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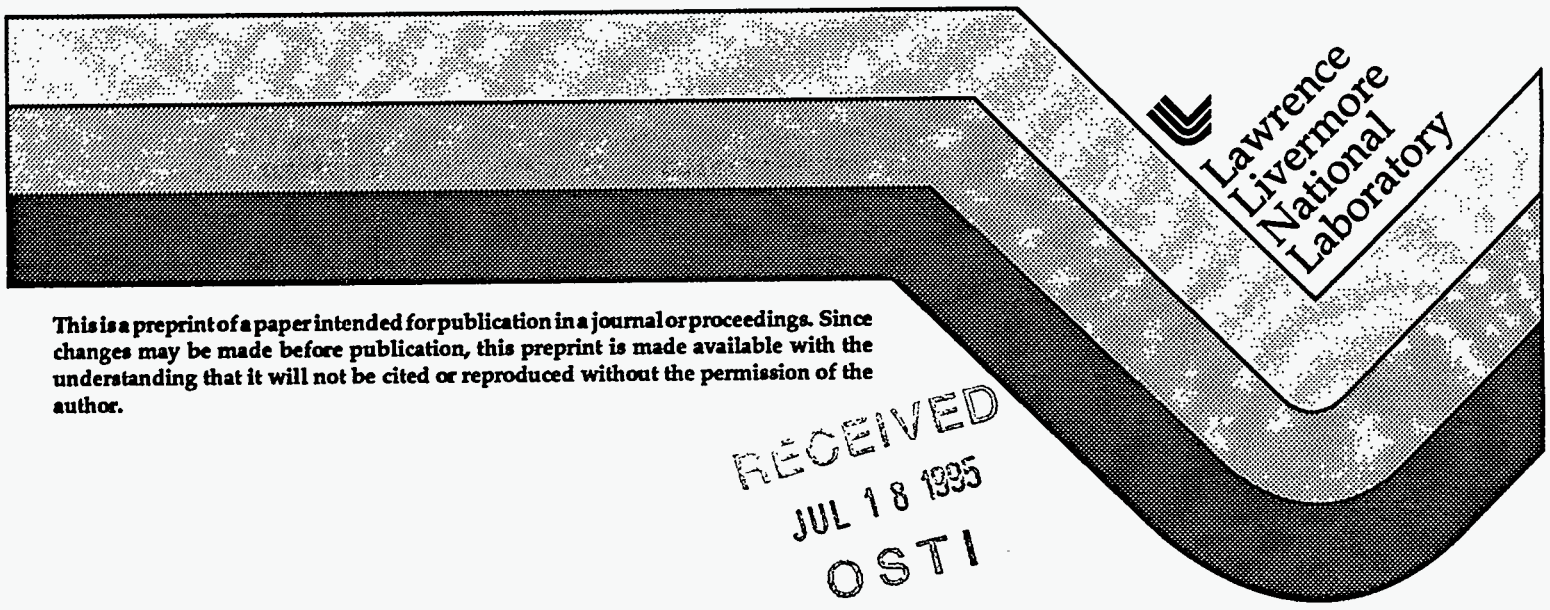

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PRODUCTION OF HOLLOW MICROSPHERES FOR INERTIAL CONFINEMENT FUSION EXPERIMENTS

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ABSTRACT

The targets used in inertial confinement fusion (ICF) experiments at the Lawrence Livermore National Laboratory are plastic capsules roughly 0.5 mm in diameter. The capsules, which typically have wall thicknesses from 20 to 60 μm, must possess extraordinary symmetry and concentricity and must have surface finishes of less than 1000 Å peak-to-valley variation over surface contours of from 10 to 100's of μm. This paper reviews the fabrication of these capsules, focusing on the production of the thin-walled polystyrene microshell mandrel around which the capsule is built. The relationship between the capsule characteristics, especially surface finish, and capsule performance is discussed, as are the methods of surface characterization and modification necessary for experiments designed to study the effects of surface roughness on implosion dynamics. Targets for the next generation of ICF facilities using more powerful laser drivers will have to be larger while meeting the same or even more stringent symmetry and surface finish requirements. Some of the technologies for meeting these needs are discussed briefly.

INTRODUCTION

The fusion of the heavy isotopes of hydrogen to form helium and a neutron requires containment of the nuclei as a plasma for a period of time which is dependent upon the density of the plasma. In a star, gravitational confinement provides both the density and the time. In the laboratory two approaches are currently being pursued. In magnetic fusion, strong magnetic fields are used to contain relatively low-density plasmas for periods of seconds. In inertial confinement fusion (ICF), the confinement times are no longer than a nanosecond, but the plasma densities are more than the density of lead. At the Lawrence Livermore National Laboratory (LLNL) these densities are achieved by symmetrically depositing 100's of kJ of energy from the powerful NOVA laser system on a small plastic capsule containing gaseous D_2 or DT over a period of a nanosecond. The deposited energy ablates the capsule wall, and the rocket-like blow off of the hot surface material compresses the interior fuel. Although the laser system that provides the energy is as large as a football field, the target containing the fuel is only about 0.5 mm in diameter. This paper deals with aspects of the production of these capsules at LLNL, with particular focus on the thin microshell mandrel upon which the multi-layer capsule is constructed, and on the characterization and modification of the outer capsule surface.

