A Method for Characterizing and Improving the Damage Resistance of the Outer Metallic Coatings on Inertial Fusion Energy Targets

Landon J. Carlson, Daniel T. Goodin, and Mark S. Tillack

***Abstract*—A very smooth highly reflective coating on inertial fusion energy (IFE) targets is essential for direct-drive ignition. A gold/palladium (Au/Pd) alloy is sputter coated onto the surface of an IFE target. This provides a larger outward momentum of the material ablated by the ignition laser and improves the energy released from the target compression and thermonuclear reaction. It is also necessary to reflect the black-body infrared radiation experienced while traveling into the chamber and preserve the delicate frozen deuterium and tritium ice inside. The coating must remain intact, without any "pinhole" defects, which requires it to be very durable and that any handling techniques, such as transferring from the layering system to the injector, must be gentle. The coatings were initially tested by physically impacting two targets together to simulate motion in a fluidized bed and were also tested in tension to better understand the adhesion of the coating. Variations in the coating parameters were explored and optimized to produce a low-stress smooth coating of Au/Pd, which was found to have better resistance to damage than the current coatings. Additionally, a titanium sublayer was coated between the plastic shell and the Au/Pd coating to strengthen the adhesion between the inner and outer layers. The initial results of the multilayered coating performed more than twice as well as the best standard Au/Pd coating previously tested. This shows promise for the use of an interlayer to promote better bonding of the outer metallic reflective coating to the plastic shell.**

***Index Terms*—Damage, fusion, gold, HAPL, impact, inertial fusion energy (IFE), palladium, target.**

I. Introduction

A COMMERCIALLY viable inertial fusion energy (IFE) power plant will need to produce and fire about 500 000 targets per day [1], [2]. This creates a challenge to mass-produce targets that maintain the stringent level of quality needed for consistent ignition. The targets are currently produced individually or in small batches, and many production and inspection techniques are not scalable to a commercial power plant. One concern with mass-production scale-up is the damage to the target's outer metallic coating. This can be caused by its interaction with other targets during processing and with machinery required for handling numerous targets. This paper will concentrate on the most damaging step when producing a target, fluidizing with hundreds or thousands of other targets during the cooling cycle. An example of the actual damage caused by fluidizing is shown in Fig. 1.

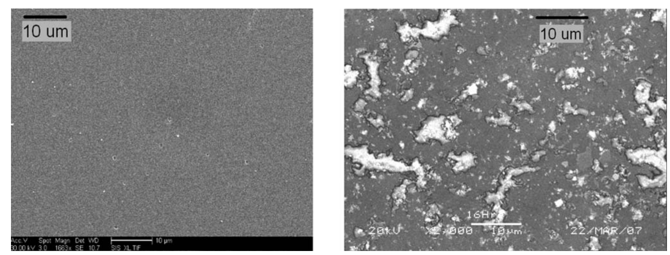

Fig. 1. SEM images of coated surfaces before fluidizing on the left and after fluidizing for 16 h on the right.

The reflective high-Z outer coating on the target is necessary for reducing laser imprinting [3] and also for reflecting the black-body radiation from the hot ignition chamber walls. The coating must be 80 nm thick and have an rms roughness of less than 50 nm [1], [4]. Gold was initially chosen as the high-Z outer layer because of its high reflectivity in the infrared region of 0.5 to 25 μm. This was replaced with a gold/palladium (Au/Pd) alloy when the addition of palladium was demonstrated to allow the D-T gas to permeate through the target more quickly, lessening the time required to fill the targets [3], [5]. The Au/Pd alloy reflects ~93%–95% of the incident IR radiation, so it still remains useful as a reflector inside the hot chamber. In this paper, a very thin sublayer of titanium has shown an improvement in the adhesion of the Au/Pd alloy. The titanium is only 5 nm thick and does not appear to pose a problem for the purpose of the outer metallic coating.

We have characterized the coating damage anticipated in a mass-production environment and have demonstrated promising techniques for strengthening the coating adhesion to improve its damage resistance. This paper will review some of the characterization techniques and some methods of improving coating damage resistance.

