

GA-A25651

# **INVESTIGATIONS TO REMOVE DOMES FROM PLASTIC SHELLS BY POLISHING**

by

**D.G. CZECHOWICZ, C.J. CHEN, J.A. DORMAN, and D.A. STEINMAN**

**JANUARY 2007**



## **DISCLAIMER**

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

# INVESTIGATIONS TO REMOVE DOMES FROM PLASTIC SHELLS BY POLISHING

by  
D.G. CZECHOWICZ, C.J. CHEN,<sup>\*</sup> J.A. DORMAN,<sup>†</sup> and D.A. STEINMAN

This is a preprint of a paper presented at the 17th Target Fabrication Specialist Meeting, San Diego, California on October 1-5, 2006 and to be published in *Fusion Science and Technology*.

<sup>\*</sup>University of California-San Diego, La Jolla, California.

<sup>†</sup>University of California-Los Angeles, Los Angeles, California.

Work supported by  
the U.S. Department of Energy  
under DE-AC03-01SF22260, DE-AC52-06NA27279, UCSD and UCLA

GENERAL ATOMICS PROJECT 30272  
JANUARY 2007



## INVESTIGATIONS TO REMOVE DOMES FROM PLASTIC SHELLS BY POLISHING

D.G. Czechowicz,<sup>1</sup> C.J. Chen,<sup>2</sup> J.A. Dorman,<sup>3</sup> and D.A. Steinman<sup>1</sup><sup>1</sup>General Atomics, P.O. Box 85608, San Diego, California 92186-5608<sup>2</sup>University of California, San Diego, 9500 Gilman Drive, La Jolla, California 92093<sup>3</sup>University of California, Los Angeles, 405 Hilgard Avenue, Los Angeles, California 90095  
czecho@gat.com

*A problem often observed for thick wall plastic targets is the presence of surface domes. We have been successful in applying mechanical polishing to remove isolated surface domes from thick wall 2 mm shells during a preliminary investigation. The background surface roughness for polished shells was dramatically improved with final values typically around 10 nm RMS as measured by WYKO patch surface profiles. The polishing sequence applied was also examined using AFM spheremapper data that was obtained for shells after each polishing step. A two-step polishing approach was able to produce shells that had significant improvement in all AFM power modes except for modes (3–10). Further polishing development is needed to reduce AFM low and mid power modes for shells. Polishing of otherwise target quality 2 mm shells that have domes could be a future treatment for NIF targets.*

## I. INTRODUCTION

A problem that often exists particularly for ICF thick (>30  $\mu\text{m}$ ) wall plastic targets such as National Ignition Facility (NIF) targets, is the presence of surface features which include domes.<sup>1–4</sup> Surface features are a general class of defects that can be either scratches, ridges, divots, or domes. A dome is typically the most common surface defect and can be formed either by protrusions on mandrel surfaces or by seed particles that are incorporated into coatings.<sup>5</sup> Letts and coworkers studied ways to help prevent formation of domes from the coating process and developed a model for dome growth.<sup>6</sup> Surface domes produced on thick wall plastic coatings have flattened hemisphere shapes and a diameter to height ratio of approximately 10 to 1.<sup>2,6</sup>

In the work described in this paper, we investigate a new way to remove surface domes from plastic shells once they are formed. Plasma etching employing oxygen, argon, and hydrogen as etching gases to improve the surface of plastic shells while removing domes has been

reported.<sup>7</sup> Recently, workers have reported that adding helium or hydrogen pulses during long coating can be successful in eliminating aggregates that are seeds for growing domes.<sup>2</sup> Removal of seed particles helps prevent dome formation. The approach taken, in this paper, is the study of the removal of surface domes once they are formed on plastic shells by polishing. Since domes are protrusions that can be micrometers in height on thick wall shells, fine mechanical polishing appeared to be a means to remove domes that formed on plastic shells.

