



## Near Net Shape of Powder Metallurgy Rhenium Parts

Todd Leonhardt and James Downs

Rhenium Alloys Inc.

Elyria, Ohio USA

### Summary:

In this paper, a description of the stages of processing necessary to produce a near-net shape (NNS) powder metallurgy (PM) rhenium component through the use of cold isostatic pressing (CIP) to form a complex shape will be explained. This method was primarily developed for the production of the 440 N and 490 N liquid apogee engine combustion chambers used in satellite positioning systems. The CIP to NNS process has been used in the manufacture and production of other rhenium aerospace components as well. Cold isostatic pressing (CIP) to a near net shape utilizing a one or two-part mandrel greatly reduces the quantity of rhenium required to produce the component, and also significantly reduces the number of secondary machining operations necessary to complete the manufacturing process. Further, the developments in near-net shape powder metallurgy rhenium manufacturing techniques have generated significant savings in the area of both time and budget. Overall, cost declined by as much as 35 % for the rhenium chambers, and manufacturing time was decreased by 30-40 %. The quantity of rhenium metal powder used to produce a rhenium chamber was reduced by approximately 70 %, with a subsequent reduction of nearly 50 % in secondary machining operation schedules. Thus, it is apparent that the overall savings provided by the production of near-net shape powder metallurgy rhenium components will be more than merely another aspect of any project involving high temperature applications, it will constitute significant benefit.

**Keywords:** rhenium, powder metallurgy, cold isostatic pressing, hot isostatic pressing, liquid apogee engine, thrusters, satellite, and aerospace.

## Introduction:

The use of powder metallurgy (PM) rhenium as the material of choice for high performance liquid apogee engines, as well as other high temperature aerospace applications, has proven to be both accurate and precise for the demanding requirements created by the very nature of their function and operation. Rhenium has a melting point of 3459 K, and has the highest modulus of elasticity of all the refractory metals (1,2). Powder metallurgy rhenium has consistently provided high yield and ultimate tensile strengths at ambient and elevated temperature, while maintaining exceptional creep and low cycle fatigue properties required for high temperature aerospace applications (1,2, 3).

Historically, rhenium has been produced in a variety of standard metal forms; rod, bar, plate and sheet, and these basic shapes underwent electrical discharge machining (EDM) methods to generate the specified shapes and sizes to indicated tolerances. The current method of consolidating powdered rhenium metal for rods has been cold isostatic pressing (CIP). In this technique rhenium metal powder (figure 1) is packed into a flexible mold with a "rigid can" to maintain the desired shape (figure 2). Then the can/mold assembly is immersed in water contained within a CIP vessel. This "wet bag" process uses hydrostatic pressures of 210 to 410 MPa, which transfers the isostatic pressure to the mold and consolidates the rhenium metal powder into a "green" rhenium compact (4).

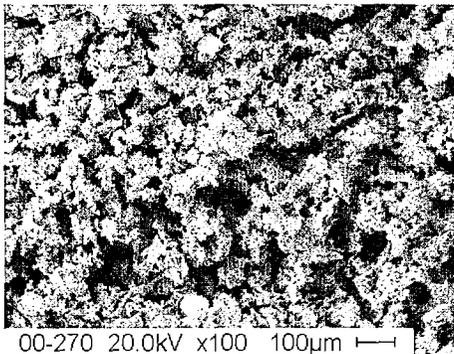


Figure 1: -200 mesh rhenium metal powder

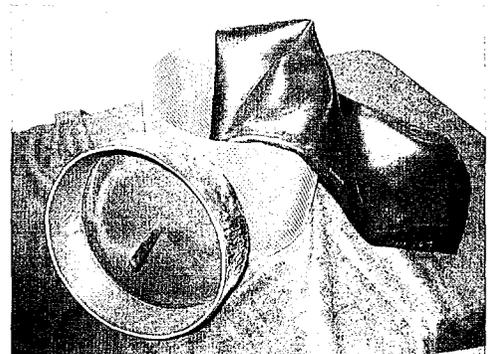


Figure 2: Typical can and mold for cold isostatic pressing

Prior to Rhenium Alloys, Inc. undertaking a National Aeronautics and Space Administration (NASA) Phase II Small Business Innovative Research (SBIR) program, all complex parts and components based on PM were manufactured using a standard rod or a large diameter rod (ingot) that involved numerous machining operations. The initial powder metallurgy apogee thrusters were made from a 15-30 kg rhenium ingot, and while this method worked well extensive machining was required to produce a high quality part (5). As an example, a 30 kg rhenium ingot (figure 3), was produced to make a finished part weighing only 2.4 kg. It was apparent that change was necessary in the PM techniques being utilized to effectively and efficiently produce components of a consistently high quality with less scrap generation. This led to our initial research into the area of powder metallurgy near-net shape manufacturing.

