

# Processing of Sintered Mo Disks Using Hydrogen Peroxide

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Chemical Science and Engineering Division

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by

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# **PROCESSING OF SINTERED Mo DISKS USING HYDROGEN PEROXIDE**

## **1 INTRODUCTION**

Dissolution studies of sintered molybdenum (Mo) disks using hydrogen peroxide have been focused on several factors that could potentially affect processing of Mo disks. It was demonstrated (Tkac 2012) that stronger pre-sinter and sinter conditions lead to the lowest dissolution rates. Milder pre-sinter, or no pre-sinter, and very strong sinter conditions usually lead to disks with the highest packing densities and lowest dissolution rates. In contrast, stronger pre-sinter conditions and very mild sinter conditions lead to higher dissolution rates. In this report, we discuss results of dissolution of sintered Mo disks provided by NorthStar Medical Technologies, LLC (NorthStar), and disks prepared at Oak Ridge National Laboratory (ORNL).

## 2 SINTERED Mo DISKS

Disks were prepared by a commercial vendor for NorthStar (which in turn provided them to Argonne National Laboratory [Argonne]), and by ORNL. Each type is discussed in a separate section below.

### 2.1 NORTHSTAR Mo SINTERED DISKS

Sintered Mo disks were prepared by pressing Mo metal powder sieved through a 400-mesh sieve (particle size  $<38 \mu\text{m}$ ) and then sintered to make a disk with specific dimensions. Mo metal powder was prepared by reduction of commercially available  $\text{MoO}_3$  (Strem Chemicals Inc.). The goal was to study the effects of pressing parameters on the dissolution rate and packing density for disks sintered under four different conditions. While information about the conditions in which the Mo disks were made is not available (proprietary information of the commercial disk manufacturer under contract to NorthStar), there is a relative measure of different conditions during pre-sintering and sintering. The disks are described by a three-digit code ABC (e.g., “741”) where the first digit ( $A = 7$ ) is the pre-sinter condition, the second digit ( $B = 4$ ) is the pressing variable, and the third digit ( $C = 1$ ) is the sintering condition. Higher numbers equate to stronger conditions (e.g., temperature of sintering or time of sintering, pressure used for pressing). Packing density was calculated from the mass of the disk and physical dimensions of the disk.

All disks were prepared from  $A = 7$  pre-sintered material and the weight of disks was  $\sim 1.07 \text{ g}$ . The pressing parameter was in the range of  $B = 8\text{--}11$ ; sintering conditions were  $C = 1, 2, 6, \text{ and } 8$ . The dependence of the disks’ packing density, thickness, and diameter on various pressing conditions is shown in Figures 1 through 3. Figures 4 through 6 show the dependence of these three parameters on sintering conditions. As expected, packing densities increase with higher pressing and sintering conditions, thickness decreases with increased pressing, and diameter increases with pressing and decreases with sintering.

### 2.2 ORNL Mo SINTERED DISKS

Sintered Mo disks provided by ORNL were prepared from multiple commercial Mo powders. The disks were pressed in steel dies without any binder or lubricant added to the powder, and sintered in a tube furnace ( $\text{Al}_2\text{O}_3$ ). During sintering, the disks were placed in an alumina boat with Ar gas flowing until the oxygen level in the exiting gas was less than 100 ppm. Then the gas was switched to  $\text{Ar}/4\% \text{ H}_2$ , and the furnace was heated at  $10^\circ\text{C}/\text{min}$  to the desired sintering temperature. The furnace was usually held at the peak temperature for 1 hour and then cooled to room temperature. The densities of the sintered disks vary with temperature, starting density (green, or as-pressed density), and powder characteristics. The densities of the sintered disks were determined using Archimedes’ method and may differ from the value obtained from the physical dimensions of the disk. The Archimedes method gives a much more accurate value for small objects.

