Decomposable Mandrel Project - Progress Report

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Summary

We report on our progress in developing a new technology to produce both Nova and NIF scale capsules using a depolymerizable mandrel. In this technique we use poly(α -methylstyrene) (PAMS) beads or shells as mandrels which are overcoated with plasma polymer. The poly(α -methylstyrene) mandrel is then thermally depolymerized to gas phase monomer which diffuses away through the more thermally stable plasma polymer coating, leaving a hollow shell. Since our last report we have concentrated on characterization of the final shell. Starting with PAMS bead mandrels leads to distorted pyrolyzed shells because of thermally induced creep of the CH coating. We found that plasma polymer coatings on hollow shell mandrels shrink isotropically during pyrolysis and maintain sphericity. We are now concentrating our efforts on the use of microencapsulated shells to prepare targets with buried diagnostic layers or inner wall surface texture.

Introduction

The purpose of this project is to develop the capability of producing both Nova and NIF scale capsules using a new technology based on the decomposition of poly(α -methylstyrene) (PAMS) mandrels at temperatures of about 300°C. There are two areas where this technology may have a major impact. First, it represents a route to large capsules and thus is of importance for both Omega and NIF. Second, this technology can impact the HEP-4 program by providing the possibility of producing Nova scale capsules with unique diagnostic or structural features, such as very thin pure metal layers buried near the inside capsule surface or by providing a route to capsules with designed inner surface roughness.

This progress report updates our previous report (TST 94-068, 5/12/94). In the remainder of this section we will briefly review where we were then, summarize our progress to date, and outline the problems yet to be solved. Technical details of the work are included in the sections that follow.

The basic method, shown in Fig. 1, can be summarized as follows. A quantity of PAMS is formed into a hollow or solid sphere of the desired size and surface finish. It is then overcoated with plasma polymer to the desired thickness and placed in an oven and heated to about 300 °C. This causes the PAMS to depolymerize to gaseous monomer which diffuses through the plasma polymer coating leaving a hollow shell.

MASTER

Figure 1. Hollow shells can be produced by starting with a poly(α -methylstyrene) bead, then overcoating with thermally more stable plasma polymer. The coated bead is pyrolyzed at 300 °C for 30 hours or more, leaving a hollow shell.

In our previous progress report we detailed our methods of forming spherical bead mandrels and also reported some preliminary results on shells that had been produced by pyrolyzing the beads after coating with plasma polymer. Virtually all of this work used commercially available 11K molecular weight (M.W.) PAMS. One advantage of this material was that it had a low softening point (~ 60 °C) which was very useful for our hot water treatment to form spherical beads. We have subsequently found, however, that bubbles form in beads made from this material when they are plasma polymer overcoated due to the slightly elevated coater chamber temperature. This has led to coating difficulties for larger beads, evidence of coating and subsequent shell asymmetry, and considerable difficulty with shell collapse during the PAMS bead decomposition process. We have now largely solved this problem by using 19K molecular weight material, but a significant effort was necessary to diagnose the problems and determine a solution. In general we continue to have some problems with the decomposition of the larger bead mandrels, especially maintaining sphericity through pyrolysis.

We have done a number of experiments with PAMS shells produced at LLE by microencapsulation techniques from monodisperse 97K molecular weight PAMS. The size range of LLE shells was from 500 to 800 μm . Use of PAMS shell mandrels is preferable since there is much less material to decompose and the polymer is in principle much cleaner. We generally found that mandrel replication was excellent with no obvious mode 2 defect. However we have found that when the LLE supplied microshells were overcoated with plasma polymer, the surface finish showed considerable roughness, suggesting that the PAMS microshells had a fair amount of surface debris. The coated shell mandrels pyrolyzed with much less sphericity distortion than the bead mandrels. If the surface problem can be solved we feel that the microencapsulated PAMS shell mandrel is the best option. To facilitate our progress in developing this route, GA will be providing us with high quality microencapsulated PAMS microshells.

