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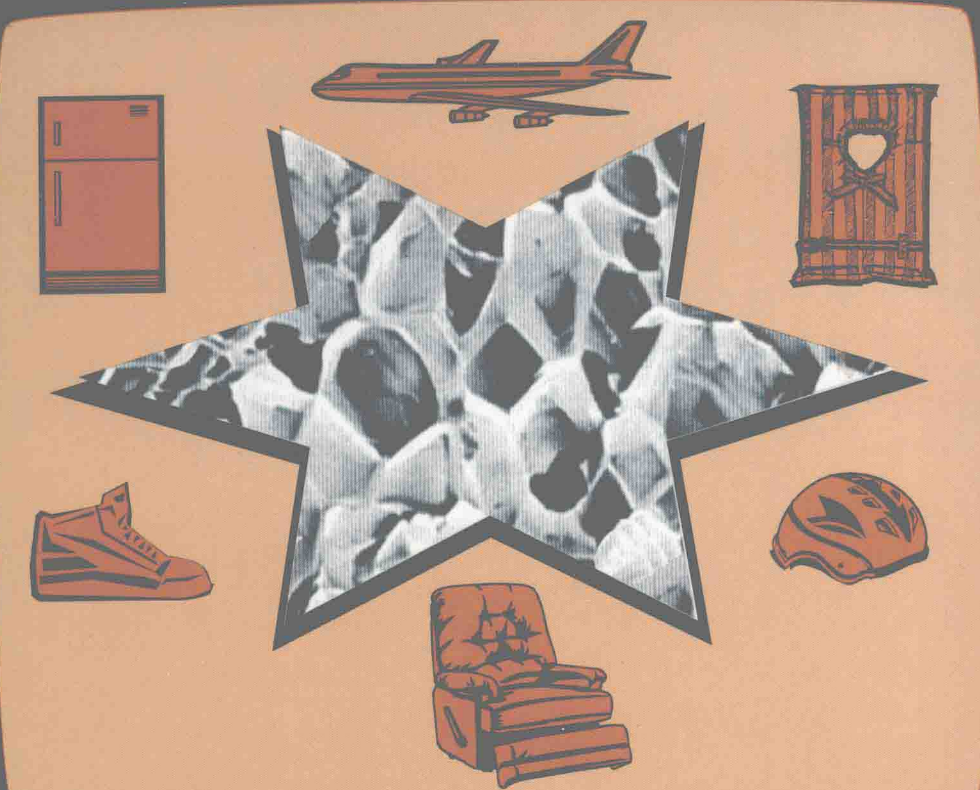
Polymeric Foams

Science and Technology



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Kishan C. Khemani

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Eastman Chemical Company

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ACS Symposium Series

Foreword

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Before a symposium-based book is put under contract, the proposed table of contents is reviewed for appropriateness to the topic and for comprehensiveness of the collection. Some papers are excluded at this point, and others are added to round out the scope of the volume. In addition, a draft of each paper is peer-reviewed prior to final acceptance or rejection. This anonymous review process is supervised by the organizer(s) of the symposium, who become the editor(s) of the book. The authors then revise their papers according to the recommendations of both the reviewers and the editors, prepare camera-ready copy, and submit the final papers to the editors, who check that all necessary revisions have been made.

As a rule, only original research papers and original review papers are included in the volumes. Verbatim reproductions of previously published papers are not accepted.

ACS BOOKS DEPARTMENT

Preface

FOAMED POLYMERS ARE USED BY THE MODERN WORLD in a variety of applications for attributes including weight reduction, insulation, buoyancy, energy dissipation, convenience, and comfort. A vast amount of research and development data on the use of foamed resins has been amassed over the last 50 years. Surprisingly, the area of foams continues to be enriched with new ideas as our understanding of the science and technology of foaming as well as the uses of foamed polymers continue to be challenged by the advent of new polymers, foaming technologies, and applications never imagined before.

This volume was developed from an international symposium presented at the 212th National Meeting of the American Chemical Society, titled "Recent Advances in Polymeric Foam Science and Technology", sponsored by the ACS Division of Polymer Chemistry, in Orlando, Florida, August 25–29, 1996. The chapters of this book are the contributions of symposium participants. The participation and attendance at this symposium, and the interactive scientific discussions that took place after each presentation, attest to the escalating interest and research and development efforts in this field. At the meeting, the need for this volume was apparent. Although it is not possible to cover the topic completely in a book of this nature, an attempt has been made to solicit manuscripts that highlight some of the main areas in this field.

