

# Nanocellular foaming with rapid depressurization system from 100 MPa

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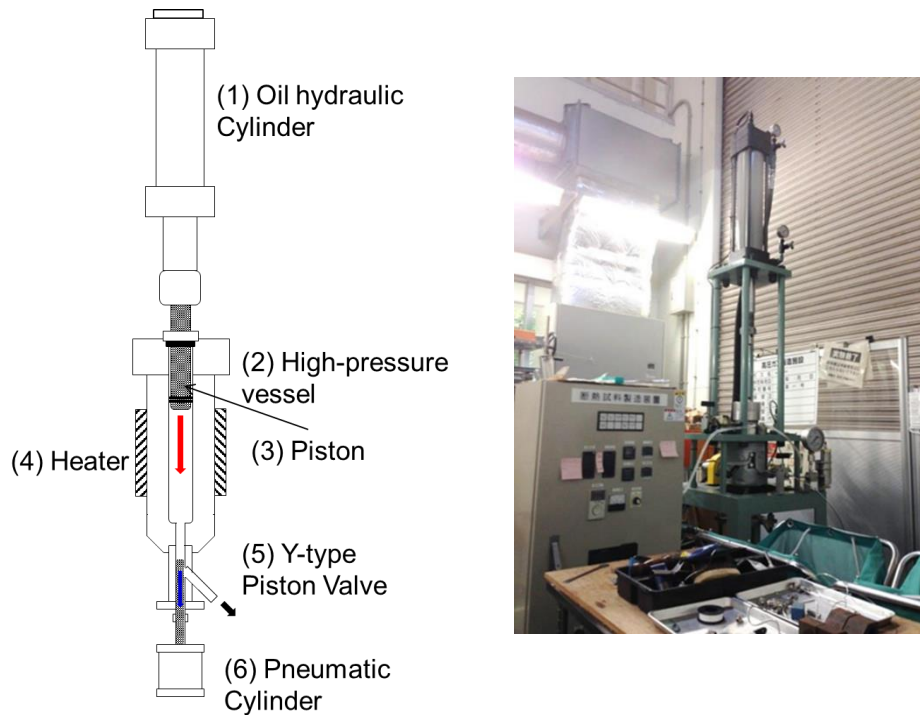
## INTRODUCTION

Polymer nanocellular foam with low density and with small cell size under 100 nm has been expected to be a transparent thermal insulating material of windows, which have a large impact to energy conservation. However, there are a lot of technical barriers to balance small cell size and low polymer density. When we follow classical nucleation model, high pressure drop rate at low is beneficial for survival of stable nuclei and for high cell density [1]. We have developed a new super high-pressure equipment with a new rapid depressurization system which is enable to operate at 100 MPa. Here we introduce our system and demonstrate nanocellular foaming by with supercritical carbon dioxide (CO<sub>2</sub>).

## EQUIPMENT and METHODS

A schematic diagram of the new system (Toyo Koatsu Co. Ltd.) is shown in Figure 1. The system is equipped with an oil hydraulic cylinder (1), volume valuable high-pressure system with vessel (340 cm<sup>3</sup>) (2) and piston (3), heater (4) and Y-type piston valve (5) for rapid depressurization and a pneumatic cylinder (6) to move the valve in very short period of time. The system is able to be used up to 100 MPa and 250°C for CO<sub>2</sub>.

