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**IMPROVING THE WALL UNIFORMITY OF RESORCINOL FORMALDEHYDE FOAM SHELLS  
BY MODIFYING EMULSION COMPONENTS**

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*Previously we have developed a production process for both standard density (100 mg/cc) and high-density (180-200 mg/cc) resorcinol formaldehyde (RF) foam shells with a triple orifice droplet generator. These foam shells are needed for direct drive inertial confinement laser fusion experiments on the OMEGA laser facility at the University of Rochester. Although this process has been developed into production mode, the yield of high density RF (HDRF) and standard density (SDRF) shells with acceptable wall uniformity has been poor. This yield depends on the type of RF shell that is being fabricated. For HDRF this yield is ~5% while for the SDRF shells the yield is ~30%. We have made improvements in the yield of these shells that meet the wall uniformity specification by modifying the composition of the outer oil solution (O2) in the microencapsulation emulsion. This improvement was achieved by a small addition (0.60 wt.%) of a styrene-butadiene-styrene (SBS) block copolymer into the outer oil (O2) solution that increased the interfacial tension of the emulsion system as well as the viscosity of the O2 solution. This modification improved the out of round and concentricity of the RF foam shells resulting in an increase in the yield of shells that meet the target wall uniformity specifications.*

## I. INTRODUCTION

Aerogel or foam shells are currently used for direct-drive inertial confinement fusion experiments at the OMEGA facility at the University of Rochester Laboratory for Laser Energetics (LLE). In the future, these shells may be used in experiments on the National Ignition Facility (NIF).<sup>1</sup> The shells needed for OMEGA experiments have diameters of ~850–900  $\mu\text{m}$ , which are scaled down versions of the targets required for foam direct

drive ignition designs on NIF.<sup>1</sup> The required wall thickness and foam density depend on the application. Cryogenic targets require a foam wall thickness of 50-100  $\mu\text{m}$  with a foam density of 50-110 mg/cc. The room temperature foam shell surrogates, mimicking the D<sub>2</sub> or DT ice layer, need to have a wall thickness of 80-120  $\mu\text{m}$  and a foam density of 180-250 mg/cc. These targets are designed to reduce the need for difficult to field cryogenic shots with the foam serving to simulate the cryogenic fuel layer.

Because wall non-uniformity adversely effects the implosions, the foam shells are required to have good wall uniformity. The mode 1 wall uniformity specifications for these resorcinol formaldehyde (RF) foam targets are usually quoted in terms of non-concentricity (NC), defined by Eq. (1),

$$NC = \frac{\Delta \text{ Wall thickness}}{2(\text{Avg. wall thickness})} \quad (1)$$

The current NC requirement for targets for experiments on OMEGA is  $\leq 5\%$ . Because the foam material is porous, the foam shell by itself cannot hold the required fill gas (D<sub>2</sub> or DT). For these foam shells to hold a fill gas, a pinhole free permeation barrier is over-coated around the shell. For room temperature surrogates an additional coating of 1000  $\text{\AA}$  of aluminum is added to assist in gas retention.<sup>2,3</sup>

Resorcinol formaldehyde was chosen as the foam material because of its transparency, facilitating the extensive dimensional characterization required by allowing the use of routine optical techniques. The optical transparency is due to the characteristically small pores (<0.10  $\mu\text{m}$ ) of the RF aerogel, first developed by Pekala,

*et al.*<sup>4</sup> The RF aerogel is formed by a polycondensation reaction of a precursor solution using a two-step (base/acid catalysis) gelation process.<sup>4,5</sup> The fabrication of RF foam shells was later developed by Lambert, *et al.*<sup>5</sup> Due to the fragile nature of the foam shell pre-form, this work only achieved a yield of ~33%-66% for intact shells. We extended this work by transforming the process into a repeatable, reliable, high yield production process to fabricate a high yield (>90%) of intact shells for both standard density RF foam shells (SDRF) which have a foam density of ~100 mg/cc and high density RF foam shells (HDRF) which have a foam density that can range from 180-200 mg/cc.<sup>6-8</sup> We have also been able to develop the required pinhole free permeation barrier in order for these shells to hold the required fill gas (DT or D<sub>2</sub>) by over-coating the shells with a glow discharge polymer (GDP).<sup>6-8</sup>

