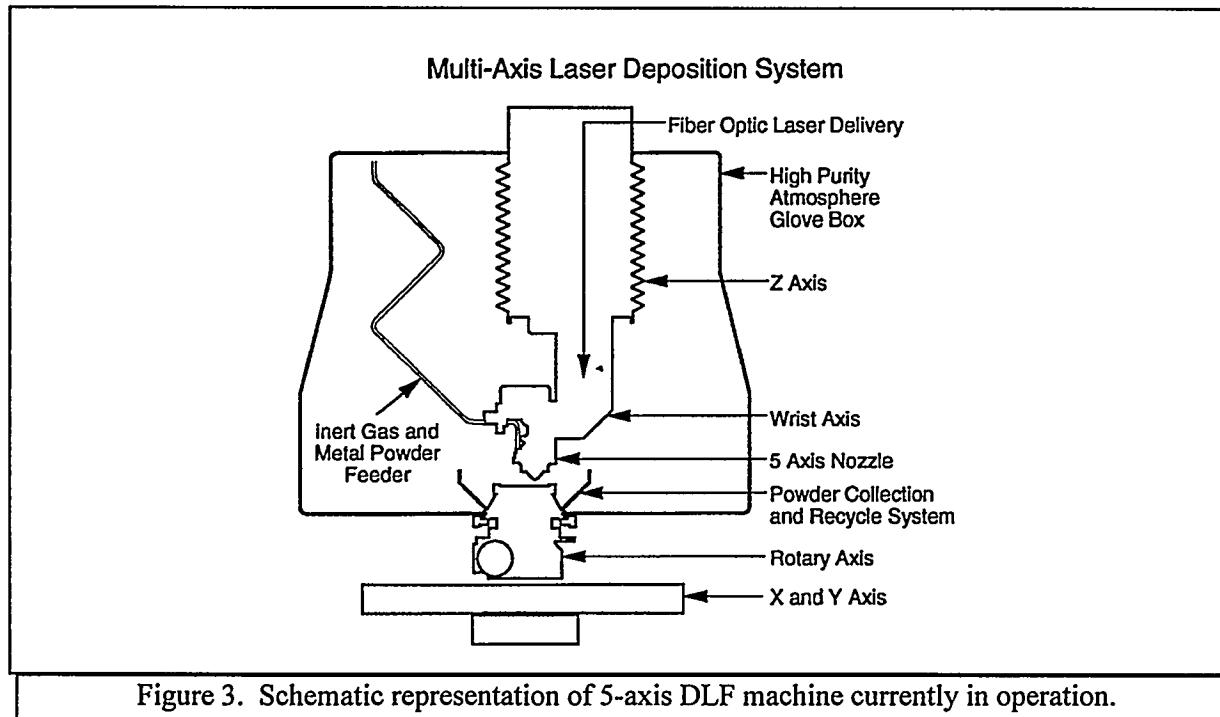


Figure 3. Schematic representation of 5-axis DLF machine currently in operation.

axis DLF deposition system where the motion of the laser beam focal point is controlled by the tool path commands. First a solid 3-dimensional model is produced using commercial computer aided design (CAD) software, and then a tool path is created using computer aided manufacturing (CAM) software. The tool path code is then post processed to produce a computerized numerical control (CNC) code specific for controlling the DLF machine.

The DLF equipment consists of the laser focal optics mounted on a vertical or wrist axis which delivers the laser beam to a focal point where deposition takes place. Metal powders of the desired composition are continuously delivered to the focal zone through the same laser focusing head where they melt into a small liquid metal pool. The molten pool moves with the focal point of the laser beam and continuously solidifies at the trailing edge of the pool forming the deposit which is built on a platform that is moved by two motion axes in the horizontal plane. The process is environmentally contained in a glove box atmosphere of argon with moisture and oxygen removed typically down to less than 5ppm. Mechanical strength of 316 stainless steel plates deposited by DLF are equivalent to annealed properties of wrought conventionally processed material. The DLF plate deposits have a yield strength of 30 ksi, ultimate strength of 70 ksi, modulus of 30×10^6 psi. Cooling rates have been calculated using secondary dendrite arm spacing and have shown 10^3 K/s to 10^4 K/s cooling for stainless steel rods and plates respectively[4].