There are many accounts in the literature of the use of chemical-mechanical polishing to decrease the surface roughness of polymers.<sup>8–11</sup> In these accounts polymer surface roughness for flat samples below 10 nm root mean square (RMS) using surface profile analysis was achieved for nominal 100  $\mu\text{m}$  x 100  $\mu\text{m}$  sample patches after using versions of a counter-rotating polisher. In addition, the use of a counter-rotating lap polisher was used to improve the surface finish of 2.0 mm diameter beryllium capsules which are candidates for NIF ignition targets.<sup>12</sup> With this knowledge we built a modified counter-rotating lap polisher that we used to mechanically polish 2.0 mm diameter thick wall plastic shells to determine if isolated domes present on shells could be removed while achieving low surface roughness.

## II. EXPERIMENTAL

A counter-rotating lap polisher was used in our work to remove domes from plastic shells similar in design to the device used for polishing beryllium capsules.<sup>12</sup> Figure 1 presents a simplified schematic of the lap polisher in the case for one shell being polished. Figure 2(a) shows how multiple shells can be held during polishing using a modified teflon holder. Plastic 2 mm diameter Ge doped GDP shells were the main samples used in our polishing studies since these shells were readily available and had thick walls (100–150  $\mu\text{m}$ ) with a rich amount of surface domes. Occasionally we used smooth shells that had few domes or smooth Si beads to show that polishing did not

worsen the surface for already smooth samples. Polishing occurs on two counter-rotating plates; a bottom plate on which the shell rests, and a top plate that polishes the shell using a side surface. Typical polishing speeds for the counter-rotating plates were 25–50 rpm. Polishing run times varied from a few hours to two days. Material was removed by using 1.0 and 0.1  $\mu\text{m}$  diamond grit paper. Lapping oil lubricant was added to the shells during the polishing procedure. A 2 mm titanium sphere was often placed on top of the GDP shell as a weight to ensure that the GDP shell remained in contact with the bottom grit paper throughout duration of polishing runs. Figure 2(b) shows a cross-section of the multiple shell holder and how the Ti sphere held the GDP shell down during polishing. Upon completion of a polishing run, lapping oil was removed from the shell by placing it in hexane for 5 min in an ultra-sonic cleaner. A final cleaning of the polished shell was then preformed for 5 min in an ultra-sonic cleaner using 0.5  $\mu\text{m}$  filtered ethanol.

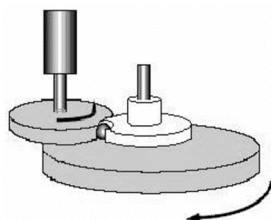


Figure 1. Simplified schematic of counter-rotating plate lap polisher showing one shell being polished.

Well identified isolated domes were often measured for height and diameter along with background surface roughness before and after polishing runs using a Veeco Instruments WYKO Surface Profiler. Shell diameters were measured by interferometry and shell weights using a Cahn Microbalance before and after polishing runs. Spheremapper AFM data was also obtained on certain shells before and after polishing runs.

### III. RESULTS

#### III.A. Removal of Isolated Dome

One of the first tests we carried out using our polishing apparatus was to try to remove a large identifiable dome from a 2 mm Ge doped shell. Figure 3(a) presents the results of this test in the form of 3-dimensional WYKO surface profiles. Figure 3(b) presents the polishing test results graphically. Figure 3(a) (A) shows the large starting dome or dome cluster that was measured to be 5.0  $\mu\text{m}$  in height and approximately 100  $\mu\text{m}$  in diameter. Figure 3(a) (B) shows the large dome after a first 4 h polish using 1.0  $\mu\text{m}$  grit paper. The dome

height decreased from 5.0 to 4.5  $\mu\text{m}$ , however the diameter did not change appreciably. Figure 3(a) (C) shows the large dome to decrease to 1.4  $\mu\text{m}$  in height with little change in diameter after a 16 h polish using 1.0  $\mu\text{m}$  grit paper. A last 45 h polish using 0.1  $\mu\text{m}$  grit paper, Fig. 3(a) (D), shows the removal of the entire dome. For successive polishing runs; (B) and (C) during this test, the background surface roughness was observed to gradually increase from the original roughness value of 35 to 44, then to 57 nm RMS. When the last polishing run (D) was carried out using 0.1  $\mu\text{m}$  grit paper the background roughness was decreased to a value of 8 nm RMS.

