The polystyrene microsphere filling with hydrogen isotopes through the fill tube with consequent freezing

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Abstract. Process of spherical polystyrene capsules filling with hydrogen isotopes through the fill tube for the purpose of a cryogenic target building is described. The scheme of the stand for researches and a technique of carrying out of experiments is represented. Results of capsules filling and subsequent freezing for protium, deuterium and protium-deuterium mixture are shown.

1. Introduction
Nowadays megajoule energy laser facility for carrying out inertial confinement fusion experiments is being built in RFNC-VNIIEF. The idea of inertial confinement fusion is based on possibility of symmetric compression and heating up to thermonuclear temperatures of small amount of the fuel contained in the target in nanoseconds. The elementary fuel target represents a polymeric spherical capsule with DT fuel cryogenic layer of the specified thickness, generated on its internal surface. Development of cryogenic targets production technology includes some steps one of which is working out of spherical capsule filling procedure with hydrogen isotopes through a fill tube [1].

2. Research stand
The stand for targets research at low temperatures consists of research cryostat, systems of simultaneous pumping out of gas channels, helium and hydrogen isotopes supplying system, the equipment for measurement and control of temperature, experimental boxes and optical system of visual control.

1.1. The research cryostat
Optical cryostat with system of cooling on the basis of two-stage pulse tube cryo-refrigerator with refrigeration capacity 1 Watt at 4.2 K is used for carrying out researches. Main parts of research cryostat are cryo-refrigerator, helium compressor, helium supplying lines [2].

The cryo-refrigerator produces different temperatures: 45 K at the first stage and 4.2 K at the second stage. The necessary temperature for liquefaction process research with the subsequent freezing of hydrogen isotopes in a spherical capsule (from 10 K to 20 K) is reached on a thermoadjustable object stage mounted at the second stage cold station.

1.2. Gas channels pumping out system, helium and hydrogen isotopes supplying system
The system of simultaneous pumping out of cryostat working volume and gas channels, with the subsequent delivery of gases into the box and the polystyrene capsule [3, 4] has been developed and installed. The system arrangement is shown in figure 1.
System pumping out is performed with the help of backing and turbomolecular pumps in three stages: after pumping out of buffer volume hydrogen isotopes supply channels, heat exchange gas (helium) supply channels and cryostat working volume are evacuated accordingly. The given scheme of pumping out allows to reach necessary vacuum at the minimum quantity of the used pump equipment for pumping out of three systems isolated from each other. Besides, the pumping out system is integrated with helium and hydrogen isotopes supply systems that allows to carry out corresponding gases puffing in experimental box and polystyrene capsule at overlapping of certain valves.

2.3. Temperature control
Control of temperature of experimental assemblages is carried out by means of two sensors. Sensor DT1 is mounted within a stage 11 mm far from the capsule center (its indications are denoted by TA). The second sensor DT2 is mounted directly on the body of experimental box (TB). Sensors run in a range of temperatures from 1.4K to 325K with accuracy ±0.009K and ±0.001K at T=20K accordingly. Time delay of sensors data reading by the controller is 0.5s. Data are displayed on a personal computer.

It is necessary to maintain defined temperature for a long time at zone of research object for carrying out of experiments. This is carried out by a heater installed at a distance of 11 mm from the centre of a capsule. Temperature control is carried out by the controller (directly from the control panel or the remote personal computer), the average dispersion of temperature data values thus does not exceed 0.002K.
2.4. Experimental box
The experimental box represents a system of isolated gas volumes with different pressure (figure 2). The polystyrene capsule is fixed in the box–container which is mounted at cryostat working zone, pumped out to high vacuum. The box is filled with helium that equalizes polystyrene capsules surface temperature. The capsule is in turn filled with gaseous hydrogen isotopes under pressure of about 105 Pa. The primary goal during assembling is maintenance of tightness of box and fill tubes for supplying of operating gases (helium, hydrogen isotopes) taking into account their use at cryogenic temperatures [5]. Thus simplicity of box installation and reliability of its connections should be provided as the size of experimental assemblages are approximately 30x20x10 mm.

Some ways of manufacturing and assembly of experimental boxes have been developed and realized. Now the most technological variant in a form of box-cell which is presented in figure 3 is used.

![Figure 2. The scheme of experimental box.](image)

![Figure 3. The experimental box-cell.](image)

2.5. Visual control system
In order to get high quality image diode illumination microscope (the aperture 0.11, working distance 90 mm) is used allowing to observe an accurate contour of polystyrene capsules and subsequent liquefied and frozen hydrogen isotopes borders. The image is registered by a digital camera and is transferred to the personal computer.

