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OAK RIDGE NATIONAL LABORATORY
MANAGED BY UT-BATTELLE FOR THE US DEPARTMENT OF ENERGY

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Materials Science and Technology Division

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Prepared by
OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37831-6283
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CONTENTS

LIST OF FIGURES

LIST OF TABLES

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ABSTRACT

Powder metallurgy approaches for the fabrication of accelerator target disks are being examined to support the development of Mo-99 production by NorthStar Medical Technologies, LLC. An advantage of powder metallurgy is that very little material is wasted and at present, dense, quality parts are routinely produced from molybdenum powder. The proposed targets, however, are thin wafers, 29 mm in diameter with a thickness of 0.5 mm, with very stringent dimensional tolerances. Although tooling can be machined to very high tolerance levels, the operations of powder feed, pressing and sintering involve complicated mechanisms, each of which affects green density and shrinkage, and therefore the dimensions and shape of the final product. Combinations of powder morphology, lubricants and pressing technique have been explored to produce target disks with minimal variations in thickness and little or no distortion. In addition, sintering conditions that produce densities for optimum target dissolvability are being determined.

1.0 BACKGROUND

NorthStar Medical Technologies, LLC has developed a new process for the production of Mo-99 which is utilized in the radio-pharmaceutical industry to obtain the daughter product, Tc-99m, the most commonly used radioisotope for medical diagnostics. The NorthStar process involves photon irradiation of isotopically-enriched Mo-100 targets in an electron accelerator to produce Mo-99 by the (γ, n) reaction. The proposed targets are thin disks of partially consolidated metal powder (~ 90% of theoretical density) with controlled open porosity to enhance dissolvability.

The targets must be fabricated employing reproducible and reliable methods to meet strict acceptance criteria which include diameter, thickness, flatness, and density. Thin molybdenum disks with tightly-controlled dimensional tolerances similar to those required for accelerator targets are typically manufactured by grinding and lapping of disks punched from sheet. Because of the high cost of Mo-100 enriched isotope powder, scrap and waste must be eliminated from the process thus the typical approaches are not viable

It has been proposed that the target disks be produced using powder metallurgy techniques in which metal powder is cold pressed and then sintered to achieve the desired density. The advantage of powder metallurgy is that very little material is wasted and at present, dense, quality parts are routinely produced from molybdenum powder. In fact, powder is the starting point for all virgin molybdenum products.[1] Substantial quantities of powder are currently being pressed and sintered into pellets which are used for melting and casting and as additives for alloys, into other simple shapes such as ingots from which all mill products such as sheet, plate, foil, rod, bar and forgings are produced, and into complex components employing a range of powder metallurgy approaches.

Since molybdenum metal products are predominately produced employing powder metallurgy techniques, the pressing and sintering of molybdenum powder has been investigated in great detail.[2-6] However, molybdenum powders can have widely varying characteristics and thus information concerning compaction and sintering varies significantly. Therefore, in an earlier study, molybdenum powders from various sources were evaluated to determine the effects of powder characteristics and processing parameters such as compaction pressure and sintering temperature on the properties of the densified product.[7] Samples of Mo-100 isotope powder were included in the study for comparison to commercially-available natural molybdenum powders and preliminary examination of processing requirements.

The conclusions from the early study were:

- Depending upon the source and grade, natural and isotope-enriched molybdenum powders possess significant variations in powder characteristics, such as particle size, particle shape, surface area, and degree of agglomeration.
- Powder characteristics affect both compaction and sintering and thus the as-pressed (green) and sintered densities of specimens.
- Certain molybdenum powders could be cold pressed to green densities of 90% of theoretical or greater.
- All molybdenum powders, including 5 different lots of Mo-100 powder, could be pressed and sintered to 90% density or greater.

As noted earlier, an advantage of powder metallurgy is that very little material is wasted and at present, dense, quality parts are routinely produced from molybdenum powder. The proposed targets, however, are thin wafers, 29 mm in diameter with a thickness of 0.5 mm, with very stringent dimensional tolerances. Although tooling can be machined to very high tolerance levels, the operations of powder feed, pressing and sintering involve complicated mechanisms, each of which affects green density and shrinkage, and therefore the dimensions and shape of the final product.[8] Typically, combinations of powder morphology, lubricants and binders, and pressing technique are optimized to produce parts with minimal variations in dimensions and little or no distortion. In addition, sintering conditions are also tailored to ensure uniform and reliable shrinkage and thus dimensional stability during densification.

The objective of this study is to determine the combinations of powder, lubricant, pressing technique and sintering conditions required to produce thin flat disks that meet the proposed accelerator target disk specifications.

2.0 EXPERIMENTAL

Molybdenum metal powders having a purity of greater than 99% molybdenum (metals basis) were obtained from commercial suppliers. Manufacturer powder specifications are given in Table 1. Additional characterization of the powders was performed to confirm properties and examine particle morphology in greater detail. A sample of each of the powders was examined by scanning electron microscopy to evaluate particle size and morphology and the extent to which the particles were agglomerated (Fig. 1). Particle size distribution was determined using laser light scattering with the particles suspended in high purity ethanol. BET analysis was used to determine surface area of the powders.

Table 1. Molybdenum Powder Characteristics

Molybdenum Supplier	Grade	Purity (% Mo)	Max. Oxygen (ppm)	Particle Size	BET (m^2/g)	Hall Flow (sec/50 g)
Climax Molybdenum	EM-NM3	99.9	1400	0.7 – 1.5 μm	2.83	No flow
Climax Molybdenum	NPA	99.95	1000	4.0 – 4.8 μm	0.45	No flow
Climax Molybdenum	PM	99.9	2000	- 200/+ 325 mesh (spray-dried)	NM	< 45
Large-batch reduction	AA995R2	NM	> 5000	4.8 ± 1.4 μm	0.46	No flow