The 15 chapters in this volume represent recent developments in different areas of polymeric foam science and technology. The first chapter is an overview of the field of polymeric foams and their markets. Each of the next 14 chapters is organized so that a comprehensive review of the respective field precedes the discussion of new results. The first five chapters (2–6) discuss new developments in the areas of siloxane, carbon, polyimide, polyester, and polyisocyanurate foams. Chapters 7 and 8 focus on the newly emerging area of microcellular polymeric foams produced via solid-state and extrusion foaming techniques, respectively. The next three chapters (9–11) discuss some recent advances in the area of polyurethane foam. Chapter 12 deals with important issues in the study of the morphology of cellular solids. Chapters 13 and 14 discuss some physical and theoretical aspects of foams and foaming processes. Finally, Chapter 15 discusses modeling studies of inherently foamable (upon exposure to heat) intumescent polymers used as fire retardants.

This volume is intended to be useful to all academic and industrial scientists, engineers, and technicians who work in research and development in the field of polymers and polymeric foams. It is the sincere hope of the contributors of this volume that the work presented here will spawn new ideas, technologies, and applications in this exciting field.

Acknowledgments

I thank all the contributors to this book for their willingness to contribute and for their timely submission of draft and final copies of the manuscripts. I have enjoyed getting to know all of them and have learned a great deal about various aspects of polymeric foams from them, for which I am grateful.

I express my appreciation to my employer, Eastman Chemical Company, for its support in this undertaking.

I also thank my mentors from the past, for it was they who shaped my present and future: the late Om P. Jha of Bhagalpur University, India; James F. King of the University of Western Ontario, London, Canada; James H. Rigby of Wayne State University, Detroit, Michigan; and Fred Wudl of the University of California, Santa Barbara, California. My transition from an organic chemist into a polymer and materials chemist was facilitated by Fred Wudl during my three years at the Institute for Polymers and Organic Solids at Santa Barbara as a Research Scientist.

Next I thank my wife, Madhu, and my children, Juhi and Ankush Raunak, for their unfailing love and support over the years, and their patience and understanding during the preparation of this volume.

Finally, I express my appreciation and gratitude to my parents Jiwat and Bimla, my brothers Jagdish and Deepak, and my sister Tripti, for their unconditional love and support in all of my earthly endeavors.

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Contents

Chapter 1

Polymeric Foams: An Overview

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It is difficult to go through a whole day without coming in contact with something which does not contain a foamed plastic part somewhere within. The most common examples perhaps are the foamed polyurethane seat cushion of the sofa or chair or the car-seat that you sat on this morning, or the polystyrene foam cup in which you had your morning coffee or tea or some other hot drink! Or the foamed polystyrene take-out boxes or trays which are used extensively at restaurants and cafeterias everywhere.

Since the first introduction of foamed rubber, phenol-formaldehyde and urea-formaldehyde resins in the early twentieth century, the foam industry has come of age, so to speak, in terms of the knowledge gained and the understanding of the fundamentals and principles governing this technology. So much so, that the science and technology of foaming of several specific polymers (for example, polyurethane, polystyrene, phenolics, polyvinyl chloride etc.) have literally become stand-alone research and development areas in themselves.

As a result of the years of research and development work in the field of polymeric foams in general, nowadays it is possible to foam virtually any polymeric material since a lot of the basic principles governing this technology and its processes are applicable to most resins. Of course, the fitness-for-use requirements, the ease or difficulty of production, and most importantly, the cost-of-goods-sold dictate the industry's choice of resins and manufacturing processes for specific applications.

Several types of polymeric materials are foamed to various low densities for applications that derive from attributes such as weight-reduction, insulation, buoyancy, energy dissipation, convenience and comfort. There are two major classes of polymeric foams: thermoplastic and thermoset foams. While thermoplastic foams can be reprocessed and recycled, thermoset foams are intractable since they are generally heavily crosslinked. Within these classes, the polymeric foams are further classified as rigid, semi-rigid, semi-flexible, or flexible, depending upon their compositions, cellular morphology and other physical and thermal characteristics such as Tg, %crystallinity, extent of crosslinking, etc.).

