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METALLIC COATING OF MICROSPHERES*

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ABSTRACT

Extremely smooth, uniform metal coatings of micrometer thicknesses on microscopic glass spheres (microspheres) are often needed as targets for inertial confinement fusion (ICF) experiments. The first part of this paper reviews those methods used successfully to provide metal coated microspheres for ICF targets, including magnetron sputtering, electro- and electroless plating, and chemical vapor pyrolysis. The second part of this paper discusses some of the critical aspects of magnetron sputter coating of microspheres, including substrate requirements, the sticking of microspheres during coating (preventing a uniform coating), and the difficulties in growing the desired dense, smooth, uniform microstructure on continuously moving spherical substrates.

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INTRODUCTION

Metal coatings on small particulate substrates have a wide variety of potential uses including substrates for catalysts, fillers for conducting epoxy, and targets for ICF experiments. The hollow glass microspheres used as fuel containers for ICF experiments are often coated with one or more layers to improve the implosion efficiency. Previous coatings have been chiefly plastics, but the current emphasis is on the larger range of properties of metal coatings. Typical metal coating applications include low Z ablators (e.g. beryllium), high Z pusher/tamper layers (e.g. gold, platinum, tantalum), and diagnostic layers (e.g. copper). This paper reviews those microsphere coating methods used to fabricate ICF targets, and discusses some of the unusual difficulties involved in sputter coating microspheres.

Typical microsphere coatings needed for ICF experiments are the single and multiple shell targets illustrated in Fig. 1. The first coating step for either target would be the deposition of the high-Z metal tamper layer on a glass microsphere. For the single shell target, the low-Z ablator layer would be coated directly over the high-Z layer after an inspection process, whereas for the double shell target, the low-Z ablator might be fabricated separately as two hemispheres, or deposited on a preassembly of the inner ball and a thin outer shell. Typical coating specifications on a high-Z tamper layer a few micrometers thick would permit a maximum variation of 10 nm in surface finish and/or thickness, i.e. relative variations in $\int \rho dr$ along any radial line must

be much less than 1%, constraining variations in both thickness and density. The thicker ablator layers would normally have more relaxed requirements, a 100 nm surface finish and uniformity of 500 nm would be acceptable in some cases. The microspheres used as substrates must meet surface finish and uniformity standards similar to the coatings, and in addition, must retain the fuel and remain intact during coating. The extreme smoothness and uniformity of these coatings is necessary to prevent the growth of Raleigh-Taylor instabilities during the implosion, which could lead to shell breakup and failure of the experiment.¹

This paper is organized in two parts. The first part is a review of established, successful microsphere coating processes reported in the literature. The second part is a discussion of the author's views on several critical aspects of magnetron sputter coating of microspheres.

REVIEW OF MICROSPHERE COATING PROCESSES

The basic process requirements for producing smooth and uniform coatings on microspheres can be lumped into several general categories:

- (1) The microspheres must be maintained in constant, random motion for coating uniformity;
- (2) The deposition method must be compatible with the motion of the microspheres and with the required coating materials;
- (3) The coating must grow with a dense, uniform microstructure.

Various attempts to meet these general requirements have lead to the three different processes, summarized in Table I, which have proven

generally effective in providing useful metal coatings on microspheres: (1) Magnetron sputtering, with the microspheres kept in motion by either continuous, mechanical agitation, or by gas levitation of a single microsphere; (2) electro- and electroless plating, with the microsphere motion usually provided by pumping the solution; and (3) chemical vapor pyrolysis on microspheres in a fluidized bed. Each of these processes has its own unique advantages and disadvantages, and none of them can be considered a universal method at this time.

Magnetron Sputtering Processes

Two methods have evolved jointly at several laboratories for keeping microspheres in motion during sputtering with magnetron Sputterguns: one suitable for batch operation, and one suitable for individual microsphere coating. The apparatus designed to mechanically bounce microspheres on a hard surface is variously referred to as a "bouncer," "shaker table," or "vibrator." This method, herein referred to as the bouncer method, is suitable for batch coating processes, but suffers from problems with dirt, microsphere breakage, and sticking effects. A non-contact, gas levitation method, often referred to as "gas jet" or "molecular beam levitation," is used to support a single microsphere during coating by momentum transfer from gas molecules preferentially striking the microsphere on the underside.