Though we are able to produce a high yield of intact shells with this production process, a major issue, which affects high volume fabrication of target quality shells, is the low yield of shells that meet NC specification. Through our previous work we have found empirically that the yield of shells that meet the NC specification depends on the density of the foam shells.<sup>7,8</sup> For SDRF shells with density of ~100 mg/cc the yield is 30% and for HDRF with density of ~200 mg/cc the yield is approximately 5%. Despite the apparent low yield, the sufficient number of target quality shells can be produced for experiments on OMEGA (typically 10 shells per campaign), because large numbers of shells (thousands) are fabricated in a production batch. However, the effort to supply these targets requires a very labor intensive sorting of shells to find those that meet the NC specification. With the possibility of these targets becoming routine direct drive experimental shots, it is important to improve the process so that a larger number of shells could be made to specification.

In our previous work on improving the sphericity and wall uniformity of full density poly  $\alpha$ -methylstyrene (P $\alpha$ MS) shells, the outer emulsion phase was modified by adding a polymer additive (poly acrylic acid) to the outer phase solution.<sup>9,10</sup> This modification increased the interfacial tension of the emulsion system as well as improving the sphericity or the out of round (OOR) of the shell as well as the wall uniformity or (NC) of the shell.<sup>9,10</sup> Although it is not immediately obvious that a large interfacial tension would result in better NC, as mentioned above this improvement on NC was empirically observed in the case of P $\alpha$ MS. As a direct extension of that work we began investigating possible polymer additives for the outer oil phase of the RF emulsion system (O2) that would produce similar results to the work that had been done with the P $\alpha$ MS shells. Because the RF

emulsion system is a reverse emulsion system of oil-water-oil when compared to the P $\alpha$ MS emulsion system of water-oil-water, the hydrophilic polymer additive that was used for the P $\alpha$ MS process could not be used. The polymer additive has to be hydrophobic and compatible in the oil phase. We used a styrene-butadiene-styrene (SBS) block copolymer (Kraton D 1102 from GLS Corp) and several other styrene based block co polymers in the outer oil phase, typically called O2, to increase the interfacial tension of the RF emulsion system, which was measured by an interfacial tensiometer. This modification to the O2 led to a higher yield of shells with NC below the 5% specification. In SDRF foam shells, this yield was increased from 30% to 90% and in HDRF shells this yield was improved from 5% to 25%. The beneficial effect on NC when the O2 phase is changed is purely empirical at this point. Despite several attempts by various authors,<sup>11-13</sup> there is not a complete theory of the physics affecting NC in the complicated systems used to cure shells. Hence, our results though empirical, clearly provide a hint for future theoretical work on NC and provide a practical guide for increasing yield of shells with low NC.

## II. EXPERIMENTAL

RF foam shells were fabricated by using the triple orifice droplet generator (Fig. 1).<sup>5,8</sup> The triple orifice droplet generator method allows precise control of shell diameters and wall thickness, so that a large quantity of similar shells of a specified size can be made. In our standard baseline process, the droplet generator creates a double emulsion of an inner oil (O1), RF precursor solution (W1), and an outer oil (O2). When introduced into the droplet generator the precursor solution has just started the acid catalysis step in the RF gelation process as described in Ref. 8. This solution will remain liquid at room temperature for >12 h, allowing consistent behavior of the RF precursor throughout the encapsulation run. The foam precursor is immiscible with the oil phases, allowing a liquid shell to form. The O1 solution is a

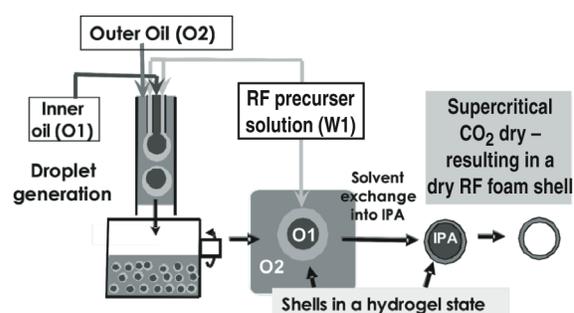


Figure 1. Diagram on how the RF foam shells are fabricated by the microencapsulation process using a triple orifice droplet generator.

mixture of mineral oil and tetrachloroethylene (TECE). The O2 is also comprised of a mixture of mineral oil and TECE with a small amount of sorbitan monooleate (Span 80). The Span 80 is the surfactant in the O2 that helps prevent the RF shells from agglomerating. The O1 solution is forced through a needle and is encapsulated by the foam precursor solution that is fed through a slightly larger concentric needle. The outer oil then strips the droplet off the needle and into the collection tube. The shells and fluid are collected in the rotating flask and placed in a water bath at the precursors curing temperature to complete the gelation. It is in this step where the NC of the shell is determined. The cured shells are solvent exchanged with isopropyl alcohol (IPA) and dried using a supercritical CO<sub>2</sub> dryer (CPD).

