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# OXIDATION OF BE AT ELEVATED TEMPERATURE

Robert C. Cook, Janelle Gunther

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## Target Area Technologies Program

Mail Station L-481

Ext: 2-3117

September 16 , 2004

To: Distribution  
From: Bob Cook and Janelle Gunther  
Subject: Oxidation of Be at Elevated Temperature

This is a brief memo to start the discussion about the extent of oxidation of our Be shells during pyrolysis to remove the mandrel. The memo has two parts. First is an analysis of the expected rate of oxidation based on data from a 1950 paper by Gulbransen and Andrew<sup>1</sup> along with some of their observations. Second is a suggestion for our own experimental study of the oxidation.

### Gulbransen and Andrew paper:

The authors expose samples of Be foil to oxygen at various temperatures and measure the mass increase. They find that the rate of mass gain follows a parabolic rate eq:

$$W^2 = Kt + C \quad (1)$$

where  $W$  is the weight gain in  $\mu\text{g}/\text{cm}^2$ ,  $t$  is the time in minutes, and  $K$  and  $C$  are constants. This parabolic form is apparently common for surface reactions and is due to the rate determining step being the diffusion of some species (in our case probably Be) through the oxide coating for reaction. One other feature of interest is that the rate of reaction seems to be independent of the pressure of  $\text{O}_2$  gas. This is consistent with the rate determining step being the diffusion of the Be through the coating. There are lots of tidbits in the paper but we simply want to focus on the expected rate of oxidation for our capsules, and this is best approached from Figure 8 of the paper, reproduced on the next page.

The is data for 500 °C and 0.1 atm of  $\text{O}_2$ . The actual data are a little difficult to see, but the lines more or less represent the data. Plotted on the ordinate is  $W^2$  in units of  $(\mu\text{g}/\text{cm}^2)^2$ , and on the abscissa is time in minutes. One can fit the line above 1200 minutes (20 hrs) with the following expression:

$$W^2 = .02083 \cdot t + 18.33 \quad (2)$$

which thus gives values of  $W$  for 1, 2, and 3 days (1440, 2880, and 4320 min) of about 7.0, 8.8, and 10.4  $\mu\text{g}/\text{cm}^2$ . For one of our 2 mm diameter shells the surface area is about

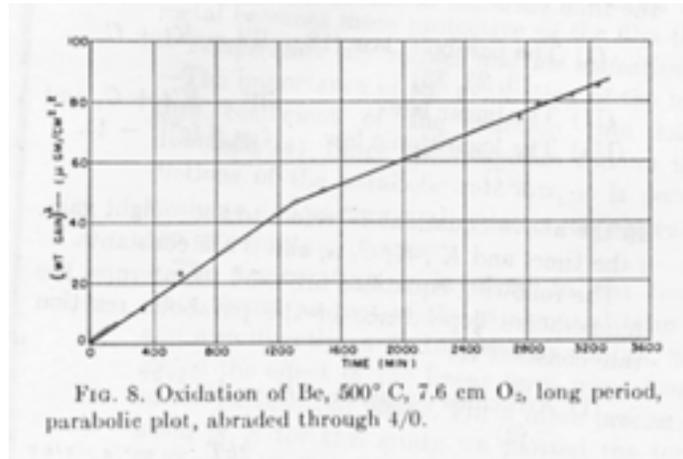
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<sup>1</sup> E. A. Gulbransen and K. F. Andrew, "The Kinetics of the Reaction of Beryllium with Oxygen and Nitrogen and the Effect of Oxide and Nitride Films on its Vapor Pressure," *J. Electrochem. Soc.* **97**, 383 (1950).

$$A = 2 \times 4\pi(0.1\text{cm})^2 \approx 0.25 \text{ cm}^2 \quad (3)$$

where the factor of 2 is for two surfaces. Thus we should expect about 1.8, 2.2, and 2.6  $\mu\text{g}$  of mass gain for 1, 2, and 3 days of heating at 500 °C.

The point of this calculation is not accuracy, but rather order of magnitude. Clearly our capsules have a different morphology than the samples used in the study. And we have been heating to 450 °C instead of 500 °C. But it would be surprising if we saw a mass gain 10 or 100 times these numbers. One last note from the paper, the authors calculated that the depth in Å of a pure BeO layer is approximately equal to 50 times the mass gain in  $\mu\text{g}/\text{cm}^2$ , thus the depth of the BeO coating for 1, 2, and 3 days at 500 °C would be 35, 44, and 52 nm.



### Experimental Plan.

We ought to explore oxidation with our materials under our conditions. This means on shells at the temperatures we are interested in, perhaps 400 to 600 °C. We should probably do the study on undoped Be, since the Cu layers are internal to the coating. It would also be useful NOT to have to worry about the mandrel. Thus we would propose coating some mandrels with perhaps 30  $\mu\text{m}$  of Be, then cooling them in liquid N<sub>2</sub> to hopefully delaminate the plastic from the Be, and then cut the shells in half, hopefully popping out the mandrel in the process. A 30  $\mu\text{m}$  thick 2 mm diameter capsule has a weight of

$$\approx 4\pi(0.1 \text{ cm})^2 \times (.0030 \text{ cm}) \times 1.85 \text{ g} / \text{cm}^3 \approx 0.00070 \text{ g} = 700 \mu\text{g}. \quad (4)$$

Thus a half a shell would have a mass of 350  $\mu\text{g}$ . Given the data above this would be subject to a few  $\mu\text{g}$  of mass gain at 500 °C over a few days, and if this is the case it might be difficult to measure accurately. However if this *is* the rate then we probably don't need to worry about it. However if the rate is 10 times (or more) as high and the weight gain is the order of 10's of  $\mu\text{g}$  then we should be able to measure it.

Thus my view is that we subject half shells to air (probably dry) at say 450, 500, and 550 °C for times up to about 72 hrs and monitor the weight gain.

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