Using the DLF process, we have made the parts shown in Figure 4. We made these plates, tubes, cones, angles, hemispheres, and cubes to demonstrate DLF capability to deposit various geometric features such as overhangs (hemisphere), straight sides, sharp corners, bulk deposits, cubes), tubes and cones. Demonstrated by the hemisphere, DLF has reached a maturity allowing deposition of 3-dimensional parts in planes other than just the horizontal xy plane used by all other processes. This capability eliminates the necessity of part support structures for overhangs, and special tooling required for "out-of-the-horizontal-plane deposition." The parts are uniform with straight sides, smooth contours and sharp corners.

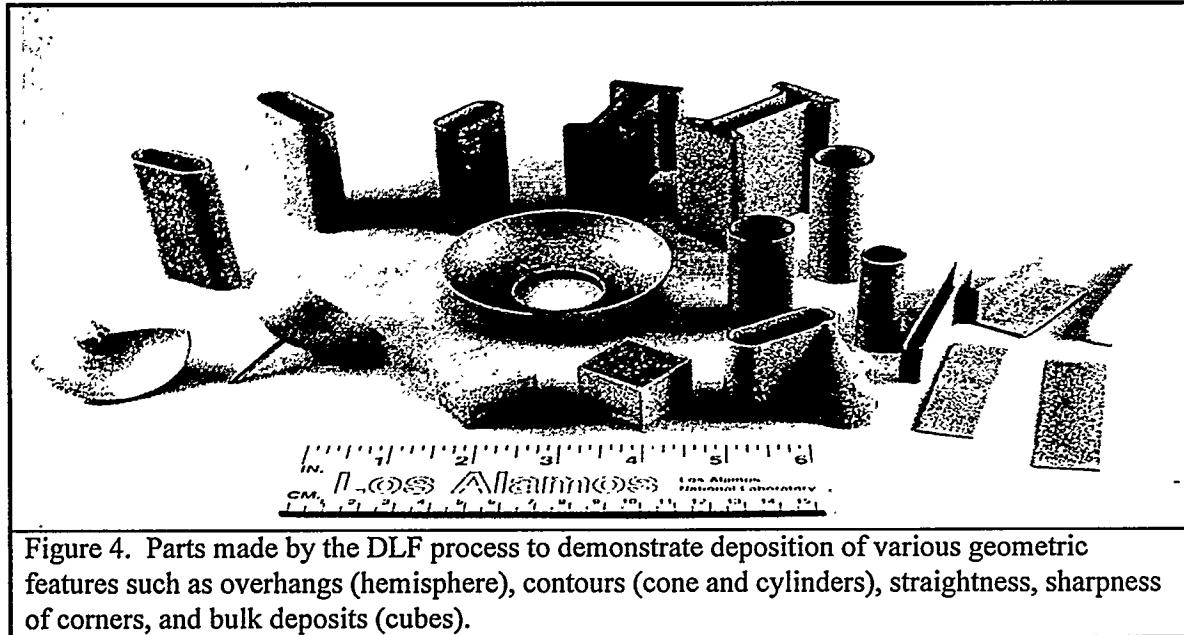


Figure 4. Parts made by the DLF process to demonstrate deposition of various geometric features such as overhangs (hemisphere), contours (cone and cylinders), straightness, sharpness of corners, and bulk deposits (cubes).

With our current DLF technology, we can produce near-net shape components, meaning that parts can be produced within a 0.010" envelope of the desired finish dimensions and a final finishing operation might typically be expected to produce the desired surface finish and meet accuracy requirements. However some parts can be formed to closer than 0.010", depending on geometric complexity, and may even be used in service as-deposited by DLF.

We have applied DLF to free-form fabrication of a broad range of metals and intermetallic compound including 316 stainless steel, 410 stainless steel, iron-nickel alloys, P20 tool steel, aluminum-copper alloys, silver-copper alloys, titanium alloys, tungsten, rhenium molybdenum disilicide, and nickel aluminide to demonstrate that the process is applicable over a wide range of melting points and compositions.

Experimental Procedure

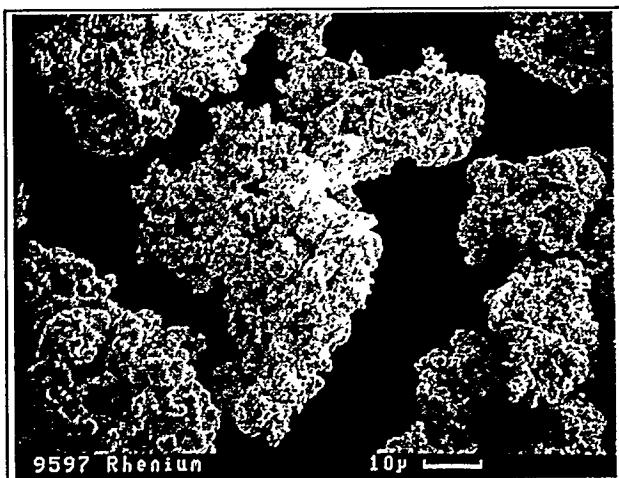


Figure 5. Rhenium powder used in this experiment.

