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## Hollow poly(alpha-methylstyrene) shells for inertial confinement fusion targets

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**Abstract.** Using a low-temperature cationic polymerization technique, a number of poly(alpha-methylstyrene) samples with molecular masses of 300000–400000 were prepared, and their structural parameters were examined via nuclear magnetic resonance and X-ray diffraction techniques. It was found that all synthesized polymers display a stereoregular syndiotactic structure of macro chains and are amorphous. Using these polymers and the microencapsulation technique, hollow shells were obtained that were 2.4 mm in diameter and had a wall thickness of 10 to 60 µm. The quality of walls and surfaces of the shells was analyzed using scanning electron microscopy and optical profilometry. The hollow shells are intended for the preparation of inertial confinement fusion targets and for the laser plasma research.

### 1. Introduction

Preparation of high-quality spherical hollow shells is one of the most important technical problems in the development of targets for the Inertial Confinement Fusion (ICF) systems. Currently, spherical beryllium hollow shells that meet very strict requirements with respect to their geometry and surface quality are considered as the most promising approach for preparing useful internal confinement targets. The preparation of these metal shells involves two major steps: deposition of metal vapors on the surface of polymeric shells and thermal elimination of the initial polymeric material. Accordingly, one must first develop a technique for preparing polymeric shells with a perfect spherical shape and a defect-free surface. Poly(alpha-methylstyrene) (PAMS) has been found [1–3] to be one of the most useful polymers. Still, it is characteristic of all polymeric materials that every batch of PAMS will exhibit its individual physical properties depending on the synthesis conditions (nature of the catalyst, temperature and other process parameters). Nevertheless, the specificity of the final targets is determined by the polymer precursor used for the preparation of hollow shells to fit into a well-defined range of thermal and mechanical properties. Thus, the polymer must degrade completely with the evolution of only volatile products during the thermal treatment at 250–300 °C, while the thin walls of the polymer shell (10–50 µm) must exhibit sufficient rigidity and withstand the process of elimination of the “internal” water that initially fills the shell.



Accordingly, the present work is aimed at the synthesis of several batches of PAMS by varying the synthesis conditions, optimization of parameters of the droplet formation stage of the fabrication process of thin-wall hollow shells composed of the obtained polymers and, finally, examination of the main quality characteristics of the shells.

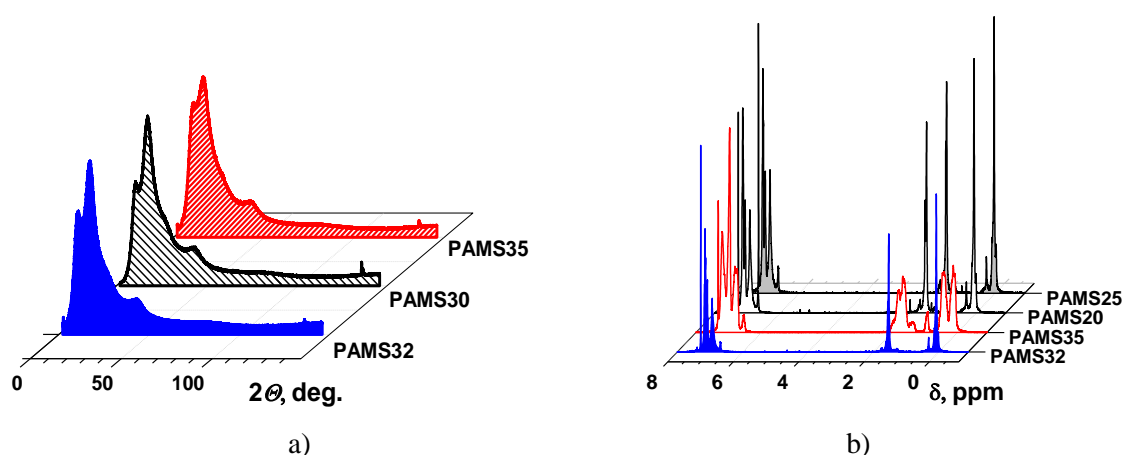
## 2. Experimental setup

High-molecular-weight PAMS is known as a useful starting material for the preparation of Inertial Confinement Fusion Targets [4–6]. To produce the material of required quality, the low-temperature cationic polymerization approach was chosen [7]. Examination of the obtained polymer structure was performed using proton nuclear magnetic resonance (NMR) and X-ray diffraction techniques. Proton magnetic resonance spectra were recorded in a deuterated chloroform solution of the PAMS samples (at a concentration of 1%) using a Bruker AV 400 instrument (400.13 MHz for  $^1\text{H}$ ). Diffraction curves of the pressed at 205 °C and 1000 atm PAMS samples were recorded using a Rigaku MiniFlex 600 diffractometer with a copper cathode in the range of 20 angles from 3 to 150 at a rate of 0.3 degree/min. The final hollow shells, which were prepared from the polymer batches, were examined using scanning electron microscopy (SEM) and optical profilometry techniques. Micrographs of the shell surface and fracture chips were obtained using an SEM instrument Jeol JSM-7001F with a Schottky cathode. Profiles of the shell surface were examined using a Zygo NewView profilometer equipped with a 10× micro interferometer.