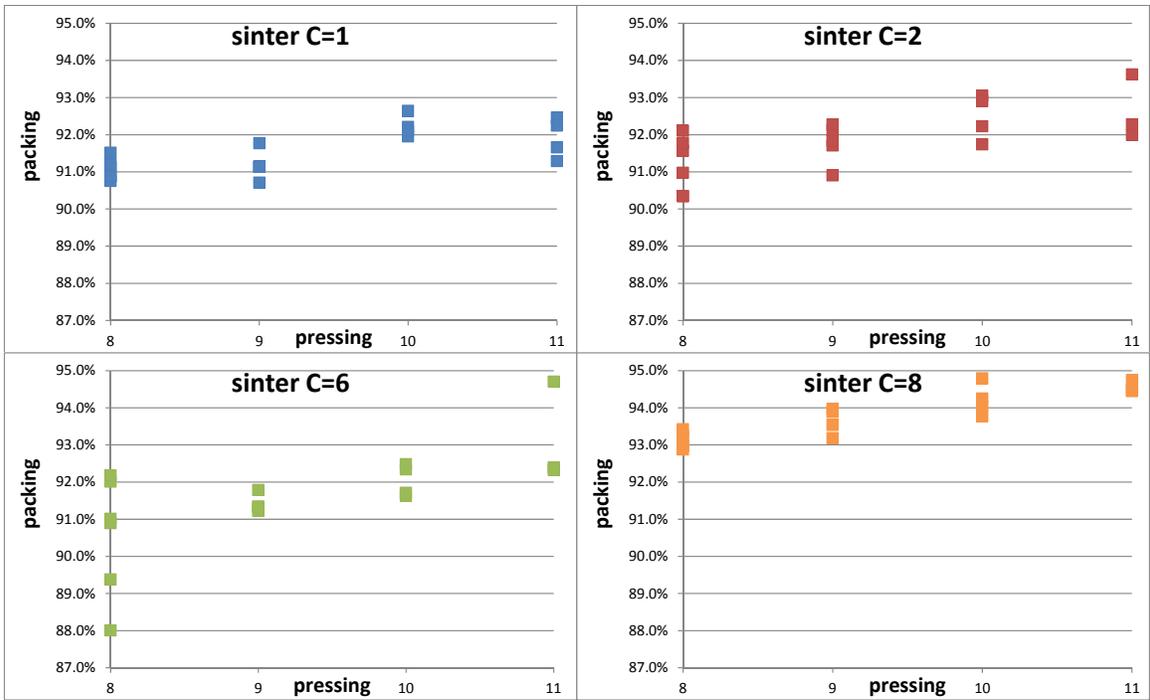


FIGURE 1 Dependence of Disks' Packing Density on Pressing Conditions

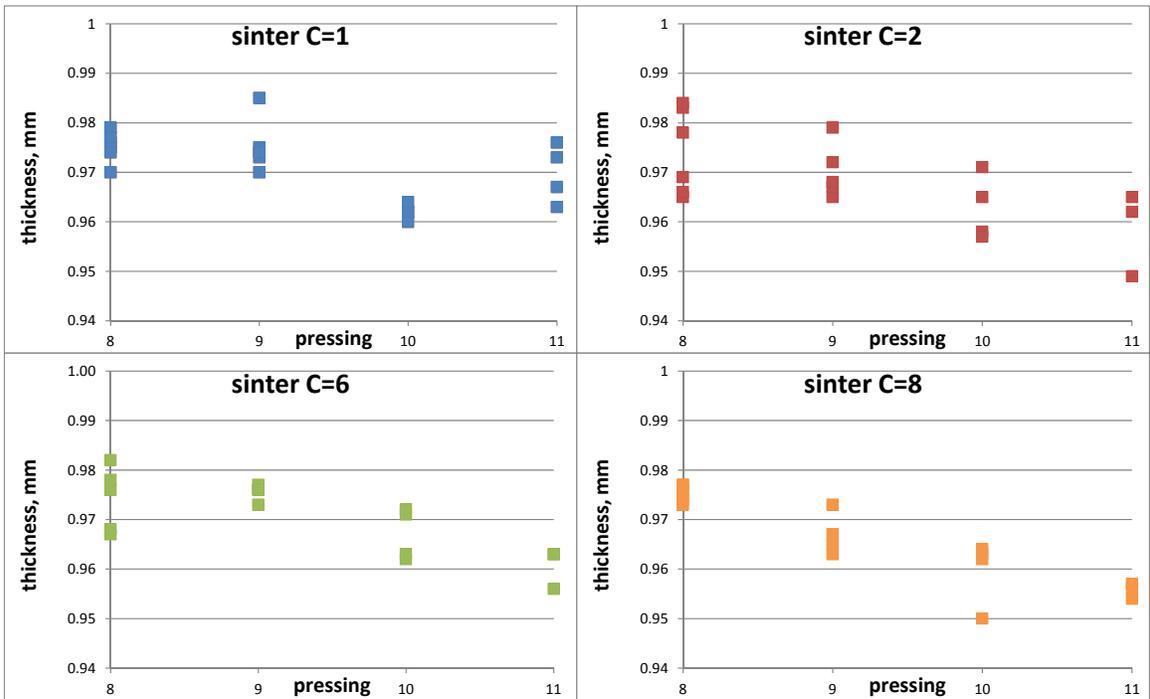
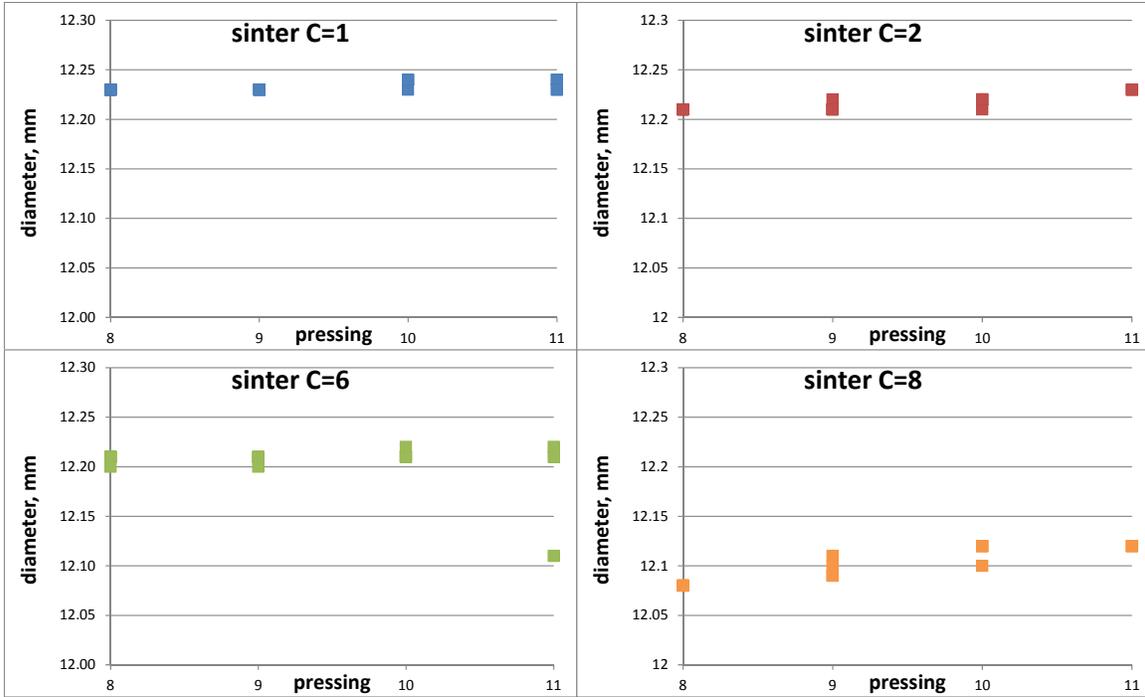
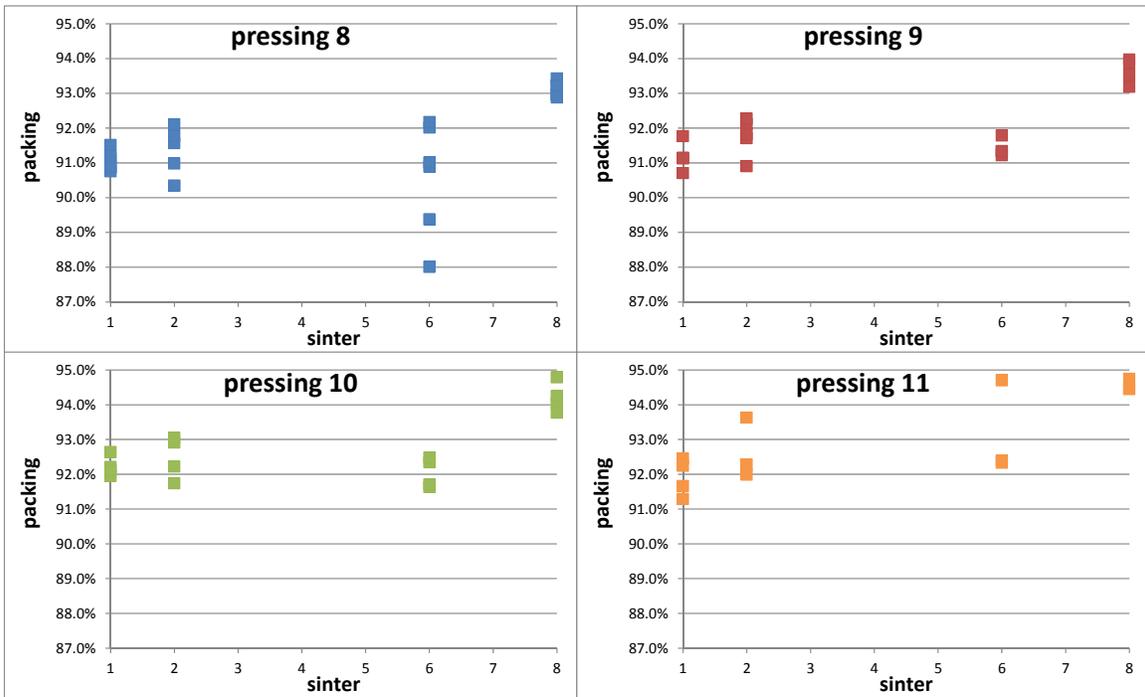