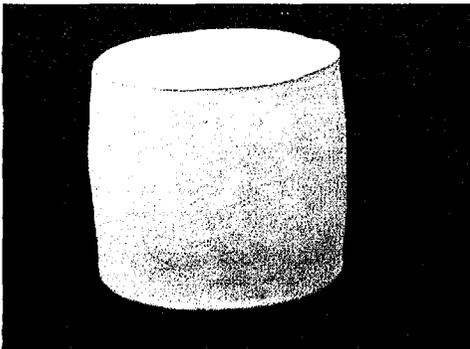


Figure 3: A hot isostatic pressed rhenium ingot

Rhenium Alloys, Inc. selected a technique that appeared to fulfill all of the criteria for near net shape processing. That method was cold isostatic pressing (CIP) to form a near-net shape part. The CIP technique achieved all of the goals that had been set forth, and established an alternative method of repeatedly producing rhenium thrusters of a consistently high quality.

## **Procedure:**

As part of the NASA Phase II SBIR, two (2) completed and “deliverable” thrusters of different and unique types were to be manufactured; one (1) each of the 440 Newton (N) and 490 N high performance bi-propellant liquid apogee rhenium/ iridium thruster. Both thrusters have rhenium as a substrate with iridium applied as an oxidation-resistant coating. Each of the thruster designs utilizes a conventional convergent-divergent design to produce thrust for use in low-earth orbit and/or a geo-stationary orbit for satellite positioning systems.

To produce a component possessing complex shape properties, the CIP tooling must have a design contour similar to that of the actual part, and will entail the use of a contour “can” and mold, as well as a one or two-part solid pressing mandrel. This method of using a pressing mandrel is known as the “collapsing bag technique” as described in ASM Handbook Vol. 7 (4). This technique was used to manufacture the thruster, with a two-part pressing mandrel, which generated a near-net shape “green” part.

Because the thrusters are of a convergent-divergent cone design, it was necessary for the mandrels to be divided at their narrowest point. Also the mandrel was manufactured with a slight taper to allow it to be removed with out scratching the inside of the “green” compacted part during the mandrel extraction process. In this method, the mandrel holds the thruster’s inside diameter contour while maintaining the dimensional tolerances, (figure 4). The flexible mold takes the contour of the mandrel, and the green compact gets a contoured outside diameter matching the shape of the mandrel. In this technique the powder fill controls the wall thickness of the near net shaped (NNS) part with contoured inside and outside surfaces, as shown in figure 5.

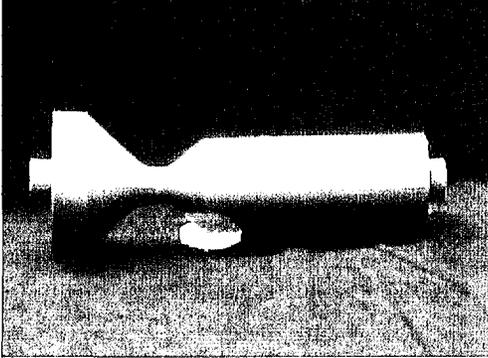


Figure 4: Mandrel used for cold isostatic pressing of NNS thruster

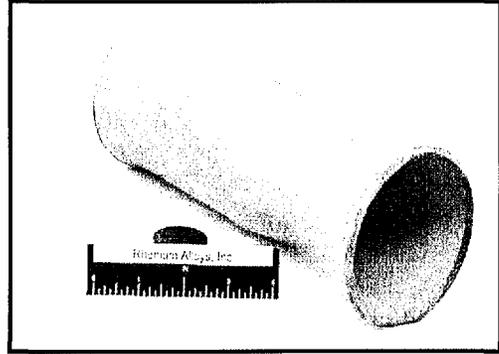


Figure 5: "Green" near net shaped rhenium thruster.

