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AUTHOR(S) AINSLIE T. YOUNG, MST-7

MASTER

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Los Alamos Los Alamos National Laboratory
Los Alamos, New Mexico 87545

MICROCELLULAR FOAMS VIA PHASE SEPARATION

by

Ainslie T. Young
Materials Science and Technology Division
Los Alamos National Laboratory
P.O. Box 1663
Los Alamos, NM 87545

EXTENDED ABSTRACT

A study of wide variety of processes for making plastic foams shows that phase separation processes for polymers from solutions offers the most viable methods for obtaining rigid plastic foams which met the physical requirements for fusion target designs. Four general phase separation methods have been shown to give polymer foams with densities less than 0.1 g/cm^3 and cell sizes of $30\mu\text{m}$ or less. These methods involve the utilization of non-solvent, chemical or thermal cooling processes to achieve a controlled phase separation wherein either two distinct phases are obtained where the polymer phase is a continuous phase or two bicontinuous phases are obtained where both the polymer and solvent are interpenetrating, continuous, labyrinthine phases. Subsequent removal of the solvent gives the final foam structure.

1. INTRODUCTION:

Typical high gain fusion targets for the laser fusion and particle beam fusion programs are simply a series of concentric spherical shells.

It is at this point that simplicity disappears. These targets are generally very small with overall dimensions of one millimeter or less and the tolerances put on these targets are on the micron scale. The components of a typical high gain fusion target are shown in Fig. 1.

One of the critical elements of such a multishell target is a cushion layer situated between tamper and ablator layers. This cushion, ideally a "structural void", can be either a full density plastic, gas, or rigid support foam. This report will attempt to provide an up-to-date review of the development of rigid support foams for these high gain target designs.

Generally, target designs have required that the density of such support foams be between 0.005 g/cm^3 and 0.1 g/cm^3 . Because foam thicknesses of 100 to $600 \mu\text{m}$ are required in these target designs, "cell sizes" have to be less than or equal to about $30 \mu\text{m}$ in order to minimize any instabilities during the hydrodynamic implosion process. These requirements eliminate virtually all commercially "blown" plastic foams for application in fusion targets. Such commercial foams can be obtained with densities down to 0.01 g/cm^3 but with cell sizes in the hundreds of micrometers. But, usually one has to prepare blown plastic foams in the density range of about 0.1 g/cm^3 before cell sizes in the desired range are obtained.

II. PHASE SEPARATION PROCESSES:

In the late 1970s several laboratories began to search for alternate routes to obtaining rigid plastic foams which would meet high gain target design requirements. Early in this study it seemed that phase separation processes which had been developed for the large scale production of anisotropic membranes¹ offered a route to rigid plastic foams with

the desired properties. In a generic process a polymer which is dissolved in a suitable solvent is induced to separate from the solvent in a controlled manner such that the original solvent accumulated in discrete zones in the polymer matrix forming either two distinct phases or two bicontinuous phases. Subsequent removal of the solvent phase usually left a rigid plastic "foam" structure with a density less than that of the bulk polymer and a void structure on a micrometer scale. Membrane technology tends to be less concerned with the final density of the rigid, porous foams than with the "pore size" of the final membrane. Herein lay the challenge to the low-density, microcellular scientist who was interested in both low-density and small cell size.

Another distinct advantage of a polymer solution phase separation process for fusion targets is that the final foam product usually has a 100% porous structure. This aspect of the foam offered the target designs the option of having either a completely evacuated polymer foam cushion layer or, in contrast, a rigid cushion layer filled with either a diagnostic or fuel gas.

Rigid support foams suitable for application to fusion target designs have been prepared by phase separation processes of polymer and solvents by four separate methods. These methods explore a variety of physical and chemical approaches which involve the formation of two phase systems, one of the phases being composed of a rigid polymeric structure. Such a variety of methods demonstrates the broad, general usefulness phase separation process have in preparing low-density, microporous, rigid foams. I will choose to label these four methods as follows:

1. Non-Solvent Induced Separation of Solvent and Polymer Phases.
2. Chemically Induced Separation of Solvent and Polymer Phases.

3. Thermally Induced Gelation/Crystallization.

4. Thermally Induced Spinodal Decomposition.

Each method will be discussed in more detail with specific examples of low-density microcellular foams prepared by the method.

III. NON-SOLVENT INDUCED SEPARATION OF SOLVENT AND POLYMER PHASES

The general technique for the preparation of thin, polymeric, porous membrane structures is by the controlled desolvation of multicomponent solutions of the desired polymer.¹ The components of the solution are generally a strong solvent for the polymer and a series of one or more weak- or non-solvents. The concentration of the solvents is adjusted such that initially a homogeneous solution of the polymer is formed. Upon removal of the good solvent by a variety of methods a so-called polymer gel is formed, the weak- or non-solvents being responsible for the formation of the eventual voids in the polymer membrane.