Magnetron sputtering with a bouncer. The bouncer, as currently used in our laboratory,² is driven by a piezoelectric crystal compressed

between a massive steel base and a low mass copper block. The microspheres are placed in a thin copper pan bolted to the water cooled copper block with two copper washers. The block is electrically isolated from the crystal electrodes and the grounded base to allow independent biasing of the pan. Of course, the microspheres are not in continuous contact with the pan so the biasing effect is rather indeterminate. The piezoelectric crystal is driven by "white noise", easily calibrated with a voltmeter, and fairly insensitive to changes in the mechanical bouncer tuning.

Other variations of this basic approach to the bouncer process have incorporated a screen over the pan², and use of a low frequency driver instead of the high frequency sensitive piezoelectric crystal.^{3,4} In general, none of the low-frequency driver technologies have received sufficient development to overcome the many bouncer problems.

All of the bouncer approaches suffer from several recurring difficulties. A successful batch process requires a large number of microspheres (10 to 500) as coating stock, often unavailable and always a potential hazard. This coating stock must be sufficiently clean to avoid distributing particulate dirt throughout the batch, causing "cone defects" on every microsphere. If the coating is a high-Z metal, all of the microspheres must be of target quality, since it is very difficult to measure glass parameters through an opaque, X-ray absorbing coating. The DT fill is also difficult to measure after coating, so leaky microspheres can cause major problems. Both of these last two restrictions, of course, apply to any process coating high-Z metal over glass

microspheres. The large number of microspheres involved in a batch process can give erratic deposition rates due to the shadowing of one microsphere by others. The repeated impact of microspheres against the pan and other microspheres can mechanically damage the coating and cause breakage, an additional source of particulate debris. Last, but perhaps most troubling of all, the bouncer processes are prone to electrostatic sticking, leading to non-uniform microsphere coatings.

Magnetron sputtering with gas levitation. The earliest gas levitator for ICF target coating, reported by Lowe and Hosford,³ used a 50 μm -dia.-hole collimated hole structure (CHS). A dimple, machined in the CHS, provided microsphere localization, and helped when starting and stopping the levitation. However, lateral stability was poor, requiring high pressure deposition (> 3 Pa) and low rates to prevent microsphere loss. An important contribution of this work was the demonstration that gas levitation can give cleaner coatings than the bouncer method, presumably from the absence of collisions which can create and transfer debris.

More recent development work produced two methods to increase the lateral stability of the CHS levitator. One method, reported by Goldstein and Varon,⁵ used a 50 μm dia focused hole structure (FHS) with the individual gas beams converging to a point 25 mm above the holes. This effectively provided a radial restoring force linear in the displacement from the symmetry axis, extending the feasibility of sputter coating levitated spheres to pressures below 1 Pa. The second method,

referred to as the Molecular Beam Levitator (MBL) by Crane,⁶ uses a CHS, but adds a beveled washer above the CHS to deflect the outer beams to give an inward directed effusive flow and a radial restoring force. The name derives from the fact that gas levitators work in the molecular flow regime levitating by momentum transfer, in contrast to the hydrostatic levitation of a beach ball by the fluid flow of an air stream.

All three of the gas levitation methods have several coating problems in addition to the general requirement for clean, non-leaky, target quality microspheres. At this time, all methods are restricted to coating a single microsphere per run, with each run often lasting many hours (efforts are underway to handle several microspheres at a time). Common electrostatic effects may cause either microsphere adhesion to the levitator structure or expulsion from the levitator. Surface defects on the microsphere can lead to preferential spinning patterns producing non-uniform coatings.^{7,8} And finally, these processes are subject to microsphere loss from vibration and transient effects during pumpdown.

