

## INTRODUCTION

Some targets proposed for the laser fusion program require coating high-Z and high-density metal on glass microspheres filled with deuterium and tritium (DT). The dimensions of some of these microspheres are 140  $\mu\text{m}$  diameter and 5  $\mu\text{m}$  in thickness. We have developed a batch coating process<sup>1</sup> to make thick and smooth Pt coatings on microspheres. However, during the development process we discovered that the coating process caused the loss of DT via thermal diffusion. The purpose of this study was to find a set of coating conditions to minimize the DT loss.

### Temperature and Resputtering Aspects on Microspheres

By comparing the DT loss rate of similar microspheres stored in temperature controlled ovens, it was established that the equilibrium temperature of the glass microspheres during coating was approximately 200°C.<sup>2</sup> Using water and LN<sub>2</sub> cooling on the bouncer pan did not have any beneficial effect on the DT loss rate. Thus we suspect that the rise in substrate temperature may be the result of resputtering, i.e. the bombardment of energetic ions or neutrals on the microspheres to cause sputtering off of the coated material. Minimizing resputtering in the coating process should reduce the substrate temperature, thus reducing the DT loss.

Resputtering is difficult to measure directly. From the physics of sputtering, resputtering is proportional to the energy of the bombarding

ions and neutrals, thus it is a function of the power used during the coating process. Under normal conditions when no resputtering takes place, the coating rate is directly proportional to power. Our experiment is to generate coating rates ( $\mu/\text{hr}$ ) at different levels and to detect resputtering if the coating rate is not linearly proportional to the power used.

### Present In situ Cleaning Procedure

The test series were performed in a clean vacuum system consisted of a Sloan-S310 r.f. sputtergun, an r.f. matching network and a vacuum general gas flow ratio controller. The spheres are loaded into a copper pan (Fig. 1) that has been pre-coated with Pt. The pre-coat is done due to the  $\text{O}_2$  in situ cleaning process that is performed prior to the actual coating of the microspheres. The in situ cleaning process seems to help prevent electrostatic<sup>2</sup> clumping of the spheres, while at the same time this pre-conditions the rest of the system before sputtering. The parameters for this cleaning process are the following:

- |             |                           |
|-------------|---------------------------|
| a) Pressure | 500 milli torr in chamber |
| b) Flow     | 200 sccm of $\text{O}_2$  |
| c) Power    | 100 watts (R.F.)          |
| d) Time     | 30 minutes                |

**DISCLAIMER**

This document contains neither recommendations nor conclusions of the United States Government. It is the property of the United States Government and is loaned to your agency; it and its contents are not to be distributed outside your agency. Responsibility for the accuracy and completeness of the information and data furnished herein is assumed by the individual or organization that furnished the information. This document is not to be distributed outside your agency without the specific written consent of the United States Government. This document is for the use of the United States Government and its agencies; it and its contents are not to be distributed outside your agency.

### Experiment Procedures:

The following is the set of conditions that were used for the experiments to determine the coating rate versus power.

1) R.F. Power	150 watts through 400 watts
2) Sputtering Pressure	$3.3 \times 10^{-3}$ torr
3) Dopant gas flow ( $O_2$ )	0.6 sccm
4) Inert gas flow (Ar)	30.0 sccm
5) Target to substrate distance	5.08 cm.

The results are shown in Table 1 and plotted in Figure 2.

### SUMMARY

The result shows that the coating rate is not linear with power used for the coating, as seen in Fig. 2. It seems that the nonlinear portion of the plot shown in Fig. 2 can logically be explained by the resputtering that is suspected. The surface morphology of the coated microspheres as shown in Figs. 3 and 4 supports this contention. At higher resputtering rate the surface should be rougher due to more severe etching. The data seem to indicate that resputtering could increase the temperature of the microspheres to cause DT loss. The future experiments will be to do coatings at the optimum rate of 250 watts and to detect improvement of DT retention.

## ACKNOWLEDGMENT

The author wishes to thank Dr. E. J. Hsieh for his invaluable contributions during the preparation of this paper.