The first ICF experiments used glass shells.[1] At LLNL these were produced from aqueous glass solutions using a heated drop tower to dry small droplets of solution to

form hollow shells.[2] Glass offered the advantages of high strength, low permeability, easy doping with diagnostic atoms, and excellent symmetry and surface finish. These last two characteristics will be discussed in more detail later. The first successful ICF experiments were conducted with commercially available 70 μm diameter shells, and as the laser drivers improved, larger and higher quality glass shells were fabricated. However it was soon recognized that the high material density and atomic mass of glass result in implosion inefficiency, and this motivated the development of carbon-based polymer shells.

Poly(vinyl alcohol) shells were prepared at LLNL as an alternative to glass.[3] These shells were also prepared using heated drop tower technology. Droplets of PVA solution with internal argon bubbles were generated using a dual orifice system. As the argon filled "tube" of PVA solution was forced out of the orifice system, acoustically assisted Rayleigh jet breakup produced uniform droplets about 200 μm in diameter containing a 100 μm diameter argon bubble. The close spacing of the droplets entering the column led to collisions during the shell formation process, and thus an electrostatic drop deflection system was developed. A frequency-controlled charging ring was placed immediately below the droplet generator to apply a positive charge to almost all droplets so that they would be deflected to a catcher by a positively charged deflection plate before entering the heated column. This allowed only the uncharged, widely spaced droplets to enter the column. In the heated column the hollow droplets dried to form high-quality spherical shells with diameters up to about 250 μm. One particularly attractive feature of PVA shells is that PVA is an outstanding barrier polymer for hydrogen, having a permeation constant several orders of magnitude lower than polystyrene. However attempts to extend this size range of PVA shells were unsuccessful, and the need for larger shells motivated the development of the currently used polystyrene-based target capsules.

CURRENT ICF TARGET CAPSULES

The current ICF target capsule is built around a roughly 0.5 mm polystyrene (PS) microshell and is composed of 3 layers as illustrated in Figure 1. The PS microshell, which is produced by solution drop tower techniques that will be expanded upon shortly, is coated with a 2 to 3 μm PVA layer. This is necessary since, as noted above, PS is a poor barrier polymer to hydrogen diffusion. The PVA layer is applied by collecting several hundred PS microshells in a 0.6 mm ID capillary tube, drawing a 10% aqueous PVA solution up around them, and then expelling them into a heated drop tower where the solution around the individual shells dries as they fall. The efficiency of this step is low, typically not more than 5% in terms of "target quality" shells recovered. Shell loss is largely due to uneven coatings and particulate adhering to the coatings. However, since each run involves several hundred shells and since the weekly demand for targets is typically 20 to 30, the inefficiency is tolerable for current capsule needs.

The outer capsule layer, called the ablator, is typically 20 to 60 μm thick and is deposited using plasma polymerization coating techniques.[4] A mixture of hydrogen and *t*-2-butene at a combined pressure of about 70 mTorr are fed through a glass tube wound with wire connected to a 40 MHz source. At appropriate power levels, a

hydrogen plasma is lit, and the organic feed gas is broken up into molecular fragments that rain down onto the PVA-coated microshells that are agitated in a bounce pan below. The coatings produced have an empirical formula of $CH_{1.3}$ and might be characterized as a heavily crosslinked polymeric material. For some ICF experiments it is useful to modify the opacity of the capsule ablator by incorporating a few atom % of higher Z atoms such as Br or Ge. This is accomplished by including in the feed gas flow small amounts of C_2H_5Br or $(CH_3)_4Ge$. Typical coating rates are from 0.3 to 1.0 μm/h, depending upon the feed gas pressure and power. The coatings produced have been shown to be completely conformal, especially over lateral length scales greater than 10 μm, a fact that will be of importance in the discussions below. The intrinsic roughness of the plasma polymer coatings has a root mean squared (rms) variation of 50 Å or less when the coater is operating properly.

As noted above, the PS microshells which form the mandrel around which the capsule is constructed are prepared using solution drop tower techniques. A 4.5 wt % solution of PS in dichloromethane which contains 3.0 wt % 2-propanol is used. The polystyrene is 98.5K and monodisperse ($M_w/M_n = 1.05$). The droplet generator uses acoustically assisted Rayleigh jet breakup to provide uniform drops with diameters of about 400 μm. The drop tower used is 4.5 m high and has seven independently heated zones. A 0.5 m 223 °C zone at the top is followed by a 3 m 200 °C zone and then a 1 m unheated zone. The tower atmosphere is quiescent air. Several thousand 400 to 500 μm diameter shells are typically made in a few-minute run.