NM = not measured

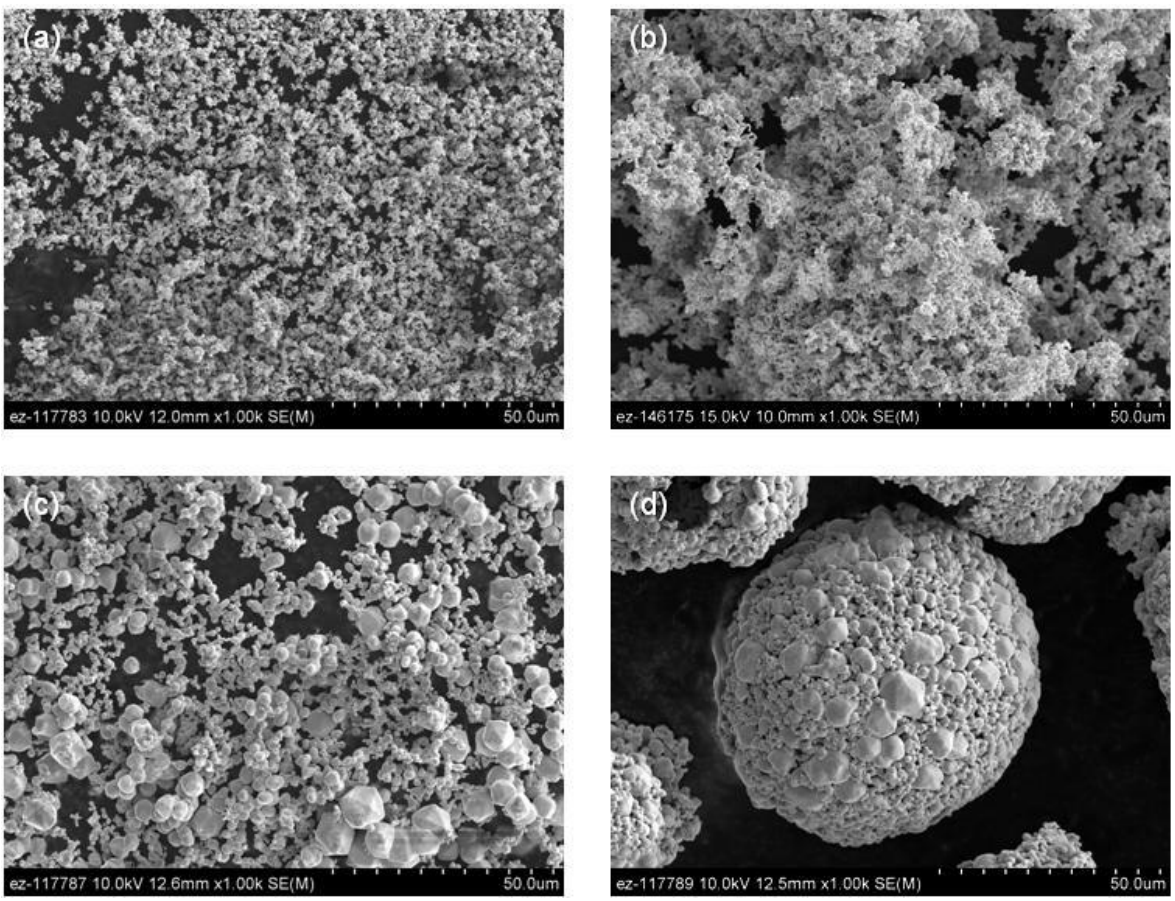

Fig. 1. Micrographs of molybdenum powders in Table 1; (a) Climax EM-NM3, (b) from large-batch reduction, (c) Climax NPA, and (d) Climax PM (spray dried).

Cylindrical tool steel and tungsten carbide punch and die sets were used to press the powders into disks. The inside diameters of the dies used to produce green disks that would sinter to 29 and 33 mm outside diameters were 30.15 and 33.2 mm, respectively. The powders (~ 4 g for 33 mm disks and 3.1 g for 29 mm specimens) were compacted in the pressure range of 690 to 1724 MPa (100 to 250 ksi) employing an automated servo-hydraulic press (Instron Model 1335). The die walls and punches were lubricated using a solution of stearic acid dissolved in methyl-ethyl ketone to reduce die wall friction. The as-pressed "green" densities of the disks were determined by weighing and measuring of dimensions.

Sintering of the pressed metal powder disks was conducted in a ceramic tube furnace with a flowing gas mixture containing 7% hydrogen with the balance being argon ($Ar/7\%H_2$). The disks were typically held vertically in a slotted carrier fabricated from molybdenum sheet. The furnace was first purged with flowing argon or nitrogen gas, after which an $Ar/7\%H_2$ flow rate of between 0.2 and 2 standard liters per minute was established and maintained for the entirety of the sintering run including heating and cooling. Sintering temperature and times varied from 1200°C to 1600°C and 1 to 4 hours. The standard heating rate was 10°C/min; however, holds at different temperatures and times were explored to reduced oxygen content and when necessary, remove binders and lubes. The densities of the sintered disks were measured by weighing and measuring of dimensions and the Archimedes method using immersion in high purity ethanol.

3.0 RESULTS

33 mm disks

Due to continuing changes in target specifications, only a limited number of 33 mm disks were produced using Climax NPA powder. The results of are summarized in Table 2. Green densities of 80 to 87 percent of theoretical were achieved for compaction pressures between 690 and 1379 MPa (100 and 200 ksi). Sintered densities above 90% of theoretical were achieved after 1 hour at temperatures of 1500°C and 1550°C. Most of the disks cupped during sintering, some quite severely.

Table 2. Compaction and sintering results for various Molybdenum powders

Powder	Compact press. (ksi)	%TD (green)	Sintering (°C/h)	%TD (sintered)	Open Porosity	Diameter (mm)	Thickness (mm)	Shrinkage (%)		Distortion*
								D	t	
33 mm disks										
NPA	100	80	1500/1	90	NM	32.0	0.59	3.9	1.7	mixed
NPA	100	80	1550/1	91	NM	NM	NM	NM	NM	severe
NPA	150	85	1500/1	92.5	NM	32.5	0.56	2.0	0	unknown
NPA	200	87	1500/1	94	NM	32.8	0.53	1.0	0	unknown
NPA	100/150	81	1200/1	91	NM	32.6	0.54	0.9	3.2	unknown
NPA	100/200	86	1400/1	93	NM	32.6	0.52	1.2	0	unknown
29 mm disks										
NPA	100	80	1500/1	89	10	29.2	0.50 ± 0.03	3.4	4.1	mixed
NPA	100	80	1550/1	93	4	29.0	0.50 ± 0.02	4.0	1.9	minimal
EM-NM3	100	43	1500/1	98	0	22.9	0.76	24.6	25	severe
LB1	100	65	1500/1	89	5	27.6	0.57 ± 0.02	8.4	9.6	moderate
LB1	100	65	1550/1	91	< 1	27.4	0.56 ± 0.02	9.4	11.4	severe
LB1-M	100	65	1500/1	88	~ 5	27.7	0.58 ± 0.03	8.3	7.3	moderate
LB1-S	100	67.5	1500/1	87	~ 9	28.0	0.56 ± 0.02	7.2	7.8	severe

* Severity of distortion, primarily cupping or bow, was ranked using the ratio of the height of the cup minus the thickness of the disk at the peak height of the cup divided by the same thickness measurement with "**minimal**" being < 0.5, "**moderate**" between 0.5 and 1.0, and "**severe**" > 1.0.

To increase the as-pressed density and potentially limited distortion during sintering, disks that were initially pressed at 690 MPa (100 ksi) were repressed at higher loads between flat steel plates, with no die body to restrict radial expansion. The repressing at 1034 and 1379 MPa (150 and 200 ksi) increased the green densities of the parts from 80% of theoretical to 81 and 86%, respectively. Sintered densities similar to parts initially pressed at these higher pressures were observed, however, at much lower temperatures of 1200°C and 1400°C. Cupping was minimized, however, only a single specimen was produced for each set of processing conditions.

29 mm disks

The most recent target specifications call for a disk 29 mm in diameter with a thickness of 0.5 mm. Tolerances are quite stringent; + 0.000 and - 0.025 mm in diameter, and a thickness of 0.504 ± 0.004 mm. Die cavity dimensions for a 29 mm sintered disk were selected based upon shrinkage results from earlier tests.

Climax NPA Powder

Initial trials for the fabrication of 29 mm targets were conducted employing Climax NPA powders and pressing and sintering conditions similar to those for the 33 mm disks. Compaction pressure was limited to 690 MPa (100 ksi) for significant deformation of the punches (tool steel or tungsten carbide) was observed at higher loads. In addition, a significant fraction of the green 33 mm disks failed upon ejection when higher pressures were employed.

The results of pressing and sintering were identical to those for the 33 mm specimens with mixed levels of cupping and warping (Table 2). Distortion of disks processed using the same processing parameters varied from minimal to severe. More detailed characterization employing multiple dimensional measurements at selected points across the sample, a computerized coordinate measuring system and radiography revealed significant variations in thickness, diameter, and density. The results for two specimens with very different levels of distortion are shown in Figs. 2 through 10. In the radiographs, thicker or denser sections are brighter while thinner and/or less dense are darker.