Furthermore, a solid polymeric foam can either consist of closed or open cells. Closed cell foams have a cellular structure in which contiguous air bubbles are entrapped within a continuous macromolecular phase. The foam polystyrene coffee cup, for example, consists entirely of closed foam cells. Open cell foams, on the other hand, have a cellular network in which continuous channels are available throughout the solid macromolecular phase for air to flow through at will. The polyurethane seat cushion is a very good example of an open cell foam. Closed cell foams are generally rigid, while open cell foams are generally flexible. All of the aforementioned attributes of foamed polymers is a direct result of the presence of these so called voided architecture.

Most polymeric foams are produced by one of the several known foaming techniques which include, extrusion, compression molding, injection molding, reaction injection molding, solid state method (where pressurized gas is forced into a solid polymer at room temperature followed by depressurization and heating to above its Tg), etc..

The gaseous phase in any polymeric foam material derives from the use of blowing agents in the foam manufacturing process. There are two types of blowing agents used to produce foams: chemical blowing agents and physical blowing agents. As the name implies, the first type are chemical compounds which give-off gases under the foam processing conditions, either due to chemical reactions or due to thermal decomposition. The second type of blowing agents are simply inert gases, such as nitrogen, carbon dioxide etc.; volatile hydrocarbons having boiling points between -40°C to +45°C, such as propane, butane, *i*-pentane etc.; and low boiling chlorofluorocarbons (CFC's), hydrofluorocarbons (HFC's) and hydrochlorofluorocarbons (HCFC's).

For some applications, it is a common practice to coextrude or laminate a thin layer of solid unfoamed polymer onto the surface(s) of the foamed material in order to improve certain physical characteristics such as the cut-through resistance, modulus, barrier to various gases, extractables, printability, and finally aesthetics.

Several excellent textbooks and handbooks have been published over the years which give good details of various foam technologies, foaming processes, and foam characterization and properties. Some such recent publications are listed in references *1-4*, and are highly recommended.

The foam industry on the whole has been challenged in recent years by issues ranging from waste disposal, recyclability, flammability, and the depletion of earth's ozone layer by the chlorofluorocarbon blowing agents. This has been further compounded by the issuance of numerous regulations and codes by the government agencies. As a result, the industry has imposed a certain level of self-regulation in several of these areas.

Although the polymeric foam market is growing worldwide, the leading producers and consumers of these products are in North America, Europe and Japan. Some Latin American and other Asian countries (such as Argentina, Mexico, Brazil, India, Taiwan, South Korea etc.) have also been developing foam products and markets in more recent years.

Specific examples of uses of polymeric foams include films, cups, food trays, containers, flooring, decorative items (ribbons, etc.), insulation boards, sound dampening, transportation, bedding, carpet padding, furniture parts, chair cushions, toys, fibers, automobile parts (seats and back-rests, bumpers, headliners, etc.), sporting goods (helmets, clothing, , etc.), flotation (boat parts, surf boards, life-vests, etc.), footwear parts (soles, inserts, etc.), insulation for appliances, and in packaging of just about all types of non-food items as well.

The statistical data reported hereafter in this overview is derived from a combination of this author's interpretation, extrapolation and interpolation of the data/information reported and published over the past ten years. This author is therefore solely responsible for any inadvertent errors in estimating these values and projections. The numbers are intended merely to give a general feeling of the markets and their trends. In addition to the past issues of various excellent plastics news magazines such as, the Plastics World, Packaging Week, Plastics Business News, Asian Plastics News, European Plastic News, Packaging News, Plastics News, Modern Plastics, Plastics Technology, Materials Engineering, Plastics Engineering, Plastics Packaging, Plastic Trends, etc., the publications listed in references *1-9* are also recommended to the readers interested in broader in-depth analyses of various foamed polymer products, and their applications / markets.

It is estimated that in 1995 close to 6 billion pounds of foamed plastics was produced and consumed in the United States alone, and it is projected that this usage will grow at 3 - 4% annual rate to about 7 billion pounds by the year 2000. Most of this growth will probably come in the automotive, construction, packaging, and consumer products markets. The three major resins used in these markets are polyurethane, polystyrene, and polyvinyl chloride.