By next October it is our expectation to bring the R&D aspects of this program to a close and begin to use it as a production tool as required. We will also transfer the technology to the GA Target Fabrication Contract for use in vacuole free microshell fabrication for LLE if other routes to these shells are not successful. For current Nova HEP-4 experiments we will attempt to produce shells with controllable inner surface roughness by late summer. If desirable we will also attempt to prepare shells with thin interior metal layers. A variant of the method will continue to be used to prepare large hollow shells for cryogenic layering experiments.

The remainder of this report provides detailed documentation of the last years work in the areas of bead and shell mandrel preparation; plasma polymer overcoating work; and pyrolysis results, including most of our final shell characterization data. The final section outlines planned activities for the remainder of the fiscal year.

PREPARATION OF PAMS MANDRELS

Preparation of the mandrel is the first step in the process of shell production. Initially we selected the most spherical pellets from commercial material. Later we learned how to control size and improve sphericity using hot water treatment. In the last year we found several problems with the 11K M.W. material that we had been using which caused us to reexamine the entire process. When we discovered that the beads were deforming during CH coating, the molecular weight of the PAMS was increased to 19K to raise the softening temperature. Later we found that deformation of the CH coating during pyrolysis was caused by the bead weight and the low molecular composition of the commercial PAMS material. This lead us to use microencapsulated shells as mandrels made from monodisperse high molecular weight PAMS. As we will show, most of the pyrolysis problems are eliminated when high molecular weight PAMS shells are used as mandrels.

Molecular Weight Analysis of PAMS

The molecular weight distribution of the 11K and 19K PAMS material was analyzed by gel permeation chromatography (GPC) to better understand its thermal behavior during pyrolysis. The manufacturer's and our own initial analysis of molecular weight showed both materials to be bimodal in molecular weight distribution. We then examined the PAMS polymers using a special low molecular weight column. Fig. 2 shows the chromatogram of the 11K and 19K PAMS materials. The high molecular weight peak is at about 28K for both polymer samples but the proportion is higher in the 19K material. Both polymer samples contain a large fraction of low molecular weight oligomer. We now realize the oligomer content plasticizes the material and allows it to soften at lower temperatures, an important aspect of the bead mandrel making process.

The low molecular weight oligomers can be largely removed by reprecipitation with methanol from toluene solution. The reprecipitated material, however, does not soften at temperatures up to 150 °C. This means that preparation of beads from this purified material in hot water would not be possible.

Figure 2. GPC analysis of 11K and 19K M.W. PAMS shows that they are multi-modal in composition. The 19K material differs only in having slightly more of the higher molecular weight material.

Thermal Properties of PAMS

The thermal decomposition behavior of 11K and 19K M.W. PAMS used to prepare bead mandrels and the monodisperse 97K material used to make shells is shown in Fig. 3. We also show plasma polymer and plasma parylene overcoating materials. Thermal gravimetric analysis (TGA) was performed on all the samples at a constant heating rate mode (5 °C/min.).

The shoulder in the thermogram starting at 150 °C and extending to 300 °C in both the 11K and 19K M.W. PAMS we believe is due to volatilization of the oligomer material. Thus, initial slow heating to pyrolysis conditions is necessary to prevent shell bursting from the lower temperature evolution of gas. The decomposition of the monodisperse 97K PAMS is by comparison very sharp.

Figure 3. Thermal analysis of PAMS is compared to plasma polymer and plasma parylene. The polydisperse (11K and 19K M.W.) PAMS, used to prepare beads, show mass loss at lower temperature. Two monodisperse (6K and 90K M.W.) PAMS samples show that higher molecular weight results in lower thermal stability.

Bead Mandrel Preparation

To achieve sphericity, PAMS beads are suspended in water heated above their softening point (60 °C for 11K and about 80 °C for 19K M.W.). When the pellet reaches the softening temperature, surface tension drives the bead to become spherical. To prevent aggregation of batches of softened PAMS beads, we added polyvinylalcohol (PVA) (13K-26K M.W., 88% hydrolyzed, 4 g/l H₂O). The water, stirred with a paddle, could be heated up to nearly boiling with no aggregation of the beads.