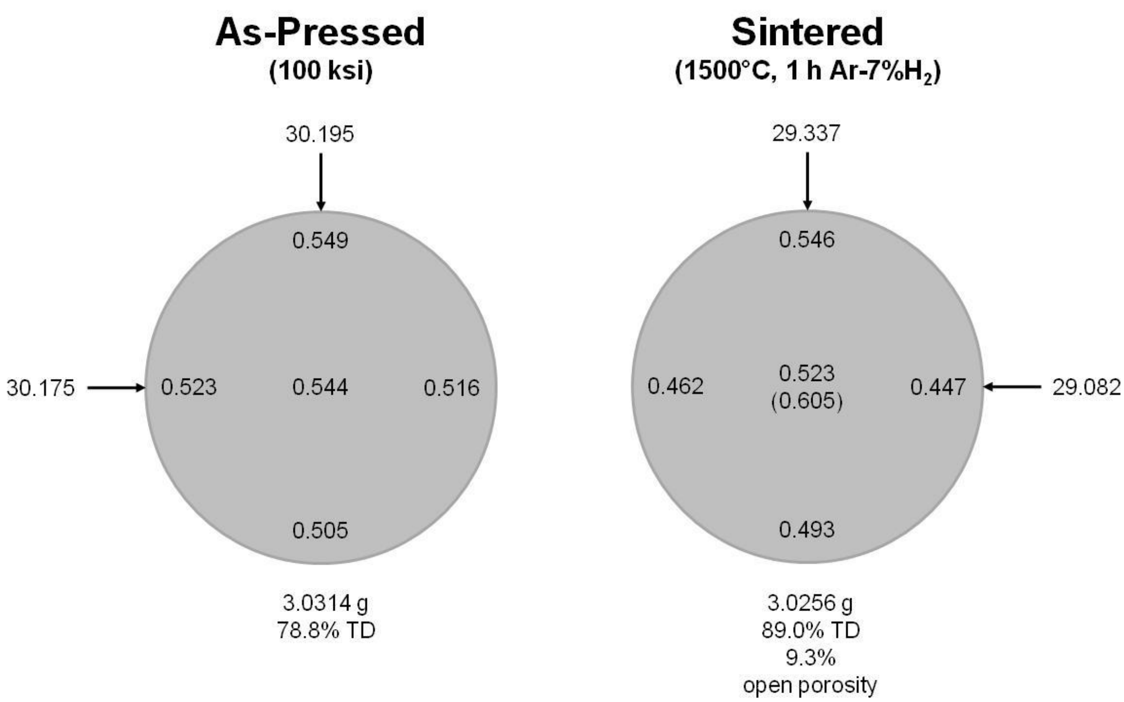

Fig. 2. Dimensions of green and sintered disk fabricated using Climax NPA powder (in millimeters, center dimension includes measurements from both sides to show cupping).

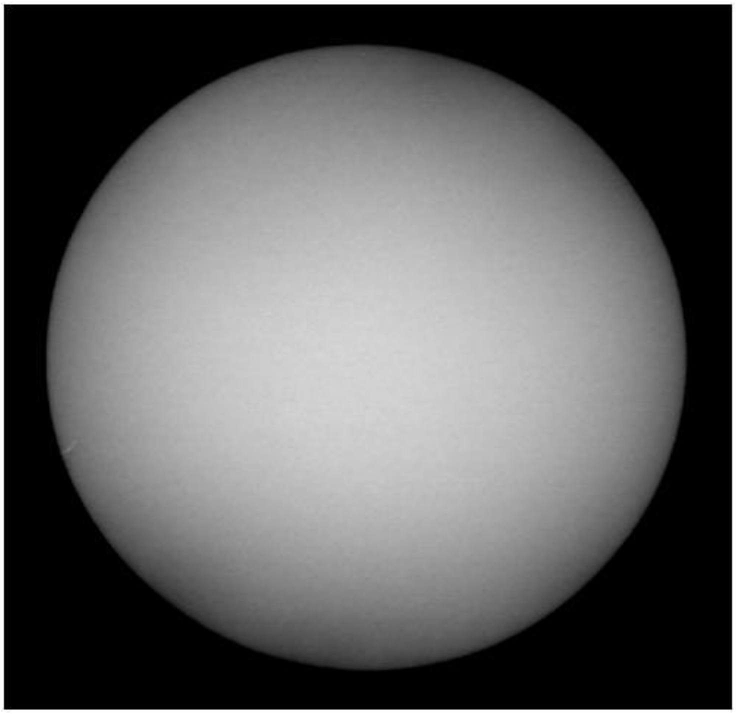

Fig. 3. Radiograph of sintered disk from Fig. 1.

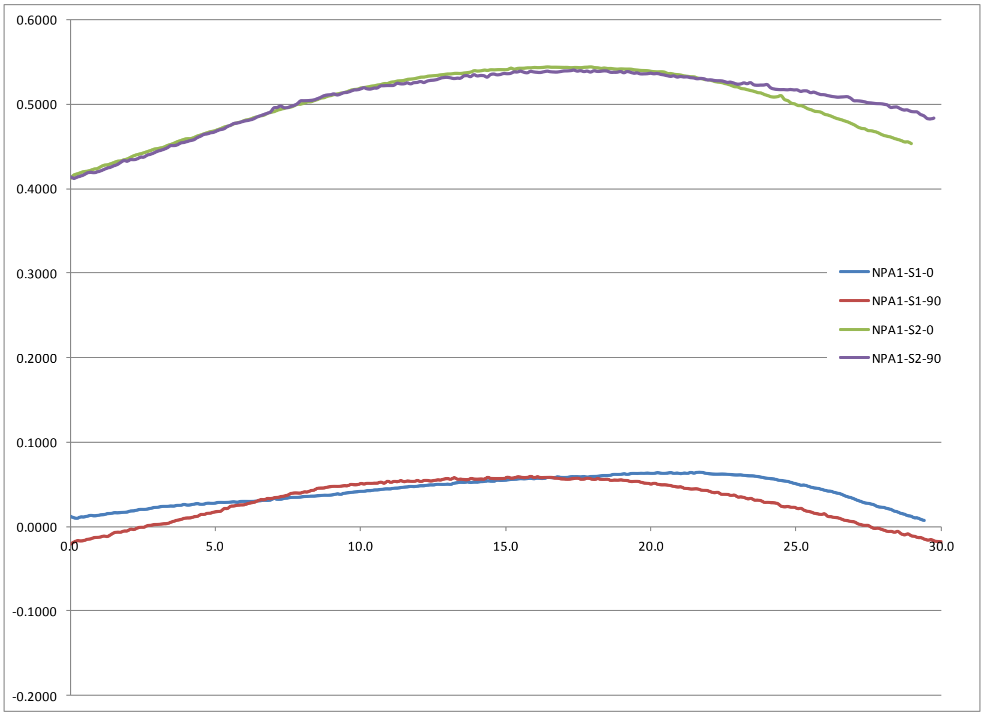

Fig. 4. Surface profiles for sintered disk from Fig. 1 (axes in mm).

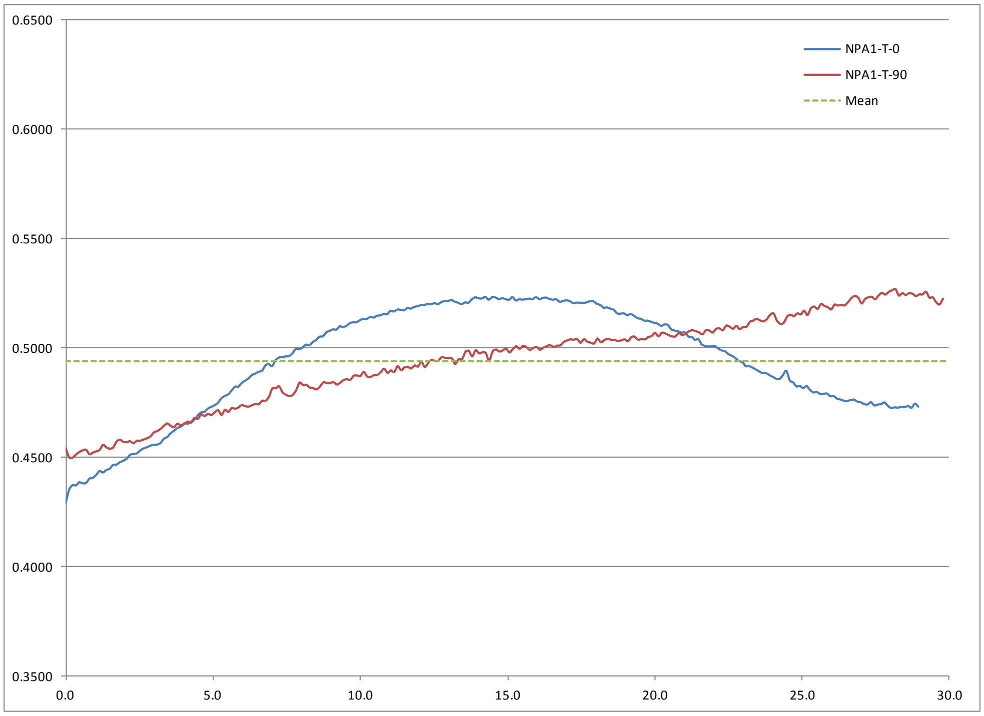

Fig. 5. Thickness profiles for sintered disk from Fig. 1 (axes in mm).