## 3. Results and Discussion

The choice of cationic polymerization approach for high molecular weight PAMS is justified by a lower sensibility of this method to eventual contaminations in the starting monomer, compared with anionic polymerization. By varying the synthesis conditions, a number of PAMS batches were obtained that differed from each other in chain structure and physical properties, mainly in the molecular weight distribution characteristics (molecular weight from 300000 to 400000). In particular, the samples PAMS20, 25, 30, 32 were obtained using boron trifluoride as a catalyst (PAMS31 – with boron trichloride) at  $-70$  to  $-76$  °C (PAMS31  $-80$  °C). The PAMS35 sample was obtained by catalyzing anionic polymerization with butyl lithium at  $-79$  to  $-85$  °C.

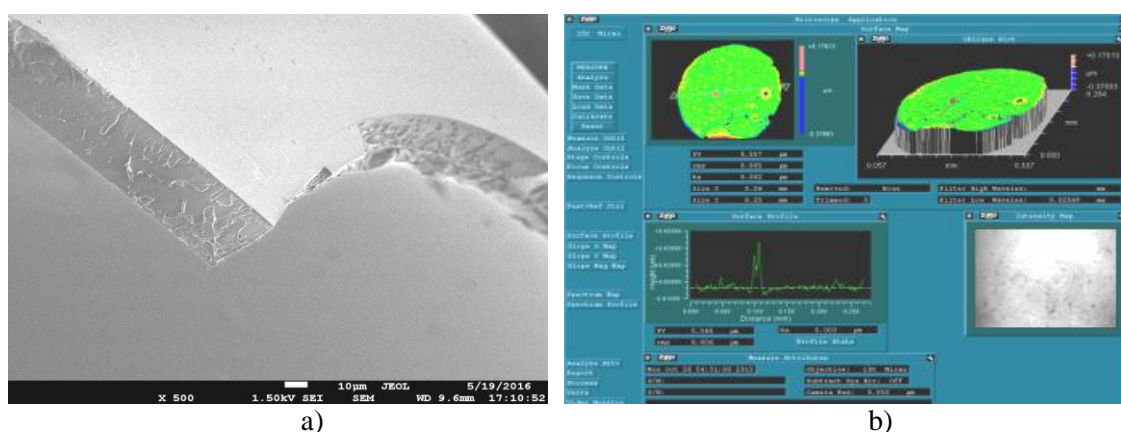
The examination of the synthesized polymer structures using the X-ray structure analysis technique (figure 1, a) resulted in a conclusion that all materials are amorphous because their diffraction curves display only two reflexes, which is characteristic of amorphous polymers of this type.



**Figure 1.** Results of structural investigations of the synthesized polymers, (a) diffraction curves and (b)  $^1\text{H}$  NMR spectra.

Besides, the  $^1\text{H}$  NMR spectra of all samples, which were obtained using the cationic polymerization approach (figure 1b), reveal a resonance signal of methyl protons near 0.3 ppm, which points to the syndiotactic arrangement of monomer units in the PAMS chain [8]. Thus, these polymers are stereoregular. This is contrary to atactic PAMS, which results from anionic polymerization [7]. The NMR spectrum of the PAMS35 sample of that type is characteristic of atactic structure of polymer chains [9].

Hollow shells that were produced from the synthesized PAMS samples were prepared using the microencapsulation technique [10]. Solutions of the polymers, which were injected into a special system of capillaries, had 10 and 20% concentrations and were based on various solvents: benzene, toluene, ortho-xylene, ethylene dichloride, tetrachloroethane, dichloropropane, fluorobenzene, and fluorotoluene. The aqueous phase, which was used as a medium to harden polymer shells by removing the solvent during the careful heating of the mixture, contained polyvinyl alcohol at a concentration of 1.5–2.0% and various ammonium salts at a similar concentration of 1–2%. The hardening of the shell droplets was carried out under a number of combinations of temperature and time conditions. Thus, the examination of solid shells, which was obtained using a profilometer, revealed a substantial difference in the surface topology and wall-forming polymer structure, which depended on the preparation protocols (figure 2).