Following removal of the mandrel from the "green" NNS compact, the parts are placed in a horizontal position and are "lightly" pre-sintered at a low temperature to ensure that no "sag" will occur. Next, the thruster is pre-sintered again, this time at a higher temperature and in a vertical position, to promote shrinkage prior to high temperature sintering at 0.8 of the melting point.

The sintering process can be performed in either a hydrogen atmosphere or in a vacuum for a period of several hours. A sintered density of 97 % of theoretical density of  $21.04\text{gr}/\text{cm}^3$  was achieved with the CIP-ing to NNS process. The NNS thruster was close to the desired dimensions as sintered. Then, hot isostatic pressing (HIP-ing) was employed at high temperature and pressure for several hours. And, due to the high as sintered density, the NNS shaped part can be made without "canning", but with lower density parts the can must be applied to avoid trapping the HIP gas in the porosities.

A "canning" operation can also be prohibitive due to the complexity of the shapes involved. The formation of a metal can around a complex shape is not only extremely difficult but also very costly. Consequently, the "can-less" or "container-less" technique becomes essential in the hot isostatic pressing (HIP) method of producing complex rhenium components of nearly full density. Further, during the HIP-ing process the NNS thruster shrank an additional 2-4 %, ensuring dimensional tolerances would be achieved.

Following the HIP-ing process, electrical discharge machining (EDM) and diamond grinding procedures were performed to produce the finished part. The EDM process was used to remove all rough surfaces in general, and on the outside diameter, where the thruster “necks down”, to remove excess material. A two-step diamond grinding process, involving both “rough grinding” and a finer “finish grinding”, was utilized to achieve final dimensions and ensure that the dimensional tolerances were met.

The 440 N thruster was the first near-net shaped chamber produced using the “CIP to NNS” process, and it reached a final density of 99.4 %. The hot processing methods applied produced a fine grain microstructure with micro-porosity inside the grains (figure 6). This thruster (Figure 7) was delivered to NASA Glenn Research Center in Cleveland, Ohio, and was coated with iridium for oxidation resistance to the propellants. After the iridium coating, the thruster will be hot fire tested for flight hardware certification.

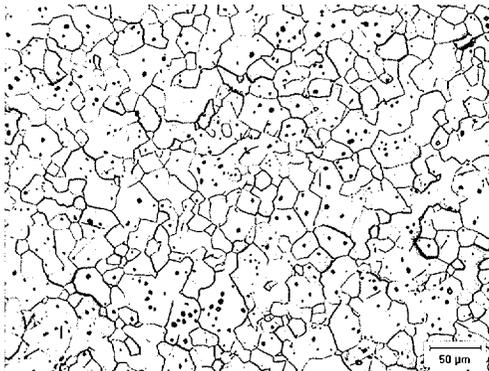


Figure 6: Microstructure of the 440 N rhenium thruster

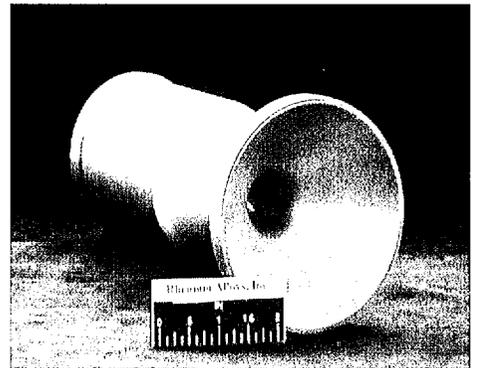


Figure 7: 440 N rhenium thruster before iridium coating

The 490 N thruster was the more challenging of the two thrusters to manufacture because of the large exit cone and smaller barrel section when compared to the 440 N thruster. The parameters for making the 490 N thruster were similar to those used for the 440 N thruster except for a higher compaction pressure. Because of the larger exit cone of the 490 N thruster, it had to be made in a two-part construction process, with a barrel section and a partial exit cone as well as a larger exit cone section (figure 8). Some

machining was performed before electron beam welding was used to join the exit cone and barrel sections together. The completed 490N thruster (figure 9) had a density of 99.9 %, with a fine grain microstructure (figure 10).