Several polymer additives were investigated in order to modify the O2 solution in the RF emulsion system. As stated earlier the additive must be hydrophobic and compatible with the other components in the O2 solution. The polymer additives were screened by first testing their compatibility with the O2 solution. This was done by adding 0.50 to 1 wt% of the polymer into 100 g of the standard O2 solution. If the polymer additive was sufficiently soluble in the O2 solution, it was then tested on how it affected the interfacial tension of the RF emulsion system. The interfacial tension of the emulsion system was measured by an interfacial tensiometer (Future Digital Scientific OCA-15) via the pendant drop method.<sup>14</sup> The tensiometer is accurate to ±0.10 dyne/cm. In addition, the viscosity of the modified O2 solution was then measured and compared to the standard O2 solution by using a rheometer, (Bohlin CVO50 from Bohlin Instruments) and compared to the standard O2 solution. These measurements were performed at 25°C on the RF precursor solution after the base and acid catalysis steps (but well before gelation).

To obtain an interfacial tension reading for a given emulsion system, the density of the emulsion components was required. The density of the emulsion components were measured with a densitometer (Anton Parr, DMA 4500). The densitometer measures the density of these emulsion components as a function of temperature. The densitometer is accurate to 0.05 mg/cc and can measure densities at temperature range of 0°C to 90°C.

The dried shells are over-coated with a glow discharge polymer (GDP) in order for the shells to hold a fill gas as mentioned above. The barrier is tested for gas retention by permeation filling the shells with 900 Torr of argon (Ar) and tracking the Ar content versus time using x-ray fluorescence (XRF) as previously described.<sup>6</sup>

### III. RESULTS AND DISCUSSION

Before modifying the O2 of the RF emulsion system, the interfacial tension of the standard RF emulsion system was measured and compared with several modified outer oil phase solutions. The results of this study shown in Table I show that the standard process for both HDRF and SDRF shells have an IFT of approximately 1 dyne/cm. The viscosities of the solutions were also measured and are listed in the table. The viscosities of standard O2 solution for both HDRF and SDRF were also measured and were approximately 10 cps at 25°C and 7 cps at its curing temperature at 70°C.

To increase the IFT of the RF emulsion system, two routes were explored. The first was a simple reduction in the concentration of the Span80 surfactant. The second route involved identifying and examining a number of polymer additives. The data on IFT and viscosity of the RF emulsion is summarized in Table I.

Table I. Lower Span 80 and addition of a Styrene-Butadiene-Styrene (SBS) hydrophobic block copolymer additive were investigated to increase the interfacial tension (IFT) of the RF emulsion in order to improve NC of the RF foam shell. This additive increased the interfacial tension (IFT) of the RF emulsion system and viscosity of the O2 solution that also resulted in shells with improved NC as well as sphericity.

Additives in O2	Amount Added in O2 (wt.%)	Amount Span 80 in O2 (wt.%)	IFT (Dynes/cm)	Viscosity of O2 (cps) at 25°/70°C
None (std O2)	NA	0.10	1	10/7
None	NA	0	7	10/7
None	NA	0.05	1.5	10/7
SBS	0.70	0	21	18/12
SBS	0.60	0	21	18/12
SBS	0.60	0.10	4	18/12
SBS	0.60	0.10	4	18/12

Decreasing Span 80 concentration indeed increased IFT as expected as shown in Table I. Span 80 was either completely removed, or its concentration was lowered from the standard 0.10% to 0.05%. IFT was increased from 1.5 dyne/cm to as much as 7 dyne/cm. The viscosity and density of the O2 solution did not significantly change which means that the viscosity and density of the O2 was not affected by the Span 80 at these concentrations. Although the desired effect was obtained, when shells were fabricated major agglomeration of shells resulted during the gelation and solvent exchange processes, which led to an unacceptably low yield (virtually 0%) of usable shells. In fact, the agglomeration made it impossible to even measure the NC of shells.