Powder

Rhenium powder used for this experiment was very fine and had an agglomerated texture as is shown in Fig. [5]. It had been stored for 28 years, without the aid of inert or vacuum packing. A high potential for surface oxide was present but the powder was not tested for oxide coatings. It had a caked resistance to flow but was vacuum baked which helped the flow characteristics. The average powder size was 3.12 microns. The powder contained enclosed porosity as evidenced by its theoretical density of 89.4% as measured by helium pycnometer .

Laser and DLF System

A three axis DLF system was used for these initial DLF rhenium trials. A Lumonics, Nd:YAG, 3 into 1, uses (3) 400W avg. power industrial lasers focused into a single 1 mm spot by the use of fiber optic beam delivery. This laser is mounted above the inert atmosphere DLF powder fusing chamber and is connected to a Z axis movement. The beam passes through a window into the powder delivery head to deliver the focused laser beams into the focused stream of powder particles. The lasers were fired in parallel (simultaneously) for 7.5 msec and at a frequency of 10 Hz for all of the tests described below. Sequential firing (in series) was insufficient to attain the peak power required to fuse the Re powders so simultaneous firing of all three lasers was utilized. An argon gas purifying system was used with deposition performed at oxygen levels of 20 ppm. The DLF chamber and feed system was cleaned using alcohol rinse and wiping to remove powder from previous experiments. Wires were grown in the Z direction beginning on a steel substrate at 0.5 mm/s at 45 to 60 J per pulse using all three lasers.

Of the 18 trial runs performed, four rods were removed from the substrate for further analysis. They displayed rough partially fused surfaces but were continuously fused at the core and strongly bonded to the substrate. They displayed a soft ductile bending behavior and ranged from 38 mm to 70 mm in length and 1 mm to 2 mm in diameter.

The largest diameter sample was cut wet with a diamond saw, ultrasonically cleaned and mounted in cross section and longitudinal section with respect to rod growth. In addition a small piece of commercial Re sheet was mounted in section along and normal to the roll forming plane. This provided side by side comparison of microstructures and hardness data with the DLF deposit. Sample preparation included a rough grind and polish to 3 micron with alumina grit. Photomicrographs were taken in the as polished condition to reveal voids, inclusions and precipitates. Scanning electron microscopy was used to image powder morphology and examine porosity and other features of the as polished surface. Vickers hardness measurements were taken from both samples. The samples were

then electroetch polished using a $0.05\mu\text{m}$ AlO_2 slurry in water with a few drops of Murakami's reagent to remove the disturbed layer. Samples were etched to reveal microstructure, examined and photographed using bright field, polarized and DIC light.

Results

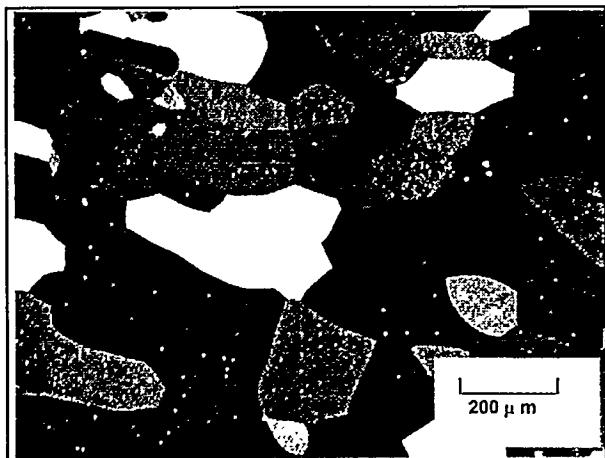


Figure 6. A transverse rod section micrograph of rhenium deposited by laser melting of powder.

The DLF deposited Re was shown to be continuously fused into large polyhedral grains ranging in size from ASTM 3.0 grain size, less than 125 microns to large grains of many hundred microns in size Fig [6]. This range of values may be a result of the variation in cooling rate inherent with the use of the pulsed laser heat source. Polarized light revealed a strong response indicative of a highly random crystallographic orientation. The commercial Re sheet sample showed a grain size of ASTM 6.5 or less than 45 microns .