The Loch-Sourirajan Wet Process for making porous membranes involves casting a solution of cellulose acetate, acetone, water and magnesium perchlorate. Evaporation of the acetone results in voids forming in the polymer gel. The voids are filled largely with water, the non-solvent for cellulose acetate. The magnesium ions act as nucleating agents for the formation of the water droplets.

In modifying this process Rinde and Stone² prepared cellulose acetate foams for fusion targets by the extrusion of an acetone solution of the polymer into a "non-solvent" environment. The non-solvent system consisted of the aqueous solution of magnesium perchlorate. Absorption of the aqueous solution into the polymer solution resulted in the separation of polymer and mixed solvent/non-solvent phases. Removal of the

solvents via sublimation of the frozen mass under vacuum (i.e. freeze-drying) yield 100% porous cellulose acetate foams with densities of about 0.5 g/cm^3 and cell sizes of 1 to $2 \mu\text{m}$.

IV. CHEMICALLY INDUCED SEPARATION OF SOLVENT AND POLYMER PHASES:

There is a large number of condensation polymerization reactions in solution which lead to the formation of "gelatinous" type dispersions of the polymer in the solvent system. A few of these systems have been explored as possible microporous foams for fusion targets. Of all the techniques described herein the chemically induced phase separation technique has yielded the finest structures yet obtained in a microporous foam. Despite this fact, this technique has not been studied to the depth of detail as the two thermally induced phase techniques discussed below.

a. SiO Foams

Commercially available silica foams with densities down to 0.08 g/cm^3 and pore sizes in the hundreds of angstroms are manufactured by Airglass AB, Lund, Sweden. These aerogels are prepared by acid or base catalyzed hydrolysis of silicates in an alcohol/water solvent system. Early work in the area³ prepared the intermediate colloidal gels of silica by the acid hydrolysis of sodium silicate solutions. The colloidal gels, called alcogels, are stabilized by the formation of the three-dimensional-Si-O-Si-O- network. The alcogel form shows elasticity and yield values, but any flow in the system tends to lead to irreversible rehydrodestruction.

The alcogel can be dried by simple evaporation of the alcohol/water systems, but the structure suffers considerable and irreversible collapse by the surface forces of the retreating solvent menisci during evaporation. Such dehydrated alcogels are widely used under the name of silica

gels as dehydrating and adsorbing agents and as catalysts or catalyst supports.

The commercial aerogels are prepared by the hydrolysis of tetra-alkylorthosilicates in methanol or ethanol solutions. Shrinkage of the resulting alcogels is minimized if the liquid system is removed above its critical temperature so that no menisci form during liquid removal.⁴ Aerogels with densities down to about 0.02 g/cm^3 have been obtained by Pearson, by exchanging the liquid in the alcogel with liquid CCl_4 and removing the CCl_4 at its critical temperature.⁵

b. Polyacetylene Foams

Foam-like structures of polyacetylene can be prepared by the polymerization of the monomer in toluene using a Ziegler-Natta catalyst at relatively low concentrations.⁶ At -78°C a red gel is formed when acetylene gas is passed over the solution of catalyst. The gel grows overnight to a thickness of 1 to 2 cm and shrinks to about 1/2 its size when the toluene is evaporated. But when the toluene is exchanged for benzene the gel retains its dimensions after the benzene is sublimed off. The resulting dull black foam is reported to have a density of 0.02 to 0.04 g/cm^3 and pores of about 200nm. The structure is made up of a fibril mate with fibril diameters of about 600-800Å.

V. THERMALLY INDUCED GELATION/CRYSTALLIZATION:

One of the most well known polymer solutions which forms a reversible gel upon cooling is common gelatin in water. This system serves as an excellent example of the mechanics of gelation of typical linear polymer systems when the solution is cooled below the critical miscibility temperature (CMT) at a particular concentration. The transition of the solution from a liquid of relatively low viscosity to an elastic gel

occurs reversibly over a narrow temperature range and, generally, the more concentrated the solution the higher the temperature at which this phenomenon happens. Compared to spinodal decomposition processes discussed below the transition is relatively slow and equilibrium conditions are hard to reach at any one temperature.

Despite the fact that, on a molecular scale, gelation of gelatin solutions results in the crosslinking of the long, polymer chains, efforts in this laboratory to preserve this structure by removal of the solvent by sublimation has resulted in an almost complete collapse of the network.