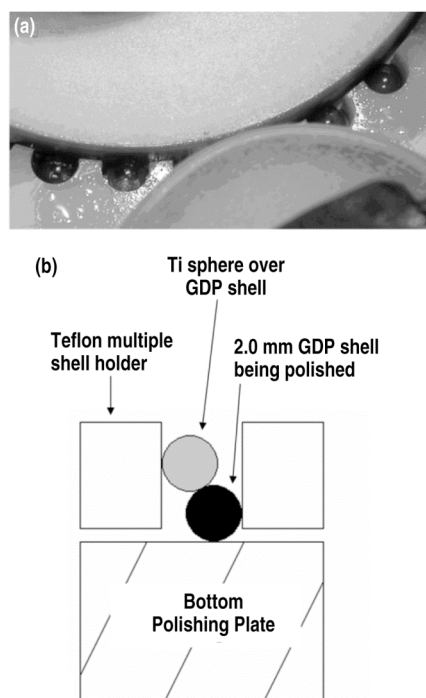


Figure 2. (a) Photo of multiple shells in modified shell holder used during polishing. (b) Cross-section of multiple shell holder showing how GDP shell is held with Ti sphere above it in shell holder during polishing.

#### III.B. Decreasing Background Roughness for Shells

Another test was carried out which focused on decreasing surface roughness for a 2.0 mm shell that had many small domes and a background surface roughness of 75 nm RMS. Figure 4 presents results from this test. After the first polishing run using 1.0  $\mu\text{m}$  grit paper for 16 h the domes were decreased in height as observed in Fig. 4, however the WYKO 3-dimensional surface appeared to have scratches. The background surface roughness was 49 nm RMS after the first polishing run. The second polishing run then used 0.1  $\mu\text{m}$  grit paper for 45 h and produced a background surface roughness of 11 nm RMS.

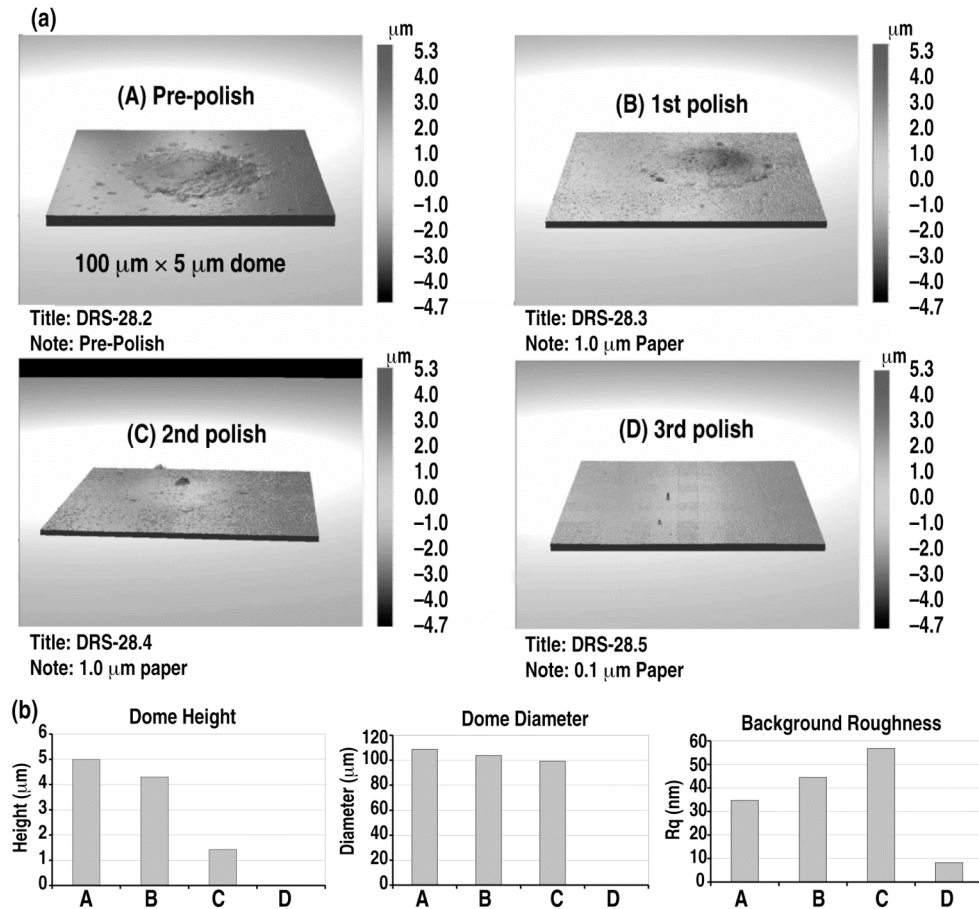


Figure 3. (a) WYKO surface profile data showing removal of identifiable dome from shell surface after three polishing runs. (b) Characterization values for dome height, dome diameter, and background roughness during removal of identifiable dome from shell surface.