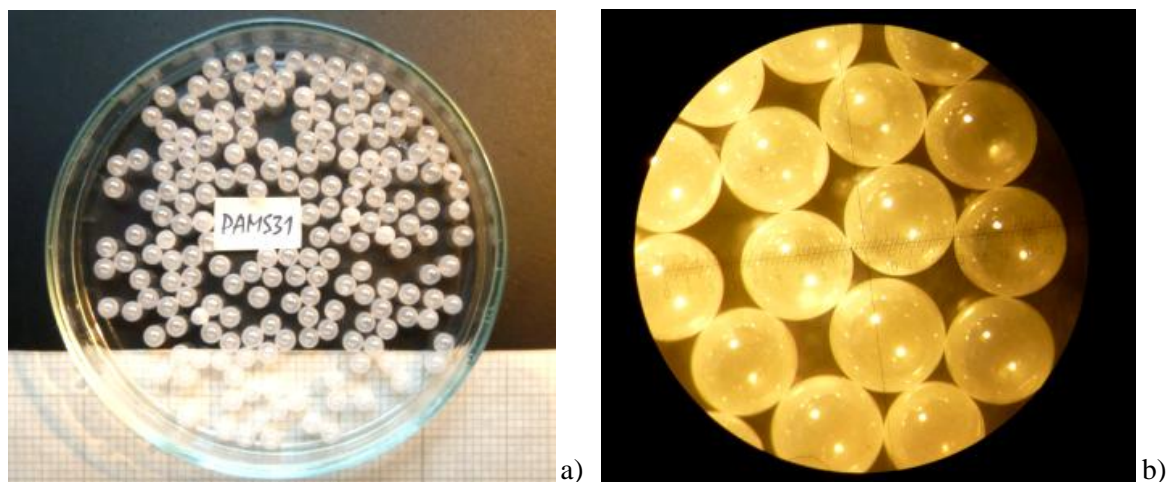
**Figure 2.** SEM micrograph of the shell fracture (a) and profilometer study of the shell surface (b) of the product PAMS30.

The shell-wall cross-section was studied to obtain uniform spherical shells. The surface of the cut edge revealed a different character of the local mechanical destruction of the polymer phase. Specifically, we came across either a ductile or a brittle character for different routes. Some shell batches have small inclusions of ammonium nitrate (as measured via Energy-dispersive X-ray spectroscopy) in the polymer layer or on its surface if ammonium nitrate was present in the aqueous phase. SEM micrographs of other batches display bubbles of various sizes in the polymer shell wall. On the surface of shells, these bubbles may form craters of different depths or distort the surface in one or another way if the bubbles remain close to the surface. There are fabrication routes that reveal a higher bubble concentration and foam structure of the wall.

Shells that have thin walls, less than 10–20  $\mu\text{m}$ , had the smoothest surface. However, the profilometer and micrography measurements of the entire shell demonstrated that shells with very thin walls, less than 10  $\mu\text{m}$ , were mostly non-spherical, rugby ball-shaped. The smallest deviation from the regular spherical form was characteristic of the shells with thick walls, approximately 50–70  $\mu\text{m}$ . Regardless of the aims of the present work, the rugby ball-shaped shells can find their use in the non-symmetric irradiation schemes and for particle source optimization.

The process of shell hardening that freezes the geometric form of the shell droplet is governed by a superposition of several factors. Specifically, these factors are the rate of diffusion of solvent molecules from the polymer solution into the aqueous environment media, rate of concomitant

changes of the rheological properties of the polymer phase of the walls, variation of the surface tension of the two-phase system, deformational resistance of the hardening droplet shell and some other factors. Therefore, it was necessary to conduct many experiments, where different compositions of initial polymer solutions and aqueous phase, temperature and time allowed for the hardening of the shells were systematically examined. Finally, rigorous optimization of the preparation protocols allowed to obtain shells with a regular spherical form and high-quality surfaces (figure 3).



**Figure 3.** General view of hollow shells prepared from poly(alpha-methylstyrene) PAMS31 (a), and the microphotograph of these shells at 16× (b).

#### 4. Conclusions

Based on poly(alpha-methylstyrene) with a molecular mass of 300000–400000 and using low temperature cationic polymerization of the monomer, hollow shells were obtained for the further use in ICF targets. By systematically varying the polymer synthesis conditions and technical protocols of the microencapsulation process, hollow spherical shells were obtained with a diameter of up to 2.4 mm and a shell thickness of 10 to 60  $\mu\text{m}$ . The rugby ball-shaped shells can also find their possible applications in laser plasma research.

#### Acknowledgments

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