II. Overview of Research Performed

The tests conducted on the targets were intended to simulate aspects of the real production environment in a controlled repeatable manner. To simplify and expedite the experiments, some parameters were modified from the real fluidized bed

Manuscript received July 9, 2009; revised December 9, 2009. Current version published March 10, 2010. This work was supported in part by the University of California, San Diego and in part by General Atomics, San Diego.
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Color versions of one or more of the figures in this paper are available online at http://ieeexplore.ieee.org.
Digital Object Identifier 10.1109/TPS.2009.2039715

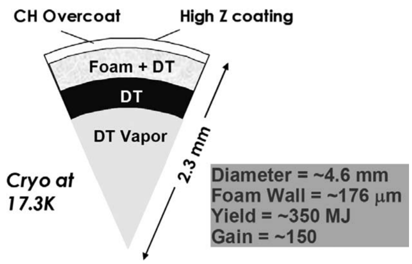

Fig. 2. Section view of a typical HAPL target. [7].

conditions for individual tests. All tests in this paper were performed at room temperature; however, experiments have shown that the strength of the coatings increases at cryogenic temperatures [6]. In order to realize an improvement in target coating strength, a baseline of current coating strengths was established from destructive testing procedures and then compared with tests on newer improved coatings. All tests, unless noted, were performed by the authors at General Atomics in San Diego.

The shells used as substrates for coating in these tests were made from poly-alpha-methylstyrene (PAMS) plastic. Once the bare shells are coated with metal, they are typically called "targets" and are not to be confused with the target of a sputter coater, which is a metal disk containing atoms to be sputtered. All the shells used were fabricated in the same batch, so each had a consistent diameter of 4.034 mm ± 0.005 mm and an average surface roughness of 29 nm rms. Each shell had an average mass of 1.9 mg and was very delicate. This specific shell material is representative of the latest shell in development, a glow discharge plasma (GDP)-overcoated resourcinol formaldehyde (RF) foam-based shell, shown in Fig. 2[7]. However, RF shells are not presently in major production and are extremely fragile, making them not feasible to use for these experiments. PAMS shells were chosen because they are prototypical of a GDP CH-based polymer, even though they are not made of the exact material planned to be used in a production plant. The similarity of sputter-coating adhesion to PAMS and GDP surfaces was tested, and both were found to perform similarly.

A. Optimization of Coating Techniques

A Cressington 108 auto/SE dc magnetron sputter coater was used to deposit a few different metals onto the small plastic spheres for analysis. The sputter coater was equipped with a planetary-rotating stage, shown in Fig. 3, which was tilted at a fixed angle of 25° and rotated at the slowest speed during sputtering. The design was crucial to give very uniform coatings by constantly rotating the shells in and out of the central region of high metal deposition. A small circular plate with an array of 8-mm holes milled 5 mm deep into it was attached to each rotating dish for coating the 4-mm PAMS shells. One shell was placed in each hole to provide a collision-free path to roll during coating, avoiding static attraction to other shells and preventing impacts while coating. This was imperative to keep the shells constantly rolling in random directions during the full coating process to evenly cover the entire surface.

Fig. 3. Planetary-rotating stage with cups for coating shells and tilted to make shells roll when coating.

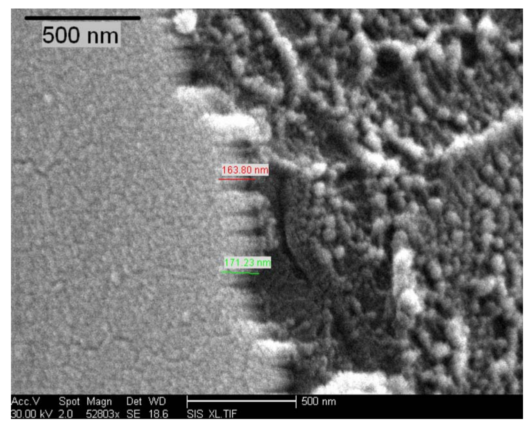

Fig. 4. Example SEM image showing coating thickness measurement technique on coated spherical shells.

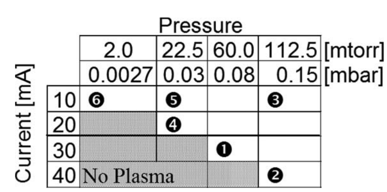

Fig. 5. Diagram identifying the parameter set choices and the region of unavailable coating parameters.