As we changed to the 19K PAMS we made some changes in the heat treatment to improve the process. Water used for the heat treatment was filtered to less than 1 μ m. Water added to make up for evaporation was preheated on a separate hot plate so that the bath temperature remained nearly constant. The stirring speed used to achieve random motion in the hot water was slowed to a minimum. Fig. 4 compares the sphericity and surface finish of an 11K and 19K bead. Good sphericity and surface finish are achieved by both processing methods although the 19K material appears rougher, indicating that longer heating time may be needed for the more viscous 19K material.

Figure 4. Good sphericity is shown for an 11K and 19K bead in AFM scans. Higher viscosity of the 19K bead may cause the observed rougher surface finish.

Shell Mandrel Preparation

Shell mandrels have three advantages over solid beads: there is much less PAMS to remove, forces exerted by bead weight and surface tension of the melted PAMS on the coating during pyrolysis are reduced, and the internal pressure generation at the start of pyrolysis is lower because of the large void volume. We have examined PAMS shell generation by three methods: drop tower blowing of PAMS solutions, frit blowing in a drop tower, and microencapsulation [1]. The drop tower method, as reported previously, produced only thin nonspherical PAMS shells. Dropping solvent-swollen PAMS beads or frit down a hot column produces some 300 μ m shells and larger shells with multiple internal bubbles. This possible shell-blowing technique will be described in a separate technical memo. Currently, microencapsulation offers the best near term promise of producing PAMS shells.

About one year ago LLE prepared a batch of microencapsulated PAMS shells by the shake and toss technique using the 97K PAMS material. The shells had a wall thickness of about 10 μ m, diameters from 500 to 800 μ m, and sphericity within about 200 nm. Both the SEM and the sphere mapper show evidence of particulate surface debris.

Very recently we attempted production of larger microencapsulated shells in our lab using a newly designed double-orifice droplet generator. This droplet generator is

composed entirely of off-the-shelf parts that is easily reconfigured to accept different needle sizes. We have found a slight mechanical vibration produced by either an electromechanical solenoid or pneumatic plunger detaches shells from the needle at regular intervals. We chose monodisperse 97K M.W. PAMS for microencapsulation of shells. The inner phase was water. Best results were achieved using 7% PAMS in 1,2dichloroethane/methylethylketone (83/17 by mass). Droplets generated using this composition fell quickly to the bottom of the container. The container, a round bottom beaker, was slowly rotated to keep the liquid droplets in random agitation and was heated to about 35 °C. Within 1 minute the organic layer began to become cloudy as drying proceeded. Within 10 minutes the shells became suspended in the external water phase and hovered in the rotating medium, appearing to be neutrally buoyant. The shape of the 2 mm droplets in the preform state, before fully hardening, was very sensitive to rotation speed. Fig. 5 shows two microencapsulated shells made using the double orifice generator. The outer surface of 5a shows no vacuole defects. Fig. 5b has a diameter of 1300 μm with an average wall thickness of 50 μm . The microencapsulation effort is now being transferred to GA for further development. They will be responsible for providing microencapsulated shells that will be used in future experiments on the depolymerizable mandrel process.

Figure 5. A microencapsulated PAMS shell (a) shows good sphericity and surface finish with no surface vacuoles. Some surface debris are visible. A fracture cross section of another shell (b) shows shell nonuniformity.

PLASMA POLYMER COATING OF PAMS MANDRELS

Coating of Bead Mandrels

For the decomposable mandrel process to work, a thermally stable coating must be deposited on the mandrel. We have found that plasma polymer is sufficiently stable to withstand the conditions of pyrolysis and is simultaneously permeable to the gaseous decomposition products. During the early phases of the decomposable mandrel project, we assumed that the mandrel was unaffected by the coating process. Recently we have found that the 11K mandrels can soften and distort during coating. Another coating issue is assuring that the coating is uniform. We have used bouncer pans to agitate Nova target microshells and have found that the coating is normally very uniform. For the decomposable mandrel work two complications arose. First, the beads used as mandrels are larger and much heavier and do not bounce as well. Second, bubbles occasionally appeared in the beads during coating. In this section, we discuss the problems encountered during bead mandrel coating and how they were solved.

Agitation During Coating

Last fall, progress was slowed by a series of experiments where the shells collapsed during pyrolysis. We have found that collapse was caused by nonuniformity in the coating. The nonuniformity is traceable to two causes: poor agitation during coating, and bubble growth in the bead mandrel during coating. Both of these factors can work together to produce badly nonuniform coatings.