### III.C. AFM Spheremapper Data for Polished Shells

Next a study was performed which examined changes in AFM spheremapper data as a 2.0 mm plastic shell was polished in successive polishing runs. Figures 5 and 6 presents the AFM power spectrum data and shell profiles from this polishing study. The first polishing run in this study used 1.0  $\mu\text{m}$  grit paper for 24 h. The second polishing run used 0.1  $\mu\text{m}$  grit paper for 19 h, and the third polishing run used 0.1  $\mu\text{m}$  grit paper for 16 h. In Fig. 5, we observe no improvement in the power spectra data after the first polish. After the second polishing run, there is an observed dramatic improvement in the power spectra, especially for modes greater than  $\sim 50$ . The third polishing run shows no further improvement and an actual increase in values for modes less than 10.

The spheremapper profiles in Fig. 6 indicate that fine structure or small domes are decreased in height after the first polishing run. However there is still a longer scale roughness and an underlying rippling or wrinkling

observed in the profiles after the first polish. After the second polish the profiles indicate a much smoother surface with very few isolated features. Finally after the third polish there is a long range waviness or wrinkling that is evident from the profile data.

Figure 7 presents a plot showing how the power modes change as a function of polishing run time based on the power spectra data given in Fig. 5. What is observed is that the first 24 h polishing run was minimally effective in decreasing all power modes. The second polishing run however was very effective in decreasing all mode values except modes (3–10). After the third polishing run, mid and high modes remained at low values, however modes less than 10 increased dramatically.

Out of curiosity we carried out one long polishing run for 45 h directly on a 2.0 mm shell that had a large amount of small ( $\sim 10 \mu\text{m}$  diameter) domes. In this polishing run we used 0.1  $\mu\text{m}$  grit paper. The resulting AFM power spectra is presented in Fig. 8. What was

observed is that after the long 45 h polishing run modes less than 10 had fairly low power values, with mode (2) = 28 nm RMS and modes (3–10) = 24 nm RMS.

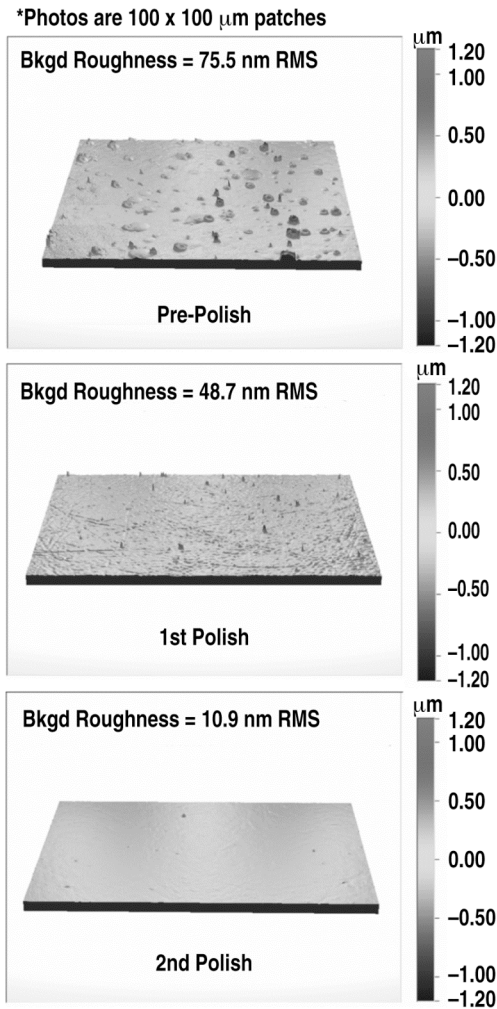


Figure 4. WYKO surface profile data showing decrease in background surface roughness after successive polishing runs.