A qualitative picture of microshell formation within the drop tower is as follows. Initially, the droplet shrinks due to evaporation of the volatile dichloromethane solvent. As the solvent evaporates, a concentration gradient builds across the drop radius with polymer concentration highest at the drop surface. During this period, the drop is being heated by the surrounding gas but is cooled by evaporation. As a polymer membrane forms at the surface, accelerated somewhat by the less volatile 2-propanol which is a non-solvent for PS, evaporative mass transport of the solvent (and thus cooling) is inhibited and the droplet begins to heat. When the droplet temperature exceeds its boiling point, an internal vapor bubble forms, and the shell inflates quickly. During shell expansion, evaporation is enhanced because the polymer-rich layer is thinned, allowing for better solvent transport, while the surface area is increased. The final size (and probably quality) will depend on the cooling rate and symmetry of the hot PS shell.

A quantitative treatment of this qualitative picture has been recently developed by Lim and Soane.[5] Due to the scope of this paper no attempt will be made to give any of the mathematical details of the treatment, but rather only an overview of the approach and a sampling of the kind of information and insight it provides. The model has a number of simplifying assumptions; key among them are: a) spherical symmetry which reduces the 3-D problem to 1-D; b) a two-component mixture of PS and solvent; c) bubble nucleation at the center at a given degree of superheating, and d) the bubble growth rate is controlled by a concentration-dependent polymer viscoelasticity. Assumptions a and c are clearly related and are perhaps the most limiting since the center of the droplet is expected to be the coolest and thus not a likely location for bubble nucleation. On the other hand these assumptions are necessary for

mathematical tractability. Also, one might argue that nucleation cannot occur too close to the edge since polymer concentration is high. In addition, the inflation stage may be self-symmetrizing since an off-center bubble will grow most rapidly in the direction of lower polymer concentration where the local solution viscosity will be lower.

The mathematical modeling of the process is broken down into three time regimes. In the first, surface evaporation of solvent predominates, and thus the process is external mass transfer controlled. As the polymer concentration builds at the surface, the process becomes dominated by internal mass transport of the solvent to the surface through the polymer-rich layer. The third time regime starts with bubble nucleation. Numerical solution of the coupled heat and mass transfer equations allows one to follow the time evolution of the drop, including quantities such as the drop diameter and the radial dependence of the drop temperature and polymer concentration. Some results are presented in Figure 2 for a 400 μm diameter droplet, initially 4.5 wt % PS (M_w = 100K) in dichloromethane, falling in a 200 °C heated drop tower. In Figure 2a, the droplet radius (left scale) and average droplet temperature (right scale) are shown as a function of time. Note that the droplet radius contracts rapidly due to evaporation until bubble nucleation. The average droplet temperature also declines rapidly initially but then remains constant until the diffusion of solvent to the surface is impeded and the droplet begins to heat. The temperature and polymer concentration gradients are shown in Figure 2b at three different times. Most notable here is that the polymer diffusion is sufficiently slow that even up to the time it takes the center of the drop to heat to 40 °C, the boiling point of dichloromethane, the inner third of the droplet has not yet sensed the high concentration of polymer at the surface. Thus the process is in fact governed by the rate of solvent diffusion through this increasingly concentrated surface layer.

It is also possible to prepare mandrels that are doped with small concentrations of higher-Z atoms that are used for diagnosing aspects of ICF implosion experiments.[6,7,8] These doped mandrels are prepared from polystyrene or polystyrene-like copolymers in which the desired diagnostic atom has been covalently bound to the polymer chain. Chlorine doping is accomplished by preparing the microshells from a copolymer of styrene and either *p*-chlorostyrene or *p*-chloromethylstyrene. Bromine and iodine doping[9] are accomplished by *para* halogenating (to the degree desired) monodisperse 100K PS with molecular bromine or trifluoroacetoxyhypoiodite . An iron-doped polymer suitable for drop tower use has been prepared by copolymerizing vinylferrocene and styrene.[10] Chromium doping involves reacting chromium hexacarbonyl with polystyrene to coordinate the chromium tricarbonyl adduct to the ring.[11] Recently we have also been able to prepare titanium-doped shells from a copolymer of styrene and a titanium triisopropoxide derivative of methacrylic acid. In all these cases the doping level can be set either by controlling the degree of reaction of the dopant group with the PS (for Br, I, and Cr) or by controlling the monomer mixture in those routes that involve copolymerization (Cl, Fe, and Ti).

CAPSULE SURFACE CHARACTERIZATION

The capsule surface finish has a large effect on the quality of an ICF implosion, and a significant fraction of the current ICF experiments conducted at LLNL are designed to study these effects. Surface perturbations on the capsule surface lead to Rayleigh-Taylor instability during the ablation phase of the implosion as the dense outer surface pushes against the less dense interior.[12] This manifests itself in a mode-dependent growth of the perturbations that feeds through to the inner wall. The mode-dependence of the growth is sensitive to capsule design (size, wall thickness, and ablator dopant level) and the laser drive, but in most cases the growth of low-mode, long-wavelength features is most severe. Figure 3 shows a cartoon version of the process. On the left side of Figure 3, the capsule before implosion is shown with an amplitude for the nth surface mode of a_n, and on the right side after compression the amplitude of that mode has grown to A_n. The mode dependence of the growth factor, $G(n) = A_n/a_n$, can be calculated from the numerical modeling of ICF implosions and generally peaks between modes 15 and 40, with magnitudes as high as several hundred for current experiments. This mode growth and related feed through leads to a mixing of the inner capsule wall with the fuel. This degrades the compression, cools the fuel, and in severe cases can lead to the complete break-up of the capsule symmetry. As an example consider that a growth factor 300 mode with a 500 Å amplitude on the outside of a 500 μm diameter capsule will grow to a 15 μm amplitude near "bang time" when the capsule diameter has been reduced to perhaps 50 μm.