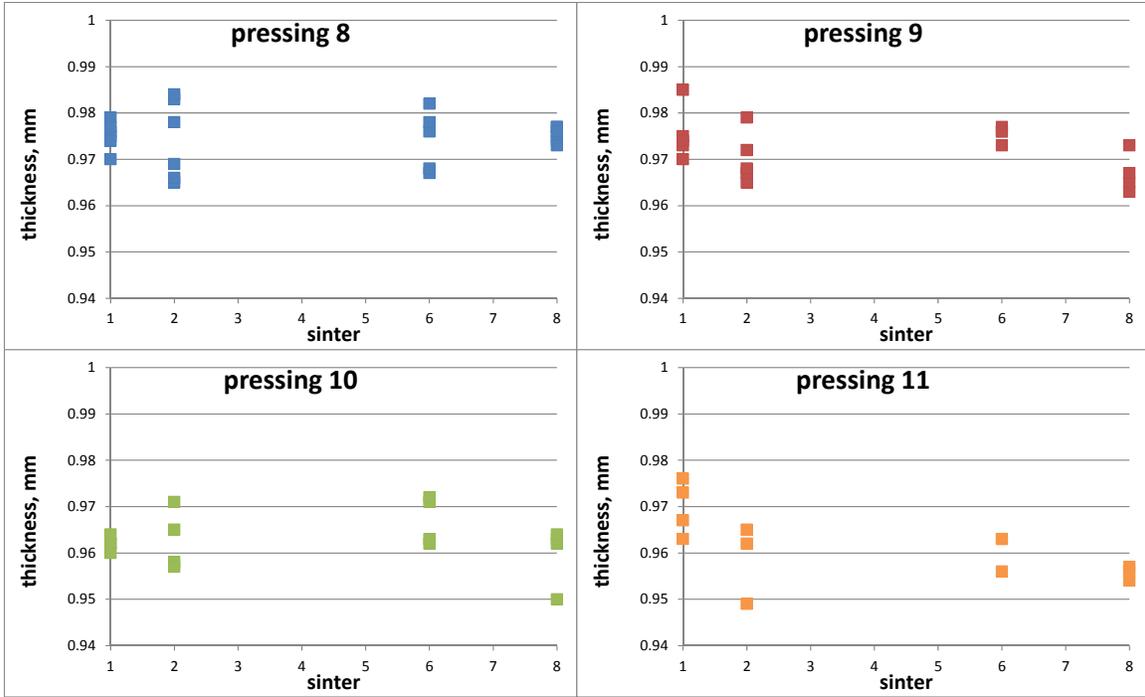
FIGURE 2 Dependence of Disks' Thickness on Pressing Conditions



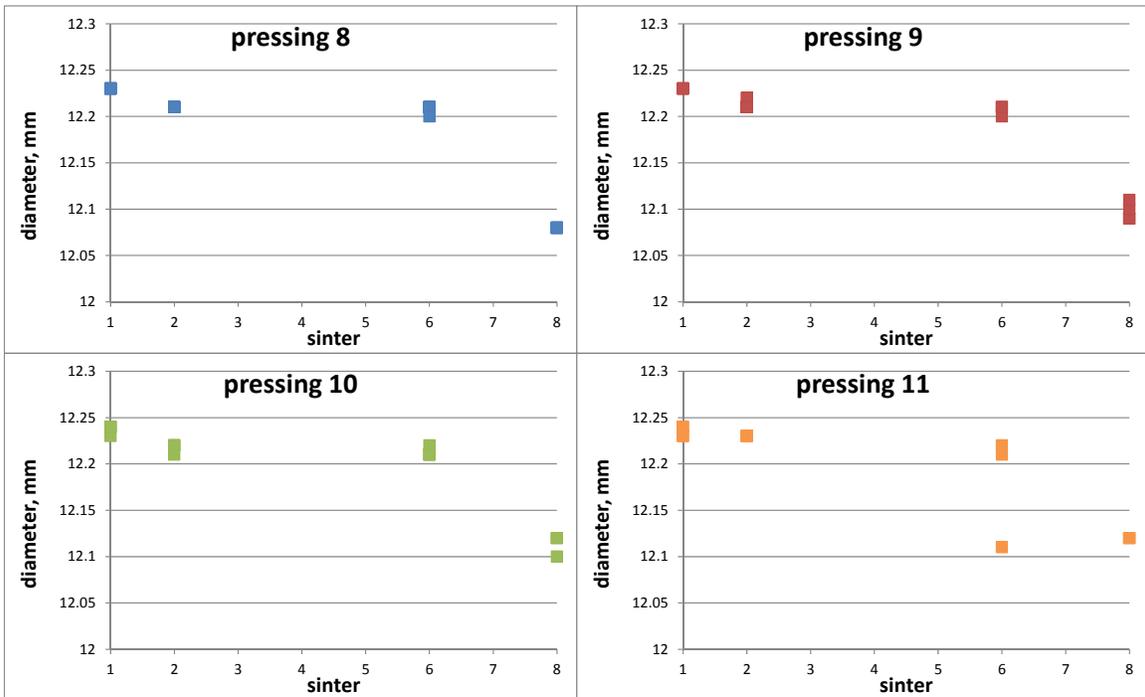
**FIGURE 3** Dependence of Disks' Diameter on Pressing Conditions



**FIGURE 4** Dependence of Disks' Packing Density on Sinter Conditions



**FIGURE 5** Dependence of Disks' Thickness on Sinter Conditions



**FIGURE 6** Dependence of Disks' Diameter on Sinter Conditions

### 3 DISSOLUTION OF SINTERED Mo DISKS

Dissolution studies were performed in a glass beaker, usually using 40 mL of 30% hydrogen peroxide heated to 70°C. In some cases, different volumes of hydrogen peroxide were used in order to achieve complete dissolution. Shortly after adding the disks into hydrogen peroxide a yellow solution forms, indicating the presence of Mo-peroxo species, and the temperature increases rapidly to about 95–100°C within several seconds. There is a significant increase in dissolution rate once the H<sub>2</sub>O<sub>2</sub> reaches ~85–90°C. This is most likely due to the catalytic destruction of peroxide and formation of oxygen in the solution, which reacts aggressively with the Mo. The reason for pre-heating H<sub>2</sub>O<sub>2</sub> solution to 70°C is to test the properties of the disks and distinguish among disks with “good” and “bad” dissolution properties. “Good” dissolving disks reach ~90°C within 10–20 seconds (if starting with 70°C H<sub>2</sub>O<sub>2</sub>). If the Mo disks do not dissolve very well, the temperature of the H<sub>2</sub>O<sub>2</sub> solution starts to decrease and the dissolution progresses very slowly without additional heating. These disks usually do not dissolve even after several hours. For the dissolution studies, if the temperature of hydrogen peroxide after adding the Mo disk did not increase above 70°C within 1–2 minutes, the solution was heated to 95–100°C in order to promote dissolution.

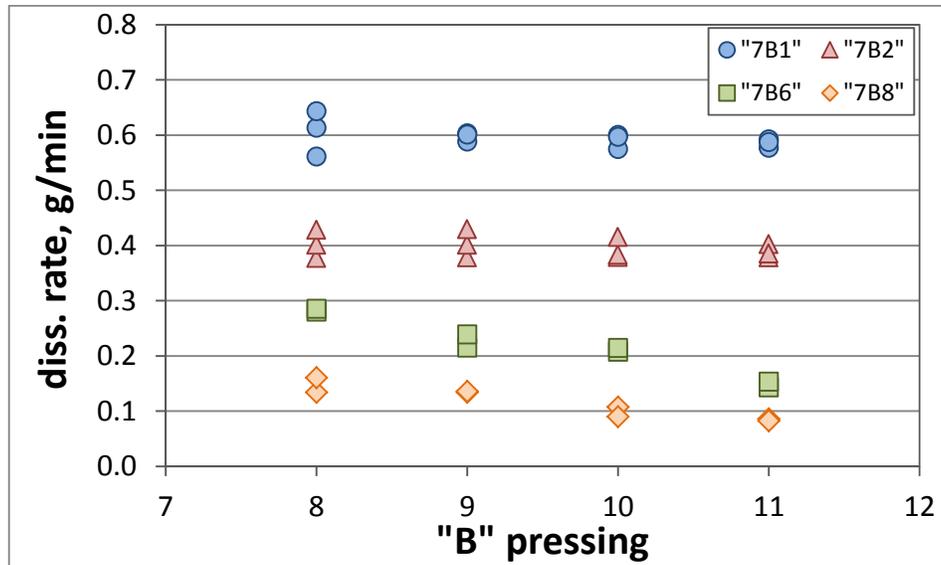
#### 3.1 DISSOLUTION OF NORTHSTAR SINTERED Mo DISKS