Chemical Reduction Plating

Electroplating. Most of the microsphere plating efforts have used proprietary bright plating baths for the levelling effect and surface properties they provide. Microsphere plating works quite successfully with nickel and gold;^{9,10} but suffers delamination and rough surfaces with copper.^{11,12} Most electroplating cells use pumps to circulate the plating solution through a cell designed to confine the microspheres.

Mayer and Catlett⁹ reported microsphere plating with a cell using two pumps, alternating every 5 to 30 seconds to force the microspheres against cathode screens at each end of a porous ceramic tube. Each cathode was energized during the half cycle when the microspheres were forced against it by the flowing solution. The electrical circuit was completed by solute flow through the porous tube to an anode in the solution tank external to the cell. The column of the cell was filled with beads larger than the microspheres to ensure random motion of the microspheres as they move from one end of the cell to the other. A more recent improvement of their apparatus¹³ eliminated the porous column, substituting a glass column with threaded ends for better microsphere recovery. The electric circuit is completed by a wire positioned behind each cathode screen to serve as an anode for the cathode at the opposite end of the cell. An external solution tank is no longer needed.

Illige, et al.¹⁴ have reported several variations of electroplating cells. One cell incorporated a mechanically driven vibrator to minimize the plating of microspheres onto the cathode screens. A second cell used a column similar to the design of Mayer and Catlett,⁹ filled with metal spheres to randomize the microspheres. However, Illige¹¹ found that continuous pumping during the plating cycle led to a drastic increase in microsphere breakage, perhaps related to the use of high pressure DT filled microspheres in his process. He achieved better results by alternately plating, then pulsing the solution to redistribute the microspheres, then plating, etc. An even more novel design allows buoyant microspheres to roll up an inclined electrode during plating,

with a stream of solution to sweep them back down and complete the cycle.¹⁴

The difficulties unique to electroplating on microspheres include: The need for a metal strike on the glass surface to make it initially electrically conducting; the limited range of suitable metals (so far only gold, copper, nickel and nickel-iron); and the design of cells to handle microspheres that initially float, and then may sink later in the deposition. Methods for providing the initial strike include the Brashear silvering process, CVD nickel, and sputtered platinum, nickel, and palladium. The sputtered strikes appear to be the most generally used, and to provide the cleanest, smoothest surfaces.

Electroless plating. Electroless or chemical reduction plating has been used by Mayer and Catlett⁹ to coat microspheres with nickel and gold. The cell design is conceptually simpler than for electroplating, since no electrodes are necessary, and simply pumping the solution back and forth with a large piston achieves sufficient motion. The reduction reaction is autocatalytic, requiring only that the microspheres be initially sensitized with sputtered Ni, Pd or Pt; CVD nickel, or stannous or palladium chloride. Respectable electroless nickel coatings have been deposited on microspheres sensitized with CVD nickel, however, electroless plating is, in general, even more restricted in applicability than electroplating. Moreover, a spontaneous reduction reaction may occur from instability of the solution in the presence of the reducing agent coating every surface and piece of debris in the system and creating a horrendous cleanliness problem.¹¹

Chemical vapor deposition

The pyrolytic decomposition of metal compounds, principally transition metal carbonyls, has been used successfully by McCreary¹⁵ to coat microspheres for ICF targets. One application was the decomposition of Mo(CO)_6 at 300°C depositing Mo_2C on microspheres in a fluidized bed. In general, CVD is carried out at too high a temperature for use on gas filled microspheres, however, the Mo_2C is permeable to hydrogen so the coated microspheres can be filled under normal microsphere filling conditions. Nickel and tungsten coatings on microspheres were made by the same process, but were impermeable to hydrogen, suggesting that CVD may be of limited usefulness for ICF target production.

Several characteristics of fluidized beds reported by Vogt¹⁶ present difficulties in microsphere coating. Bubbling of the gas stream in the bed can cause significant inhomogeneities in the coatings, and reduce the coating rate from loss of reactant. Fluidized beds tend to be sensitive to the relative density of the particles, and the deposition of heavy metals rapidly changes the bed behavior of lightweight glass microspheres. Work is in progress to characterize the dynamics of CVD/fluidized bed reactor to develop a reliable microsphere coating process.

