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# RF MAGNETRON SPUTTERING OF THICK PLATINUM COATINGS ON GLASS MICROSPHERES

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# RF MAGNETRON SPUTTERING OF THICK PLATINUM COATINGS ON GLASS MICROSPHERES*

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Thick platinum coatings on glass microspheres are needed for proposed Laser Fusion targets. The spherical nature of these substrates coupled with the small dimensions (∿100 μm OD) make it difficult to achieve a smooth and uniform coating. Coating problems encountered include a rough surface and porous microstructure from the oblique incidence and lack of temperature and bias control, "clumping" of the microspheres causing non-uniformities, and particle accumulation causing "cone" defects. Sputtering parameters significantly affecting the coatings include total pressure, DC substrate bias, and the addition of doping gases. Using an ultrasonic vibrating screened cage and RF magnetron Sputtergun, we have successfully batch coated microspheres with up to 6 μm of Pt, with a surface roughness of 200 nm, thickness non-concentricity of 300 nm, and density greater than 98% of bulk Pt.

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## INTRODUCTION

High Z, high density coatings on glass microspheres are needed as the tamper layer in proposed double shell laser fusion targets. The tamper specifications are 30 nm surface smoothness, 100 nm thickness uniformity, and thicknesses in the 5 to 10 μm range. The spherical substrates and small dimensions (~100 μm OD) make it difficult to achieve the uniformity and surface finish required using conventional physical vapor deposition processes. Previous attempts at sputtering onto moving microspheres with a batch process in our laboratory, at Los Alamos[1] and KMS Fusion,[2] have suffered from severe microsphere sticking problems, preventing the continuous motion needed for coating uniformity.

There are several coating problems that are unique to the coating of small microspheres. First, the continuous microsphere motion required for coating uniformity prevents substrate cooling, leading to large grain growth with rough surfaces. Second, the high degree of oblique incident flux inherent in coating a spherical substrate promotes a porous, rough film growth. Third, the deposition rate on a rotating spherical substrate is reduced by a factor of four from geometry. Fourth, the lack of continuous electrical contact prevents direct substrate bias sputtering. These problems lead us to use a high incident energy, low pressure, RF magnetron sputtering process, which allows a moderate deposition rate without excessive microsphere heating. Other problems inherent in coating glass microspheres include: "clumping" of the microspheres causing thickness variations, and cone defects nucleated from accumulated particles.

## EXPERIMENTAL

We conduct our microsphere coating experiments in a stainless steel, diffusion pumped vacuum system with servo controlled gas flow and a Sloan S-310 RF Sputtergun. The sputtergun and screened cage are illustrated schematically in Fig. 1.

The screened cage for bouncing and confining the microspheres is driven by a piezoelectric crystal compressed between a massive steel base and a low mass water cooled copper block. The block is electrically isolated to allow DC biasing of the cage. An axial bolt provides the compression and acts as the spring in the mechanical spring-and-mass system. Three copper rings bolted to the copper block clamp the screen and thin copper pan to form the cage, with the pan acting as a mechanical amplifier to bounce the microspheres. The crystal is driven by a "white noise" source so that all resonant modes of the block and pan are excited. The 300 mesh screens are electroformed Ni with an initial transmission of approximately 65%.

All cage parts including the screen go through a series of solvent ultrasonic cleaning and rinsing steps to remove all organic contaminants and loose particles. The final assembled cage containing the microspheres is hydrogen plasma cleaned.

## RESULTS AND DISCUSSION

### I) Microsphere Sticking Problem

Continuous free random motion of the microspheres is absolutely necessary to ensure a uniform coating. However, the microspheres often tend to stick to each other, the pan, and the screen. Vacuum cold welding[3] is probably the main cause of sticking for soft metals, such as gold and aluminum, which often results in patches being pulled off from the microspheres. The other sticking effect, believed to be electrostatic in origin, causes a clumping of the microspheres which is usually reversible when the coating conditions are changed. This sticking usually takes the form of microspheres moving up to the underside of the screen and either sticking motionless to the screen, or moving around on the screen intermittently. Sometimes the microspheres clump together and stick to the pan without motion. Increasing the mechanical bouncer drive or reducing the sputtering power releases the microspheres, which then return to normal bouncing in the pan. However, excessive bouncer drive can cause cracking and breakage of the Pt coating. Keeping the microspheres in constant motion without breakage is a major problem.