## IV. DISCUSSION

### IV.A. Removal of Isolated Domes and Decreasing Surface Roughness

From the initial polishing tests carried out, the results of which are summarized in Figs. 3 and 4, we demonstrated that mechanical polishing of thick wall 2.0 mm shells can be effective in removing isolated domes. For shells with large domes or surfaces having many isolated domes we chose to use a coarse (1.0  $\mu\text{m}$  grit) polishing paper to first decrease the height of the domes present. We followed this initial polishing with the use of finer

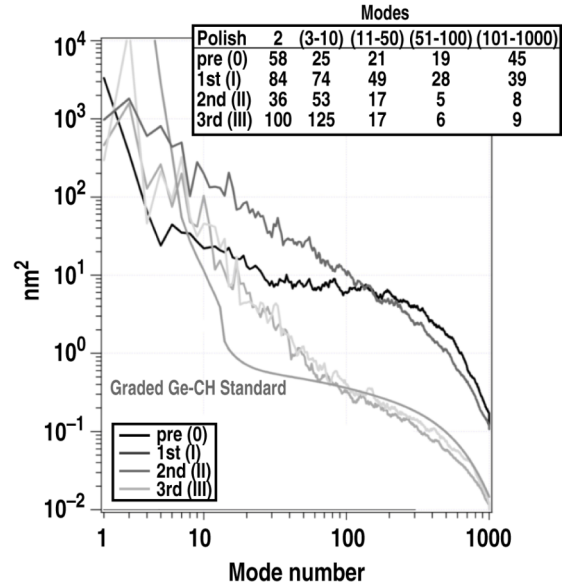


Figure 5. AFM power spectrum for same 2 mm shell before and after three successive polishing runs.

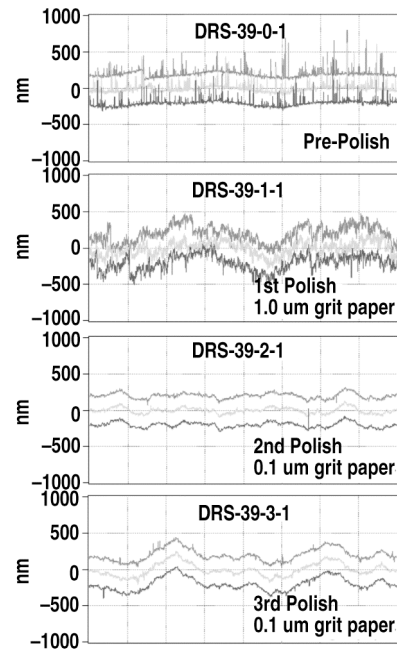


Figure 6. AFM spheremapper profiles for same 2 mm shell before and after three successive polishing runs.

(0.1  $\mu\text{m}$  grit) paper to improve the background surface roughness for the shells. This approach appeared to work as indicated in Figs. 3 and 4. The final background surface roughness, which in our work was  $\approx 10$  nm RMS as analyzed by WYKO 20  $\mu\text{m}$  x 20  $\mu\text{m}$  surface patches, is similar to the lowest surface roughness obtained for high quality thick wall 2.0 mm plastic shells that are being

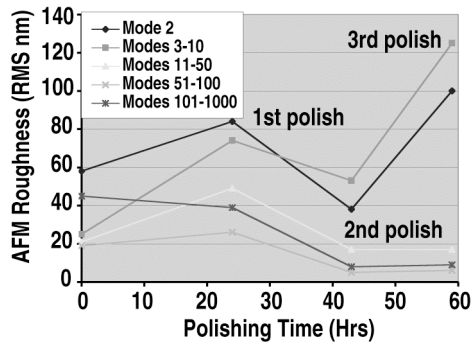


Figure 7. Changes observed in power modes as a function of polishing time for the same 2 mm shell.