CRITICAL ASPECTS OF SPUTTER COATING MICROSPHERES

Microsphere Substrates

The microspheres used for ICF targets are currently made of glass with typical sizes of 100 to $300\ \mu\text{m}$ in diameter and wall thickness

ranging from 1 to 20 μm . These glass microsphere may serve as a major component in simple targets, however, in more recent target designs the principle use is merely as a fuel container and spherical mandrel for the first coating layer. In fact, a high-Z metal fuel container would be preferable in some designs. These tiny glass microspheres weigh as little as 60 ng for a 100 μm OD by 1 μm wall microsphere, and present a difficult handling problem. With such small masses, gravitational force is small compared to the random electrostatic forces normally present, even in a humid atmosphere.

Microspheres are normally filled with DT fuel by diffusion for several hours at elevated temperature and pressure.¹⁷ After fill, a careful washing of the filled microspheres is necessary to remove dirt and shards of broken microspheres picked up in the fill system. Of course, the DT fuel makes these microspheres potentially toxic and due concern must be exercised in the handling at all stages of their use. (Fragments of tritiated glass microspheres are a potential inhalation hazard.¹⁸)

We have found that it is impossible to obtain smooth coatings unless the microspheres are prepared and maintained in an unblemished condition. Without careful vacuum storage, defects of the order of 100 nm will sometimes grow under the influence of atmospheric moisture in a matter of days, more than sufficient to nucleate cone defects in a subsequent coating. Moreover, the surface condition of the microsphere influences all aspects of the deposition, including coating morphology and the microsphere motion. To achieve starting conditions as

reproducible as possible, we plasma strip the microspheres and fixturing in the deposition system before starting the coating process. (We use either hydrogen or oxygen for the plasma strip depending on the coating and fixturing materials.) A contaminated surface on a microsphere can produce interesting defects in the coating, such as the bubbles under the 2 μm thick Pt coating shown in Fig. 2. The microsphere probably warmed up after the coating had formed an initial "skin" to confine the outgassed vapors in a bubble. A similar growth of a sputtered coating over an outgassed bubble was reported earlier by Meyer and Hsieh¹⁹ in the deposition of SiO_2 on Kovar mandrels. Adequate plasma stripping to clean and outgas the microspheres before deposition should prevent such defects.

Microsphere sticking during sputtering

Vacuum welding. Soft metals, such as copper, gold and aluminum, are prone to vacuum welding on impact,²⁰ whether in a bouncer or upon hitting part of the levitator during gas levitation. Harder materials, e.g. Ta, W, Ni, Ti, Cr, Pt, are not as subject to vacuum welding, but still exhibit sticking from electrostatic effects. The severity of the vacuum welding problem for a gold coating in a bouncer pan process is graphically visible in the SEM micrograph in Fig. 3. The thin gold coating on the two microspheres has stuck together in a patch, nearly pulling the patches off of both spheres. The adhesive strength of the gold to the glass is clearly less than the vacuum weld strength. A large number of roughly circular patches where the coating was completely torn

away exist on this and other spheres from the coating run. Such vacuum welding produces unacceptable defects and prevents uniform coating.

Alloying or reactive sputtering can be used to harden the soft metals and prevent vacuum welding, however, there is at present no satisfactory way to use the bouncer method to deposit the pure metals. These soft metals can be coated with gas levitation techniques provided the electrostatic effects are controlled.