Because of the mode sensitivity to growth, it is important to be able to characterize the capsule surface perturbations over length scales up to 100's of μms with a vertical resolution of at least 10 Å. At LLNL an atomic force microscope (AFM) based "Sphere Mapper" has been developed for this purpose. The details of the device have been reported elsewhere[13] and will only be briefly summarized here. The capsule is supported on a vacuum chuck connected to an air bearing rotor. Its equator is positioned next to a stand-alone AFM head and the capsule is rotated while the AFM records height measurements at 3600 evenly spaced points. Generally three equatorial traces are taken, at the equator and at 40 μm above and below it, and then the capsule is rotated 90 degrees and the process repeated. One additional 90 degree rotation gives us three orthogonal sets of three traces. Typical trace data for a 1000 μm diameter precision silicon nitride ball bearing and a 435 μm diameter titanium-doped microshell are shown on the left side of Figure 4. Note that the ball bearing is extremely spherical but relatively rough on a short length scale. In contrast, the microshell shows a significant (by comparison) asymmetry but is locally extremely smooth. Also notable on this microshell are some intermediate length scale perturbations, 20 to perhaps 100 μm in breadth, with amplitudes of 10 to 50 nm. It is worth noting that these are the kind of surface perturbations that are associated with large growth factors, and that the mode 2 asymmetry with perhaps a 200 nm amplitude has little effect on an ICF implosion.

To properly analyze the trace data to obtain the amplitudes of the different surface perturbation modes, each trace is Fourier transformed and the squares of the amplitudes of each mode are averaged for the (typically) nine traces. The resulting mode spectrum of the squared amplitudes is called the power spectrum, and the data

from the ball bearing and titanium-doped microshell (nine traces each) are displayed in this form on the right side of Figure 4. The short-length-scale roughness of the ball bearing manifests itself in the power at higher modes in contrast with the microshell which shows higher power at low modes due to its asymmetry and long-lengthscale roughness.

Another illustrative example of the power of this type of analysis is displayed in Figure 5. Before the development of the Sphere Mapper, chlorine-doped mandrels for diagnostic experiments were produced by blending pure polystyrene with pure poly(*p*-chlorostyrene) to produce a blend at the desire dopant level. The top left of Figure 5 shows representative traces from some of these shells. The visible oscillations have amplitudes of 100's of nm, but with wavelengths of 100's of μm, and thus were not detectable using standard optical techniques. In the bottom left of Figure 5 are shown representative traces from a microshell prepared from a styrene - *p*-chloromethylstyrene copolymer which had the same (1 atom %) level of Cl doping as the blend. Clearly the long wavelength roughness is absent, and the traces in fact look indistinguishable from traces one obtains from undoped PS shells. This conclusion is more clear on the right side of Figure 5, which shows the power spectra from these shells along with the spectrum from an undoped shell. Note that the blended shells exhibit more than a factor of ten more power in the critical low modes between 10 and 40.

SURFACE MODIFICATION

To study the effects of capsule surface finish on capsule performance, it was necessary to develop ways of controllably varying the outer capsule topology. This was initially done by making use of the fact that the plasma polymer coating is conformal, and that deliberate perturbations on the surface of the PVA layer would manifest themselves in perturbations on the outside of the finished capsule. Because of the omni-directional nature of the conformal plasma polymer coating, perturbations on the surface of the PVA layer of height a give rise to bumps on the outer capsule surface of height a but with width of approximately $2\sqrt{2at}$, where t is the thickness of the plasma polymer coating and the initial defect width is small compared to the coating thickness.[14] Our approach to roughening the PVA layer prior to coating was to include 2 to 4 μm diameter solid polystyrene microspheres in the PVA coating solution. These microspheres were thus incorporated into the PVA coating providing a bumpy texture which produced a bumpy surface on the completed capsule. A cartoon version of the capsule structure is shown on the left side of Figure 6.

The incorporation of a dopant in the polystyrene mandrel allows one to diagnose spectroscopically the degree of "mix" of the inner capsule wall with the fuel due to the growth of surface perturbations. This is shown on the right side of Figure 6. For a smooth capsule there is little mix and the inner wall is kept largely free of the hot fuel, resulting in only a small chlorine emission relative to the emission from the Ar dopant in the fuel. However for the rough capsule the feed through leads to significant mix of the inner capsule wall with the hot fuel, resulting in a much greater chlorine emission signal.

