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GENERAL ATOMICS

# DISCLAIMER

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# REMOVAL OF GDP MANDRELS FROM SPUTTER-COATED BERYLLIUM CAPSULES FOR NIF TARGETS

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*Ablative targets for the National Ignition Campaign (NIC) have been fabricated by sputter coating spherical mandrels made of glow discharge polymer (GDP) with graded copper doped beryllium (Be) layers. The inner mandrel must be completely removed to meet specific ignition design requirements. The process of removing the mandrel requires elevated temperature in the presence of oxygen. However, elevating the temperature in air also oxidizes the Be and can cause blistering on the inner surface of the Be shell. This paper will discuss a refined technique, which removes the GDP mandrel without compromising the integrity of the inner Be surface. The oxygen gradient that develops during the mandrel removal and the impact of its presence will also be discussed.*

## I. INTRODUCTION

The current baseline ignition design for the National Ignition Facility (NIF) is a graded copper doped beryllium ablator.[1,2] These ablators, or shells, are fabricated by sputter coating beryllium onto GDP shells.[3,4] The mandrel is then removed and the inner surface of the beryllium is characterized to confirm that it meets the surface finish requirements. The procedure for mandrel removal has been under development for some time,[5] but the protocol has evolved with our understanding of the mandrel removal process.

Removal of the glow discharge polymer from the ablator occurs when the CH-based GDP mandrel reacts with oxygen at elevated temperatures. The specific reaction that occurs is temperature dependant. Combustion of the GDP ($CH_{1.3}$) forms $CO_2$ and $H_2O$ at temperatures above 450°C. When the temperature is below 450°C, the polymer decomposes and forms a variety of intermediate organic compounds, which then evaporate.[6]

A small hole, typically between 5 and 10 μm in diameter, is laser drilled[7] through the Be ablator and the GDP mandrel through which the gases can flow. The pressure is cycled to force the gases to circulate in and out of the shell. It is well documented that when beryllium is exposed to heat and oxygen, a combination of stress and gas diffusion can cause blisters to form on beryllium surfaces.[8,9] The temperature protocol has been modified to provide enough heat energy and oxygen for the reaction of GDP without compromising the integrity of the inner beryllium surface. To meet the NIF residual contaminant specifications, the method must be further refined to decrease the oxygen contamination that occurs during the mandrel removal process.

## II. EXPERIMENTAL

The GDP mandrels were fabricated using previously developed methods described elsewhere.[10,11] The beryllium sputter coatings were done in one of four separate sputter deposition systems, all of which are routinely used for development of Be shells.[3,4]

Two furnace systems were used to conduct these experiments. The first system was designed when the mandrel removal experimental work began.[5] It was specifically developed to maximize the gas flow through a 5 μm hole. This furnace housed a tubular pressure vessel into which the samples were inserted. Filtered and dried air was pressure cycled through the vessel from ambient pressure for 30 seconds, then to four atmospheres for 30 seconds. This pattern was repeated for 60 hours as the mandrel reacted with the air and was removed. The second system was a standard muffle furnace that ran the specified temperature protocols in air without pressure cycling. This standard muffle furnace was later modified to have the ability to cycle the pressure.

To verify that the mandrel had been removed, contact film radiography was used. The shells were then fractured and the inner surface was inspected using an Hitachi S-800 scanning electron microscope (SEM). It was necessary to look at a large area of the inner Be surface to verify that there were not isolated defects. The curvature of the shell made it impossible to view an acceptable sized area under an AFM microscope system as the AFM does not have the capability of moving up and down enough to accommodate the curvature of the shell. Interferometry was, therefore. used to generate RMS data on the inner surface. A phase shifting diffraction interferometer (Spherical Interferometer) system capable of providing data over 300 $\mu m^2$ has been developed[12] and was used to characterize these parts. Thermogravimetric analysis and quantitative radiography[13-15] methods were also used to study the decomposition of the plasma polymer mandrels.