Electrostatics Sticking. The single most troubling problem with sputtering metals on microspheres with either bouncer or gas levitation technology has been the tendency of the microspheres to stick to the adjacent fixturing during coating. The sticking may arise from the plasma sheath which exists over the fixturing in most sputtering systems.²¹ A microsphere in the fixturing sheath experiences several different electrostatic interactions simultaneously. First, the time dependent net charge on the microsphere interacts with the fixturing surface charge, and with its image charge induced in the conducting fixturing. Second, the non-uniform electric field in the sheath always exerts an attractive force on the dipole moment induced in the microsphere.²² The electric field strength in the sheath decreases rapidly with distance away from the bouncer surface because of the positive ion space charge, and vanishes at the edge of the plasma sheath. As illustrated in Fig. 4, this field gradient always exerts a force on the induced dipole in the direction of the stronger field. Unfortunately, in contrast to net charge attraction, polarization

attraction is not neutralized by contact between the microsphere and the fixturing. Thus, the polarization attraction can account for the observed sticking of microspheres to the fixturing and other microspheres, although direct experimental confirmation is lacking.

As a result of the interplay of mechanical drive and the electrostatic forces, the motion of a microsphere in a bouncer system can occur in two different modes. In the first mode, the microsphere is bounced sufficiently hard that it escapes the sheath region near the bouncer surface, and becomes surrounded by the plasma. Because of shielding by the plasma, the microsphere experiences no net electrostatic forces (see Fig. 4) and its motion is completely ballistic while in the plasma. In the second mode, the microsphere does not bounce high enough to escape into the plasma, but remains within the plasma sheath. The electric forces in the sheath are much stronger than gravity and completely dominate the microsphere motion. (We have even been able to bounce microspheres downwards on the underside of a screen in this dynamic mode.²) The transition between these two dynamic modes is a quite abrupt function of both the fixturing bias and the bouncer drive. Negative bouncer bias voltage (with respect to the plasma potential) has the effect of repelling the microsphere immediately after contact, to provide a form of "plasma bias drive." Either sufficiently negative bias ($V_p - V_b$ of a few tens of volts) or strong mechanical bouncer drive will propel the microspheres high into the plasma in the first dynamic mode. With weak mechanical and plasma drive, the polarization force and microsphere charging will overcome the initial microsphere velocity,

pulling the microsphere back to the surface in the tightly coupled bouncing of the second dynamic mode. We have fewer problems with mechanical damage to the coating and loss of microspheres when we use this second low amplitude mode.

The gas levitation of a microsphere in a plasma discharge, on the other hand, represents an equilibrium problem. Since with most gas levitators the microsphere sits of order 100 μm above the CHS, the microsphere and levitator have a common sheath. When depositing non-conducting films on an insulating microsphere, the polarization force is small, even with a grounded CHS, and levitation during sputtering is not difficult.⁷ However, with a metal deposition, the highly polarizable microsphere is strongly attracted to the fixturing, resulting in sticking or a high probability of loss if increased gas flow is used to overcome the attraction.

We have reduced this polarization induced sticking with the bouncer coating process in two ways, by floating all of the fixturing near the microspheres to minimize the electric fields, and by the use of oxygen doping during platinum sputtering.²³ Empirical methods used by various researchers to avoid this sticking problem with gas levitation processes have included high pressure sputtering with a screen in front of the Sputtergun,³ and a large separation between the levitator and the Sputtergun.⁵ All of these methods work by reducing the plasma density and the sheath voltage, but may adversely affect the coating structure and deposition rate.

Coating microstructure and surface finish

The structure of a growing sputtered coating appears to be predominately controlled by four factors: the deposition pressure, the amount of surface and volume diffusion (parameterized by T/T_m where T_m is the bulk material melting temperature in Kelvins), the degree of oblique incidence, and the amount of ion bombardment. The different structures obtainable on planar substrates with moderate amounts of oblique incidence and ion bombardment are illustrated in the Structure Zone Model of Thornton²⁴ shown in Fig. 5. At deposition temperatures above about $0.3T_m$ (zone 2 and 3), surface diffusion and bulk diffusion processes can produce rough surfaces. The zone 1 structure is generally less desirable than zones 2 and 3 due to porosity and lack of structural continuity. However, the smooth surfaces and dense microstructures in zone T (low pressure and low T/T_m) are most desirable for ICF target coatings.