All disks were pre-sintered at A = 7. Figure 7 shows the dissolution rates of Mo disks sintered at C = 1, 2, 6, and 8 conditions versus pressing conditions in the range of B = 8–11. In general, increased pressing does not have a significant impact (and density, see Figure 1) for disks sintered at C = 1 and C = 2 (7B1 and 7B2). Very good dissolution rates of ~0.6 g/min for 7B1 and ~0.4 g/min for 7B2 disks were observed for all pressing conditions. On the other hand, disks sintered at C = 6 and 8 dissolved significantly slower, and these disks showed a noticeable trend of decreased dissolution rates with pressing and density (Figure 7). It should be noted that all disks sintered at C = 8 needed to be heated in order to achieve complete dissolution. The solution was heated to ~95°C.

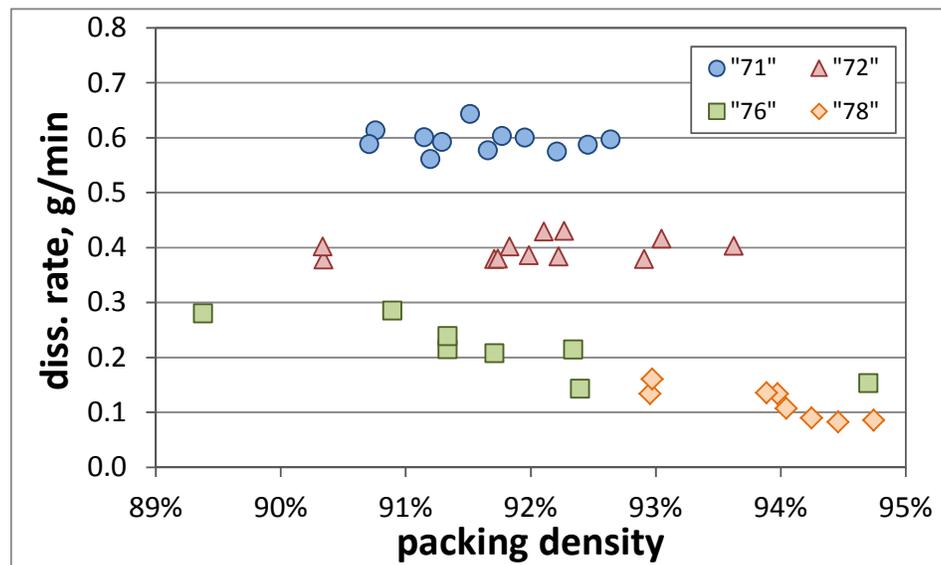
Disks with up to ~95% packing density can be easily prepared; however, data in Figure 8 show that the dissolution properties strongly depend on pre-sinter and sinter conditions. By increasing pressing conditions, even lightly sintered disks with C = 1 and 2 had packing densities in the range of ~90–94% but had significantly higher dissolution rates compared to disks sintered at stronger conditions (C = 6 and 8).

Figure 9 compares the dissolution rates for disks with packing densities in the range of 92–94% and sintered under four different sets of conditions. It shows the benefit of using lighter sinter conditions to achieve very good dissolution rates for disks with densities >90%.

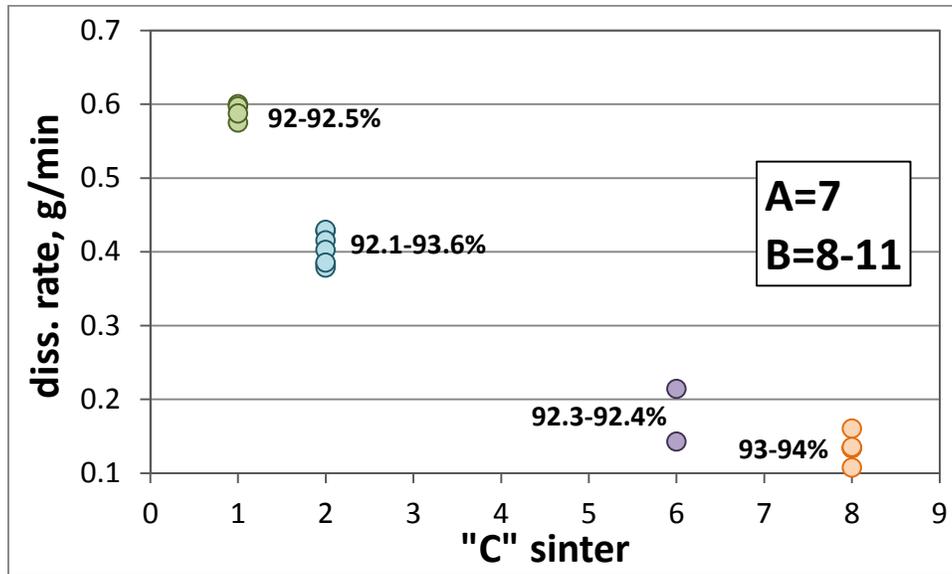
Optical micrographs of disks sintered under four different conditions (Figure 10) show significant changes in disk microstructure due to different sintering conditions. It should be noted that most of the apparent porosity for disks sintered at C = 1 is due to removal of poorly bonded particles during the grinding and polishing operations. The disks sintered under stronger



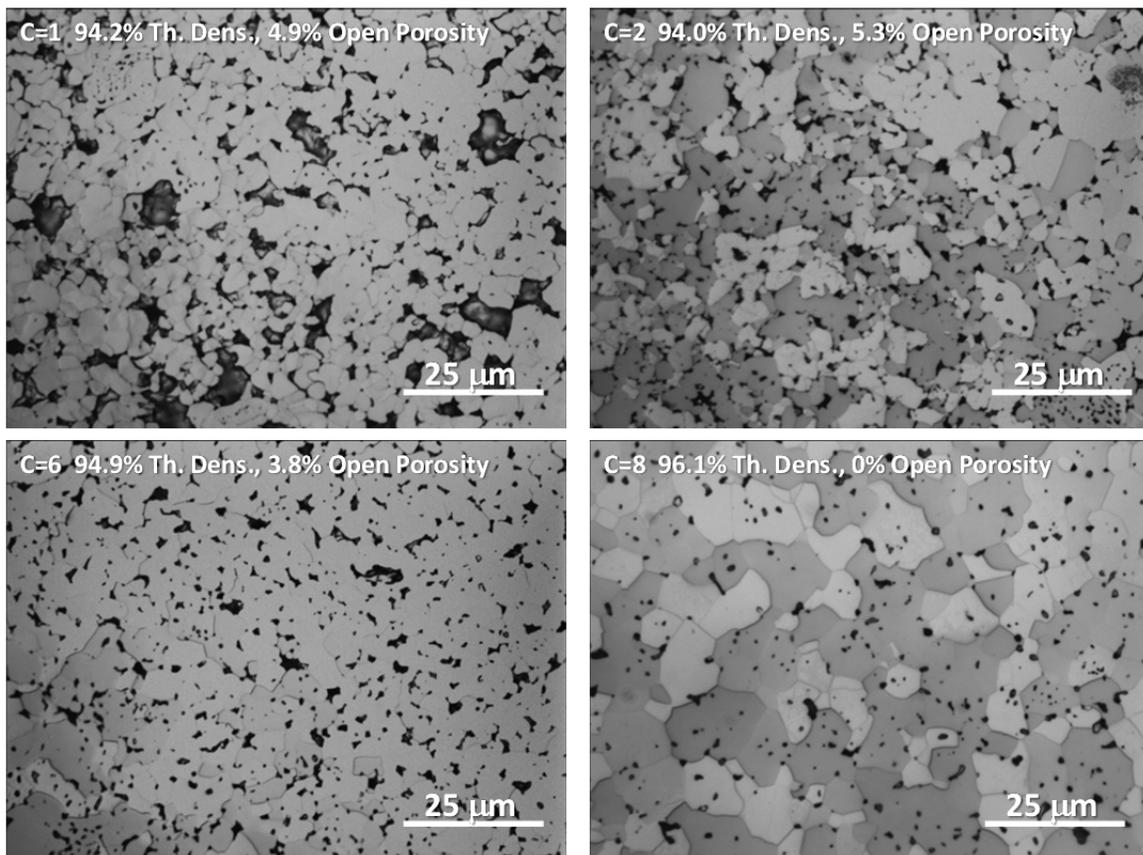
**FIGURE 7** Dependence of Dissolution Rates on Pressing Conditions for Disks Sintered at Four Different Conditions, C = 1, 2, 6, and 8.