## III. TEMPERATURE STUDIES

Previous work on mandrel removal and capsule characterization used the first pressure cycling furnace described above and a treatment profile that consisted of a temperature ramp from ambient to 200°C at a rate of 5°C/min. The temperature ramp then slowed to 1°C/min to a final temperature of 500°C. The system was set to dwell at this temperature for 60 hours before cooling to ambient temperature as the furnace turned off. The inner surfaces of the Be shells that underwent this process were inspected, and what was believed to be some type of residue was observed.[5]

The initial thought was that the residue was caused by incomplete mandrel removal. To expedite the analysis, shells were quartered and treated in a variety of different environments. The furnace that did not cycle the pressure was used because the pressure cycling was intended to assist with flow through a small hole and it was thought to be unnecessary with the open-faced sections. Subsequent visual microscopy from shells processed in both stagnant and pressure cycled environments showed an increase in the amount of residue. Characterization with low angle SEM [Fig. 1(a)] images revealed that the residue was actually blistering of the beryllium. First, it was necessary to resolve if the blistering was caused by an interaction between the Be and the GDP, so the same analysis was performed using a bare beryllium sample in the absence of GDP. The bare beryllium also blistered [Fig. 1(b)].

Next, it was critical to understand the temperature at which Be blistered as well as the temperature necessary to facilitate the removal of the GDP. To study the effects of temperature on sputtered Be, the inner surfaces of many beryllium ablators were inspected after the mandrel was removed at various temperatures. Additionally, Be was sputtered onto mandrels of poly(α-methylstyrene) (PαMS) which can be removed from the Be with solvent. The surface of the Be after the PαMS mandrel was removed with solvent was compared to the surface of the samples which were heat treated to remove the GDP. As shown in Fig. 2, the roughness of the inner surface increased with the temperature used to remove the mandrel. At 400°C there was no discernible difference between the bare Be from the PαMS method and the heat treated Be. To verify the minimum temperature necessary to remove the mandrel, samples of plain GDP were also analyzed using a thermogravimetric analysis system (TGA). This system can measure the mass of samples held at a constant temperature as a function of time. Results for GDP samples held at three different temperatures are plotted in Fig. 3. Mass loss occurs as the mandrel decomposes and is

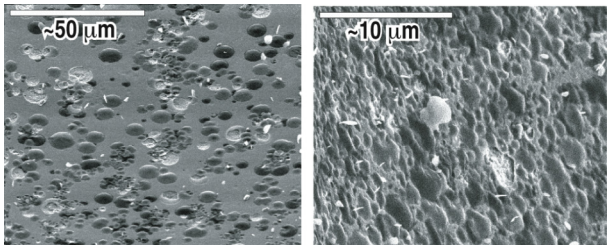


Figure 1. (a) Be on GDP mandrel to 500°C. (b) Bare Be to 500°C. Although the blisters appear differently on the unattached sample, there are clearly blisters present.

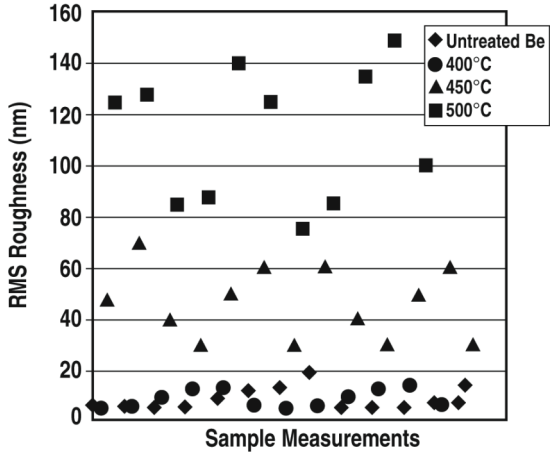


Figure 2. Surface interferometry verified that roughness increased with removal temperature.

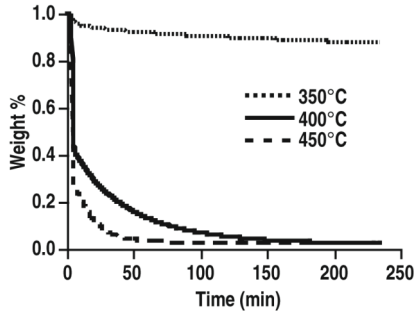


Figure 3. TGA data showed that at 400°C there is complete removal of the GDP mandrel.

reflected as a decrease in the weight percent. The data confirmed that uncoated GDP mandrels were not completely removed at temperatures less than 400°C. In an attempt to break down the GDP through other mechanisms, the mandrels were heated in the presence of inert gases, but without oxygen large amounts of GDP residue remained in the TGA.

Based on the results of the TGA work, the varied temperature tests on beryllium ablators, and the blistering tests on pure beryllium, the following temperature protocol was implemented. To remove the mandrel without inducing blisters, the samples were brought from ambient to 200°C at a rate of 5°C/min the dwell temperature was decreased to 400°C for 60 hours. Both the pressure cycling and the standard muffle furnace described in Sec. II were used for these experiments.