We next pursued identifying and examining the effects of several styrene based hydrophobic block copolymer additives on the RF emulsion system IFT as well as the OOR and NC of the RF shells. Hydrophobicity of the polymer is essential to prevent its mixing into the aqueous RF precursor solution. These polymer additives were also aimed as possible replacements for the Span 80 in analogy with the P $\alpha$ MS system where a single additive, PAA replaced the polyvinyl alcohol (PVA) and increases IFT to simplify the emulsion.<sup>9</sup> Some of these polymers were not compatible with the O2 because they were not soluble in the O2 solution. The polymer additive that increased IFT while being compatible with the O2 oil phase was a styrene-butadiene-styrene (SBS) block copolymer also referred to as Kraton D 1102 from GLS Corp. The effects on the IFT on the RF emulsion system and the viscosity of the O2 solution by adding the SBS into the O2 are shown on Table I. Both HDRF and SDRF foam shells were fabricated with 0.60% SBS replacing the Span 80 in the O2 solution. This modification will be referred to this as modification 1. This modification did not change the density of the O2 solution but it did increase the IFT of the RF emulsion system from 1 to 21 dynes/cm and the O2 viscosity increased from 10 to 18 cps at room temperature and at its curing temperature of 70°C the viscosity increased from 7 to 12 cps.

The shells fabricated from modification 3 were able to survive through the curing step without any observed agglomeration of the shells. This non-agglomeration of shells was also observed during the solvent exchanged step into isopropyl alcohol (IPA). The shells in IPA visually appeared to have better wall uniformity than shells fabricated with the standard process; this is shown in Fig. 2. But when the shells were dried in the CPD agglomeration was once again observed.

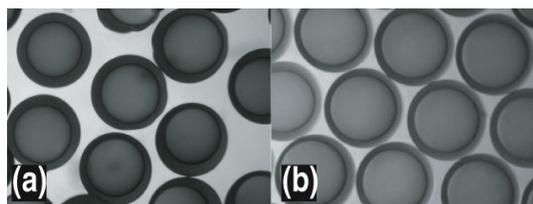


Figure 2. HDRF shells fabricated with the traditional fabrication process. These shells visually are less concentric than the shells shown in (b). Shells from (b) are fabricated by simply eliminating Span 80 to increase IFT. Measurements of the dry shells confirmed the improvement, however, these shells agglomerated after drying resulting in a low yield of usable shells.

Although it is hard to explain the observed agglomeration beyond the solvent exchange step into IPA, Span 80 must form a thin coating on the shells that form. This observed agglomeration necessitated addition of

Span 80 back into the O2. RF shells were fabricated using 0.60 wt% SBS and 0.10% Span 80 in the O2 solution. This will be referred to as modification 2. Again both SDRF and HDRF foam shells were fabricated in this manner. Again the density of the O2 was not affected when compared to the standard O2, but the IFT of the RF emulsion system and the viscosity of the O2 was modified. The IFT of the RF emulsion system was 4 dynes/cm, which while lower than without Span 80, it was still higher than the standard emulsion system at 1 dyne/cm. The viscosity of the O2 with this modification was similar to the viscosity change without Span 80. A visual improvement of the wall uniformity of these shells was qualitatively observed upon drying. The shells did not agglomerate rendering usable batches of shells and allowing measurement of NC after drying. This wall uniformity improvement was confirmed by interferometry. The yield of shells that met the NC specification increased from 30%-90% for SDRF shells, and for HDRF the yield was improved from <5% to 25%. These results shown on Fig. 3 are from three separate runs done on different days. The sphericity of shells was also examined, as increased IFT should have a natural beneficial effect of sphericity. The sphericity as measured by out-of-round of shells was also improved from an average of 4  $\mu$ m to 1  $\mu$ m as expected for both HDRF and SDRF foam shells. Although the previous 4  $\mu$ m value was below the current 5  $\mu$ m specification, the decrease in sphericity is beneficial as the specifications are expected to become tighter in the future.