Unfortunately, the growth of the desirable smooth, dense microstructure on microspheres is complicated by several factors not normally present in planar coatings. First, a spherical substrate has an area four times the area of the circular crosssection presented to the vapor flux, giving an effective deposition rate only 1/4 that of a planar substrate. Second, the degree of oblique incidence in microsphere coating is inherent in the spherical geometry, and the small size precludes the use of masking techniques. Third, the need for constant motion prevents direct control of the microsphere temperature. Fourth, the electrical biasing parameters are often dictated by the need to

prevent electrostatic sticking, and are unavailable for microstructure modification. The only deposition parameter truly available to the experimenter is sputtering pressure.

Oblique incidence. Any particular point on a randomly moving sphere has a probability, $P(\theta)$, of being at an angle θ to the flux at any given time, which is equal to the surface area, $A(\theta)/2\pi$, at that angle to the flux. Assuming that the flux direction defines the Z axis, then it is clear that $P(\theta) = A(\theta)/2\pi = \sin\theta/2\pi$, and that the deposition rate, $R(\theta)$, relative to a normal incidence planar coating is given by

$$R(\theta) = P(\theta) \cdot \cos\theta = \sin\theta \cdot \cos\theta / 2\pi, \quad 0 \leq \theta \leq \pi/2.$$

Thus, each point on the sphere receives a deposition flux equivalent to a source peaked at 45° incidence as a result of the rapid time average by the moving microsphere over all deposition angles.

This oblique incidence has the effect of promoting zone 1 growth through geometric shadowing processes (high spots receive more coating flux than the surrounding area). If adatom mobility is insufficient to transport the excess material away from the high spots, the bumps grow rapidly, increasing the probability of void formation.²⁴ The initial high spots may come from substrate defects, particulate contamination, and adatom agglomeration in the presence of adsorbed gases. The initial bump formation can be minimized by energetic atom and ion bombardment to increase the adatom mobility, resputter adsorbed gases and create additional nucleation sites.^{25, 26} Higher deposition pressures increase the gas adsorption and reduce the bombardment energy through gas

phase collisions and ion-atom charge exchange.²⁷ Of course, increasing the substrate temperature increases the adatom mobility and reduces gas adsorption, but also increases grain growth. Therefore, for a given degree of oblique incidence, the most favorable conditions for zone T growth are a relatively high energy, low pressure deposition.

To suppress the tendency for porous growth resulting from the combined effects of oblique incidence and low deposition rate, our work with platinum microsphere coating has shown that the deposition pressure must be less than 1 Pa to prevent zone 1 growth.² For pressures above 1 Pa, the Pt film is extremely rough and has a density about 14 g/cc, whereas lower pressure coatings tend to form a columnar microstructure with a rounded cap on each column. This "cobblestone pavement" effect is illustrated in Fig. 6 for a platinum coating with greater than 98% of bulk density. With a substrate less smooth than this microsphere, the resulting columnar structure can easily trap voids between the columns even at low pressures.

There is some very preliminary evidence that interfaces in the growing coating may disrupt the columnar growth and produce a smoother surface, in a manner similar to the oxygen pulsing technique reported by Springer²⁸ during aluminum evaporation on planar substrates. Interfaces apparently occur in the Pt coatings as a result of transient periods of sticking to the bouncer pan with attendant changes in bias and temperature.

Temperature. A microsphere will reach thermal equilibrium between the energy input terms (plasma radiation; vapor atom, electron, and ion kinetic energy; heat of condensation) and the energy loss terms (radiation and gas conduction). Experiments comparing the rate of fuel loss between uncoated microspheres in an oven and those undergoing sputter coating with platinum for an equivalent time, suggest that the coated microspheres reached a temperature of order 200°C ($T/T_m \sim 0.23$ for Pt).²⁹ Under similar conditions, thick beryllium coatings ($200^{\circ}\text{C} = 0.3T_m$) have apparently formed a porous coating, but with large crystallites indicating substantial surface diffusion, as shown in Fig. 7. This porous, yet highly crystalline structure, is not represented in Thornton's Structure Model, since this structure is the result of very oblique incidence not present in his experiments. This large grain growth (but not the porosity) was predicted from beryllium depositions on water cooled and thermally floating planar substrates.³⁰

Efforts to reduce the equilibrium temperature by cryogenic cooling the fixturing, and by using an opposing magnet to reduce electron bombardment have had little apparent effect on either the Pt microstructure or DT loss rate. However, the adverse effects of the relatively high microsphere temperatures during sputtering can be partially avoided by selecting materials with high binding energies, or by hindering diffusion and crystallization by suitable reactive deposition to obtain smooth, dense coatings. For example, oxygen doping during platinum coating produces a harder, smoother, finer grained microstructure, as should reducing T/T_m .