## IV. SHELL RESULTS

After establishing a protocol on shards, the work continued on shells with 5-10 μm holes. Contact radiographs showed incomplete mandrel removal when temperatures were very near 400°C. When the temperature was increased to 425°C there was no residual mandrel evident when the pressure cycling system was used. When shells were treated in the standard muffle furnace without pressure cycling, small amounts of residue were seen upon inspection with contact radiography, as shown in Fig. 4.

It was then verified that at 425°C the inner surface of the Be, when treated as a shell and not a shard, did not blister. To characterize the blistering, the shell was fractured and analyzed with SEM images. In addition to qualitative SEM images, RMS roughness measurements provided a quantitative analysis of the surface. The phase shifting interferometer was used to characterize the shards [Fig. 5(a)]. The region monitored by this method finds the isolated defects that can occur infrequently and be missed in small patch scans, which

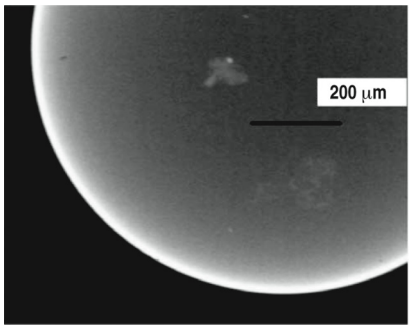


Figure 4. Contact radiography image of residue in Be shell (in boxed area) after mandrel removal.

are the measurements generally seen with atomic force and interference microscopy. The power spectrum, which describes how the amplitude of the roughness is distributed with frequency, is shown in Fig. 5(b). These spectra are from a shell that underwent the new GDP mandrel removal method, and it closely matches the NIF specification.

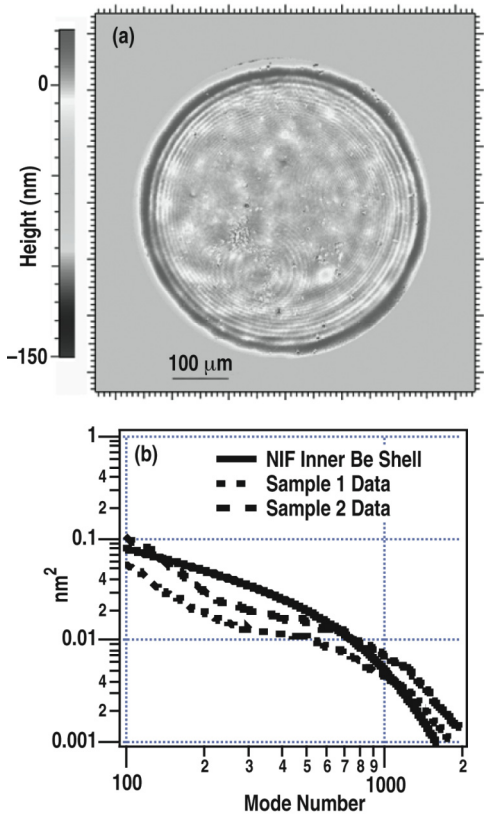


Figure 5. (a) Image of inner topography of ablator. (b) Power spectrum of inner ablator surface in the region defined by the NIF specifications. Spherical interferometry of the inner surface of shards from a Be capsule treated at 400°C shows a close match to the NIF specifications.

After the successful removal of the mandrel, quantitative radiography was used to characterize the residual impurities in the shell. Oxygen uptake that exceeds the allowable level of impurities in the NIF specifications was measured. The oxygen contamination increased from low levels (under 1 atom%) on the shell's interior surface to high values (near 5 atom%) on the exterior layer. This uptake of oxygen poses a significant challenge in production of Be shells to meet the current NIF specifications.[1,2] Experiments are underway to further understand the oxygen uptake mechanism and solve this problem. A possible route using Be mandrels is under investigation.[16]

## V. CONCLUSION

A method has been developed that completely removes the GDP mandrel from the full thickness (about 165 μm) beryllium ablator through a 5-7 μm hole while maintaining an inner surface finish that meets the requirements of the NIF point design. Mandrel removal at too high a temperature leads to oxide formation and blistering of the inner beryllium surface. The blistering had previously been attributed to carbon residue from incomplete mandrel removal. The optimum temperature for mandrel removal is 425°C, where the GDP mandrels are completely combusted with minimal oxidation of beryllium. Mandrel removal studies are continuing to determine the reproducibility of this process.

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