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ASSESSMENT REPORT

A.J. Hunt

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ASSESSMENT REPORT

MICROPOROUS TRANSPARENT MATERIALS FOR INSULATING WINDOWS AND BUILDING APPLICATIONS

by

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1. INTRODUCTION

This report examines the properties and uses of microporous optical materials as insulating apertures in buildings. Significant energy savings can result if conventional window glazings are replaced by high R-value insulating materials. New glazing materials with microporous structure can be made that combine high transparency and good visibility with excellent insulating characteristics. These microporous materials may be made of silica (pure glass) or other materials, but are fundamentally different from conventional glazings because they are made in the form of a porous, low density solid. Microporous materials are transparent rather than translucent because the *pore size is much smaller than the wavelength of light*; therefore they transmit rather than scatter light. These materials are excellent insulators because of the high ratio of volume of trapped air to volume of solid matrix and the extremely small pore size.

Microporous materials offer an alternative to conventional glazings in that they can replace double or triple pane glass with a package of similar thickness that possesses a higher R value. Microporous glazings may be manufactured as a single unit with a transparency that exceeds that of multiple glazings. A form of microporous silica called aerogel that is both transparent and possesses good insulating properties has been produced in the laboratory. However, this material requires development to improve its properties to the point that it can be used as a glazing.

There are two main obstacles standing in the way of commercialization of this material. First, methods need to be developed to improve the physical and optical properties of the material to the point of commercial acceptability. Second, preparation techniques need to be developed that are more suitable to mass production than is the present laboratory technique. Research and development is needed to overcome these obstacles.

A joint research effort between the LBL Solar Program and the LBL Windows and Daylighting Program was initiated with the support of the DOE Passive Solar Program; its objective is to develop techniques to produce transparent microporous materials and investigate their properties for insulating applications.

Related work has thus been underway in two closely cooperating groups at LBL, and the author would like to acknowledge significant contributions to the understanding of aerogels as well as data supplied by Mike Rubin and Steve Selkowitz of the Windows and Daylighting Group of the Energy & Environment Division.

This report is divided into five chapters that attempt to answer the major questions about the material and its development. Chapter Two is devoted to the description of the properties and methods of production of the material, with an indication of the types of applications for which it is suitable. Chapter Three treats the questions of energy savings, technical and economic barriers to obtaining private sector involvement, and the degree of work necessary to bring the concept to bench scale tests. The fourth chapter reviews the current research and identifies groups involved in the development of microporous optical materials and groups that are likely to express an interest in commercializing this material. Chapter Five briefly discusses the future applications and possible spinoffs of the development of this material.

2. STATE OF THE ART

Microporous optical material consists of a rigid matrix of transparent material with a pore size considerably less than a wavelength of light. The density of this composite may be less than two percent of the density of the bulk material of which it is made. Most familiar porous materials such as foamed plastic or rubber have pore sizes on the order of millimeters or fractions of millimeters. Cells of this size in a transparent material quickly scatter incoming light so no trace of the original propagation direction[1] of the light remains. Basically, light diffuses through these large cell materials rather than propagating though them as it would in a transparent solid. If the pores are made very small, much less than the wavelength of light, the amount of scattering decreases rapidly, and the transparency is restored.

A. Background and History

The first transparent porous materials were made about 50 years ago by Kistler using a process based on colloidal gels[2]. Materials prepared in this way are referred to as aerogels. In this process, a colloid of the desired material is made sufficiently dense so that the individual colloidial particles begin to link together. When the necessary cooling or aging has taken place, the material changes from a liquid state to that of a gel or jelly. The material is then a semi-solid matrix of linked solids permeated by a liquid[3]. Attempts to drive off the liquid by heating or drying inevitably lead to the collapse of the matrix and the material shrinks to a fraction of its original size. This shrinkage occurs because, during drying, both liquid and gaseous states coexist in the matrix. Because of the very small size of the pores, surface tension effects between the gas and liquid are very large, and cause the structure to collapse during drying.

Kistler recognized that, if the distinction between liquid and gas could be eliminated by raising the temperature and pressure of the liquid above its critical point, the resulting fluid could be released from the gel without the surface tension effects that would destroy the matrix. Thus the procedure is to make a dense colloid of the desired material, allow it to form into a gel, then heat the gel under pressure till the liquid is above its critical point, and finally slowly release the pressure to allow the fluid to escape from the matrix. This process is referred to as supercritical drying.