**FIGURE 8** Dependence of Dissolution Rates on packing Density for Disks Sintered at Four Different Conditions C=1, 2, 6 and 8.



**FIGURE 9** Dissolution Rates of Disks Pre-sintered at A = 7 and Sintered at Different Conditions with Densities ~92–94%



**FIGURE 10** Optical Micrographs of Polished Sections of Mo Disks Pre-sintered at A = 7, Pressed at B = 9, and Sintered at Four Different Conditions C = 1, 2, 6, and 8.

conditions (C = 2, 6, and 8) had stronger inter-particle bonds and do not show removal of Mo particles. The disk sintered at C = 8 clearly shows coalescence of particles and significant grain growth. The voids in this disk are located internally within the grains. On the other hand, the porosities of disks sintered at C = 1, 2, and 6 are located at the interfaces between the original powder particles. These images show the importance of disk microstructure toward dissolution properties. A decrease of open porosity and an increase in bonding of Mo particles for disks sintered at stronger conditions explain poor dissolution properties.

### 3.2 DISSOLUTION OF ORNL SINTERED Mo DISKS

The first set of disks represented samples prepared from three different starting Mo materials (Table 1). It is clear from the data that non-sintered disks 1 and 5 dissolve very fast. A very good example of how sintering affects the properties of the disk is to compare the dissolution rates of disks 1 and 2. Dissolutions were performed in ~40 mL of hydrogen peroxide pre-heated to 70°C.

The green packing density of disk 2 is 80.8%; this increases to 87.2% by sintering at 1600°C. A similar trend is apparent for other disks sintered at high temperatures (e.g., disks 3, 4, and 6). However, the dissolution rates between disks 1 and 2 are significantly different despite very similar packing densities, with the non-sintered disk dissolving more than two times faster. The same relationship is observed for disks 5 and 6. It is also interesting to see a significant difference in dissolution rates for disks 9 and 10. Both disks were prepared from the same material, with the same pressing and sintering conditions. In addition, disk 10 was prepared from coarser Mo powder and treated at a higher temperature, which resulted in a higher dissolution rate, most likely due to significantly higher open porosity and lower packing density.

Another set of disks (disks 11–37) was prepared from six different Mo powders, and several parameters were tested to study packing density and dissolution rates (Table 2). All disks were sintered under Ar/4% H<sub>2</sub> atmosphere. Four different Mo powders were used to make cold-pressed disks. The densities were in the range of 89.7–94.3% and depend mostly on the starting material used and the sintering temperature. Very good dissolution rates were observed for non-sintered samples (~1 g/min). However, it should be pointed out that for some materials, including enriched Mo-100 material (discussed later), disks needed to be sintered in order to achieve >90% packing density.

Sintered disks (Table 2) were made of three different Mo powders, all pressed at 100 ksi and treated and sintered at different temperatures. Dissolution rates differ significantly for different starting materials. In general, the densities for disks made of CLIMAX EM2 material did not exceed 91% even if sintered at high temperature (1600°C). A significant difference between CLIMAX NM3 and Atl. Eq. Eng. (AEE) powders is that the disks made of CLIMAX NM3 sintered at 1400°C produced very high density (>94.5%), while to make disks with 94% density from AEE powders, sintering needed to be take place at 1600°C. The best dissolving sintered disks were disks made of Atl. Eq. Eng. powders. A comparison of dissolution

**TABLE 1 Characteristics of Sintered Mo Disks and Corresponding Dissolution Rates**

Disk	Powder			Green Dens. (%)	Sintering			Sinter Dens (%)	Open Porosity (%)	Dimensions (mm)		Mass (g)	Diss. Rate <sup>b</sup> (g/min)
	Type <sup>a</sup>	Treat (°C)	Press (ksi)		Temp. (°C)	Time (hr)	Atm			Diameter	Thickness		
1	AA	None	200	89.4	–	–	–	–	6.3	9.57	1.16	0.75	0.69, GS
2	AA	None	100	80.8	1600	1	Vac	87.2	11.4	9.25	1.29	0.74	0.32
3	AA	None	100	80.5	1600	1	Vac	89	4.2	12.25	2.38	2.46	0.37
4	AA	None	100	79.5	1700	1	Vac	91.6	3.7	12.1	2.33	2.46	0.28
5	EM2	None	200	90.9	–	–	–	–	8	9.57	1.15	0.75	0.69, GS
6	EM2	None	100	83	1600	1	Vac	90.3	5.6	9.18	1.29	0.75	0.19 <sup>c</sup>
7	NM3	60	200	67.4	1200	1	Ar/4% H <sub>2</sub>	90	3.2	8.63	1.1	0.58	0.33
8	NM3	800	100	61.6	1100	1	Ar/4% H <sub>2</sub>	87.3	11.3	8.45	1.22	0.60	0.38
9	NM3	800	200	72.8	1100	1	Ar/4% H <sub>2</sub>	92.7	0.6	8.81	1.06	0.60	0.26
10	NM3	1000	200	81.1	1100	1	Ar/4% H <sub>2</sub>	86.2	13.9	9.37	1.02	0.60	0.60

<sup>a</sup> AA = Alfa Aesar; EM2 = Climax Em2; NM3 = Climax NM3.

<sup>b</sup> GS = green solution formed.

<sup>c</sup> Additional heating applied.

rates versus packing density for non-sintered and sintered disks made of different starting material is shown in Figure 11.

It is clear that dissolution rates are very sensitive to sintering temperature, but the starting material can also affect properties of the disks. Figure 12 shows SEM images of two Climax Mo powders in the as-received, as-pressed, and sintered conditions. After sintering, the powder particles have coalesced to form large grains that are joined to each other with diffusion bonds. The remaining porosity becomes trapped within the grains, and, therefore, the dissolution rates are very low. The effect of sintering temperature on disk properties, which significantly affect the density and dissolution rates, is shown in Figures 13 and 14.