As mentioned above, one does not necessarily expect an improvement in NC by increasing IFT. Our wall uniformity data (from several batches) shows a clear improvement from the SBS addition. This improvement cannot be due to better density matching, since the densities of the solutions with high and low IFT were virtually the same ( $\pm 5 \times 10^{-4}$  g/cc) as described above. These differences are much less than the changes which had previously been shown to affect NC ( $\pm 5 \times 10^{-3}$  g/cc for SDRF and  $\pm 1 \times 10^{-2}$  g/cc for HDRF).<sup>7,8</sup> Another factor that can affect NC and OOR is the agitation of the shells during the curing process.<sup>6,9</sup> The agitation of the shells did not cause the observed NC improvement because the RF shells (with or without SBS) were agitated the same way. Although changes in viscosity in the O2 were observed with the SBS addition, previous work on PAMS shell fabrication by Takagi shows that viscosity only affected shell survival during the curing process, while IFT played a major role in affecting the OOR of the shell.<sup>9</sup> However, the viscosity changes in the O2 can not be completely ruled out from having an effect on the OOR and NC of the shells given our data set. We hope to distinguish the effects of viscosity and IFT separately in future work.

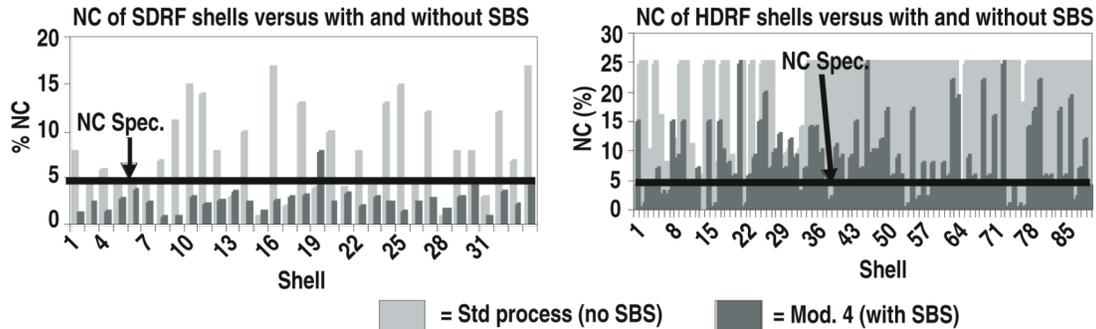


Figure 3. Shows the NC for SDRF and HDRF shells fabricated with the traditional process (No SBS) VS the NC of SDRF and HDRF foam shells fabricated with the addition of 0.60% SBS in the O<sub>2</sub> (Mod. 2). The addition of the SBS to the O<sub>2</sub> solution made a dramatic improvement to the yield of shells that meet the NC specification.

Besides improving the NC of the RF shells, the shells fabricated with this process are required to hold a fill gas. These shells were successfully coated with 3-4  $\mu\text{m}$  of GDP, the same 3-4  $\mu\text{m}$  over-coating that served as the pinhole free permeation barrier for the standard HDRF and SDRF foam shells. As stated earlier the shells were tested for gas retention by filling them with argon (Ar), and analyzing the shells by XRF. Approximately 24% of the shells over-coated with GDP were gas retentive, this is a lower yield when compared to the GDP coated traditional high density and standard density RF shells that are approximately 50% gas retentive. More gas testing must be done to confirm these results. It may appear that the lower yield in gas retentive shells could be due to the SBS changing the foam structure or surface of the RF foam. More studies must be done to determine the surface structural change of the RF foam by using the SBS process and whether this plays a role with the gas retention of these shells. Rough substrate surface has been shown to lead to a reduced effectiveness of permeation barrier coatings.<sup>15</sup> Simple washing of the surface may reduce the isolated features observed in the surface of the shells and lead to a smoother surface for GDP coatings and hence improved gas retention.

#### IV. CONCLUSION

Addition of 0.60% SBS block copolymer to the O<sub>2</sub> phase of the RF emulsion system increased the IFT of the emulsion system and the viscosity of the O<sub>2</sub>. This modification was implemented for both HDRF and SDRF foam shells that led to a process to fabricate a higher yield of spherical and concentric RF foam shells that met the NC and OOR specifications. The yield of shells that met the target specification for NC for SDRF foam shells improved from 30% to 90%, and in HDRF foam shells the yield improved from less than 5% to about 25%. The sphericity of the shells was improved by increasing the IFT of the RF emulsion system. These results though

purely empirical, are of importance in high yield production of RF shells of 100 and 200 mg/cc.

#### ACKNOWLEDGMENT

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