The sticking seems to be caused by the microspheres being charged by the plasma. The evidence for an electrostatic mechanism includes: a negative bias voltage on the cage reduces the sticking; RF sputtering has less sticking than DC sputtering at similar power levels; addition of an electronegative gas such as oxygen to the plasma atmosphere minimizes sticking; and lastly, the degree of sticking is proportional to the plasma power density.

Adding oxygen as a dopant gas in our sputtering process is one of the most effective methods for preventing sticking. Once oxygen is introduced into the plasma during coating, the microspheres immediately become unstuck, and bounce freely. As the oxygen flow is gradually reduced, at a critical flow the microspheres clump back together again. The clumping and unclumping actions are reversible and controlled strictly by the oxygen.

II) Platinum Coating

Several coating parameters such as operating pressure, cage bias, and oxygen doping have a substantial influence on the Pt coating quality. In particular, the effect of the total sputtering gas pressure is in good agreement with Thornton's Structure Zone Model.[4] The transition region, Zone T, produced by low deposition pressure and low substrate temperature, is the desired region for the very smooth, uniform coatings needed on laser fusion targets.

Our best coatings were achieved with the following deposition parameters: total pressure in the vicinity of 0.8 Pa (6 mTorr), with oxygen flow of 0.5 SCCM, argon gas flow of 30 SCCM, bias of -100 V, and RF Sputtergun power of 300 W. The resulting Pt coating on microspheres shown in Fig. 2 is ∿ 98% bulk Pt density and has a ∿ 100 nm surface finish. Deposition pressures of 0.8 Pa and lower typically produce dense, smooth coatings which are typical of Zone T growth. However, the deposition rate of 1 μm/hr appears to decrease monotonically with decreasing pressure, so operating at the lowest possible pressure is not necessarily desirable. Different deposition parameters produce coatings with characteristics which fall predominately into three other classes. We will discuss in general terms how each coating parameter influences the coating structure.

The quality of the Pt coating decreases with increasing deposition pressure, in agreement with the Structure Zone Model. Pressures above about 1 Pa (7.5 mTorr) produce the rough coating shown in Fig. 3, which is indicative of Zone 1 growth. These Zone 1 coatings appear rough, porous or loosely packed, and are of low density, typically ∿14 g/cc. An inert gas pressure below 0.8 Pa will suppress this Zone 1 growth in spite of the high degree of oblique incidence.

The oxygen dopant also affects the Pt coating characteristics. The addition of about 1% oxygen to the argon sputtering gas refines the grain structure in the growing Pt coating, and hardens the metal against cold welding. With a large amount (> 5%) of oxygen, the Pt coating becomes very hard and brittle. These extremely fine grained coatings are characterized by cracks and broken layers from impact damage. On the other hand, when we use insufficient oxygen, (< 1%), the Pt coating is soft and forms the knobby structure shown in Fig. 4. Accretion by the microspheres by cold-welding of loose particles within the cage probably accounts for the patchy surface. The bouncing of the microspheres against the cage appears to peen the soft Pt particles into a semi-smooth surface. The desired oxygen dopant level falls between these two extremes, and depends, of course, on both sputtering rate and system gas throughput.

The bouncing action is the third most important parameter (after total pressure and oxygen dopant) in controlling the Pt coating characteristics. The gross non-uniformity seen in some depositions is obviously the result of inadequate bouncing action. On the other hand, a too vigorous bouncing action causes cracking and breakage of the Pt coating, particularly with higher oxygen flows. A not so obvious effect of vigorous bouncing action is that the impact of the microspheres onto the cage parts, the Ni screen in particular, can knock off some micrometer sized particles from those parts. On impact, the small particles adhere to the Pt coating producing "cone defect" nuclei. We achieved our cleanest coatings by reducing the bouncing action to barely moving the microspheres around with minimum collisions with the screen.

With our cage configuration, we do not observe a pronounced difference in the coating between biased and unbiased runs, possibly because the microspheres are not at bias potential most of the time. However, as already mentioned, the bias definitely helps prevent sticking.

## CONCLUSIONS

We have demonstrated a method for batch coating microspheres with a smooth, uniform layer of platinum of micrometer thicknesses. The essential ingredients in the process include low pressure RF magnetron sputtering, oxygen doping of the sputtering gas, and confining the microspheres in a biasable, water cooled, vibrating, screened cage. The oxygen doping refines the grain size for a smoother surface finish, and alters the plasma to reduce the microsphere sticking for a more uniform coating. Work is in progress to determine the oxygen content of the platinum films, and to understand the basic mechanisms involved with the microsphere sticking effects.