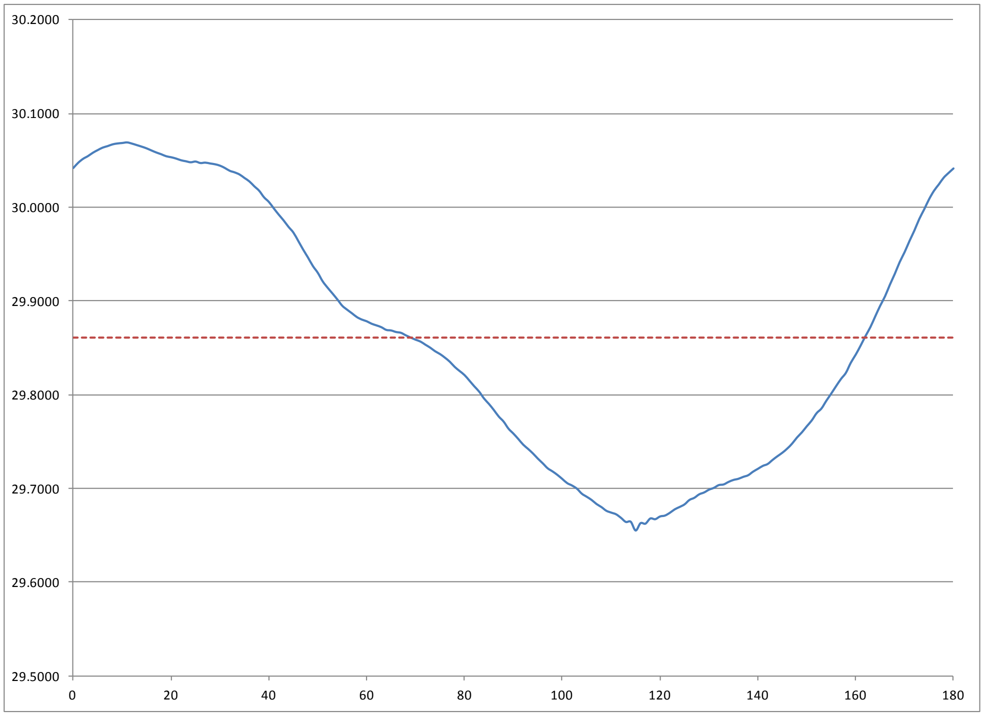

Fig. 6. Diameter profile for sintered disk from Fig. 1 (x-axis in mm, y-axis distance in 2° increments).

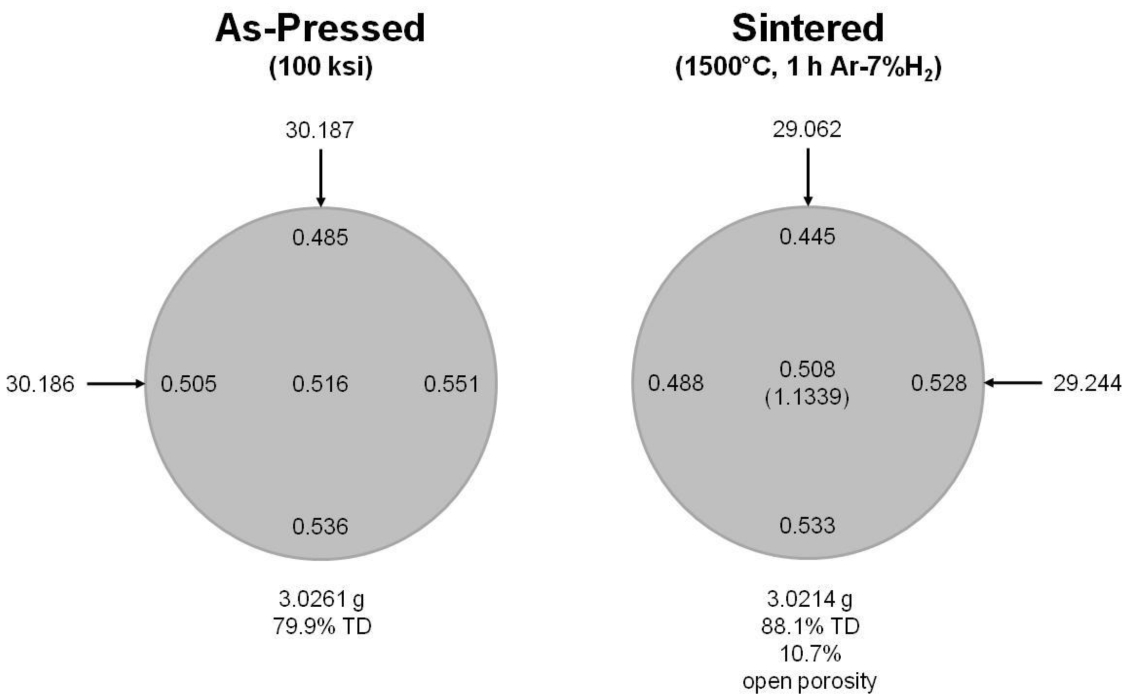

Fig. 7. Dimensions of green and sintered disk fabricated using Climax NPA powder (in millimeters, center dimension includes measurements from both sides to show cupping).

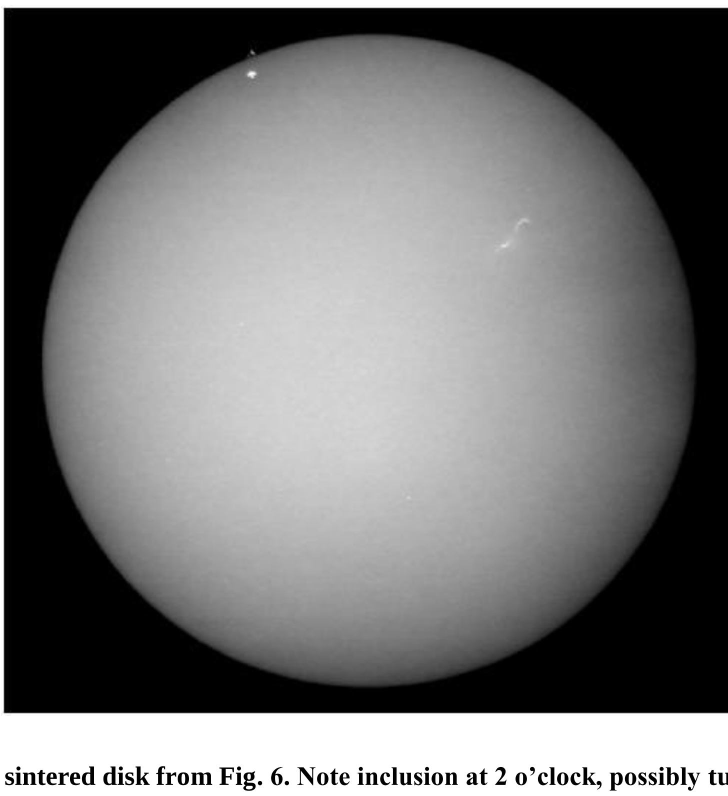

Fig. 8. Radiograph of sintered disk from Fig. 6. Note inclusion at 2 o'clock, possibly tungsten. Bright spot at 11 o'clock appeared to be surface contamination.