For linear polymers such as the polyolefins like polyethylene, polypropylene, polystyrene, etc. gel states can also be obtained when cooling solutions of the polymer in appropriate solvents. In these cases intertangement of polymer chains and attractive forces between segments are the primary interactions in gel formation. But not all linear polyolefins form gels in the same solvents. For example, bibenzyl (1,2-diphenylethane) serves as an excellent solvent for both TPX (poly 4-methyl-1-pentene copolymer with <5% polyoctene and polyhexene) and polystyrene at temperatures above their respective melting points (T_m). Fig. 2 shows a plot of the viscosities of the two polymers vs temperature at 5 wt% concentrations.⁷ Phase separation of the systems occurs at about 150°C for the TPX solution and at about 85°C for the polystyrene system. The marked increase in the viscosity of the TPX system at the phase separation temperature for a cooling rate of about 1°C per minute is indicative of gel formation whereas a gel state is not observed in the polystyrene system.

Foam researchers have concentrated in polyolefins gels in order to determine if these delicate structures can be isolated as microcellular

foams for fusion target applications. Early in the studies it was found that, like gelatin, many polyolefin gels collapsed when an attempt was made to remove the liquid phase. Those systems in which a minimum amount of dimensional change occurred during solvent removal happened to occur among those polyolefins which showed a high degree of crystallinity in the foam state. It is felt the formation of a gel state followed by crystallization of the polymer has contributed to the success in the isolation of low-density, microcellular foams from these crystalline polymers. Figure 3 is a DSC cooling curve for the same bibenzyl solution of TPX described in Figure 2.⁷ A distinctive exotherm is observed at the phase separation temperature indicative of polymer crystallization. Two of the more successful systems will be discussed in detail although it is not clearly understood how crystallization of the polymer gels effects the final foam structure.

a. TPX Foam

Low-density, microcellular stable TPX foams can be readily prepared by a phase separation procedure involving gel formation from a solvent followed by crystallization of the polymer.⁸ Currently, the foams prepared by this process are routinely made for fusion target studies at Los Alamos National Laboratory and have been the workhorse foams for the past five years. Unlike the spinodal decomposition process in which homogeneous solutions of polymers are cooled rapidly into an instability zone in the phase diagram, the process by which these foams are made involves cooling homogeneous solutions of the polymer in a slower, more controlled manner. The purpose of this controlled cooling is to assure that the gel stage is formed at the CMT and to avoid super cooling. Crystallization may or may not occur after gel formation depending on the

polymer/solvent system but it appears that it is essential in order to obtain a stable foam structure. TPX is a commercial polyolefin material manufactured by Mitsui Petrochemical Co. in Japan and consists of predominantly poly(4-methyl-1-pentene) with <5% polyoctene and polyhexene. Table I contains a list of typical solvents used in preparing TPX microcellular foams. For a typical density foam of 0.05 g/cm^3 the cell size¹³ varies from solvent system to solvent system. For example, maximum cell sizes of $30\mu\text{m}$ are obtained from solvents such as diphenylmethane, phenylether and bibenzyl whereas durene yields a foam with a $10\mu\text{m}$, cell and diisopropylbenzene produces a $6\mu\text{m}$ cell.

The process by which foams are obtained from TPX polymers is relatively straightforward. Homogeneous solutions of the polymer are obtained at elevated temperatures by heating the mixture for an extended period of time with mixing. This solutions are then cooled to just above the temperature at which gelation occurs. Once the system is brought to temperature equilibrium, all mixing or other forms of agitation are stopped and the solution is slowly cooled below the gelation temperature. For the TPX/bibenzyl system^{8b} this rate is roughly 1°C/minute . Complete phase separation is indicated when the solution develops a solid, cloudy or gelatinous appearance. Cooling then to room temperature results in a non-fluid TPX foam matrix whose cells are filled with solvent.

If a solvent is selected which is a solid at room temperature the process offers another distinct advantage in that the solid solvent dispersed in the TPX polymer foam matrix acts as a reinforcing agent for the delicate cell walls and, thus, the filled-foam structure can be machined to relatively high tolerances before the former solvent is removed.^{8b}

Removal of the former solvent from the foam matrix is accomplished in several different ways. For solvents such as diphenylmethane, phenyl ether and bibenzyl leaching with a low molecular weight alcohol such as methyl alcohol works well. Durene can be evaporated under vacuum at about 50°C to yield the foam matrix and diisopropylbenzene is leached from the smaller cellular structured foam with liquid CO₂.