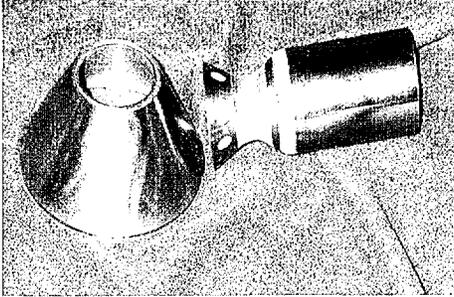


Figure 8: Two-part construction of the 490 N thruster

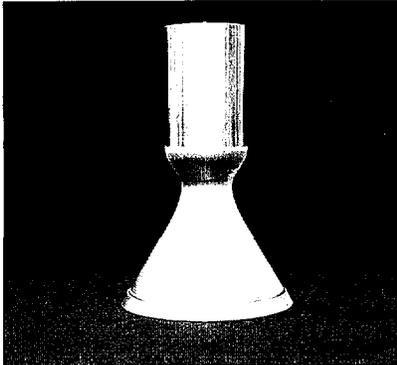


Figure 9: 490 N rhenium thruster

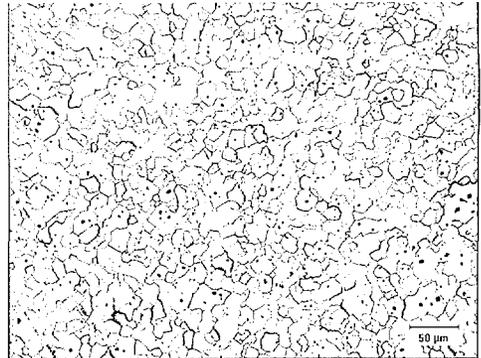


Figure 10: Microstructure of the 490 N rhenium thruster

### 100N thruster

After completing the 440 N and 490 N thrusters, Rhenium Alloys began work on several small 100 N liquid apogee engines for NASA Glenn Research Center. The thrusters were produced by the “CIP to NNS” process. The production process was shortened by using only a, light pre-sintering cycle, followed by a shorter sintering cycle. Two (2) 100 N thrusters could be hot isostatic pressed simultaneously, reducing both the production time and cost

of the 100 N thruster (figure 11). Previously, the 100 N thrusters were machined from a 76 mm rod, with a considerable amount of rhenium scrap being generated.

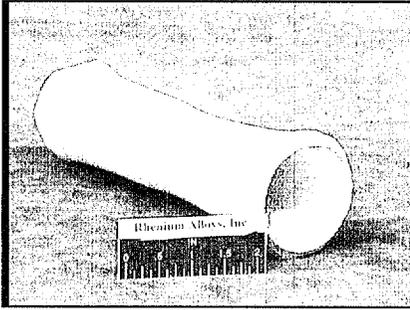


Figure 11: 100 N NNS rhenium thruster before machining

### Hemispherical domes

Two 38 mm-radius hemispherical domes (figure 12) were produced using the "CIP-ing to NNS" process. A one-piece mandrel (figure 13) and complex CIP tooling were designed to produce dome shaped objects. The retaining can and mold were contoured to produce the inside and outside diameters and to control the wall thickness of the hemispherical dome. The final wall thickness of the domes was 4 mm.

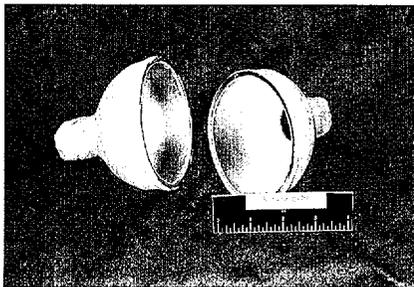


Figure 12: Two rhenium domes



Figure 13: One-piece mandrel for NNS domes

## Discussion

To produce consistent, high-quality NNS parts, a more in-depth understanding of the consolidation process was needed, and therefore, several experiments were performed to achieve a greater understanding of the flow characteristics of RMP under hydrostatic pressure. A simple tube mandrel would indicate the interactions between the powder, the mandrel and the mold. When designing CIP tooling, mold thickness, powder-fill, and mandrel design are critical elements for the success or failure of the project. That is why a solid and basic understanding of a cold isostatic pressing model is essential. The thickness of the mold provides a significant contribution to the "as-pressed" density and the shape of the outside diameter of the CIP-ed part. If the mold is too thick, it will wrinkle, or if the part has small diameter the "green" part will fail to consolidate, or have a very low "green strength".