Additionally, a smooth flat polystyrene shim and a glass slide with a piece of tape on it to create a coating step were also sputter coated at the same time to use as witness specimens for examining other properties of the coating. The coating step on the glass slide was used to verify the linear time dependence of coating thickness. It was also used to correlate the coating time required to deposit an 80-nm thick coating on the small rolling spheres by comparing the step thickness, measured with a stylus profilometer, and the cross-sectional coating thickness on the sphere, measured using an SEM. An example of this cross-sectional thickness on a spherical target is shown in Fig. 4.

The shells were meticulously coated using the procedure outlined in the Cressington's user manual [8]. A matrix of coating parameters was selected using Thornton's surface deposition model [9] to test the reasonable extremes of both the sputtering current and argon pressure variables, shown in Fig. 5. Initially, the surface morphology of the coating was improved by lowering the argon backfill pressure during sputter coating. This reduced the intrinsic stress developed during sputtering, allowing it to remain as a stable continuous sheet rather than causing it to grow as many less stable columnar structures. The coating was strengthened by a decrease in grain size from about 100–200 nm to 20–50 nm, depending on the specific parameter set used, and is indicated by the established Hall–Petch equation: $\sigma_y = \sigma_o + k_y/d^{1/2}$[10].

After successfully reducing the grain size of the coating, other innovative coating methods were considered to further strengthen the coating. A thin titanium sublayer was sputter coated onto the PAMS shell before the Au/Pd was coated to promote adhesion between each layer. In addition, a colleague at General Atomics coated GDP on top of the Au/Pd layer, which improved the target's resistance to damage, yet not enough to be acceptable for mass production [11]. The hypothetical process of gold–ion implantation into the PAMS surface was modeled as a possible method to strengthen its bonding with the Au/Pd coating, and that process has hope of improvement [12]. It must be recognized that all works described in this paper was done with shells at room temperature, whereas actual fluidization and handling of IFE targets will be done at cryogenic temperatures of less than 20 K.

III. Characterization Methods and Procedures

In an effort to determine which sputtered coatings were more durable than others, many methods of characterization were used to measure the coating adhesion. There are many aspects of the coating that influence its resistance to damage such as the resistance to tensile, compressive, and shear forces. The morphology of the surface contacting the coated target also plays a large part in the damage resisted by the coating. It is important to recognize that the durability of a coating is partly influenced by its adhesion to the substrate and partly influenced by the shear strength within the coating itself.

The most common and simple coating durability test measured the adhesion of the coating to the substrate with a sticky tape or glue. This was difficult to quantify and perform consistently on 4-mm targets. Another test was devised to test the coating durability in a destructive manner more closely representing the actual types of forces experienced during the fluidization of many targets. A mechanism was developed to simulate the impact of two targets against each other, investigating the combination of compression and shear forces involved. An automatic mechanical "tapper" was designed to consistently impact two sputter-coated targets against each other a specified number of times and is shown in Fig. 6. The speed of the impact was 2.2 m/s $+/-0.5$ m/s, which is characteristic of targets in an actual fluidized bed. The combined mass of the impact mechanism was more, so the tested shells experienced more energy transfer than typical and would be a worst case scenario. An average impact of a target at 2.2 m/s would experience $5^{*}10^{-6}$ J of energy transfer, and the automated impact mechanism imposed roughly ten times more energy than that. That increase was allowed because the test was designed to determine which coatings were more durable than others and not meant to exactly duplicate the impacts inside the fluidized bed. The targets were tapped for a specified number of times and then examined with an SEM microscope, and the damage was quantified using a variety of observable and measurable parameters.

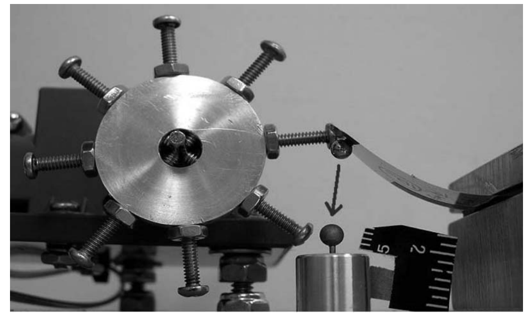

Fig. 6. Picture of automated tapping mechanism.

Fig. 7. Example picture of scratch created by a diamond stylus during nanoscratching.