Foamed and unfoamed polyurethane is used in a very wide range of commercial applications. The main applications in the foam area are those requiring cushioning and insulation. Flexible polyurethane foam uses include, for example, seat cushions, furniture, carpet underlayment, mattress padding, appliances etc., and those of rigid polyurethane foams include insulation material for construction (as boards) and in appliances (injection molded). About 3 billion pounds of foamed polyurethane was used in these markets in 1995, and it is estimated that this number will grow to over 3.5 billion pounds by 2000. A large part of this growth might be in the newer area of reaction injection molded (RIM) polyurethanes for automotive components. Polyurethane has seen some market erosion in the past 10 years due to the shift from chloroflurocarbon (CFC) blowing agents which were extensively used in the past. However, it is no longer used in most places and presently more than 75% of the volume is blown with water and carbon dioxide. The three current largest producers of polyurethanes are Bayer, Dow, and BASF.

Polystyrene is another very widely used resin for the manufacture of foamed products. However, it is heavily dependent on packaging markets, although demand in construction markets as boardstock used in insulation is poised to take off. In 1995, about 1.7 billion pounds of polystyrene was consumed in these markets. In the packaging markets, general concerns related to waste disposal and recyclability are considered as impediments for this resin, and thus its growth potential is somewhat diminished as compared to polyurethanes. It is expected that the market share for this resin will grow to about 1.8 - 2.0 billion pounds by the year 2000. Foamed polystyrene products are typically manufactured by either molding of the expandable polystyrene (EPS) beads, or the extrusion foaming and thermoforming technology. The major producers of EPS are ARCO, BASF, and Huntsman, and those of extruded polystyrene foams are Amoco, ARCO, and BASF.

Foamed vinyl (polyvinyl chloride) is perhaps the first thermoplastic foam commercialized during World War II and was soon followed by the polyurethane and polystyrene foams. However, it ranks third in terms of volume as compared to the latter two, but still has a strong market share. It topped 300 million pounds in 1995, and is growing at a 2 - 2.5% rate. Some of the major current markets for this product are the construction (flooring, fittings, wire insulation, molding and pipe, conduit, etc.) and the automobile industry (mainly seat covers), with two major suppliers being BF Goodrich and 3M. Both flexible and rigid foamed vinyls are used in these markets.

As mentioned above, a large growth rate is forecast in the reaction injection molded polyurethane foam area. In 1995, about 250 million pounds of this material was consumed. And it is expected to grow at a rate of 7% or better over the next few years mainly due to high demand from the automobile industry where it has found uses in exterior body panels, trims and bumpers.

Foamed polyethylene (low and high density), phenolics, polypropylene, ABS, acrylics, cellulose acetate, urea-formaldehyde (UF), polyimides, polyetherimides, polyphenylene oxide (PPO), polychloroprene (PCP), silicones, epoxy foams, etc. all added up to over half a billion pounds of volume in 1995. Growth for these niche players is expected at anywhere between 4 - 6% per annum for the next few years, with perhaps the most growth being in the phenolics area due to their flame retarding capabilities. The markets served by these resins are quite diverse and include: packaging (HDPE); insulation (cellulose acetate, UF, silicones, polyetherimides); construction (HDPE, LDPE, phenolics, ABS); household products (HDPE, LDPE, acrylics); toys (HDPE, LDPE, ABS, PP); sporting goods (HDPE); floatation (UF), office products (LDPE, PPO); aircraft (phenolics, polyimides, PPO, PCP, silicones); electrical (epoxy, silicones), furniture (HDPE, ABS, PP); appliances (ABS); bottle labels (LDPE, PP); decorative items (LDPE, PP, UF); medical products (PP, UF)); artificial wood (UF, epoxy), etc..

Finally, there has been some progress in the recent years in the development of biodegradable foam materials for several of the same applications as described above. Different approaches developed by various groups include the use of a wide range of resin/material compositions. Some examples include, the compounding of non-degradable polymers such as the polyolefins with degradable materials such as starch, woodflour, jute, hemp, etc., and/or the use of inherently degradable materials such as the partially substituted cellulosics, starch, aliphatic polyesters, aliphatic-aromatic polyesters, polylactic acid, plasticized polyvinyl alcohol, polyesteramide, polycaprolactone, etc.. However, large scale commercial applications in these areas are still years away as much more work is needed. Recently, EarthShell Corporation of Santa Barbara, CA, has been test marketing a potato-starch/cellulose-fiber/calcium-carbonate based foamed food-packaging material (molded into clam-shells, cups, plates and trays, etc.) through McDonald's, which is claimed to degrade readily into its natural ingredients in a compost environment (*10*).