Data in Figure 14 show the importance of powder treatment before pressing and sintering the disks. The negative effect of sintering temperature on dissolution rates is clearly visible; however, increasing the temperature of powder treatment before pressing in general has a positive effect on dissolution rates. From the data presented, it is clear that sintering temperature affects the dissolution rates due to a change in the microstructure of the Mo powder. However, increased packing density may play an important role as well. The effect of packing density on dissolution rates can be seen for non-sintered disks pressed at different conditions in Figure 15.

**TABLE 2 Characteristics of Sintered Mo Disks and Corresponding Dissolution Rates**

Disk	Powder		Press. (ksi)	Green Dens. (%)	Sintering		Sinter Dens. (%)	Open Porosity (%)	Dimensions (mm)		Mass (g)	Diss. Rate <sup>b</sup> (g/min)
	Type <sup>a</sup>	Treat.			Temp. (°C)	Time (hr)			Diameter	Thickness		
11	AA	None	200	89.7	–	–	–	6.5	12.75	0.996	1.14	1.14, GS
12	EM2	None	200	91.0	–	–	–	8.3	12.75	0.989	1.15	1.15, GS
13	EM2	None	250	92.9	–	–	–	5.7	12.76	0.955	1.15	1.09, GS
14	HDFM	None	200	90.8	–	–	–	8.1	12.75	0.986	1.14	1.52, GS
15	NPA	None	200	91.9	–	–	–	7.4	12.75	0.972	1.15	1.08, GS
16	NPA	None	250	94.3	–	–	–	5.1	12.75	0.944	1.15	0.93, GS
17	AEE	800°C	100	77.6	1400	1	89.7	7.1	12.13	1.10	1.14	0.62
18	AEE	None	100	77.4	1500	1	91.7	0.3	11.95	1.12	1.15	0.38
19	AEE	800°C	100	77.6	1500	1	91.9	0.2	11.97	1.12	1.14	0.36
20	AEE	1000°C	100	77.8	1500	1	92.0	0.5	11.98	1.11	1.15	0.41
21	AEE	1100°C	100	77.5	1500	1	91.5	4.4	11.96	1.11	1.15	0.38
22	AEE	None	100	77.4	1600	1	93.8	0.1	11.87	1.12	1.15	0.18 <sup>c</sup>
23	AEE	800°C	100	77.6	1600	1	94.0	0.0	11.86	1.10	1.14	0.19 <sup>c</sup>
24	AEE	1000°C	100	77.8	1600	1	94.3	0.0	11.85	1.10	1.15	0.2 <sup>c</sup>
25	AEE	1100°C	100	77.5	1600	1	93.9	0.0	11.89	1.09	1.15	0.21 <sup>c</sup>
26	EM2	None	100	81.3	1500	1	89.7	8.8	12.28	1.09	1.15	0.27 <sup>c</sup>
27	EM2	1000°C	100	81.0	1500	1	89.5	7.2	12.28	1.09	1.15	0.27 <sup>c</sup>
28	EM2	None	100	81.3	1600	1	90.5	0.1	12.20	1.09	1.15	0.18 <sup>c</sup>
29	EM2	800°C	100	81.0	1600	1	89.5	2.9	12.24	1.10	1.16	0.21 <sup>c</sup>
30	EM2	1000°C	100	81.0	1600	1	90.4	0.4	12.21	1.09	1.15	0.19 <sup>c</sup>
31	EM2	1100°C	100	80.1	1600	1	89.5	5.1	12.18	1.10	1.15	0.2 <sup>c</sup>
32	NM3	800°C	100	59.3	1300	1	90.1	2.6	10.95	1.35	1.15	0.36
33	NM3	900°C	100	62.0	1300	1	90.6	3.7	11.10	1.33	1.15	0.41
34	NM3	1000°C	100	65.4	1300	1	89.5	8.1	11.32	1.30	1.15	0.46
35	NM3	800°C	100	59.3	1400	1	94.6	2.7	10.71	1.35	1.13	0.1 <sup>c</sup>
36	NM3	900°C	100	62.0	1400	1	95.4	1.9	10.85	1.32	1.12	0.12 <sup>c</sup>
37	NM3	1000°C	100	65.4	1400	1	96.5	0.8	11.02	1.28	1.15	0.12 <sup>c</sup>

<sup>a</sup> AA = Alfa Aesar; AEE = Atl. Equip. Eng.; EM2 = Climax Em2; NM3 = Climax NM3; NPA = Climax NPA.

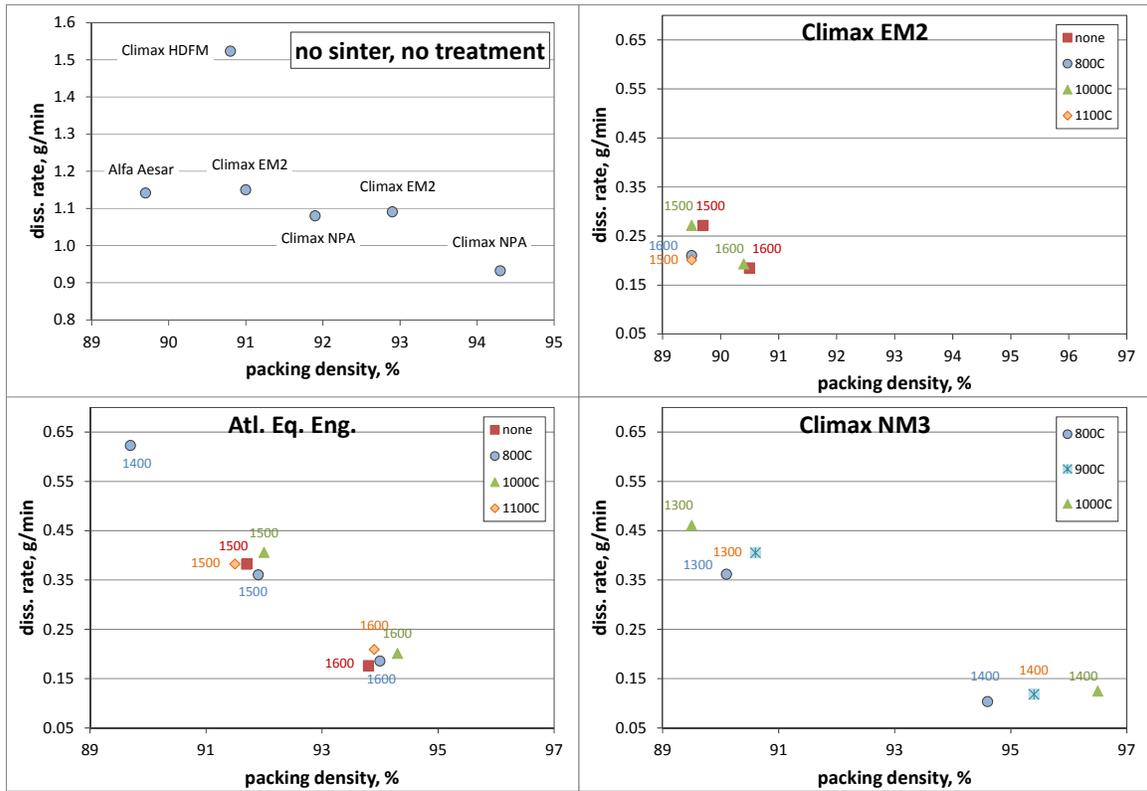
<sup>b</sup> GS = green solution formed.

<sup>c</sup> Additional heating applied.