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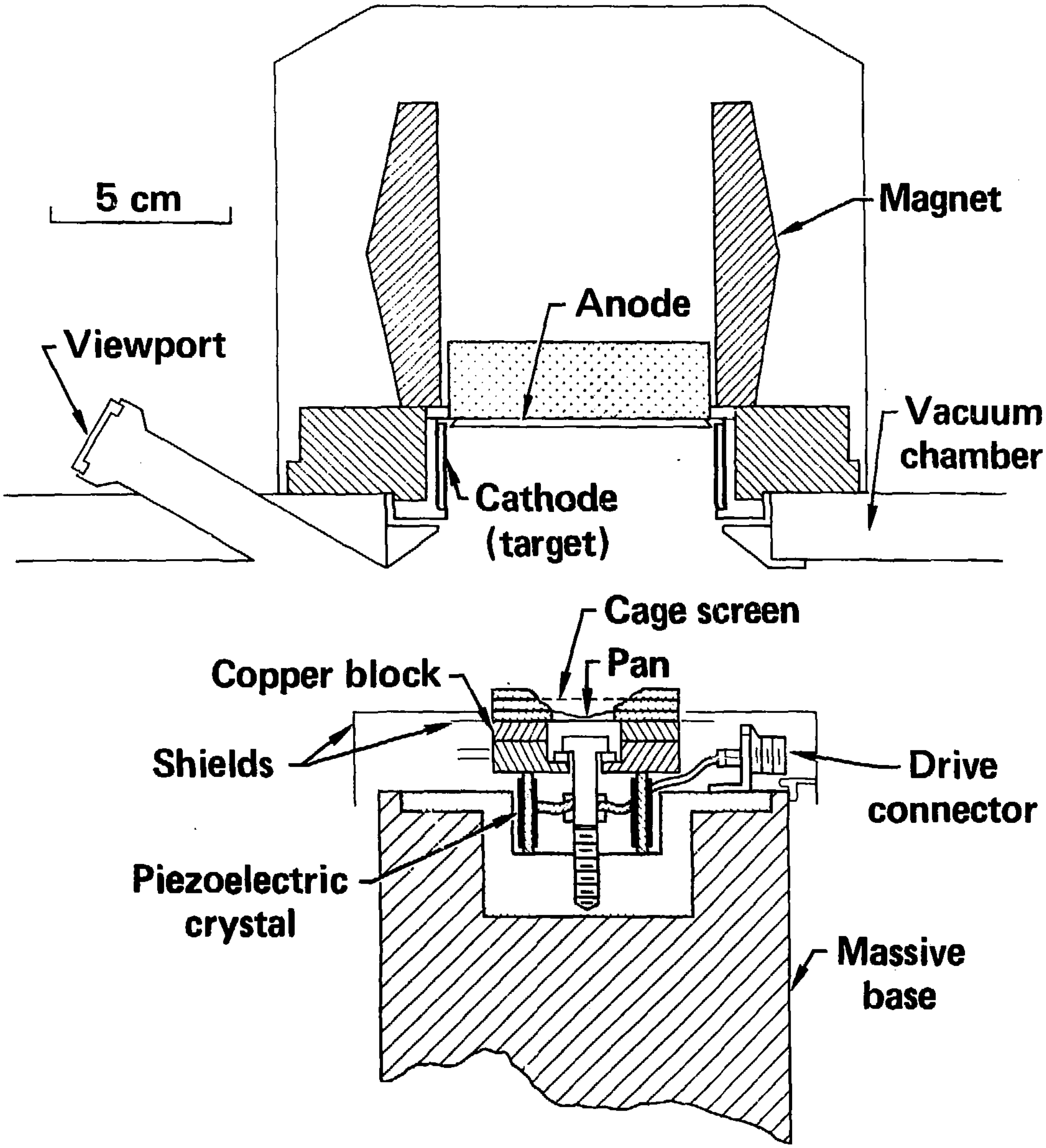
## FIGURE CAPTIONS

Fig. 1 Schematic illustration of Sloan S-310 Sputtergun and screened cage showing their relative orientation and major components.

Fig. 2 Smooth platinum coating (100 nm surface finish, bulk platinum density) grown on microsphere under best sputtering conditions. The "cobblestone" surface texture appears to be related to the columnar microstructure.

Fig. 3 Low density, porous, Zone 1 platinum coating on microsphere. Sputtering pressure above 1 Pa (7.5 mTorr) promotes this growth.