The use of polystyrene seeds in the PVA layer as a mechanism to produce roughened capsule surfaces offers only modest control over the degree of roughness. Recently we have developed the capability of using an Ar-F excimer laser to precisely ablate pits with depths from 0.1 to 2 μm and widths up to 100 μm on capsule surfaces.[15] The capsule positioner allows placement accuracy to ± 2.5 μm. Knowledge of the size and location of the ablated pits on the capsule surface allows one to calculate the expected power spectrum, which can be checked with the Sphere Mapper. In Figure 7, we show in the inset a 0.5 mm diameter plasma polymer-coated capsule with 200 randomly-placed ablated pits, each roughly 75 μm in diameter. The power spectra shown are for two capsules, one with 0.3 μm deep pits and one with 1.0 μm deep pits. Both the measured (data) power spectra obtained with the sphere mapper and the expected (calculated) spectra based on the known placement of the pits are shown. The experimental and calculated spectra agree well except for the 0.3 μm pits at very low modes, where the capsule asymmetry is dominant. The success of this controlled surface ablation technique for roughening capsules to a predetermined extent for studies of mix has proved extremely valuable in developing our understanding of hydrodynamic instability during an implosion due to capsule surface perturbations.

FUTURE ICF CAPSULE NEEDS

As the ICF community develops more powerful laser drivers, the capsules used in these experiments will need to be larger. The Omega Upgrade ICF facility at the University of Rochester's Laboratory for Laser Energetics will come on line in the spring of 1995 and will require capsules that are roughly 1 mm in diameter. The National Ignition Facility, with a planned completion date shortly after the year 2000, will require 2 mm diameter capsules. For both of these capsules the surface finish and symmetry requirements will be at least as stringent as for the current capsules. The production of these new capsules depends on the development of new technology, since the existing drop tower technology is limited to approximately 500 μm capsules, due largely to the large heat and mass transfers necessary.

There are, however, a number of alternative technologies that hold promise for delivering larger capsules. Perhaps the most widely developed is microencapsulation.[16] In this method a water droplet is encapsulated by a polystyrene solution, and this encapsulated droplet is suspended in an aqueous phase. The organic solvent containing the polystyrene slowly dissipates into the aqueous phase leaving behind a polystyrene shell. This approach to shell manufacture has been used for a number of years for microshell production at the Institute for Laser Engineering (ILE) at Osaka University and also for the Omega system at the University of Rochester. In both cases the typical shells produced for ICF targets are significantly less than 1 mm. Work aimed at extending this technology to 1 to 2 mm capsules using a controlled mass encapsulation technique is reported by Boone, *et. al.*[17] elsewhere in this volume.

The target fabrication group at the Lebedev Institute in Moscow has historically made plastic shells by drop tower techniques for Russian ICF experiments using particles of solid plastic frit infused with a volatile organic blowing agent. One advantage of this approach for larger shells is that there is much less mass and heat

transfer necessary compared with solution drop tower techniques. Using these techniques they have been able to routinely prepare 1 mm capsules with good symmetry and surface finish. For larger shells there is concern that the hydrodynamic interaction of the falling hot capsule with the surrounding atmosphere will lead to distortions. To remedy this problem, as well as to maximize heat transfer in the minimum column length, they have developed the concept of a "Ballistic Furnace." The polymer seed material is injected up into the column so that it blows into a hollow shell at the trajectory apex when the hydrodynamic interactions with the surrounding media are at a minimum. Some of the details of this approach are reported in this volume.[18]

A route to larger shells as well as unique capsules for current ICF experiments is being developed at LLNL based on the use of a decomposable mandrel.[19] Briefly, the method makes use of the fact that poly(α-methylstyrene) thermally decomposes to monomer at a relatively low temperature. It is possible to prepare very symmetric and smooth solid beads of this polymer at sizes up to several μms, overcoat them with a thin layer of plasma polymer, and then heat them to drive off the polymer mandrel leaving a symmetric shell of the desired size. This method also provides a route for the production of "designer" capsules for the current ICF program. For example, a 30 Å thick Ti metal layer can be applied to the polymer bead by sputtering before plasma polymer overcoating to yield a shell with a thin metal layer on the inside of the capsule . The method may also be useful for preparing capsules with inner surface roughness by using the laser ablation technology discussed earlier to precisely roughen the polymer bead before overcoating.

Future ICF target designs for both the Omega and NIF facilities call for cryogenic targets in which a 100 μm thick, symmetric, solid or liquid hydrogenic fuel layer is present on the inside of the capsule wall. One route to accomplishing this is to prepare capsules with a low-density organic foam liner to hold the fuel inside the full density plastic shell. Capsules of this type have been developed at Osaka University using microencapsulation techniques.[20] The method involves microencapsulating a water droplet with an oil layer containing a multifunctional polymerizable monomer at 3 to 5 wt %. Polymerization in the oil phase is then initiated resulting in the formation of a heavily crosslinked foam network in the oil phase. A full density overcoat is applied by filling this foam shell with a solution of an organic diacid chloride and suspending it in an aqueous bath of a polymer with pendant alcohol groups such as hydroxyethylcellulose or poly(vinylphenol). This results in an interfacial reaction crosslinking the aqueous polymer at the capsule surface and thus the formation of a full-density layer. Attempts to modify this technology to meeting our national ICF needs has recently been reported,[21] and elsewhere in this volume a report on the current capabilities at Osaka University are reported.[22]

SUMMARY

The production of ICF capsules, with their very stringent symmetry and surface finish requirements, and the characterization of these capsules represent major materials science challenges. The quality of the capsule is largely dependent upon the quality of the thin-walled plastic microshell mandrel around which it is built. The

characterization requirements for the capsules have led to the development of new and unique capabilities for measuring their surface topology, and the scientific interest in studying the effects of surface topology on the implosion dynamics has led to methods of precisely modifying the capsule surface finish. Future ICF targets will require larger capsules, and technologies designed to meeting this need are now being developed.