Initial work with the depolymerizing mandrel process concentrated on mandrels in the 500 μm diameter range. These beads were easily coated using the piezoelectric bouncer pans normally used for Nova shell targets. We later experimented with 1 to 2 mm diameter beads and found that the agitation action of the bouncer pan was ineffective. Another agitation technique, originally developed to coat silica aerogel spheres, was found to provide what appeared to be random agitation for the beads. It utilized a tilted rotating pan in which the beads tumbled down the sloping pan as it rotated. A series of coating runs were made using a range of bead diameters. When these coated beads were pyrolyzed, we found that the coating collapsed on itself yielding a cup-shaped structure. This failure was found to correlate with the agitation technique. Bounce-coated shells survived pyrolysis while rolled coatings collapsed.

The rolled coatings were investigated for uniformity by drilling a 100 μm hole in the coating and dissolving the inner PAMS with solvent. Radiography of the resulting shells showed that the coating thickness was not uniform, with the wall thickness varying by more than 50%. We now believe rolling causes a spinning motion that results in coating the top side of the bead preferentially. Any imperfection in the bead, such as a bubble, enhances the imbalance of the bead and adds to nonuniform coating.

Bubble Growth

A second source of failures was traced to elevated temperature during plasma coating. After coating, we occasionally observed bubbles within the PAMS material. When plasma polymer coating was conducted at high power (and thus at higher

temperature), the beads developed many internal bubbles and the coating cracked as the outer surface expanded. The discovery of bubble growth caused by coater thermal effects showed that the conditions PAMS beads are exposed to during coating can alter the homogeneity of the bead and lead to coating nonuniformity. Clearly this problem needed to be solved to have a successful shell-making process. Two approaches were taken to prevent growth of bubbles within a PAMS bead: deposition at lower power and use of slightly higher molecular weight PAMS. When the deposition temperature remained below 40 °C for 11K M.W. and 60 °C for 19K M.W. PAMS beads, we found that no bubbles or surface deformations were observed. Fig. 6b, discussed in the pyrolysis section, shows that coating at lower coater power preserves the sphericity of the bead through the plasma polymer coating process.

Coating of PAMS Microencapsulated Shells

We have successfully coated microencapsulated PAMS shells produced at Soane Technologies, by D. Schroen-Carey (using droplet generator), and by LLE (using shake and toss). The shells tested were from 500 μm to 2 mm in diameter. The CH coating thickness was varied from 10 to 50 μm . We have had no difficulties handling the shells. While bouncing activity decreases with coating time for beads, microencapsulated shells have behaved in the bouncer pans remarkably similar to the Nova sized shells. We have observed no deformation or change in appearance. Monodisperse high molecular weight polymer shells show no deformation or change in appearance from exposure to plasma coater conditions.

PYROLYSIS OF COATED MANDRELS AND CHARACTERIZATION OF THE RESULTING SHELLS

Pyrolysis of Coated Beads

During pyrolysis of PAMS beads, we encountered four problems: bursting, thermal deformation of the mandrel, shrinkage of the coating, and collapse. Collapse resulted from nonuniform coating thickness, as was discussed in the previous section.

Our first attempts at overcoated bead pyrolysis resulted in shells that burst from heating too rapidly. After measuring the rate of PAMS depolymerization with several isothermal TGA runs, we constructed a temperature program that attempted to achieve a constant rate of mass loss to avoid the bursting problem. This temperature program consisted of raising the sample temperature to 200 °C at 10 °C /min, holding at this temperature for 30 minutes to allow low temperature volatiles (low molecular weight oligomers) to escape, and then ramping at 0.2 °C/min to 300 °C where the sample was held for 30 hours. This final hold time was adjusted depending on the mass of the mandrel. All mandrel pyrolysis was done in a flowing nitrogen stream in the center of a quartz tube furnace (1-inch diameter with a 12-inch long heated zone).