Fig. 4 Platinum coated microsphere showing "soft" coating grown with insufficient oxygen doping.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Magnet
Anode
Viewport
Vacuum chamber
Cathode (target)
Cage screen
Pan
Copper block
Shields
Drive connector
Piezoelectric crystal
Massive base

FIGURE 1

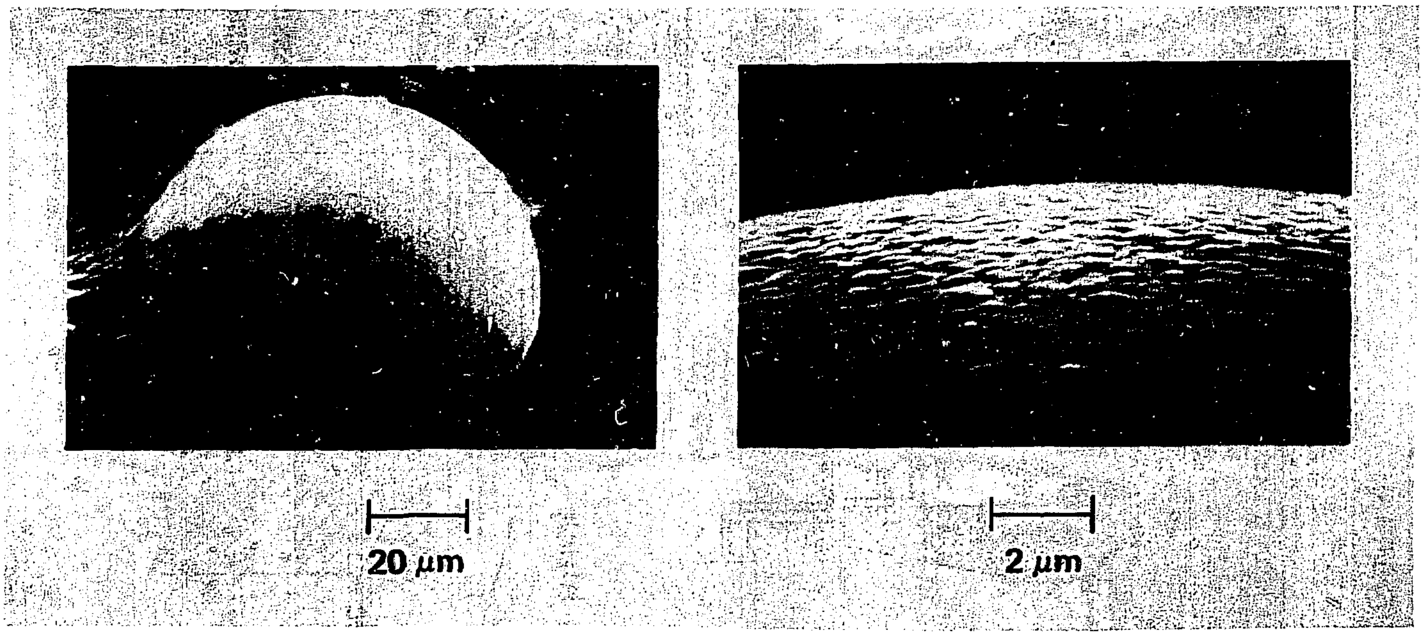


FIGURE 2

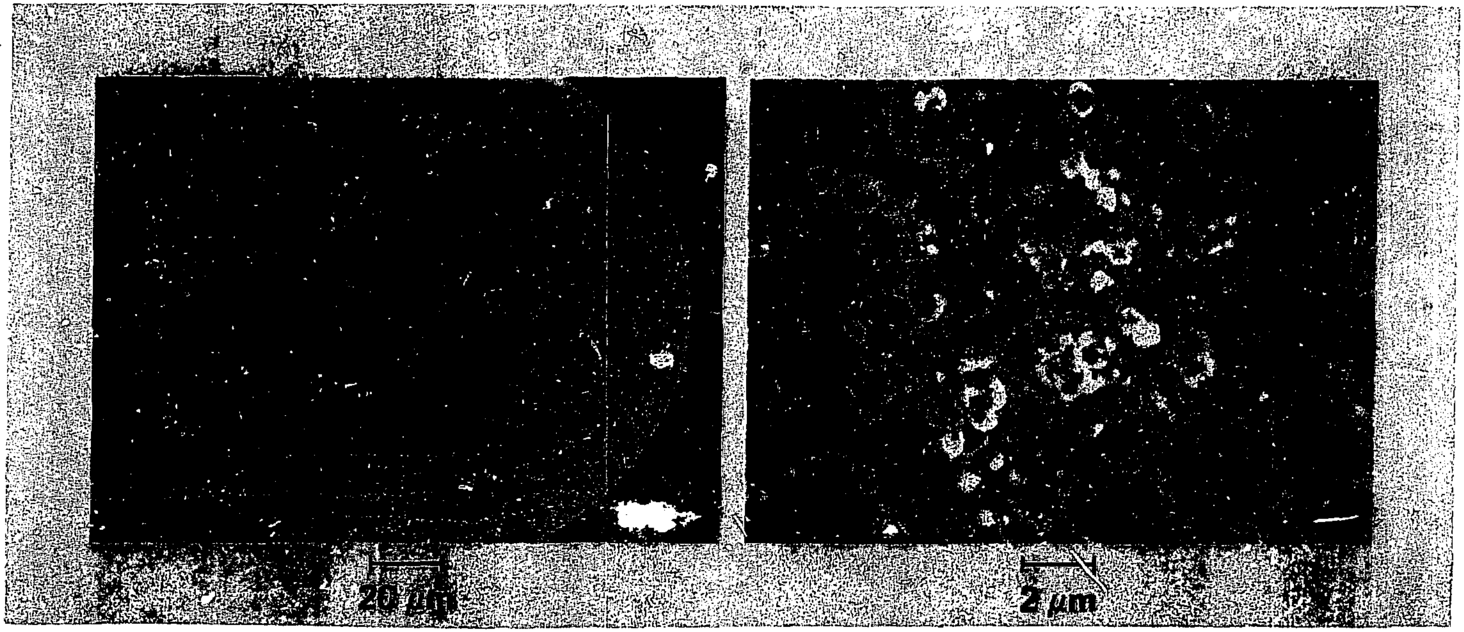


FIGURE 3

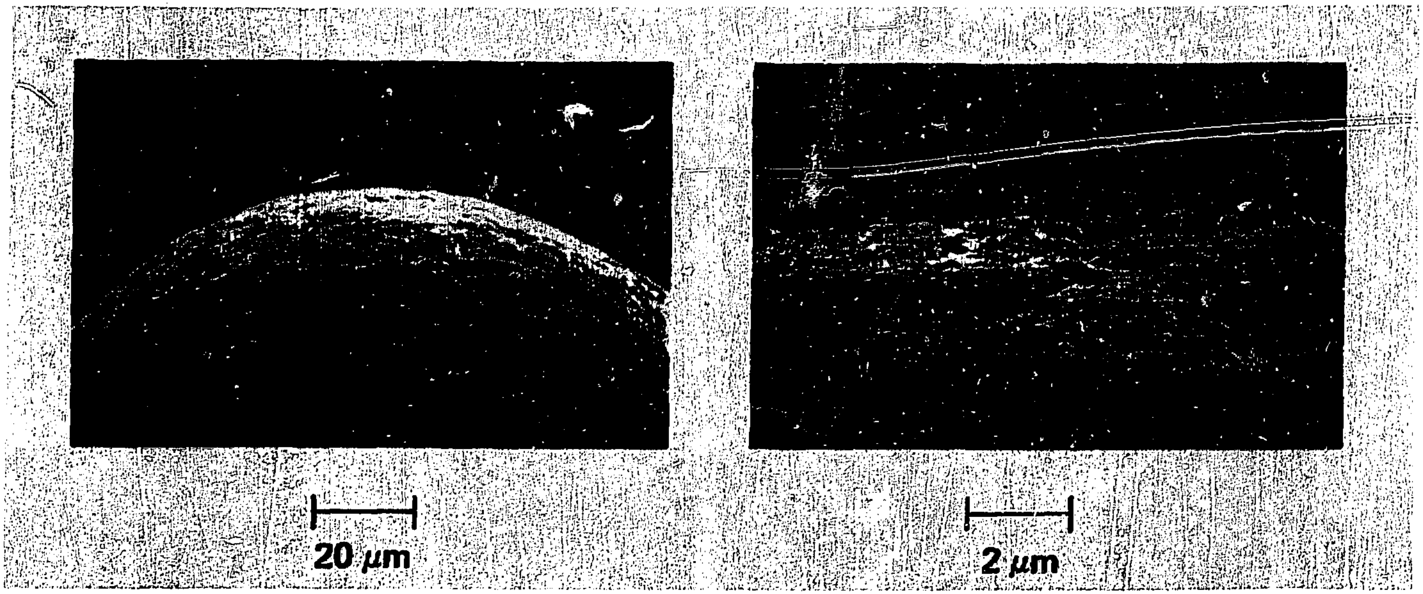


FIGURE 4