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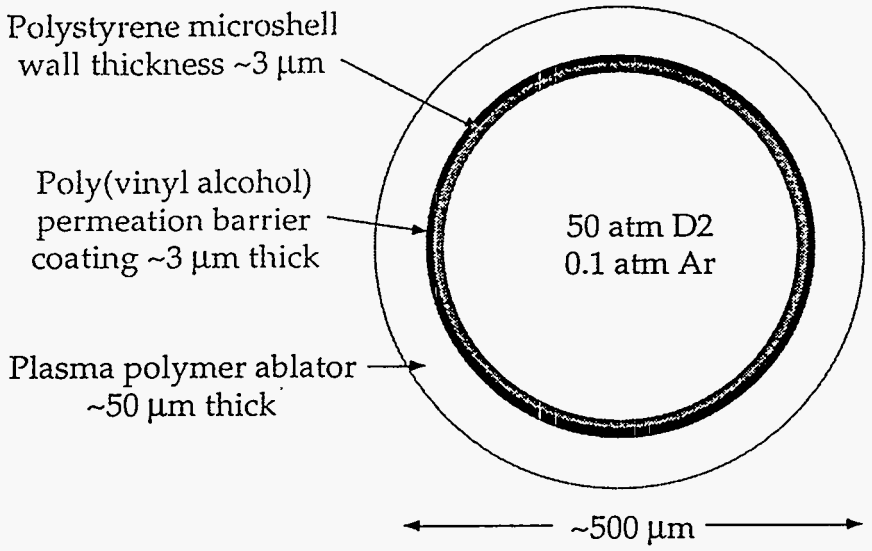

Figure 1. A typical ICF capsule is composed of three layers.

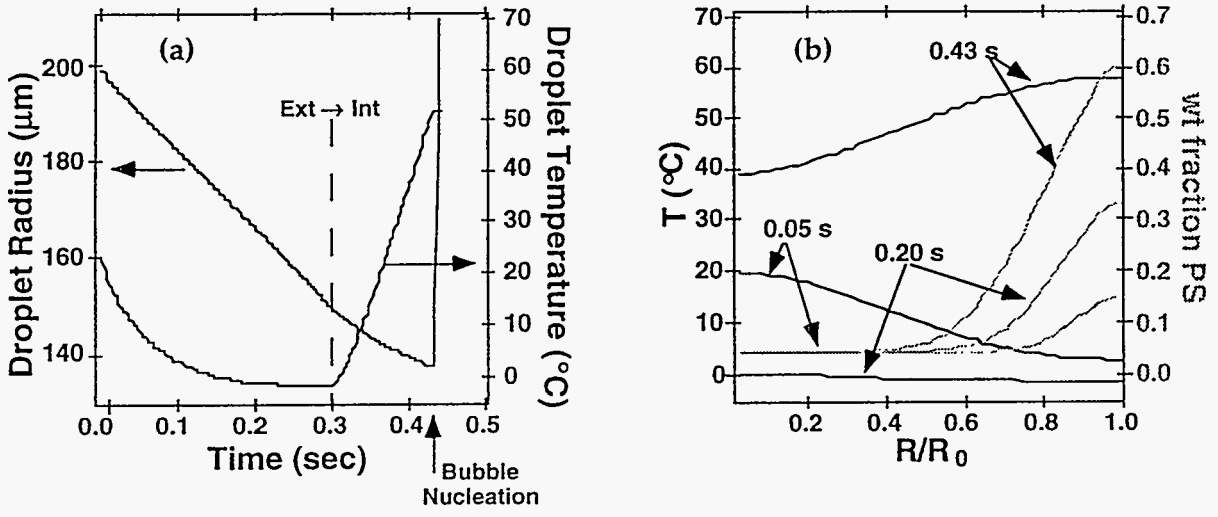

Figure 2. On left (a) is shown modeling results for droplet radius and average temperature as a function of time. The modeling transition between external and internal mass transfer control is indicated by the dashed line. On the right (b) is shown the radial temperature (black, left scale) and concentration gradients (gray, right scale) at 3 different times.

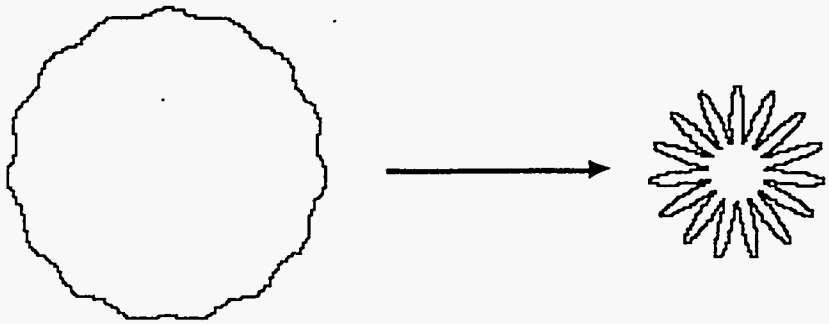

Figure 3. During an implosion there is a mode-dependent growth of surface perturbations. The growth factor for mode n is $G(n) = A_n/a_n$.