In many of his preparations[4,5,6], Kistler took advantage of the fact that one liquid may replace another in the gel without causing damage to the gel matrix. Thus liquids with lower critical points may be substituted for the colloidal solution, reducing the temperatures required for supercritical drying.

Aerogel may be transparent or translucent depending on the details of the preparation. Later investigators improved Kistler's techniques to yield uniform, transparent aerogel samples[7,8]. The density of aerogels may vary significantly, depending on the relative proportions of chemicals in the starting solution.

Very little work was done on solid aerogels for the following 25 years. In the early 1970's there was considerable interest in developing new types of Cherenkov radiation detectors for high energy physics. What was needed was a transparent solid with a very low index of refraction, an ideal application for aerogels. After a time the early aerogel work was rediscovered and new work was started, largely in Europe[9,10,11]. The detector work has been almost exclusively on silica aerogel. Recently, an aerogel window was fabricated by sandwiching aerogel between two sheets of glass to improve its physical properties[12,13,14].

While aerogels represent the most promising near term technology for producing microporous optical materials for glazing applications, several other techniques have been used to make low density transparent solids. Very fine silica powders (fumed silica) were compressed to obtain transparent solids[15]. Selective etching of thermally disaggregated glass has been used to produce very thin porous regions on the surface of glass to produce excellent antireflection coatings[16]. Glass has been dipped in colloidial solutions of silica to obtain antireflection coatings[17,18]. Animal tissue has been converted to microporous structure for electron microscope studies[19]. It seems likely that, if there is an interest in this material, other methods will be discovered to produce porous transparent solids. If a microporous material with good transparency can be made with a closed cell structure (possibly just near the surface) it would reduce some of the problems associated with sealing the aerogel-type materials.

B. Properties of Microporous Materials

Optical Properties

The most striking characteristics of microporous materials are their transparency to visible light and very low mass density. In particular, aerogels can have very good visibility; that is, they do not produce blurring of images observed through them. However, most aerogels produced so far have some slight but noticeable color effects associated with them. These color effects result almost exclusively from light scattering[20]. Shorter visible wavelengths are scattering more strongly. This leads to a slight bluish cast to the material when it is viewed against a dark background. Correspondingly, there is some enhancement in the relative transmission of the longer wavelengths. This enhancement leads to slight reddening of the transmitted light. These effects, while slight, are sufficiently noticeable to affect their use as viewing glazings (although some window coatings in use have roughly equivalent effects). However, an ongoing investigation at LBL into the cause of the scattering[21] leads us to believe that it can be reduced by changing the production process. This is the first of two research areas needed to develop and improve the properties of microporous materials.

There is another unusual visual characteristic of pure, uncoated aerogels that results from their extremely low index of refraction. The surface reflectivity is extremely low, rendering them almost invisible. This effect would be further enhanced if the scattering in the material were reduced. This invisibility could have positive or negative impact on their use. Windows that extend to the floor and patio doors would have to be decorated to make them visible. Alternatively, there may well be a market for exceptionally clear windows. This is probably a moot point for early application of microporous materials, because they will require some form of encapsulation that will at least partly restore their reflectivity and appearance as normal glass.

Physical Properties

The physical properties of porous optical materials result mainly from their very low density. Aerogels are an open cell network of fused particles. The typical size of these particles is 0.005 to 0.01 micrometer (50 to 100 Angstrom units) in diameter[9]. In low density materials the average size of the open cell must be significantly larger than the particle diameter. Thus the materials are very open networks that, if cut through, would expose very small solid cross sections. This openness and small cross section necessarily result in materials that are weak compared to the bulk material. Aerogels are not strong materials (they crumble when pinched moderately hard between the fingers) but show significantly more strength in compression than in tension or sheer. This suggests that, if the faces of the material are protected, it will have adequate strength for glazing applications.