## REFERENCES

1. E. J. Hsieh and S. F. Meyer to be published in J. Vac. 18, 1981.
2. S. F. Meyer to be published in J. Vac. 18, 1981.

TABLE 1

<u>R.F. Power (watts)</u>	<u>Time (Hrs.)</u>	<u>Thickness (<math>\mu\text{m}</math>)</u>
150	1.0	0.5
150	2.0	1.0
200	0.5	0.5
200	1.0	1.0
250	0.5	1.0
250	1.0	2.0
250	2.0	4.0
300	1.0	1.7
400	1.0	1.2

## FIGURE CAPTIONS

Fig. 1. Schematic diagram of the fixturing for batch magnetron sputter coating.

Fig. 2. Coating rate versus power showing non-linear relationship.

Fig. 3. Surface morphology of coatings on microspheres where resputtering is negligible.

Fig. 4. Surface morphology of coatings on microspheres where resputtering takes place.

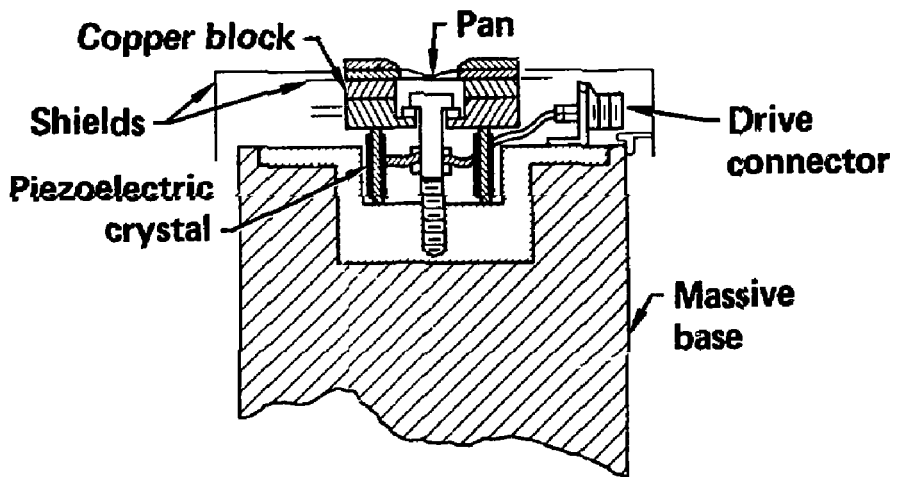
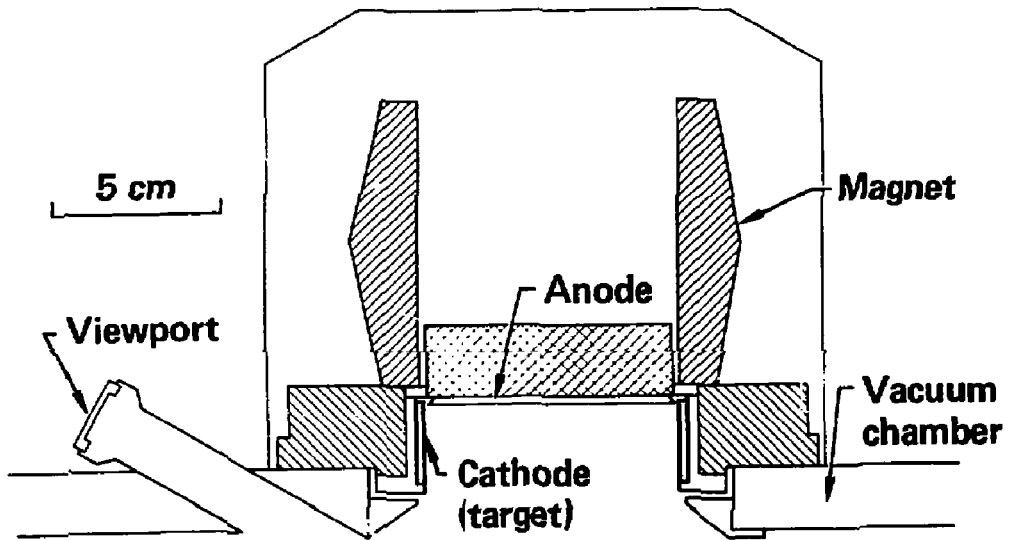
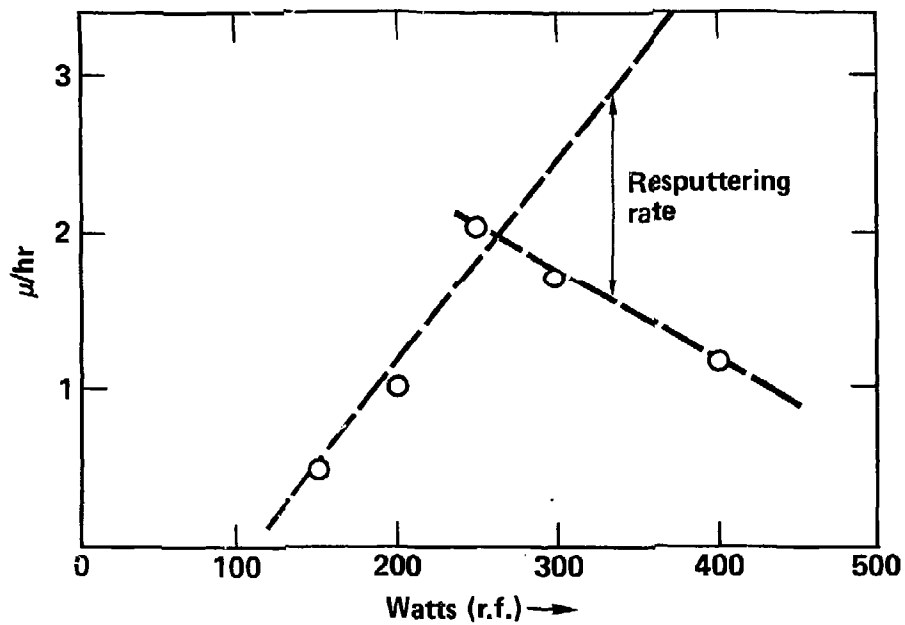