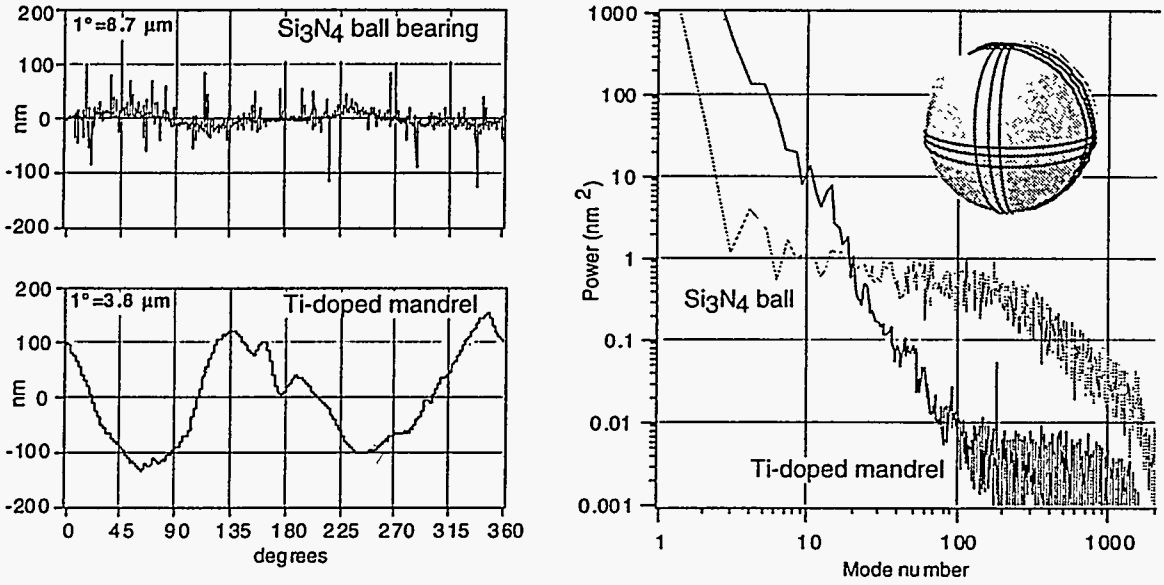

Figure 4. On the left are shown representative Sphere Mapper traces of a Si_3N_4 ball bearing and a Ti-doped mandrel. On the right are the respective power spectra averaged over 9 traces per ball as shown in the inset.

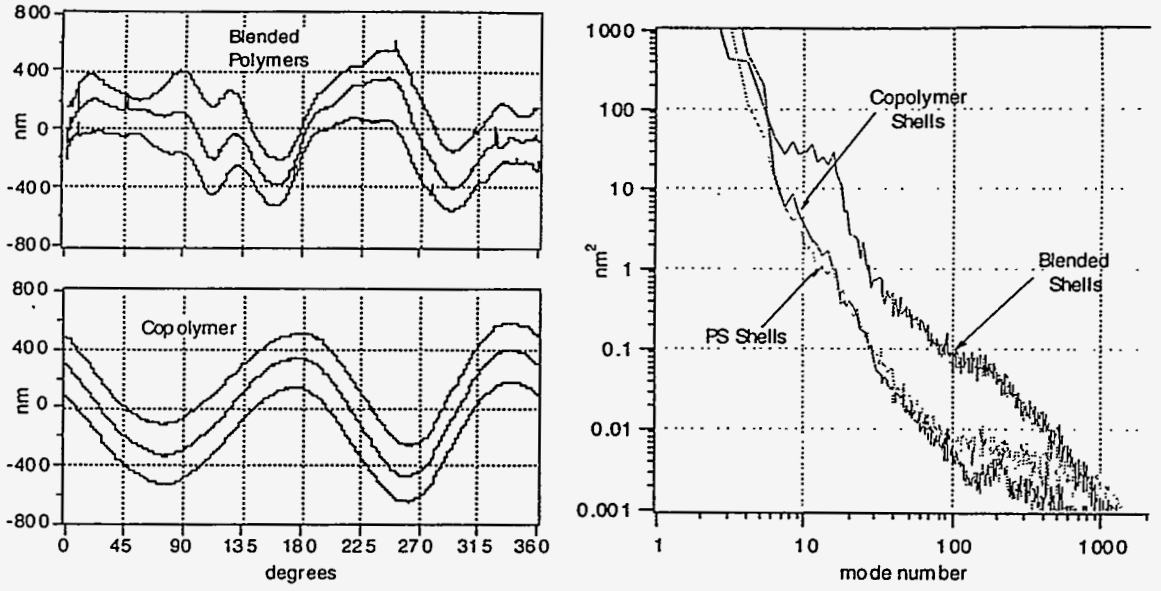

Figure 5. On the left are shown Sphere Mapper traces for Cl-doped microshells made from blended polymers (top) and a single copolymer. The power spectra for these shells, as well as for an undoped PS shell, are shown on the right.