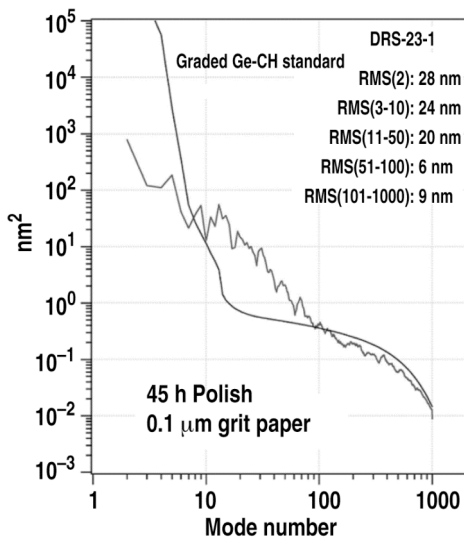


Figure 8. AFM power spectrum for 2 mm shell after long 45 h polishing run using 0.1  $\mu\text{m}$  grit paper.

developed for NIF Ignition Targets.<sup>13</sup> There exists 0.05  $\mu\text{m}$  grit polishing paper that could be used to attempt to further decrease surface roughness for polished shells.

One major concern we had during our initial polishing tests was if contaminants were being incorporated into the plastic shell wall from the polishing process. Based on optical microscopy and SEM analysis no signs of carbon from the diamond grit paper were observed incorporated into polished shells. Titanium spheres often used as weights on top of shells to assist in the polishing process were another potential source of contamination. These spheres could contact the upper polishing surface of our polishing device. Examination of polished shells by x-ray fluorescence and SEM EDAX analysis for titanium showed no titanium present in shells before or after polishing runs. The use of ultrasonic cleaning using solvents could have only helped remove contaminants from shell walls if they had been present.

#### IV.B. AFM Spheremapper Analysis of Polished Shells

Results from spheremapper data presented in Figs. 5 through 7 indicate that polishing of plastic shells having domes can be a very effective way of improving the power spectrum for shells, particularly in the high mode region (modes  $> 50$ ). In our study, the first polish using 1.0  $\mu\text{m}$  grit paper appears to have decreased the height of the original  $\sim 0.5 \mu\text{m}$  tall domes present while scratching or roughening up the underlying surface based on the spheremapper profiles. The first polish result also appears to have a longer-range waviness that suggests wrinkling of the surface. The second polish using 0.1  $\mu\text{m}$  grit paper was very effective in smoothing the surface roughness (modes  $> 100$ ), in addition to lowering all other modes except modes (3–10) below their original values. However the profiles suggest that the second polish may have produced some surface wrinkling based on their wavy shape. The third polishing run using 0.1  $\mu\text{m}$  grit paper did not improve or worsen any of the mid and high modes (modes  $> 10$ ), however low modes (modes  $\leq 10$ ) were significantly worsened (Fig. 7). The spheremapper profiles after the third polishing run indicate that an exaggerated wavy or wrinkled surface was produced on the shell.

In this study it would have been best to stop polishing after the second polishing run when low modes (modes  $< 8$ ) and high modes (modes  $> 50$ ) were below the NIF Ge doped GDP standard curve (Fig. 5). The spheremapper data suggests that polishing using a counter-rotating lap polisher as we currently use it, may not be effective in decreasing low and mid modes. The challenge of decreasing mid modes was also observed during polishing of beryllium capsules.<sup>12</sup> The reason mid modes may pose a problem with our current polisher is because our polisher does not work like a lathe. With a lathe you have a fixed piece that is rotated around a precise axis while removing material. This produces a uniform radius. In our polisher the shells tumble freely in somewhat random motion while contacting the polishing surfaces. This shell motion against the polishing paper can smooth surfaces by removing domes as our data has indicated. However our polishing technique does not seem to be able to correct longer-range non-uniformities for shells if these are present prior to polishing.

The AFM power spectra results presented in Fig. 8 are encouraging. We did not obtain relative before and after polishing AFM data for the shell whose data is shown in Fig. 8. However the absolute RMS values for the various modes are quite low after a long 45 h polishing run using 0.1  $\mu\text{m}$  grit paper. The results indicate that mode (2), modes (3–10), and modes (11–50) remained fairly low with values of 28, 24, and 20 nm RMS respectively after the 45 h polish. This is an



indication that polishing does not always produce high values for mid and particularly low modes as was observed in the polishing data of Fig. 5.