Another consequence of the open structure is that porous materials are very susceptible to liquids. Water will penetrate aerogel and the surface tension effects on the small pores effectively cause it to dissolve. Therefore, it is imperative to protect the surfaces by sealing them from the environment. This can be done by sandwiching the material between two glass plates and sealing the edges. It may also be done in a number of other ways, including increasing the surface density by local melting, evaporating a protectant on the surface, or spatially varying the density of the original gel by changing the ph or applying electric fields[22]. This protective sealing process is the second key area in materials research and development that needs to be addressed.

Thermal Properties

Thermal conductivity is an important parameter of microporous materials for insulating applications. Aerogel materials exhibit excellent thermal insulating properties. Room temperature measurements in air give values of thermal conductivity for aerogel[12] as low as 0.019 W/m·K (0.011 BTU/hr·ft·°F). A three inch thick aerogel window would have an R value of about 15. These values may be compared with the conductivity of stagnant air (0.022 W/m·K) or typical single and double glazings with R values of one and two respectively. Reasons for the conductivity being so low are that the mean free path of the air molecules is comparable to the pore size, and that silicon dioxide tends to inhibit radiative transfer[23]. If the material is sealed, it is possible to permeate the matrix with other gases that will result in lower heat conductivity. Using carbon dioxide or dichlorodifluoromethane reduces the conductivity by 10 or 25 percent, respectively[24].

Chemical Properties

Pure silica aerogel is silicon dioxide and therefore not very chemically reactive to most common substances. However, the very large surface area and small pore size lead to significant degradation when a liquid permeates the material. This will be true of liquids that can wet pure glass (silica). Therefore, this once again calls for sealing the material from the environment.

C. Applications of Microporous Materials

Insulating glazings based on microporous materials may be used to replace existing windows where heat loss or gain is to be minimized. This represents a very large conservation market. Because of the unitary nature of the glazing, the first applications will probably be in new construction. However, window unit replacement is quite common, and commercial systems incorporating this technology could be introduced by existing window manufacturers as a distinct product line and be marketed in similar ways to other window systems.

The most cost-effective applications will occur in regions of the country where energy loss through windows is largest. Thus, areas where triple glazing is being installed would be natural markets for insulating glazing. These glazings may be used for residential, commercial, and industrial applications.

Adopting insulating glazing for residential use involves several considerations. The ability to open a window is probably most important in residential applications. A composite window unit that is two to three inches thick and required to open, will require engineering a new system but will require no new technological breakthroughs. Strength and resistance to damage is another important consideration in all applications. While the basic strength of microporous materials does not compare with conventional glazings on a per thickness basis, an aerogel window sealed with microglass or standard glazing should have adequate strength for most applications. The greater thickness-to-span ratio of insulating glazing should result in less flexing than with conventional glazings. The damage resistance will also depend on the method of sealing the surface, but will probably be comparable to conventional glazings.

The availability of a well sealed, low scattering, high R value glazing material of moderate cost will trigger the development of a number of new fenestration systems for many conservation applications. For many applications the residual scattering exhibited by present silica aerogel will have little or no consequence. In many daylighting applications such as skylights and atrium lighting the faint bluish cast will not be objectionable and may even be pleasing.

3. ECONOMICS AND DEVELOPMENT

This chapter treats the possible energy savings associated with the adoption of insulating glazings, and the developmental activities that will be necessary to convince potential manufacturers that the production technology is practical.

A. Energy Savings

There will be a positive economic benefit from replacing conventional glazings with microporous insulating glazings if the value of the energy savings outweighs the added cost. The exact energy savings will depend on details regarding the performance of the insulating glazings, assumptions about climate, building construction, personal tastes, and many other factors.

An approximate value for the energy savings associated with space heating may be obtained by assuming that an aerogel window replaces an existing single or double glazing. The energy savings may be calculated by using the expression:

$$E = D(U_g - U_a)$$

Where E is the energy savings per year per square foot, D is the heating requirement in degree days per year, U_a and U_g are the U values for the aerogel window and the conventional glazing, respectively, in BTU per hour per square foot (U = 1/R). A typical value for D in much of the United States is 5000 °F days/year. If an aerogel glazing with an R-10 rating replaces a single glazing (R value of 1.0), the energy savings can be calculated as:

$$E = 5000\,°F days/year \times 24 Hrs/day \times (1.0 - .1) BTU/hour/\,ft^2$$

or about 108,000 BTU/year/ft^2. Assuming natural gas is the energy source at $.40/therm and an 80% fuel efficiency, the yearly cost savings is $.54/year/ft^2. If a ten year payback is required, the maximum allowable cost of the glazing would be $5.40/ft^2 to pay for the investment. The savings would be about half this amount if the aerogel were replacing a double glazed window (R value of 2). The savings would be significantly higher if the heating source were electricity instead of natural gas. Financing and fuel escalation costs were not considered in this rough estimate.