Coating shrinkage was found to be related to the pyrolysis conditions. The gradual shrinkage of the plasma polymer is controlled by both time and temperature. We measured many coated beads before and after pyrolysis to gather shrinkage data. We have observed diameter shrinkage in the range of 3-15% on CH shells which appeared to maintain sphericity. Table I summarizes the results of shrinkage experiments. We

found that as either time or temperature increased, the amount of linear shrinkage increased. This information is needed to be able to predict the size of a completed shell. Simultaneous with the shrinkage measurements, we made residue assessments by weighing the individual shells. The mass was compared to a calculated value from the outside diameter and the expected wall thickness. From these experiments we found that pyrolysis at 300 °C for 2000 minutes is needed to achieve a residue free shell from a 700 μm to 900 μm starting bead diameter.

Temperature	Pyrolysis time (min)		
	1000	2000	4000
290°C	3	6	10
300°C	5	10	15

Table I. Shells were characterized for percent diameter shrinkage after pyrolysis. Longer pyrolysis time and higher temperature caused greater shrinkage.

We felt at this point that we had solved many of the bead process problems and focused our efforts on a detailed experimental investigation of the sphericity of the beads through the three major steps of the process: mandrel preparation, plasma polymer overcoating, and pyrolysis. We selected a batch of PAMS beads that were in the 600 to 900 μm diameter range with good surface finish. A total of 14 beads were measured for diameter and mass and then were sphere mapped on the AFM. All of the selected beads were found to have sphericity of 200 nm in three orthogonal axes. The beads were next coated with 40 μm of plasma polymer and sphere mapped again. Because we chose a range of bead sizes, we were able to establish the identity of each bead with its original size. Finally, seven of the original beads were pyrolyzed, measured for mass and diameter, and sphere mapped. While all the beads selected from the original batch showed excellent sphericity before and after coating, we found that after pyrolysis most shells were distorted. Of the seven pyrolyzed shells, three were mappable in one or two directions, three were not mappable at all. The shells typically showed one scan axis that measured as spherical and was distorted in the other two directions. Only one shell could be sphere mapped in three directions without exceeding the 4 μm travel limit of the AFM. Fig. 6 shows the scans of the best bead from this series for the starting bead mandrel, after coating, and after pyrolysis. From these results we concluded that deformation in the range of 4 to 10 μm occurs during pyrolysis. Such deformation could be driven by the weight of the molten PAMS bead or by the surface tension of the puddle of liquid PAMS inside the shell. From our investigation of collapse, we found that the plasma polymer may become soft under the conditions of pyrolysis. Close inspection of the collapse fold shows no cracking. The softening may be caused by both temperature and solvent effects of the escaping α -methylstyrene monomer.

Figure 6. Mandrel and shell are characterized with an AFM scanning profilometer. The first traces (a) are a PAMS bead treated in 96°C water for 7 hours. The next traces (b) show the effect of adding a 30 μm overcoating. After pyrolysis (c), approximately 3 μm of eccentricity develops.

To reduce the effects of distortion during pyrolysis, we attempted to distribute the mass of liquid PAMS by random tumbling of the bead during heating. A rotating fixture was installed in the furnace consisting of a glass tubular holder driven by an external motor. Some problems were initially encountered with the beads falling out of the rotator. The entrance aperture was reduced to a 5 mm opening in a 1 cm diameter tube which prevented beads from falling out. We found that the beads did roll in the tube up to a temperature of 260 °C when the bead would stick to the wall of the tube. Despite the sticking, several beads were pyrolyzed and measured for sphericity. Of 23 shells, five that were successfully rotated were measurable on the AFM, having a deformation about 1 to 3 μm . This was an indication that rotation can improve the sphericity. However, some deformation was still occurring beyond the starting bead. All of these beads stuck to the tube and thus had one axis of rotation and were not randomly tumbled. On some of the pyrolyzed shells, we saw a contact point anomaly where the bead was held to the glass surface.