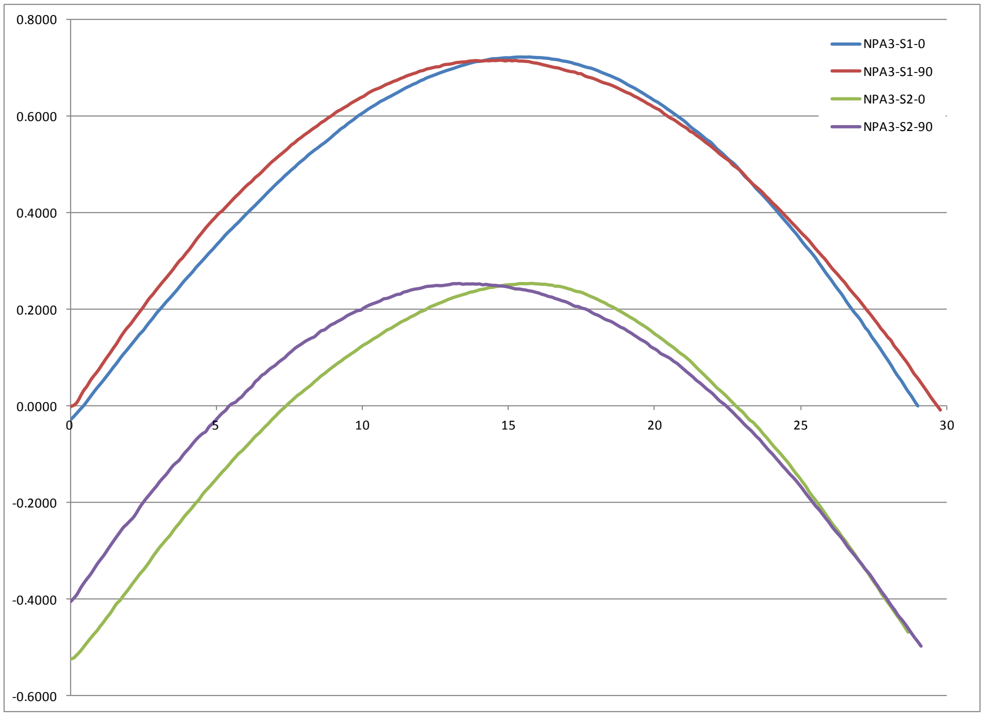

Fig. 9. Surface profiles for sintered disk from Fig. 6 (axes in mm).

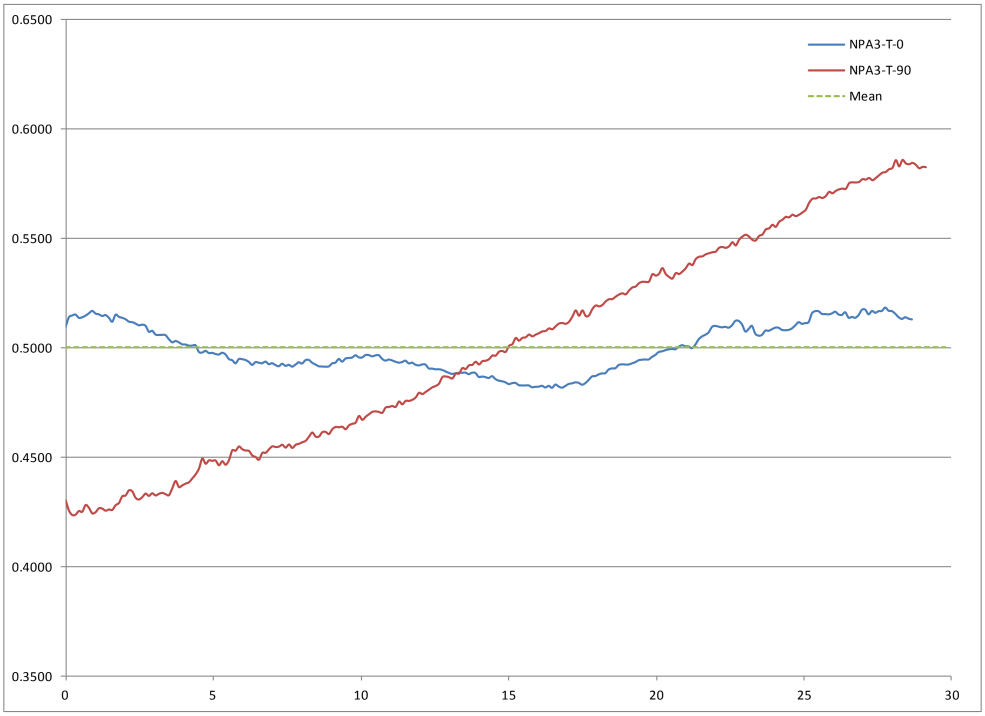

Fig. 10. Thickness profiles for sintered disk from Fig. 6 (axes in mm).

Climax EM-NM3 Powder

Since the Isoflex Mo-100 powders have characteristics between Climax NPA and EM-NM3 materials, a small number of 29 mm disks were produced using Climax EM-NM3 molybdenum powder. The powder was difficult to press achieving green densities of 43% of theoretical at 690 MPa (100 ksi) compaction pressure. Sintering for 1 h at 1500°C resulted in densities of 98% theoretical with no open porosity. The disks exhibited extreme shrinkage (25%) and were extremely deformed, cupped and warped, with significant variations in thickness and diameter.

Molybdenum Powder from Large-Batch Reduction

Once in full production, much of the isotopically-enriched molybdenum used in the fabrication of targets will be recycled from the process. Recovery of molybdenum is being examined including large-batch reduction of molybdenum trioxide to metal powder. Molybdenum powder for this study was produced by the single-stage reduction of 500 g of commercially-available MoO_3 powder (Alfa Aeser 99.5% MoO_3) in a hydrogen furnace. Details regarding the reduction process and characterization of product powders will be discussed in a future report.

The powder reduced from trioxide possessed properties comparable to Climax NPA, therefore pressing and sintering conditions similar to those for the commercially-available material were utilized. Again, compaction pressure was limited to 690 MPa (100 ksi) which produced green parts with much lower densities than expected, 65% versus 80% of theoretical (Table 2). The disks sintered to 89% and 91% of theoretical in 1 hour at 1500°C and 1550°C, respectively, with moderate to severe levels of distortion. A 4-hour hold at 800°C was included in the sintering schedule to reduce oxygen levels in the powder before reaching final sintering temperature.

Due to the unpredictability in cupping and warping of disks processed employing the same parameters, an array of approaches to minimize distortion during sintering were applied. These included:

- Milling and/or sieving of the powder to narrow particle size distribution and eliminate agglomerates.
- Repressing of green disk between flat plates at higher pressures.
- Sintering disks lying horizontally on a bed of fine milling media.
- Restraining the disks between metal plates during sintering.
- Repressing sintering disks at room and elevated temperatures.

Small samples of the powder (50 g) from the large-batch reduction were separately sieved and milled and disks pressed and sintered using standard parameters; 690 MPa compaction pressure and 1 h at 1500°C. Only minor differences in pressing and sintering properties were observed (Table 2) with no improvement in flatness or uniformity. No changes in dimensional stability were shown for disks repressed at 1550 MPa (225 ksi) or sintered lying horizontally on a bed of 500 μm spherical zirconia milling media (radiograph shown in Fig. 10).