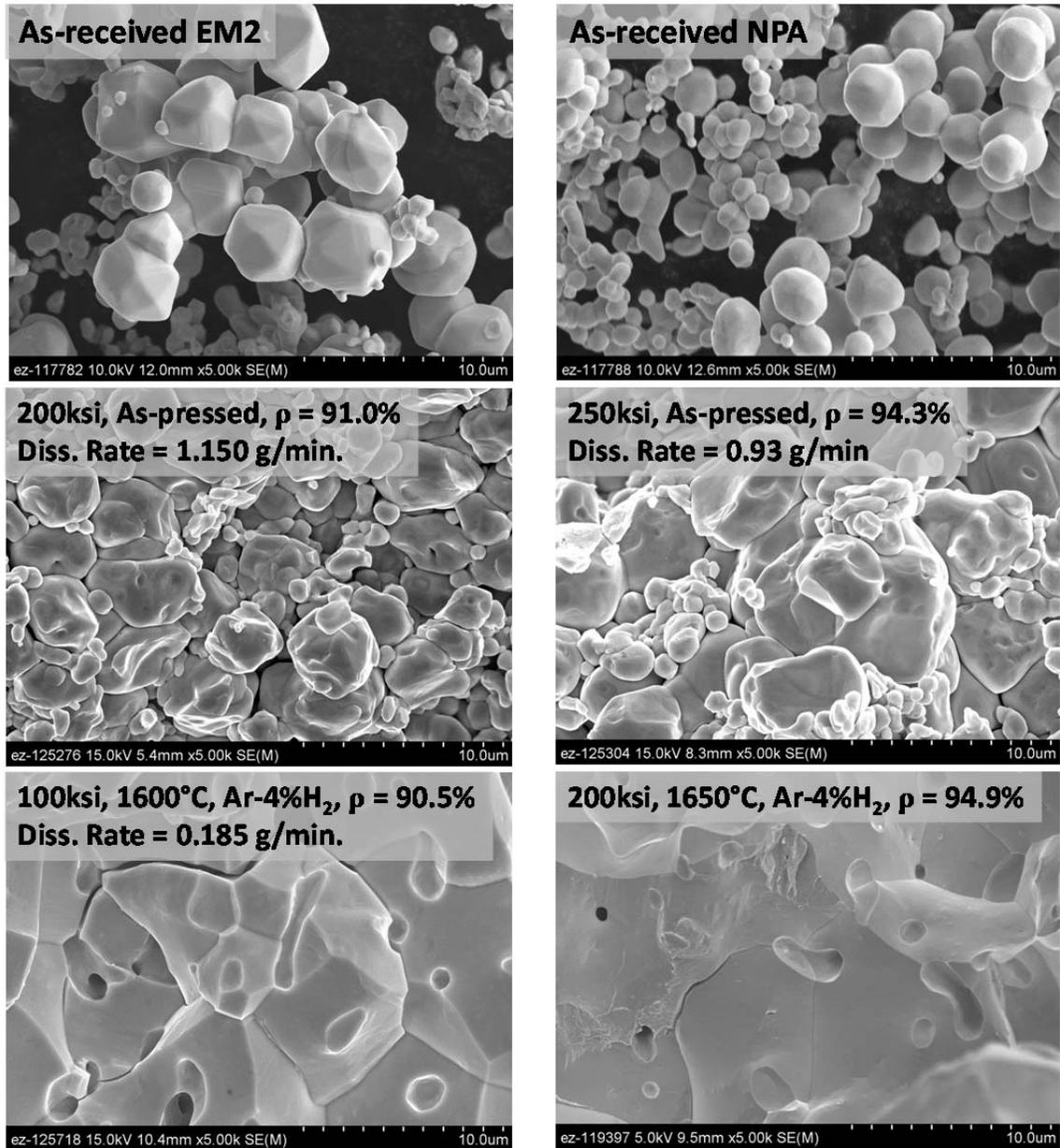
Although the dissolution rates observed for non-sintered disks are very high for all packing densities in the range of 91.5–95.3%, there is a noticeable effect of packing density on dissolution rate. The increase of 3.4% in packing density reduces the dissolution rate by 20%. This trend could be even stronger for sintered disks.

Table 3 shows the characteristics for sintered disks made from IsoFlex >99% enriched Mo-100 powder. Surprisingly, despite relatively high open porosity and low sintering temperature, the dissolution rates for all Mo-100 enriched disks were relatively low, and additional heating was required for complete dissolution; this could be due to smaller surface area. However, it should be noted that the dissolution was very slow only at the beginning, when the temperature of the H<sub>2</sub>O<sub>2</sub> was 70°C; once the temperature of H<sub>2</sub>O<sub>2</sub> was increased to ~95–100°C by additional heating, the dissolution started to progress very quickly, with complete dissolution occurring in 1–1.5 min. This is very promising, because actual irradiated Mo targets

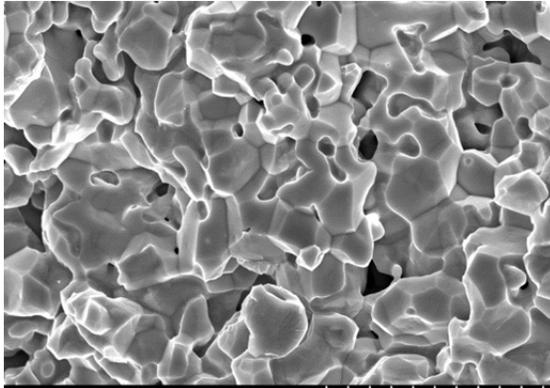
will be dissolved at higher temperatures, which could lead to significantly shorter dissolution times. At the same time, the dissolution data for Mo-100 enriched disks demonstrate the importance of starting material on the characteristics of the final disks. Future studies should therefore focus on the preparation of disks from powder that was obtained by reduction of MoO<sub>3</sub> powder. This is important because after recovering spent Mo-99 source solution from the generator, enriched Mo-100 material needs to be recovered and purified from potassium molybdate solution and recycled by conversion to MoO<sub>3</sub> that can be reduced to Mo powder and used for production of new disks. Therefore, it is important to understand that the dissolution of sintered Mo disks and the recovery of Mo-100 are directly interconnected, since one produces a starting material for the other.



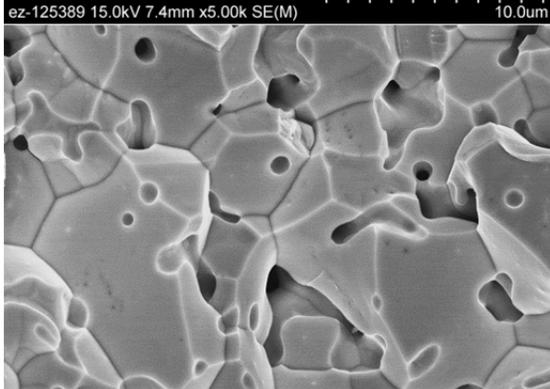
**FIGURE 11 Comparison of Dissolution Rates Versus Packing Density for Non-sintered and Sintered Disks Made of Different Starting Material (Data points for sintered disks indicate temperature of sintering.)**