To achieve greater randomness, we tried a "fluidized bed" approach using glass beads. The glass beads, which are used for bead blasting cleaning, are about 100 μm diameter. The beads were cleaned and filtered prior to use. A sufficient quantity of beads to cover the bottom of the rotating tube was used. We found that the tumbling motion of the coated mandrel in the beads was good even at 300 °C. However, upon examination at the end of pyrolysis, we found the shell encrusted in glass beads. The beads were easily removed by immersing the shell in toluene in an ultrasonic bath. Unfortunately, the glass beads left an impression on the plasma polymer and gave the

shell a golf ball appearance. Again the softening of the plasma polymer during pyrolysis appears to be the cause of the problem. Some of the impressions on the plasma polymer were rings of residue on the outside where glass beads had stuck. Possibly α -methylstyrene oligomer is able to diffuse through the coating and form a puddle at the contact point. Being of low molecular weight, its thermal stability would be higher, as we had found previously (see Fig. 3), and would tend to adhere the shell to external substrates.

Pyrolysis of PAMS Microencapsulated Shells

Deformation during pyrolysis was largely eliminated by using a microencapsulated thin shell mandrel made from 97K M.W. PAMS material. Fig. 7 compares the AFM circumferential scans of a starting shell mandrel and a pyrolyzed coating done without rotation on a shell mandrel. The sphericity after pyrolysis is within 200 nm and is comparable to the starting mandrel. Using microencapsulated shells has also reduced sticking to the substrate in the furnace.

Figure 7. The first AFM sphericity traces (a) are scanned from a PAMS microencapsulated shell. The second traces (b) are scanned from a completed shell after pyrolysis, showing sphericity has been maintained.

The degree of shrinkage of the plasma polymer coating was essentially the same for coatings on either beads or shells. The controlling factors are temperature and pyrolysis time. In principle, shorter pyrolysis times should be needed for shells because of the reduced mass. We found that 400 minutes at 300 °C left a puddle of residue, but 1000 minutes at 300 °C left a residue-free shell. This is somewhat longer than expected based on the times used for beads.

NEAR TERM PLANS

The critical issues remaining that must be resolved to use the decomposing mandrel process for target production are: preparation of smooth, spherical shell mandrels, development of surface texturing, and deposition of buried diagnostic layers. Shell mandrels, at this time, seem to offer the surest route to production of both Nova and Omega sized targets primarily because of the much better sphericity of the finished shells. We will rely on GA to prepare high quality microencapsulated shell mandrels. Our own efforts will focus on pyrolysis and characterization of the final shells. We will confirm that there is no distortion of shells after pyrolysis. We will determine the required time to completely pyrolyze the mandrel as a function of mandrel size, measure the shrinkage as a function of time and temperature, and examine the range of coating thickness that produces shells with good sphericity.

To provide a more uniform thermal and stress free environment for conducting the pyrolysis, we have begun working on a gas levitator that is based on a gas rotameter tube. We expect that a gas levitated environment will avoid the point contact loading that may be one of the causes of deformation.

We have begun working on textured PAMS mandrels. Several tests with PAMS beads were conducted to determine the feasibility of using laser photoablation. Laser ablated pits 1 to 2 μm deep and 50 μm wide have been cut in several PAMS beads and shells. We are investigating for both flat and spherical samples how the surface topology changes with the deposition of thick plasma polymer layers.

Early in the development of the depolymerizing mandrel process, we fabricated shells with 0.1 μm thick titanium layers on the inside surface. No difficulties were encountered during pyrolysis. The titanium layers were somewhat rough, as were the surfaces of the PAMS beads used at that time. Diffusion through the titanium may have been through grain boundaries in the titanium or through shadowed areas caused by the rough PAMS. We have recently attempted to deposit thin palladium layers in a sputter coater. When the coatings were examined we found tears in the thin metal coating. The metal apparently cold welds to itself in the bouncer pan and tears the coating. If cold welding is a persistent problem, we will attempt to overcome it by applying thin metal layers to static beads and bouncing only after oxidizing the surface.

At this point no technique has been developed for applying a PVA overcoating to shells produced by the depolymerizing mandrel process. One difficulty is the small number of shells (less than 10 at a time) expected to be produced. For Nova sized shells we believe a modification of existing overcoating techniques may work and will be tried soon.

We feel confident that within the next several months, a recipe will be defined for production of unmodified CH shells from microencapsulated PAMS mandrels. By the end of the fiscal year, development of texturing and layering techniques should be completed.

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