Historical data on compaction, tap and apparent densities proved invaluable when determining the powder fill requirements. If the wall thickness were too thin or too thick, the "green" compact would crack. The first consideration is the mold interaction due to the "spring back" of the powder compact and the adhesion of the powder to the mold itself. When the part was too thin, the mold would break the compact. If the wall thickness was too great, the density was too low. A part produced with a consistent wall thickness and a contoured mold provided the best solution to both the problems of cracking and low "green strength".

Most metal powders are abrasive, and under pressure are forced into the mold material and around the mandrel, where frictional forces play a significant role. During these experiments, the mandrel became warm; indicating substantial frictional heat was generated when the powder was flowing around the pressing mandrel during the compaction process. Additionally, particle re-arrangement and compaction play a role in producing a "green" part possessing good "green" density.

One major observation was that cold isostatic pressure in a CIP unit with a complex mold and mandrel, is not truly isostatic when acting on a component with a complex mandrel design and a flexible mold. This non-uniform pressure caused density variations in the "green" part that in turn caused additional distortion problems during sintering.

Due to the compaction variation from the non-uniform isostatic pressure, predicting and controlling shrinkage and distortion was the major technical hurdle. In early experiments, slump and distortion were observed with the walls distorting or becoming oblong, and during sintering, slump would cause a thickening of the walls at the bottom of the part.

To predict and control the distortion from the consolidation process, the CIP tooling compaction and thermal processing steps had to be controlled through the CIP pressure, pre-sintering time and temperature. Further, the length of the sintering cycle needed to be understood and quantified for each distinct part. It is now possible to predict, for a given part, the shrinkage rate and thereby determine the dimensions of the tooling and mandrel.

## Conclusion

A research program was established which was funded under a Phase I and II SBIR grant from NASA. The goal of the project was to establish an alternative manufacturing method for the production of powder metallurgy rhenium combustion chambers. The task of reducing the quantity of rhenium required to produce combustion chambers was undertaken to lower the cost. This paper has focused on the evolution of manufacturing techniques utilized in the production of rhenium thrusters. One of the major benefits of the "cold isostatic pressing to near net shape method" is the ability to make parts with a density of 95-98 % after sintering and 98.5 to 99.9 % after hot isostatic pressing.

The developments in the production of near-net shape powder metallurgy rhenium combustion chambers has reduced the manufacturing cost of such chambers by as much as 35 %, and shortened the manufacturing time by 30 - 40 %. The quantity of rhenium metal powder used to produce a chamber is reduced by approximately 70 %, and the subsequent reduction in machining schedule and costs is nearly 50 %. Cold isostatic pressing to near net shape has provided significant cost savings, and also increased the quality and density of rhenium components. The overall benefits provided by this process have become an important aspect when competing in the international market for satellite propulsion components and other high temperature components made of powder metallurgy rhenium.

## Acknowledgements

The authors would like to thank Nancy Moore, Eric Blankenship and Mark Hamister for their significant contributions to the preparation of this paper. The authors acknowledge Jan-C. Carlén for providing his unwavering support, as well contributing his technical expertise in the research of cold isostatic pressing to form a near net shape rhenium part. The authors would like to acknowledge James Biaglow for his support and funding of Rhenium Alloys Inc. NASA Phase II SBIR.

## References

1. Chazen, ML, "Materials Property Test Results of Rhenium", AIAA Paper 95-2938, July 1995
2. Chazen, ML, and Sicher, D. " High Performance Bipropellant Engine", AIAA paper 98-3356, July 1998
3. Biaglow, JA, " High Temperature Rhenium Materials Properties", AIAA paper 98-3354, July 1998
4. ASM Handbook Vol. 7, Powder Metal Technology and Applications, 1998
5. Leonhardt, TA, Hamister, M. and Carlén, JC, " Near-Net Shape Powder Metallurgy Rhenium Thruster", AIAA paper 2000-3132, July 2000