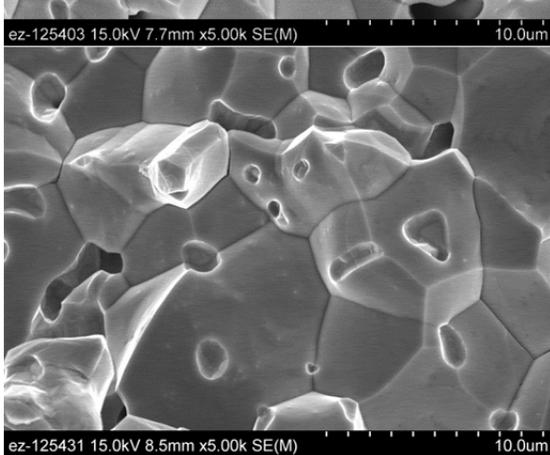
**FIGURE 12 SEM Images of Two Climax Mo Powders As-Received (Top), and Fracture Surfaces of As-Pressed (Middle) and Sintered (Bottom) Disks**



**1400°C**  
**Density = 89.7%**  
**Open Porosity = 7.1%**  
**Diss. Rate = 0.623 g/min**

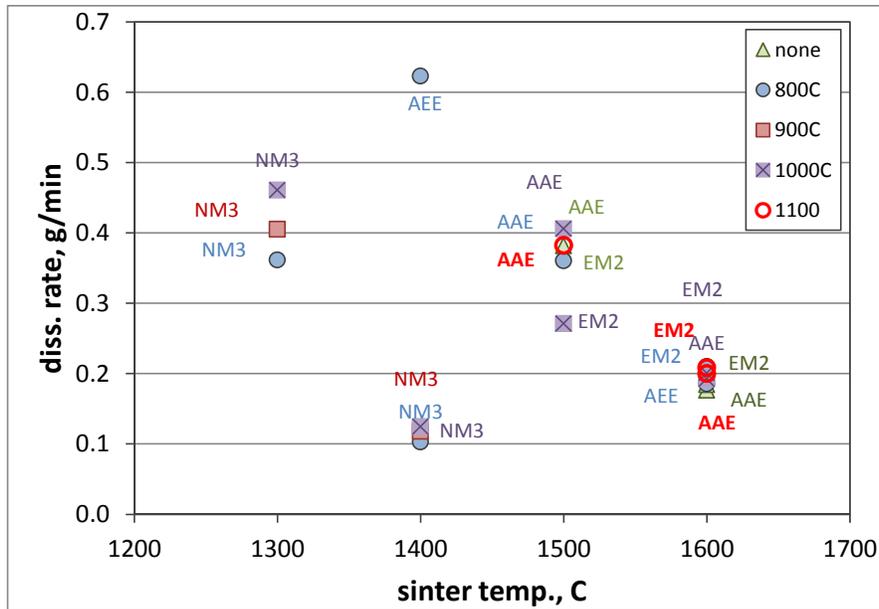


**1500°C**  
**Density = 91.9%**  
**Open Porosity = 0.2%**  
**Diss. Rate = 0.361 g/min**

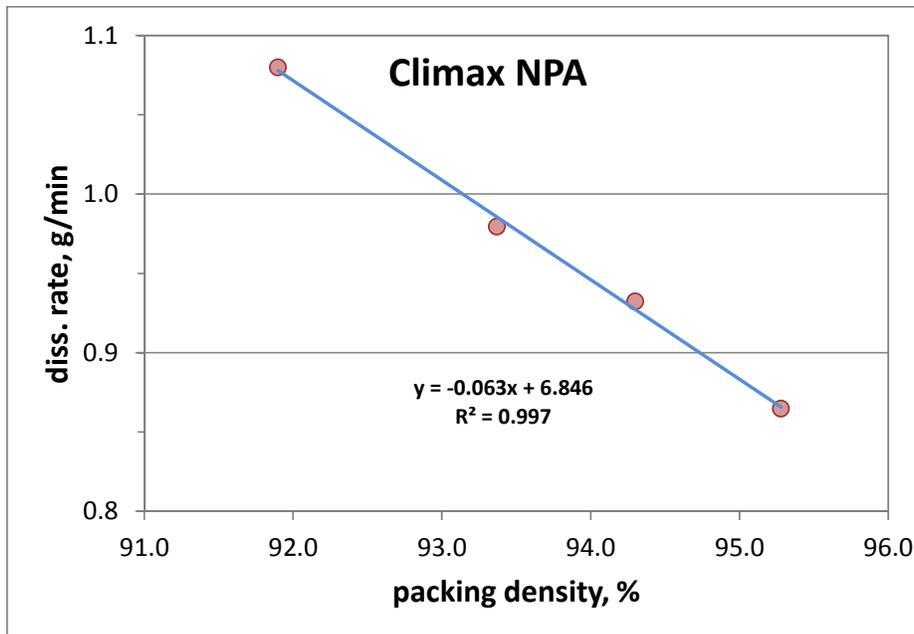


**1600°C**  
**Density = 94.0%**  
**Open Porosity = 0%**  
**Diss. Rate = 0.186 g/min**

**FIGURE 13 SEM Images of Fracture Surfaces of Sintered AEE Mo Disks Show the Effect of Sintering Temperatures on the Microstructure (As the temperature increases, grain size and diffusion bonding increase while the open porosity and dissolution rates decrease.)**



**FIGURE 14 Comparison of Dissolution Rates Versus Sintering Temperature for Different Mo powders Pretreated at Different Conditions (EM2 = Climax EM2; AEE = Atl. Equip. Eng.; NM3 = Climax NM3)**



**FIGURE 15 Dependence of Dissolution Rates of Non-sintered Mo Disks on Packing Density**

**TABLE 3 Characteristics of Sintered >99% Mo-100 Enriched Disks and Corresponding Dissolution Rates**

Disk	Powder		Press. (ksi)	Green Dens. (%)	Sintering		Sinter Dens. (%)	Open Porosity (%)	Dimensions (mm)		Mass (g)	Diss. Rate (g/min)
	Type <sup>a</sup>	Treat.			Temp (°C)	Time (hr)			Diameter	Thickness		
43	IF 4663	None	100	69.2	1200	1	91.7	3.3	8.56	1.11	0.5849	0.10 <sup>b</sup>
44	IF 4178	None	100	65.1	1300	1	89.8	1.5	8.68	1.05	0.5915	0.14 <sup>b</sup>
45	IF 4663	None	200	81.6	1000	1	89.7	8.4	9.25	0.89	0.5575	0.22 <sup>b</sup>
46	IF 4663	1000	200	84.4	1000	1	89.5	9.5	9.43	1.03	0.6446	0.25 <sup>b</sup>

<sup>a</sup> IF = IsoFlex 99+% Mo-100 enriched Mo powder.

<sup>b</sup> Additional heating applied.

## 4 SUMMARY

Dissolution studies performed on NorthStar and ORNL disks show that an increase in sintering temperature has a negative effect on dissolution rates in 30% hydrogen peroxide. This is due to changes in the microstructure of sintered Mo powder. At higher sintering temperatures, the powder particles coalesce, form diffusion bonds, and grow into larger grains, which reduce the open porosity of the disks. The remaining porosity becomes trapped within the grains, and therefore the dissolution rates are very low. We also observed that different starting materials lead to different dissolution properties, and in some cases, even high sintering temperature at 1600°C does not produce disks with high packing densities. Studies with NorthStar disks showed that increased packing density can be achieved by increased pressing conditions without affecting the dissolution rates if mild sintering conditions are used.

Future experiments will focus on producing sintered Mo disks produced from recovered Mo material and on optimizing the sintering process to produce disks with >90% packing density and good dissolution rates.

## 5 REFERENCE

Tkac, P., G.F. Vandegrift, and J. Harvey, *Dissolution of Sintered Mo Disks*, ANL/CSE-13/19, Argonne National Laboratory, July 2012.





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