## V. SUMMARY/CONCLUSION

We have successfully used a counter-rotating lap polisher to remove isolated domes from thick wall plastic shells. Along with the removal of domes we were able to consistently achieve improved surface roughness for shells. Final background surface roughness values for polished shells were approximately 10 nm RMS from WYKO analysis, similar to values obtained for the best quality NIF development shells. AFM spheremapper data for 2.0 mm shells having a rich amount of domes indicated dramatic improvement in power spectra after polishing. Typically after polishing all AFM power mode values were lowered except modes (3–10). As our polishing device is currently used it does not necessarily improve problems with mid and low modes. Further study is needed to reduce AFM low and mid power modes for shells that are polished.

We present a preliminary investigation. Contaminants did not appear to be a problem during this work, however further polishing development must be vigilant of problems such as contaminant incorporation to shells from polishing. SEM and microprobe analysis should be used in the future to continually check for contaminants. The size or quantity of domes present appears to only affect the polishing time required to obtain polished dome-free shells. It is believed that plastic shells having good long-range uniformity (low values for low and mid AFM power modes) will have final good long-range uniformity after polishing. Future studies are planned to determine if this is true. Optimization of low modes for shells having long-range non-uniformity will require a significant redesign of our current polishing apparatus.

Since polishing has been shown to be effective in removing isolated domes from thick wall plastic shells, polishing may be a future treatment for otherwise target quality 2 mm NIF shells that have domes.

## ACKNOWLEDGMENTS

Work supported by the U.S. Department of Energy under DE-AC03-01SF22260 and DE-AC52-06NA27279. The authors acknowledge General Atomics staff members, J. Gibson and T. Lee for providing timely AFM spheremap data, E. Castillo for assistance during construction of the counter-rotating lap polisher, and K.C. Chen for providing 2 mm plastic shells. The authors also thank T. Bernat and K.C. Chen for helpful discussions.

## REFERENCES

1. K. C. CHEN, *et al.*, "Fabrication of Graded Germanium-Doped CH Shells," *Fusion Sci. Technol.* **49**, 751 (2006).
2. M. THEOBALD, *et al.*, "Gas Etching to Obtain Germanium Doped CH<sub>x</sub> Microshells Compatible with the Laser Megajoule Target Specifications," *Fusion Sci. Technol.* **49**, May 2006, p.757.
3. K. C. CHEN *et al.*, "Reduction of Isolated Defects on Ge Doped CH Capsules to Below Ignition Specifications," *Fusion Sci. Technol.*, this issue.
4. S. W. HAAN, *et al.*, "Update on Specifications for NIF Ignition Targets, and Their Rollup into an Error Budget," *Fusion Sci. Technol.*, this issue.
5. A. NIKROO and D. M. WOODHOUSE, "Bounce Coating Induced Domes on Glow Discharge Polymer Coated Shells," *Fusion Sci. Technol.* **35**, 202 (1999).
6. S. A. LETTS, D. W. MEYERS, and L. A. WITT, "Ultrasooth Plasma Polymerized Coatings for Laser Fusion Targets," *J. Vac. Sci. Technol.* **19**, 739 (1981).
7. R. L. CRAWLEY, "Surface Improvement and Thickness Adjustment by Plasma Etching," KMS Fusion, Inc. 1988 Annual Technical Report on Inertial Fusion Research, p. 58, (1988).
8. A. A. YASSEEN, *et al.*, "Chemical-Mechanical Polishing for Polysilicon Surface Micromachining," *J. Electrochem. Soc.* **144**, 237 (1997).
9. G. NANZ and L. CAMILLETTI, "Modeling of Chemical-Mechanical Polishing: A Review," *IEEE Transactions on Semiconductor Manufacturing* **8**, 382 (1995).
10. G.-R. YANG, *et al.*, "Chemical-Mechanical Polishing of Parylene N and Benzocyclobutene Films," *J. Electrochem. Soc.* **144**, 3249 (1997).
11. C. BORST, *et al.*, "Chemical-Mechanical Planarization of Low-k Polymers for Advanced IC Structures," *Transactions of the ASME* **124**, 362 (2002).
12. M. L. HOPPE and E. R. CASTILLO, "Polishing of Beryllium Capsules to Meet NIF Specifications," *J. Phys. IV France* **133**, 895 (2005).
13. K. C. CHEN, private communication (2006).