A green disk was placed between two molybdenum plates, 5 cm in diameter and 0.6-cm thick, and weighing about 110 grams. Pieces of 0.005" tungsten sheet were placed between the molybdenum plates and the green disk to prevent interaction during sintering. No clamping or other device was used to restrict movement of the assembly so that only the mass of the top plate would affect shrinkage and deformation of the disk during sintering. Densification was hindered with the specimen reaching a density of 85% of theoretical in contrast to 89% for parts that were not restrained. The sintered disk was very flat but severely deformed across the diameter (Fig. 11). Shrinkage in thickness was greater than for unrestrained disks, 14% versus about 10%, however, in diameter was less, ~ 4% as compared to ~ 8%.

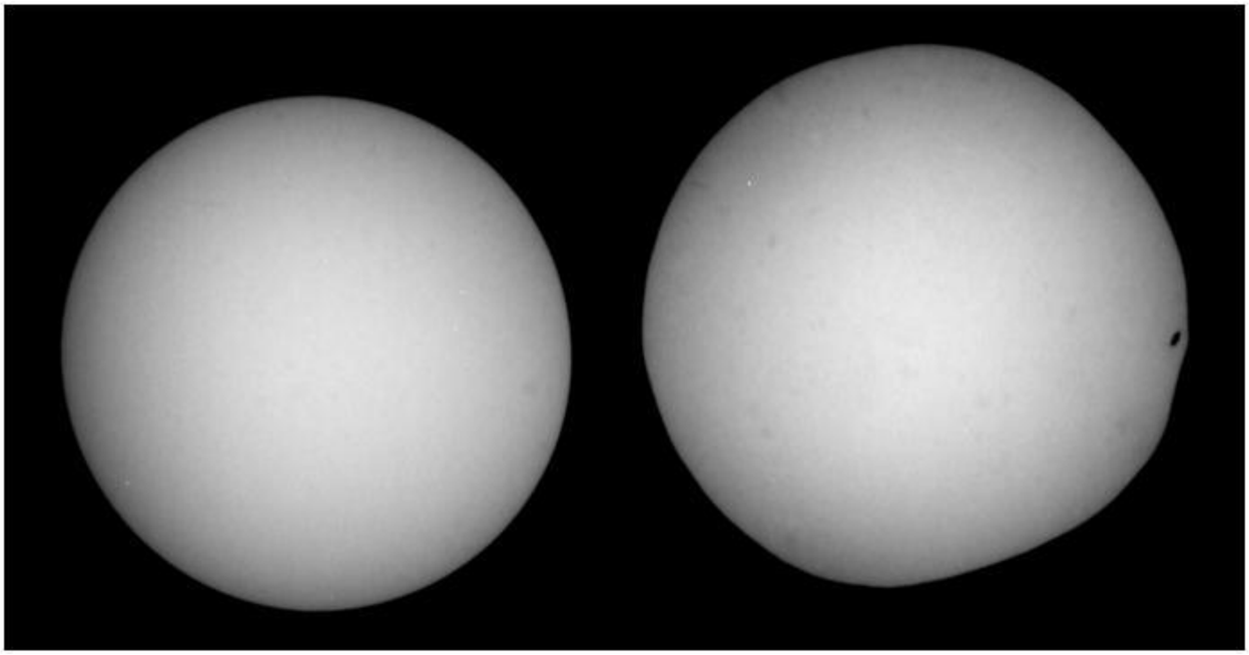

Fig. 11. Radiographs of disks fabricated using powder from a large-batch reduction. Disk on left sintered lying horizontally on a bed of 500 μm zirconia milling media. Disk on right was sintered while restrained between molybdenum plates.

Cupped sintered specimens were placed between flat hardened steel plates and repressed to plastically deform and flatten the metal disks. Sintered powder metal molybdenum is brittle at room temperature thus the disks failed catastrophically, i.e. shattered. The brittle-to-ductile transition temperature for molybdenum is affected by many factors including processing history, work added, and crystal structure. Additional repressing of sintered disks was conducted at temperatures to 400°C with all specimens failing in a brittle manner.

Climax PM Spray-Dried Powder

The Climax NPA and EN-NM3 powders and the material from large-batch reduction exhibited poor flow characteristics. The powders were sticky and clumpy causing difficulties in uniformly filling the shallow cavity of the die and hindering particle movement during pressing. Most powders used in pressing operations are intentionally formed or treated to enhance flow behavior. Most of the techniques such as plasma or gas atomization, spray drying, and granulation produce a spherical product that flows well during feed and compaction.

Climax PM powder is a spray-dried spherical product with excellent flow characteristics (Table 1). As-received Climax PM powder was pressed into disks at a pressure of 690 MPa which resulted in structurally-sound green bodies with densities of 76% of theoretical. Disks sintered at 1500°C for 1 h possessed densities of 85% of theoretical with 12% open porosity (Table 3) with minimal distortion. Sintering temperature was increased to 1600°C with times of 1 and 4 hours. Density increased to 87% with 10% open porosity and 90% with 7% open porosity, respectively. Shrinkage was relatively uniform at a given time and temperature helping to minimize distortion. Compaction pressure was increased to 1000 MPa (145 ksi) producing green densities of 79% and densities of 92% and 93% when sintered at 1600°C for 2 and 4 hours, respectively.

Table 3. Compaction and sintering results for climax PM spray-dried powder

Lubricant	Compact Press. (ksi)	%TD (green)	Sintering (°C/h)	%TD (sintered)	Open Porosity	Diameter (mm)	Thickness (mm)	Shrinkage (%)		Cupping
								D	t	
As-Received	100	76	1500/1	85	12	29.3	0.55 ± 0.02	3.0	2.6	minimal
As-Received	100	76	1600/1	87	10	29.1	0.54 ± 0.02	3.7	3.4	minimal
As-Received	100	76	1600/4	90	7	28.8	0.54 ± 0.02	4.7	5.2	moderate
As-Received	145	79	1600/2	92	4	29.3	0.53 ± 0.02	2.9	1.1	minimal
As-Received	145	79	1600/4	93	1	29.1	0.52 ± 0.03	3.1	2.1	moderate
0.25% EBS	100	77	1600/4	90	5	28.7	0.52 ± 0.005	4.7	4.9	minimal
0.25% EBS	145	80	1600/2	93	2	29.3	0.49 ± 0.01	3.0	8.1	minimal
0.25% EBS	145	80	1600/4	93	1	29.1	0.50 ± 0.007	3.5	5.1	minimal
0.5% EBS	100	76	1600/2	89	9	29.0	0.53 ± 0.006	4.2	6.3	minimal
0.5% EBS	100	76	1600/4	90	8	28.7	0.53 ± 0.006	4.9	5.3	minimal
0.5% EBS	123	80	1600/2	89	8	29.2	0.52 ± 0.006	3.4	5.0	moderate
0.5% EBS	145	81	1600/2	90	8	29.4	0.50 ± 0.01	2.8	5.5	moderate
0.25% SA	100	75	1600/4	87	8	28.8	0.53 ± 0.02	4.6	5.8	moderate
0.25% PVB	100	73	1600/4	87	11	28.8	0.54 ± 0.01	4.5	7.2	moderate

Minimal to moderate cupping as well as variations in thickness across each specimen was observed for the disks produced using the spray-dried molybdenum powder. The cupping is likely a result of differences in compaction and thus density and shrinkage as frictional forces at the die wall and across the faces of the punches prevent transfer of load and movement of particles. Combinations of part and die design, pressing technique and lubricants are generally employed to minimize variations in green density and reduce distortion during sintering. Three lubricants; stearic acid (SA), ethylene bis-steramide (EBS), and polyvinyl butyral (PVB) were blended with Climax PM powder at a concentration of 0.25 weight percent to reduce frictional forces during compaction and improve part dimensional stability.