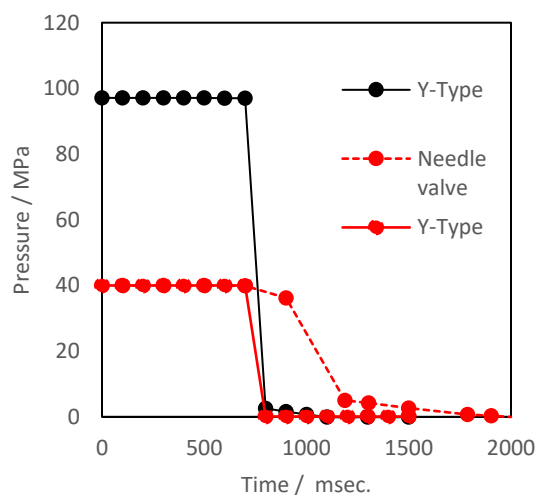
Poly (methyl methacrylate) (PMMA)(Aldrich, Mw=120,000 and 350,000) were employed for demonstration of polymer foaming with the system. Polymer foams were prepared between the range of 20 to 100 MPa at 60 °C. Foaming with small high pressure vessel (15 cm<sup>3</sup>) with needle valve (Swagelok SS-3NBS4) was used for comparison. Microstructures of foaming samples were evaluated by scanning electron microscopy (SEM).



**Figure 1 Schematic diagram of the high-pressure systems**

## RESULTS

Figure 2 shows a measured value of depressurization rate of the system. That rate on the small vessel with needle valve from 40 MPa is shown as a reference. The depressurization rate was confirmed to be large even though the system has a large volume of the vessel. The rate came up to 950 MPa/s at 100 MPa..



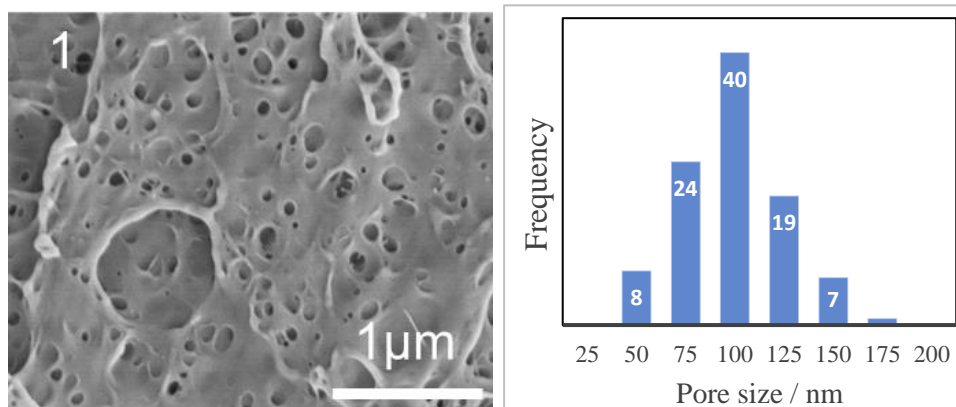
**Figure 2 Pressure profiles of the vessel when the Y-type valve is opened.**

Table 1 shows results for PMMA foaming with this system at 60°C from 40-100MPa.

Figure 3 shows SEM image of sample No. 1 with histogram of pore size distribution. Large solubility of CO<sub>2</sub> and plasticization effect at high pressure enabled to foam PMMA under low temperature condition. PMMA foam with 63% porosity and with mode pore size of 100 nm in diameter was successfully prepared by foaming from 100 MPa.

**Table 1 PMMA foaming with the high pressure system**

Sample No.	CO <sub>2</sub> Pressure MPa	Temp. °C	Bench Time hr.	Depress. Rate MPa/sec	Magnification ratio	Porosity
1	100	60	3	950	2.7	63%
2	80	60	3	800	2.1	52%
3	60	60	3	600	1.9	47%
4	40	60	3	400	1.8	44%



**Figure 3 SEM image of sample No. 1 in table 1 with histogram of pore size distribution (n=98)**

## CONCLUSION

We developed a new high-pressure system for rapid depressurization on polymer foaming process which is enable to operate at 100 MPa. Rapid depressurization rate up to 950 MPa / second was observed. Nano-cellular PMMA foams were prepared without using any nucleation agents. We will take advantage of the high pressure system on research and development of nano-cellular foaming.

## ACKNOWLEDGEMENTS

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## REFERNCES

[1] S. Costeux, CO<sub>2</sub>-Blown Nanocellular Foams, J. Appl. Polym. Sci., Vol. 131, 2014, p. 41293