3. Technique of carrying out experiments
Stages of carrying out experiments on hydrogen isotopes inlet through a fill tube in spherical polystyrene capsule with the subsequent freezing:

- cryostat volume and gases (helium, hydrogen isotopes) supplying systems pumping out with the help of backing and turbomolecular pumps to $10^{-3}$Pa;
- the procedure of experimental assemblage cooling to 30K;
- the puffing of helium as heat exchange gas into the box cavity;
- the puffing of hydrogen isotopes into the spherical polystyrene capsule;
- capsule cooling down to temperature below triple point with «gas-liquid-solid» phase transitions recording process on video.

In experiments on hydrogen isotopes liquefaction with the subsequent freezing hydrogen ($H_2$), deuterium ($D_2$) and mixture of hydrogen-deuterium (HD) are used [6]. Gas filling occurs in the capsule already mounted in the cryostat, fixed in the copper box. Temperature data is obtained by means of the
sensor placed on a surface of the box. Before gas puffing required temperature (T=30K) is achieved and is maintained by the controller. Then there is heat exchange gas puffing into the box and hydrogen isotopes puffing into the capsule. Gradual decrease of temperature (1K in 3-4 minutes), set by the controller program, prior to the beginning of liquefaction process in the capsule occurs. Process of liquid phase increase in the polystyrene capsule as a result of filling through a fill tube is presented in Figure 4.

![Figure 4. Process of spherical polystyrene capsule filling with liquid phase hydrogen isotopes.](image)

4. Results of experiments

Dynamics of hydrogen cryogenic layer behavior at different temperature near triple point is shown in Figure 5.1-5.3 (hydrogen), 6.1-6.3 (deuterium), 7.1-7.3 (hydrogen-deuterium mixture). Experiments were conducted in “box-cell” experimental assemblage. Polystyrene capsule diameter is $\varnothing 1,325$ mm, diameter of the glass fill tube is 56 microns.

Hydrogen liquefaction process in the capsule is shown in figure 5.1. Gas is condensed at $T_B=14.7K$ that corresponds to saturated vapor pressure value $P=10.6kPa$ in the capsule (the temperature of triple point is 13.96K). Rough estimate gives us that mass of condensed gas is $m_\text{H}_2=0.07$ mg. At temperature $T_B=14.6K$ we can observe transition of liquid phase in a solid one (figure 5.2) which has completely come to the end at $T_B=14.2K$ (figure 5.3).

The experiment with deuterium was carried out in the same way. The temperature of deuterium triple point is 18.7K and it is higher than that of hydrogen, therefore deuterium liquefaction process has begun at higher temperature $T_B=19.4K$, $P_{\text{sat},B} = 23.4kPa$ (figure 6.1). Calculated condensed gas mass value is $m_\text{D}_2=0.168$mg. With decrease of temperature to $T_B=19.27K$ deuterium freezing process on capsule internal surface has come to the end at $T_B=19.17K$ (figure 6.2, 6.3).
In a series of the experiments distinctions in protium and deuterium behaviour have been noticed. Freezing of heavy hydrogen isotope occurred all around on sphere inner surface while in a case of protium rather the most part of ice was formed in the bottom of microsphere. It was revealed that in experimental conditions uniformity of deuterium distribution in triple point between the top and the bottom pole of the spherical capsule is considerably higher. It is connected, possibly, with higher than of protium viscosity, more magnitude of surface tension coefficient near triple point ($\sigma_{D2}(18,5K) = 4,006 \cdot 10^{-3} \text{Н} \cdot \text{м}$, $\sigma_{H2}(13,95 = 3,004 \cdot 10^{-3} \text{Н} \cdot \text{м})$.

Temperature of hydrogen-deuterium mixture triple point is 16,6K, thereby phase transformations occur around that temperature. In figure 7.1 condensation beginning is shown at $T_A=16,05K$, $T_B=16,97K$, with temperature decrease to 16,74K process of crystallization has begun and at $T_B=16,6K$ all the liquid has transformed into solid phase (figure 7.2, 7.3). Calculated mass of condensed mixture is $m_{HD}=0,025$ mg.

5. Conclusion
The technology of spherical polysterene capsules filling by hydrogen isotopes through a fill tube has been developed.

Experiments on liquefaction of protium, deuterium and protium-deuterium mixture with the subsequent freezing on internal surface of the capsule are made. The technics of carrying out the experiment is worked through.

The given work is the initial stage of developing the technology of cryogenic targets development with smooth defined thickness homogeneous layer of solid fuel for carrying out experiments on laser confinement fusion.
References

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