Disks were initially pressed at 690 MPa producing green densities of 75% for the stearic acid addition, 77% for the EBS and 73% for the PVB (Table 3). Sintering at 1600°C for 4 h produced disks with densities of 87% with 8% open porosity, 90% with 5% open porosity and 87% with 11% open porosity, respectively. The specimens fabricated using EBS exhibited uniform shrinkage and minimal cupping while the others showed moderate levels of distortion. Additional specimens were pressed at 1000 MPa using the EBS-containing blend producing green densities of 80% and sintered densities of 93% with 1% open porosity and minimal cupping (Table 3).

Due to the positive results for the EBS lubricant, a mixture containing 0.5 wt% was blended and evaluated. Disks were pressed at 690, 850 and 1000 MPa resulting green densities of 76%, 80% and 81%, respectively. The specimens were sintered at 1600°C for 2 and 4 hours producing densities of 89% – 90% and open porosities of 8% – 9% (Table 3). Cupping was minimal for disks compacted at 690 MPa but moderate for those pressed at 850 and 1000 MPA. Variations in thickness within each disk were also minimized but not completely eliminated.

4.0 DISCUSSION

Disks with sintered densities greater than 90% of theoretical can be fabricated from most any molybdenum powder. Fabricating thin, flat, uniform disks that meet accelerator target specifications by pressing and sintering of powdered metal is challenging. ALL aspects of the process affect the quality and reproducibility of the final product.

Powders with small particle sizes do not compact well but are readily sintered to high densities even at relatively low temperatures and short processing times (e.g., 1200°C to 1400°C for 1 hour. These types of powders do not flow creating difficulties when filling the cavity of the die and in particle movement during compaction due to particle-to-particle and die and punch surface frictional forces. This results in significant gradients in pressure transfer and thus green density through the thickness and across the diameter of a thin disk. Higher compaction densities typically occur very close to the perimeter of one flat surface and at the center of the opposite surface (Fig. 12). The higher density around the perimeter has the added effect of increasing die wall friction during ejection, further stressing the green part. Non-uniform shrinkage based upon density distribution and stress results in cupping during sintering. And when overall green density is low, shrinkage is more significant, further complicating dimensional control.

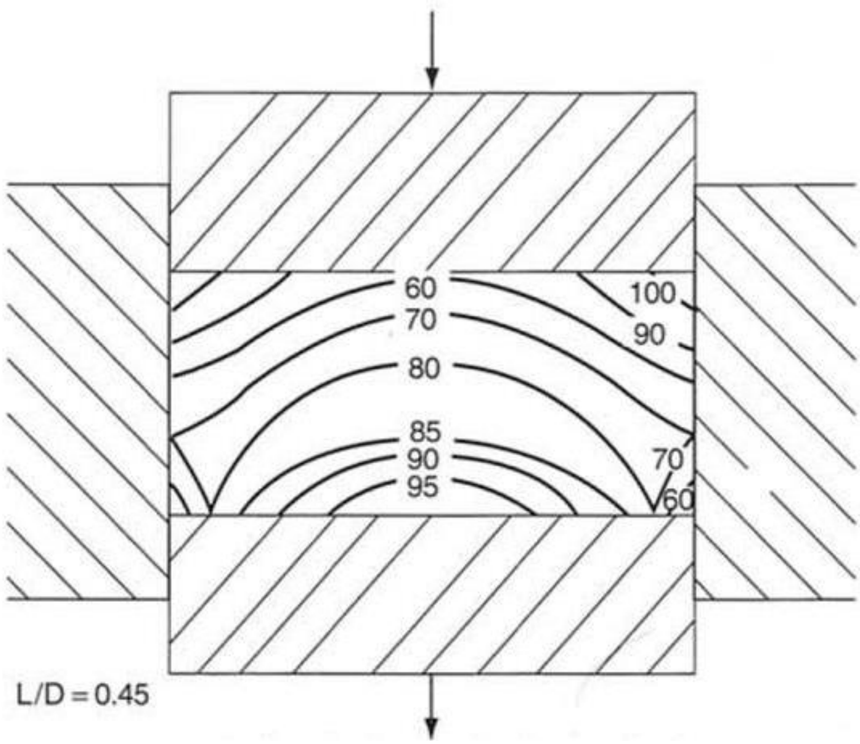

Fig. 12. Typical density gradients in a cylindrical powder metal green body fabricated\ employing single-sided compaction (in % theoretical density). [9]

Coarse powders compact more easily but are more difficult to densify requiring higher temperatures and longer times, e.g. 1500°C or greater and times to 4 hours or more. Unless treated, these powders also do not flow well creating the same problems with uniformity as for finer powders. Shrinkage thus distortion during sintering is reduced for higher green densities which are more easily attained with coarse powders.

Spherical powders such as the Climax PM spray-dried product flow well and thus more uniformly fill the cavity of the die. These powders are larger agglomerates of smaller particles with particles sizes in the range of 45 to 80 μm. Spherical powders compact well and sinter under conditions similar to those for coarse powders but with improved control of shrinkage due to improved uniformity in green density.

Lubricants reduce particle-to-particle and part-to-tooling friction allowing the powder to move more freely during compaction and thus minimizing pressure and subsequently density gradients in the powder compact.[10] Little or no increase in green density was achieved by the addition of ethylene bis-steramide (EBS) lubricant to the Climax spray-dried powder; however, significant improvement in uniformity between disks and within each was observed (Table 4). Dimensions and densities for green and sintered disks were much more consistent as was shrinkage (~ 5% for diameter and thickness). The densities for five specimens with 0.25% EBS that were sintered in the same run were 90.5 ± 0.2% of theoretical density with 6.4 ± 0.1% open porosity. Distortion was minimal for these and other disks fabricated using the EBS addition.

Table 4. Properties of Disks Fabricated From Climax PM with EBS Lubricant

Lubricant (wt%)	Green Density (%)	Sintered Density/ Open Porosity (%)	Average Diameter (mm)	Thickness (mm)		
				Average	All Disks	Each Disk
None	76	90/7	28.8	0.54	± 0.02	± 0.04
0.25	77	90/5	28.7	0.52	± 0.005	± 0.01
0.5	76	90/8	28.7	0.53	± 0.006	± 0.01

(3.1 g of powder pressed at 690 MPa and sintered at 1600°C for 4 h)

All of the disks were fabricated using one-sided compaction (Fig. 12). The bottom punch was fixed and thus only the top punch was able to move as pressure was increased and the powder compressed. One-sided compaction exacerbates pressure and density gradients. Two-sided compaction reduces variations in pressure and thus density gradients resulting in a more homogenous green body (Figs. 13 and 14). Although wall friction is not as significant a factor for thin disks, punch surface friction must be considered.

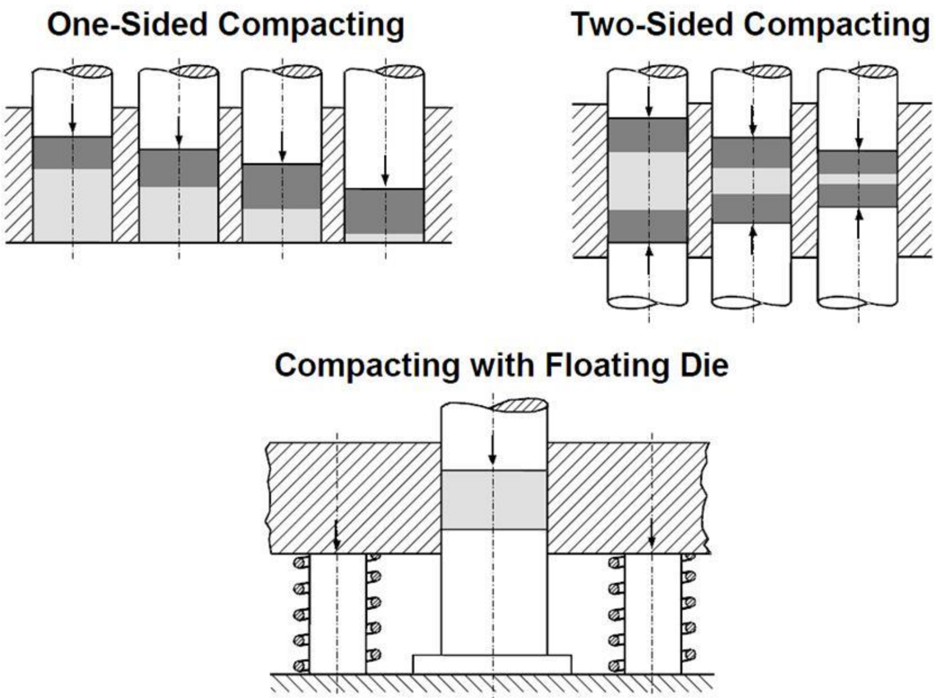

Fig. 13. Various approaches for compaction of powders.[11]