b. Polyethylene Foams

Polyethylene gels can be made from a wide variety of organic solvents such as decane, dodecane, and decalin. Letts, et. al.²¹ have studied ultra-high (~3 million) molecular weight polyethylene in these and other solvents such as tetralin and 1-octene to explore the possibility of preparing low density foams by a thermally induced gelation/crystallization. For example, Letts was able to obtain rigid polyethylene foams from densities down below 0.01 g/cm³ to above 0.1 g/cm³ in tetralin by the controlled cooling of the homogeneous solutions at rates of from 1 to 50°C per minute. For targeted densities of about 0.05 g/cm³ gelation of the polymer solution was determined by viscosity measurements to occur at about 100°C. DSC cooling runs on the solutions showed crystallization of the polymers to occur just below 100°C. Extraction of the phase separated system with liquid CO₂ gave a polyethylene foam which showed no shrinkage during the backing process and had void dimensions from 3 to 5µm.

VI. THERMALLY INDUCED SPINODAL DECOMPOSITION:

Sudden quenching of homogeneous polymer solutions much below the cloud point of the solution forces polymer solutions to develop unstable fluctuations leading to phase separation. This process is very similar to the spontaneous phase separation of amorphous glass and metal mixtures via a spinodal mechanism under rapid quenching conditions. Figure 4 shows a

typical temperature versus composition phase diagram for a polymer/solvent system. The upper solid curve is the binodal. Due to kinetic effects the experimental cloud point curve does not necessarily correspond to the binodal line. The binodal line is established by taking into account polydispersity factors in the system and extrapolating to infinitely slow heating and cooling rates.

The upper dashed curve in Fig. 4 is the spinodal or unstable region of the phase diagram and it is into this region one wants to reach rapidly in order to get the desired foam structure. Between the binodal and spinodal curves polymer solutions will undergo liquid phase separation by either homogeneous or heterogeneous nucleation. Below the spinodal curve spontaneous liquid-liquid phase separation occurs without the requirements of nucleation. The solid curve below the binodal and spinodal curves is the freezing point curve of the solution.

Since the initial work of Cahn and Hilliard¹⁴ the spontaneous phase separation of amorphous glass and metal mixtures via a spinodal mechanism has been extensively studied. In contrast polymer/solvent and polymer/polymer mixtures have received little attention. Attempts are being made to study the thermodynamics of polymer/polymer systems¹⁵ and polymer/solvent systems¹⁶ applying the concepts of the Flory-Huggins^{17a} approximation for polymer solutions and the Cahn-Hilliard treatment describing spinodal structure of a liquid phase separating binary liquid at early times.^{17b} It will be shown in the following discussions that cell sizes of plastic foams prepared spinodal phase separation of polymer/solvent systems average from approximately equal to the wavelength predictions of Cahn to being more than an order of magnitude larger than prediction.

Aubert explains this broad range of different cell sizes as being due to a quench-rate dependent process allowing fine wavelength structures to coarsen if they are not "trapped" rapidly enough.¹⁶

a. Water-Soluble Polymer/Water Systems

Coudeville, et. al.¹⁸ first described a method of producing a low-density, microcellular foam for fusion targets by a rapid quenching technique. This method involved the spinodal decomposition of a dextran/water solution by rapidly freezing the solution in an isopentane bath at 163°K. Incorporation of a co-solvent of dioxane in the system prevented the expansion and cracking of the resultant cooled dextran/water-ice structure and, thus, fusion target foams could be readily made. After rapid cooling the solvent system was removed by freeze-dry evaporation yielding foams of about 0.05 g/cm³ density and cell sizes of 1 to 2μm (sizes close to the Cahn-Hilliard prediction of 0.8μm).

In a parallel effort Young, et. al.^{18a} prepared plastic foams from water solutions of the ammonium salt of carboxymethyl cellulose (CMC) and of methyl cellulose by rapidly freezing the solutions in liquid nitrogen at 78°K (Figure 5). Via this spinodal decomposition process CMC foams could be prepared over a range of densities of 0.05 g/cm³ with 2μm cells to 0.016 g/cm³ with 10μm cells. Methyl cellulose foams were prepared with densities down to 0.007 g/cm³ and a cellular structure of up to 40μm.