As mentioned in the beginning, foamed polymers offer unique advantages over non-foamed polymers in terms of specific properties such as weight-reduction, heat-transfer characteristics, buoyancy (for closed cell foams), physical force dissipation via cushioning, and comfort. On the other hand, the technology and process of foaming a polymer is a challenging one and encompasses the effective utilization of the knowledge base of several different scientific fields including polymer, materials and organic chemistries, physics, chemical and mechanical engineering, process engineering, equipment design and operation, and so on.

This volume deals with some recent developments in several of the different areas of polymeric foam science and technology in fourteen independent chapters. Each chapter is organized such that a comprehensive review of the respective field precedes the discussion of the new results.

The first five chapters (*2* through *6*) discuss some new developments in the areas of Siloxane foams, Carbon foams, Polyimide foams, Polyester foams and Polyisocyanurate foams. The next two chapters (*7* and *8*) focus on the newly emerging areas of microcellular foams produced by solid-state and extrusion foaming techniques respectively. The next three chapters (*9*, *10* and *11*) discuss some recent advances in the polyurethane foams area. Finally, chapters *12* through *15* discuss some physical and theoretical aspects of foams and foaming processes.

Specifically, chapter *2* discusses two different approaches to producing injectable elastomeric siloxane foams. The first, a synthetic approach, utilizes a reversed emulsion technique, and the second consists of blending thermally labile particles into the crosslinkable elastomer followed by curing and forced degradation of the labile particles to generate voids.

Interest in carbon foams has been high due to its potential in a wide range of specialized applications. Chapter *3* discusses several of these applications and reports the preparation of carbon foams by the pyrolysis of a series of rigid hypercrosslinked polyaromatic polymers such as the ones obtained from benzene, biphenyl, m-terphenyl, diphenylmethane, and polystyrene monomers and p-dichloroxylene as the crosslinking agent.

In chapter *4*, polymerization of nadimide end-capped oligobenzhydrolimide (BBN) blended with 3% of a linear thermostable polymer to produce rigid thermostable foams is described. During the thermal curing of the BBN, a reverse Diels-Alder reaction takes place with the evolution of cyclopentadiene which acts as an *in-situ* blowing agent. The resulting cellular network has good mechanical properties and other interesting features.

Polyester foams form the content of chapter *5*. Some new approaches for rendering linear polyesters and copolyesters foamable are discussed in this chapter. These include the use of monomeric branching agents, polymeric branching agents, and polymeric concentrates containing monomeric multifunctional branching agents, in order to enhance the rheological properties of the linear resins which is necessary for extrusion processing of foams from these materials. A good discussion of the basic requirements for foaming of a polymeric material in general, is also presented in the background review section of this chapter, and the readers may find Scheme 3 of this chapter particularly useful for this purpose.

Chapter *6* deals with novel polyisocyanurate foams which are modified by the incorporation of thermally stable linkages such as amide, imide or carbodiimide. This yields foams which exhibit improved thermal stability, smoke generation characteristics and have higher inherent flame retarding capacity.

Solid-state microcellular foams typically have cells which are 10 microns in diameter; consequently these materials have a very high cell density as compared to a more conventional foam of equal density in which the cell diameters range from 50-500 microns. Since their discovery in early 1980's at the Massachusetts Institute of Technology, a lot of development work has been done at other academic and industrial institutions and several different plastics have been foamed *via* this technique to relative densities of 0.1 to 1.0 and containing 10^8 to 10^{11} cells per cm^3. Very simply, the method involves impregnating and saturating a solid plastic material with a suitable gaseous blowing agent under high pressures at below its Tg (glass transition temperature), and followed by releasing the pressure and heating the gas-saturated plastic to a temperature higher than its Tg. Chapter *7* discusses the *pros* and *cons,* and various other issues surrounding this new technology in some detail.