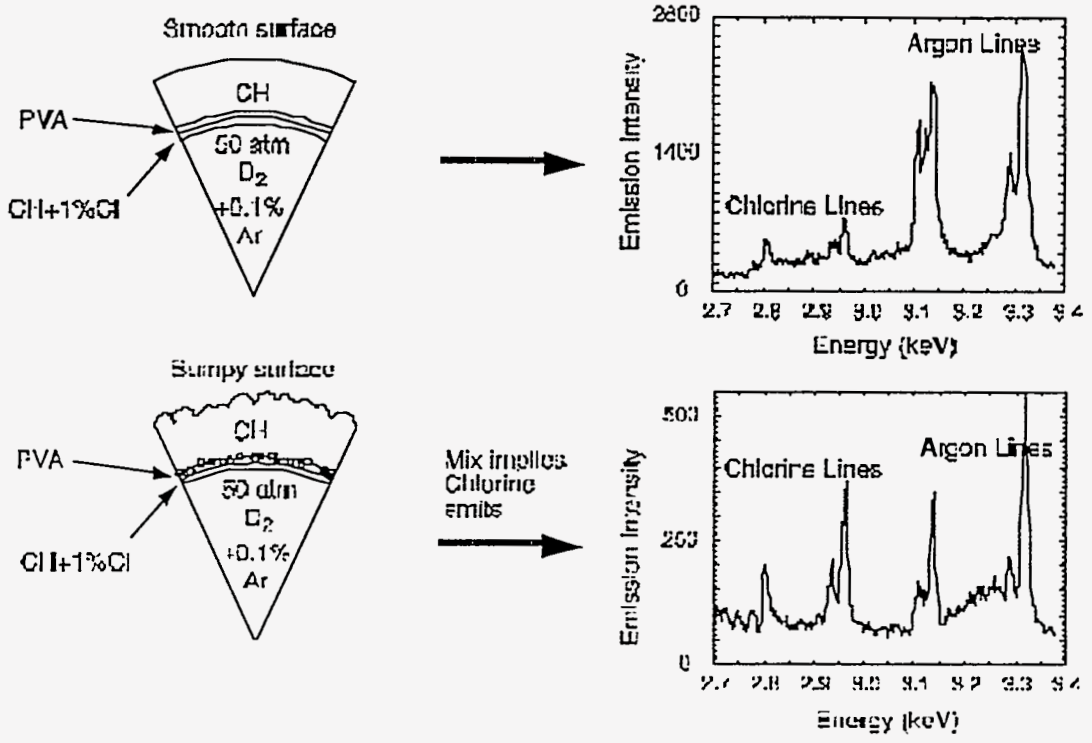

Figure 6. On the left are shown sketches of a smooth and bumpy capsules. On the right is shown the spectra obtained during the implosion of these capsules. The bumpy target leads to more mix and thus a more intense chlorine emission.

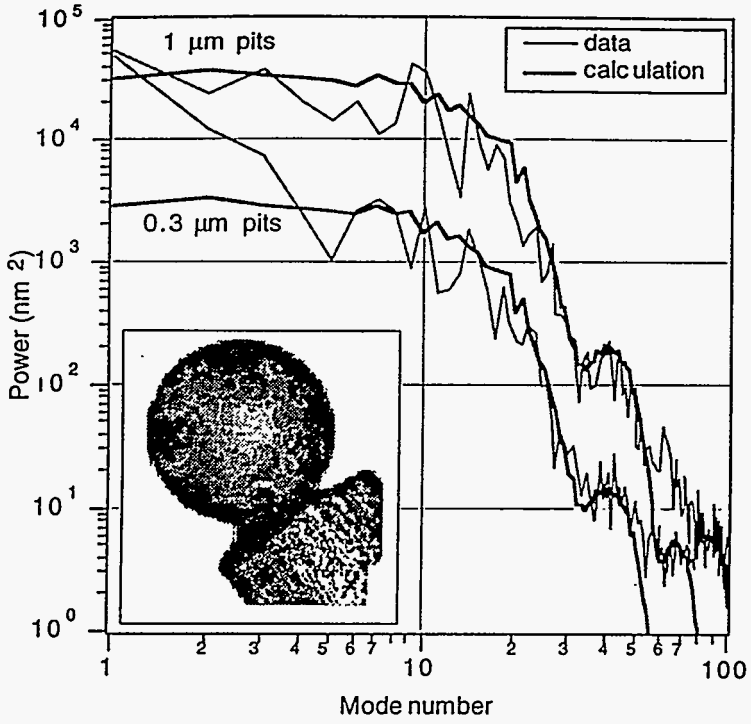

Figure 7. Shown are the measured and calculated power spectra for a capsule with 200 randomly placed ablated pits. The inset shows a SEM picture of the capsule.