Fig. 1

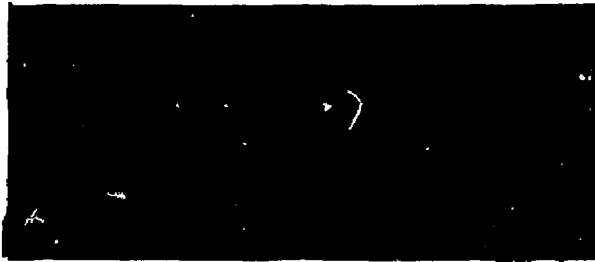




10-00-0581-1183

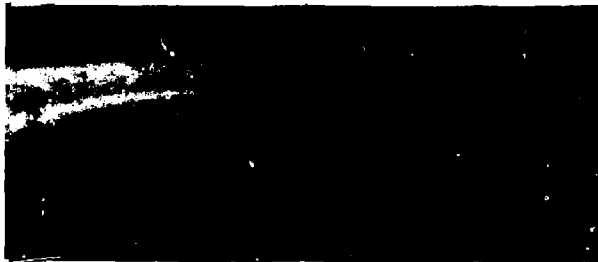
Fig. 2

## ETCHED SURFACE SUGGESTING RESPUTTERING



- 2  $\mu\text{m}$  thick
- 140  $\mu\text{m}$  diameter

2  $\mu\text{m}$



- 1.7  $\mu\text{m}$  thick
- 140  $\mu\text{m}$  diameter

2  $\mu\text{m}$



- 1.2  $\mu\text{m}$  thick
- 140  $\mu\text{m}$  diameter

4  $\mu\text{m}$

## SMOOTH PLATINUM COATED MICROSPHERES



- 0.5  $\mu\text{m}$
- 140  $\mu\text{m}$  diameter

2  $\mu\text{m}$



- 1  $\mu\text{m}$  thick
- 140  $\mu\text{m}$  diameter

4  $\mu\text{m}$

Fig. 4