More recently, it has been demonstrated that microcellular foams can also be produced by an extrusion process. Whereas the solid-state process must by its nature be a batch process or a semi-continuous process at best, the extrusion process is a continuous one. It is therefore perhaps more cost effective, and consequently has been the focus of a lot of the recent attention and research and development effort. The technique simply involves the continuous formation of a polymer/gas solution inside an extruder followed by the nucleation of a large number of bubbles using rapid pressure drop and the control of the foaming step *via* pressure control in order to induce a desired volume expansion. Chapter *8* describes some intricacies involved in such a process in the extrusion of microcellular high impact polystyrene foams (HIPS).

Some recent advances in polyurethane foams is covered in Chapters *9*, *10* and *11*. In chapter *9*, stabilization of the bubbles during the foaming of flexible polyurethane foams using polymeric silicone surfactants is reported. The structure of the silicone surfactant used has significant effect on the final air flow through the foam. A relationship between the surfactant structure and foam openness is provided which suggests some basic noteworthy trends.

Chapter *10* deals with an *in-situ* study of structure development during the reactive processing of water blown polyurethanes. The kinetics of the reaction-induced phase transformation, its mechanism, and the resultant cellular morphology, all suggest that microphase separation in polyurethane foam occurs *via* spinodal decomposition at a critical conversion of isocyanate functional groups.

Imaging of the three-dimensional cellular network of flexible polyurethane foams is done using a new technique, the laser confocal microscopy, in chapter *11*. This technique acquires sharp 2D images which are then used for 3D reconstruction of the cellular structure. Use of this technique for studying foam compression under different mechanical compressions is also discussed in this chapter.

Chapter *12* discusses the relationships between foam properties and foam cellular morphology and cautions against the inherent limitations and pitfalls of the various methods of quantifying the cellular structure.

Chapters *13* and *14* deal with the fundamental aspects of the key areas of the extrusion foaming processes. Whereas chapter *13* looks at the overall foaming process using the physical blowing agents, chapter *14* focuses on the dynamics of the bubble growth.

Finally chapter *15* discusses the modeling of mechanisms that determine the fire-resistant properties of intumescent polymers, which are designed to swell into thick robust foams upon exposure to heat and thus protect the underlying materials from fire.

The foam densities reported in this volume are either in g/cm^3 or lb/ft^3 or kg/m^3 units. The interconversion of these units is related by 1 g/cm^3 or g/cc = 62.37 lb/ft^3 or pcf = 1000 kg/m^3 or kcm.

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Chapter 2

Siloxane Elastomer Foams

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New approaches to produce injection moldable elastomeric foams with porosities in the micrometer range have been developed. In the first approach, water is dispersed within the siloxane network by first preparing a reverse emulsion in the presence of an appropriate surfactant. Porosity is then generated by evacuation of the water. Supercritical drying was used to avoid surface tension effects and prevent collapse during drying. The measured porosities were in good agreement with the water incorporated into the initial emulsion. The second approach surveyed as a means of generating controlled porosity in elastomers involves blending thermally labile particles into the siloxane resin. Upon network formation, the particles can be selectively degraded to leave voids, the size and shape of which should be identical to that of the initial dispersion morphology. PMMA particles with deliberate low thermal stability were synthesized using a combination of dispersion polymerization and chain transfer. The resulting particles showed a significant decrease in degradation temperature. These two techniques for the preparation of elastomeric foams are described.

Elastomer foams are generally produced using gas blowing techniques. By such techniques, it is very difficult or even impossible to produce narrow pore sizes distributions and to control pore compositions, due to coalescence and ripening phenomena. New applications demand a precise control of both the volume fraction of porosity and pore size. It is also often necessary to injection mold which is not compatible with the gas blowing techniques requiring vented molds to allow for expansion. Based on these considerations it is necessary to find new ways to produce injectable elastomer foams with controlled porosity. The purpose of this paper is to present a new approach to synthesize porous siloxane elastomers with a controlled porosity in the μm-range.

Despite the numerous existing techniques to prepare polymeric foams, few are suitable to prepare porous elastomers with the desired closed porosity. It is not in the scope of this paper to review in detail the existing techniques to porous polymers, but the general strategies of the most important techniques to prepare porous polymers will

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