A more effective test on a much smaller scale is called nanoscratching, which consists of dragging the sharp diamond tip of an atomic force microscope (AFM) stylus across the target's coated surface. This puts a very high stress onto the coating using a small force, so the PAMS shell does not crack or deform excessively. The AFM stylus used had a spherical Rockwell C diamond tip with a 5-μm radius and was drawn across the surface at a constant speed of 0.26 mm/min for a distance of 0.25 mm. The loading was progressively increased from 1 to 25 mN along the length of the scratch, and one sample is shown in Fig. 7. Each coating was scratched three times using the same parameters to produce a statistical average, and the test was performed by a specialized laboratory. It would be impossible for a shell to experience a scratch like this during normal IFE target fluidization, so the actual critical delamination load has no real meaning and is only useful in comparing between the different samples that were tested. This method was used to accurately determine the combined adhesion and shear strengths of various coatings, effectively measuring their durability.

Lastly, the coating composition was measured as a function of depth and found to be consistent through the entire coating thickness, as desired. The coated targets also showed good permeation to deuterium with a time constant of 10 min, only a small increase compared with a time constant of 8 min for an uncoated shell [5]. The coated targets were subjected to gentle handling with a variety of tips on a vacuum chuck, and a hard metal tip was found to badly damage the coating. A silicone tip did not produce any observable damage to the coating. All these characterization methods focused on the three main components of possible damage: compression from impact, shear from sliding during impact, and tension from adhesion during impact. Considering the combination of these three components allows one to understand better how to improve the durability of the metallic coating.

TABLE I
NANOSCRATCHING RESULTS FROM MICROPHOTONICS

Nanoscratching results				
Sample	**Initial Delamination [mN]**	**Std. Dev. [mN]**	**Continuous Delamination [mN]**	**Std. Dev. [mN]**
Au-Pd	8.53	0.68	20.74	2.05
Au-Pd	7.02	0.30	18.81	1.26
Au-Pd then Ti	11.53	0.48	18.51	0.71
Ti	19.90	1.21	32.11	1.21
Ti Then Au-Pd	13.37	0.17	19.53	1.22

IV. EVALUATION OF INITIAL RESULTS

Obtaining clear results from the aforementioned tests and interpreting them correctly were crucial to understanding what improvements could be made to the target coatings. A Veeco optical profilometer was used to measure the rms surface roughness of all the different types of coatings, and all prospective coatings had an rms surface roughness that is less than the 50-nm requirement. This indicates that there were no defects on the surface and verifies that the coating was sputtered smoothly and consistently. The initial adhesion tests did not provide any useful data because the adhesives could not delaminate a coating without damaging the substrate. However, the impact tests were very successful as they showed a clear distinction between poorly adhered and well-adhered coatings. The results illustrated that the coatings with the smaller grains, created by coating with a lower argon pressure, were much stronger than the coatings with larger grains. This also underscores the importance of maintaining clean shells and sputtering dishes to ensure that the sputtered coating adheres well directly onto the PAMS shell surface. The coating with the titanium sublayer showed a very high resistance to impact damage, which was also verified by the nanoscratching tests.

The nanoscratching results confirmed that the smaller grains created stronger coatings, as shown by the impact tests. It also showed that the interface between the PAMS shell and the titanium layer and the interface between the titanium layer and the Au/Pd layer were both stronger than the strongest Au/Pd coating directly on the PAMS surface, as shown in Table I. The damage-resistance results of the coatings using the sublayer are promising for the use of a metallic sublayer to promote the strength of the outer metal reflective coating.

V. ACHIEVEMENTS, CONCLUSION, AND RECOMMENDATIONS

The results from the aforementioned tests led to insightful changes that showed improvements in coating durability for the initial experiments. This work has successfully accomplished the goals of characterizing and improving the outer metallic layer on targets for use in an IFE power plant. Simulated damage tests confirmed that the current production and handling techniques are not scalable to a mass-production facility. Changes in the sputtering parameters were explored and the strength of the coating was optimized by producing smaller grains using a lower argon sputtering pressure. In addition, a very thin sublayer of titanium was sputter coated below the final Au/Pd coating, and the outer coating showed a great improvement in durability compared with a single layer Au/Pd coating. This suggests a possible path for creating a more robust target coating for a mass-production IFE power plant.

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