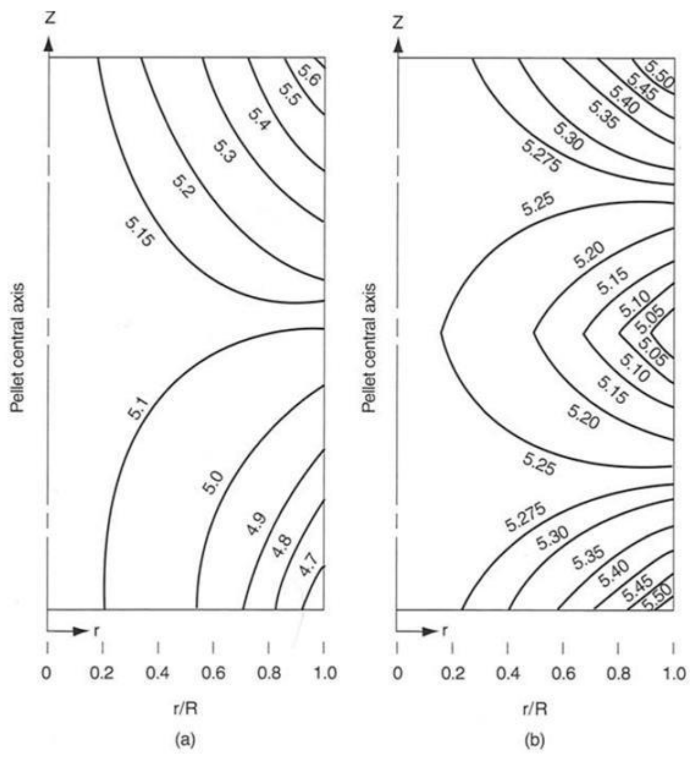

Fig. 14. Density gradients in a cylindrical powder metal green body: (a) single-sided compaction and (b) double-sided compaction.[12]

Tooling tolerances also play an important role in achieving desired dimensions in a sintered component. Tolerances for the 29 x 0.5 mm accelerator target disks are quite stringent; +0.000 and -0.025 mm (-0.001”) for the diameter, and 0.504±0.004 mm for thickness (actual specification for thickness is 0.0197” to 0.020”). A trend was observed with regard to thickness variations for individual disks. For many disks, green and sintered, one edge was thicker than the opposite edge, and in some cases the difference was quite significant (see Figs. 1, 4, 6, and 9).

The die and punch set used to produce the 29 mm disks were fabricated with a specified punch-to-die diameter clearance of 0.001”. The die is short, only 1.3”, thus the cavity has an aspect ratio close to unity (L/D = 1). Based on these tolerances and ignoring the taper included on one side of the cavity to aid with ejection, each punch has the potential to skew at an angle that could produce a variation in thickness across the diameter of 0.0035” or about 0.09 mm. If both punches were misaligned, the total possible variation in thickness could be 0.007” or 0.18 mm. Variability in target thickness if either or both punches were skewed would exceed currently specified acceptable limits. This effect would be minimized by tightening the tolerances for the tooling and lengthening the body of the die.

5.0 SUMMARY AND RECOMMENDATIONS

Disks with sintered densities greater than 90% of theoretical can be fabricated from most any molybdenum powder. Fabricating thin, flat, uniform disks that meet all accelerator target specifications by pressing and sintering of powdered metal requires optimization of a combination of powder characteristics, lubricants, pressing technique and sintering schedule. Spherical spray-dried molybdenum powder with small amounts of lubricant produced the most uniform target disks. Remaining challenges including small levels of distortion, primarily cupping, and variations in thickness will be addressed through changes in tooling design and tolerances and utilization of double-action or two-sided compaction.

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APPENDIX A

NEW TOOLING

New tooling for improved compaction of the 29 mm target disks was designed and procured (Fig. A.1). The diameter of the die cavity was increased to accommodate current shrinkage (~5% and produce disks with a nominal diameter of 29.0 mm. Commercially-available spray-dried molybdenum powder with 0.25 wt% ethylene bis-steramide lubricant was used in the initial tests. Disks were pressed using two different techniques; single-acting where one punch is fixed and thus pressure is applied from one direction, and double-acting or floating die in which the die body is suspended allowing pressure to be applied from two directions.

Green densities of 77% of theoretical density were achieved using both techniques (Table A.1). After binder removal, all specimens were sintered at 1600°C for 4 h in an argon-hydrogen mixture. Sintered densities of 89% with open porosities of 6% were measured for disks pressed using both approaches. However, the dimensions of the specimens pressed using the double-acting technique were much more uniform and the samples exhibited almost no distortion.

The disks pressed using the single-acting approach had an average diameter of 28.98 ± 0.04 mm and an average thickness of 0.52 mm with a part-to-part variation of ± 0.005 mm. The average variation in thickness across each specimen was +22 μm/-47 μm with a maximum deviation from the median thickness of -79 μm. The disks cupped significantly with an average distortion of the surface being > 400 μm.

The disks pressed using the double-acting technique had an average diameter of 29.04 ± 0.005 mm and an average thickness of 0.53 mm with a part-to-part variation of ± 0.005 mm. The average variation in thickness across each specimen was +9 μm/-14 μm with a maximum deviation from the median thickness of -25 μm. The disks were very flat with an average distortion of the surface being < 20 μm.

Additional quantities of target disks for testing and evaluation were prepared using the improved tooling and double-acting compaction. Again, commercially-available spray-dried molybdenum powder with 0.25 and 0.5 wt% EBS lubricant was used and the weight adjusted to produce disks that will meet current specifications. A set of 20 disks were compacted and characterized in the green state. The disks had as-pressed densities of 77.7 ± 0.3% of theoretical, diameters of 30.54 ± 0 mm, and a nominal thickness of 0.526 mm with part-to-part variations in average thickness of ± 4 μm and average in-part variations in thickness of -5/+8 μm. Distortion of the specimens was minimal.

Fig. A.1. New tooling for double-acting compaction of 29 mm disks.

Table A1. Target disks fabricated employing new tooling

Specimen Number	Material	Compact Pressure (MPa)	%TD (green)	Sintering (°C/h)	Weight (g)	Density (g/cm^3)	%TD (sintered)	Open Porosity (%)	Diameter (mm)	Thickness (mm)
Single-Acting Compaction										
SA-1	Climax PM +.25%EBS	675	76.5	1600/4	3.1106	9.03	88.5	5.8	29.0	0.52
SA-2	Climax PM +.25%EBS	675	76.3	1600/4	3.1121	9.07	88.9	5.5	29.0	0.53
SA-3	Climax PM +.25%EBS	675	76.8	1600/4	3.0786	9.18	90.0	5.9	29.0	0.52
Double-Acting Compaction										
DA-1	Climax PM +.25%EBS	675	76.8	1600/4	3.1120	9.13	89.5	6.1	29.0	0.52
DA-2	Climax PM +.25%EBS	675	77.3	1600/4	3.0873	9.04	88.6	5.8	29.0	0.53
DA-3	Climax PM +.25%EBS	675	76.7	1600/4	3.0800	9.00	88.2	6.5	29.0	0.53