Porosity was shown to form in two distinct sizes, ranging from $2\mu\text{m}$ to $5\mu\text{m}$ for the small porosity to $\sim 50\mu\text{m}$ to $75\mu\text{m}$ in size for the large porosity Fig. [7]. The small porosity was shown to form

at grain boundaries and within the grains when examined under 500X magnification and differential

interference contrast (DIC) illumination Fig. [8]. No porosity was found in the sample of commercial plate. SEM examination of an internal pore surface revealed a striated twinned surface indicative of basal plane slip due to solidification stressed cooling Fig. [9].

The large grain size of the DLF deposited material produced a large variation in hardness values due to an insufficient number of grains per indent, a wide variation in grain orientation and elastic recovery. The mean hardness of the DLF deposit was HV 190, with a standard deviation of 29. The mean hardness values for the commercial sheet was HV 227 in the edge section and HV 371 in the top section, with standard deviations of 10 and 36

respectively. No precipitates were observed in either samples.



Figure 8. Microporosity observed at grain boundaries and within grains.

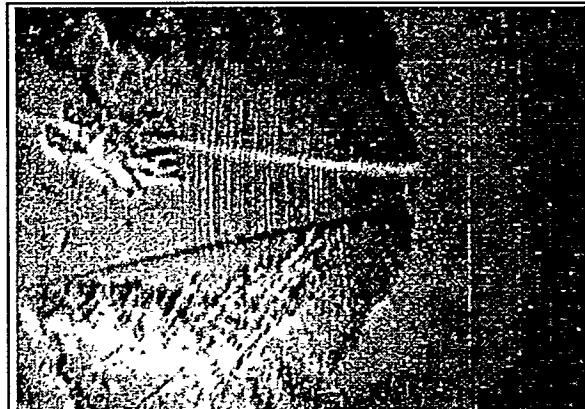


Figure 9. SEM of slip lines inside a large pore due to solidification stresses.

Discussion

The laser energy density was only just sufficient to melt and fuse the Re powder into continuous rod like shapes. Pre-trials using the laser fired in a sequential manner did not fuse the powder due to insufficient energy density. Past experiments demonstrating the fusing of tungsten powder into wires showed a much smoother deposit [7]. These experiments however used a higher energy density (less energy but much smaller spot size). A higher power continuous wave Nd:YAG system with a smaller focal spot size would be attractive for further experimentation. Porosity was evident but may have been introduced into the melt pool by the porous powder and trapped within the rapidly solidifying structure due to the pulsed energy (high cooling rates) used. The powder used may also have been oxidized, though no attempt was made to ascertain this condition. The bimodal size and spatial distribution of pores suggests more than one formation mechanism. Slower cooling rates associated with CW laser processing may provide opportunity for porosity to be replaced by liquid within the solidifying deposit. New powder without the potential for oxidation would be recommended. A larger and more spherical powder would be attractive with respect to better flow characteristics. Commercial powder feeders made specifically to feed finer powders are available and may serve to alleviate the need for a second powder processing step such as ball milling or rolling. Future work may include use of the recently completed and tested 5-axis DLF system (Figure 3) in the LANL Materials Science Laboratory which features both improved powder feeding capability, 2 kW of laser energy, a smaller spot size and a new DLF powder head with significantly improved powder focus in horizontal and vertical orientations. Tantalum metal shapes have been successfully deposited using the 5-axis DLF system in the vertical and horizontal positions and demonstrated surface finishes as good as stainless steel.

DLF technology has demonstrated the fabrication of complex shapes which would be difficult to fabricate out of rhenium using conventional technology. Under optimal conditions, no contamination should enter the fabrication sequence,(i.e.; oxides, carbon or steel pickup or forming lubricants) and the powder, not fused, could be used over and over again without loss. Aerospace and space applications could directly benefit from the development of this near net fabrication technology. Commercial applications such as crucibles, furnace hardware, high temperature contact surfaces and electron sources such as emitters or sputtering targets could find immediate applications

Conclusions

DLF direct laser deposition of rhenium powder was demonstrated. A free standing rod structure was formed which demonstrated a continuously solidified microstructure displaying ductile behavior. Comparison with commercial rhenium plate showed a large grained structure having the hardness of annealed rhenium vs. a small grained recrystallized structure in the plate sample. This research combined with existing DLF technology demonstrates the feasibility of forming complex rhenium metal shapes directly from powder.

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