More recently Rand at Sandia National Laboratories has prepared lower density plastic foams from aqueous solutions of polyacrylic acids and guar gums for use in Narya gas-puff x-ray laser experiments¹⁹. Rand rapidly cools the polymer solutions with nitrogen gas passed through liquid nitrogen and removes the solvent by freeze-drying. Rand has obtained foams from guar gum with densities down to 0.003 g/cm³ and

routinely makes polyacrylic acid foams for Narya experiments with a density of 0.006 g/cm^3 and maximum cell sizes of $20\mu\text{m}$.

b. Plastic Foams from Thermoplastic Polymers in Organic Solvents

1. In one of the most extensive studies ever undertaken to produce plastic foams from thermoplastic polymers by the spinodal decomposition of the polymer solutions was reported by Castro.²⁰ Castro studied the spinodal decomposition products of some 29 different polymers and blends of the polymers in a wide variety of solvents by rapidly freezing the solution in, for example, a dry-ice/acetone bath. Castro was able to prepare a host of polymer foams with claimed cell sizes from $0.5\mu\text{m}$ to $100\mu\text{m}$ and with densities down to about 0.1 g/cm^3 .

2. In an elegant study Aubert was able to prepare polystyrene foams by a spinodal decomposition of solution of the polymer with densities down to about 0.02 g/cm^3 and with cell sizes in the $10\mu\text{m}$ region.¹⁶ In his approach a homogeneous solution of polystyrene in a suitable solvent system was poured into a mold and the mold cooled rapidly with liquid nitrogen until the solution was frozen. The solvent was then removed by freeze-drying.

Aubert was able to control the morphology of the foams by selecting the solvent system from which to do phase separation. If an isotropic morphology was desired Aubert picked a solvent or co-solvent system; in which liquid-liquid phase separation occurred before the solvent was frozen. A liquid-solid phase separation yielded an anisotropic foam structure when the solvent began to freeze first, inducing dissolution of the polymer. For example, Aubert obtained an isotropic cellular structured foam when a cyclohexane solution of polystyrene ($M_n = 1.8 \times 10^6$) was rapidly frozen. In stark contrast to this structure Aubert obtained a

pleated-sheet like morphology when a benzene solution of the same polymer was rapidly cooled. In the later case benzene crystallizes during cooling, the crystal growth of the solvent excluding polystyrene at the crystal grain boundaries yielding spacings between the sheets folds in the final foam of up to $100\mu\text{m}$.

Aubert was able to control the cell size of isotropic polystyrene foams by narrowing the temperature range between spinodal decomposition of a polystyrene solution and the freezing of the solvent. The solvent system was dioxane/isopropanol. At a co-solvent ratio of 55/45 an isotropic foam structure with cell sizes of around $10\mu\text{m}$ was obtained from the same polystyrene. But when the co-solvent ratio was adjusted such that liquid phase separation occurs just before the solvent freezes, i.e. 60/40, a finer structure with cell sizes around $1\mu\text{m}$ is obtained. The Cahn-Hilliard predicted cell size is about $0.7\mu\text{m}$. Thus, the higher concentration of isopropanol allowed time for the spinodal fluctuations to coarsen before solidification of the polystyrene rich phase occurred, wherein a ratio of 60/40 the solution frozen right after spinodal decomposition occurred "freezing" the early structure in place.

VIII. CONCLUSIONS:

Phase separation processes of polymer solutions have been very useful for the preparation of low-density microcellular plastic foams for fusion target designs. The technology is still very young and a lot is yet to be learned about the physical chemical aspects of the process. Continual study of these processes should provide fusion target designers with a broad selection of low-density microcellular foams to choose from in the near future.

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TABLE I
SOLVENTS USED IN PREPARING TPX FOAMS

Solvent	Physical State at Room Temperature	Boiling Point or Melting Point	Ref.
Cyclohexane	Liquid	81.4°C/6.5°C	9.
Diphenylmethane	Liquid	264°C	8a.
Phenylether	Liquid	259°C	8a.
Cyclohexylbenzene	Liquid	239°C	8a.
1-Phenyloctane	Liquid	261°C	8a.
Diisopropylbenzene	Liquid	(mixed isomers)	10.
Bibenzyl	Solid	49.6°C	8.
Hexamethylbenzene	Solid	162°C	8a.
Durene	Solid	80°C	12.

POLARIS PRIME

Figure 1. Typical Design of
A High Gain Fusion Target

Figure 2. Plots of the viscosities of 5% solutions of TPX(▲) and 1.8m Polystyrene(●) in bibenzyl solutions vs temperature. At these concentrations the phase separation temperature for TPX is about 150°C and for polystyrene is about 180°C.

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5%TPX IN BIBENZYL COOL 20C/MIN

BP3

Figure 3. DSC of the cooling
of a 5% TPX in bibenzyl
solution

Figure 4. Typical temperature versus composition phase diagram for a polymer/solvent system.

Figure 5. Carboxymethyl
cellulose foam,
Density = 0.05g/cm^3