SUMMARY

The deposition processes reviewed in this article represent a tremendous improvement in the technology of microsphere coating over the last two years. However, an even greater increase in coating technology is necessary to meet future ICF target requirements, and to improve the current products. Better understanding of the electrostatic sticking mechanism operating in the sputtering processes should permit bias sputtering for desperately needed modification of the coating microstructure. Substrate temperature remains a problem for both sputtering and CVD processes, placing severe constraints on possible DT fill pressures and microsphere permeation rates. Desirable advances in coating technology include development of processes compatible with new materials such as lithium hydride and beryllium, with deposition on high pressure or cryogenic fuel fills, and with deposition of outer layers on sophisticated, multishell assemblies.

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FIGURE CAPTIONS

- Fig. 1. Schematic diagrams of two target designs for inertial confinement fusion experiments. Both designs require one or more metal coating operations on glass microspheres.
- Fig. 2. Microsphere with bubbles under a $2\text{ }\mu\text{m}$ platinum coating. The bubbles apparently formed by outgassing under an initial "skin" of platinum during sputter coating in a bouncer system.
- Fig. 3. Gold plated microspheres with patches torn off by vacuum welding between two microspheres during collisions. The microspheres were in a bouncer system during coating.
- Fig. 4. Schematic illustration of one microsphere in a plasma and one microsphere in the plasma sheath near an electrically biased, conducting surface. The non-uniform electric field in the sheath exerts a polarization force on the left-hand microsphere, attracting it towards the surface. The microsphere surrounded by the plasma experiences no net electric forces.
- Fig. 5. Zone model for the different microstructures which occur in sputtered coatings (after Thornton²⁴).
- Fig. 6. An example of the "cobblestone" surface found on microspheres sputter coated with Pt at $\sim 200^{\circ}\text{C}$ ($T/T_m = 0.23$) and 0.8 Pa pressure. The rounded cobblestone caps form on the columnar microstructure as a result of the inherently oblique nature of microsphere coating.
- Fig. 7. An example of the porous, yet highly crystalline surface found on a microsphere sputter coated with $18\text{ }\mu\text{m}$ of beryllium at $\sim 200^{\circ}\text{C}$ ($T/T_m = 0.3$) and 0.4 Pa pressure.

TABLE I

DEPOSITION METHOD	MICROSPHERE MOTION METHOD	METALS COATED SUCCESSFULLY	MAJOR DRAWBACKS	REFERENCES
Magnetron sputtering	Bouncer	Pt, Mo, Cr, Ni, Ta, W, Ti Most transition metals possible	Electrostatic sticking Vacuum welding Particulate debris Requires batches of target quality microspheres	2,3,21
Magnetron sputtering	Gas levitator	All of above + Au, Al, Ag, Cu	One-at-a-time Electrostatic sticking Loss of microsphere	3,5,6
Electroplating	Solution pumped against electrodes	Au, Cu, Ni, Ni-Fe	Conducting strike needed Microsphere sticking Rate control	9,10,13,14
Electrolessplating	Pumped solution	Ni, Au Very limited	Sensitizing strike needed Spontaneous reduction	9
Chemical vapor pyrolysis	Fluidized bed	Mo, Mo ₂ C, W, Ni	High temperature Bed behavior changes with coating thickness	15,16

FIGURE 1

20 μm

FIGURE 2

FIGURE 4

FIGURE 5

2 μm

FIGURE 6

1 μm

FIGURE 7