These figures represent the allowable cost *differential* between using high performance glazings and conventional glazings for heating applications. Thus, in this example, if a glazing of 4 by 6 feet were specified for a new structure, an additional cost of about $130 over a conventional installation would be justified.

The above analysis only addresses the question of the value of the heat saved by using insulating glazings in cold weather. A similar analysis may be performed for cooling loads in the summer. In this case the dollar savings would be large when air conditioning is used because electricity is displaced instead of natural gas. Again, these savings will vary considerably with climatic zone, personal preferences, and costs for electricity.

To obtain an accurate evaluation of the energy savings, it is necessary to model a specific structure and calculate the total energy required on a yearly basis including all of the above factors. However from the above it can be seen that significant energy savings can be obtained.

B. Economic and Technical Barriers

To gain private sector support for the development of porous optical materials into commercial products, several obstacles must be overcome. First, alternative cost effective techniques for mass production of insulating glazings need to be explored. Next, the cause of the residual scattering must be determined and the preparation parameters changed to reduce the scattering to a minimum. Finally, methods of sealing the faces and edges of the material to improve its environmental resistance and increase its strength must be developed. If private industry becomes convinced that these are solvable problems, it is anticipated that significant private interest will develop. Potentially, there are several approaches to produce porous optical materials. The silica aerogel process is the most developed, and therefore the following discussion is based on the assumption that it is the process to be commercialized. The nature of these problems and approaches to their solution are outlined in the remainder of this section.

Aerogel Production

To produce an aerogel in the laboratory, first a colloidal solution of silica must be prepared. An ethyl or methyl silicate compound is mixed with an alcohol solvent and water, then catalyzed to form a colloidial suspension. The colloid is poured into a mold and the silicate continues to hydrolyze until the gel is formed. Either the starting chemicals or the mixture is usually cooled to facilitate the setting of the gel. After setting, the gel is placed in a chamber, pressurized, and heated. Once supercritical drying conditions are achieved, the pressure is slowly released and the fluid allowed to escape. After cooling, the aerogel is removed from the chamber and the process is complete.

The finished cost of an aerogel window will depend on the costs of materials and the costs of processing. The material costs for off-the-shelf chemicals to produce an R-10, two-inch-thick window in the laboratory is about 10 dollars per square foot. However, the cost of laboratory chemicals for research purposes should not be taken as representitive of the mass production costs. The material cost is dominated by the chemical, tetraethyl orthosilicate. Ethanol is also used but may be recovered in an industrial process. It is almost certain that the cost of tetraethyl orthosilicate will drop if there is a significant market for the chemical, because the costs of the materials that go into the chemical are quite low.

To evaluate the cost of the aerogel processing with any degree of accuracy requires performing a conceptual design for a production facility. However, the key factors that impact the cost may be identified. If the procedure is adaptable to mass production, the cost component for the process can be minimized.

To evaluate the processing costs, consider the steps one at a time. The mixing, cooling, and setting requirements can be adapted to mass production techniques. The supercritical drying step in the laboratory procedure takes place at pressures over 100 atmospheres and temperatures of about 270 °C. While these conditions are not difficult to obtain in the laboratory, the pressure requirement would be expensive to implement for mass production. The problem is compounded by the need for large area glazings that will require big pressure vessels. Approaches to dealing with this problem are either to simply accept the requirement and the associated costs, or to look for ways of reducing the pressure requirements. The former approach, while straightforward, may result in a prohibitively expensive production process. Alternatively, if the solvent in the highly permeable gel is first replaced by a fluid with a lower critical point, the pressure requirements for the supercritical drying may be reduced significantly. This latter approach should be investigated to assess its feasibility.

The time for each of these steps to take place varies with the details of the process, but does not seem to present serious difficulties for mass production processes. Note that the temperatures are much lower than those required for traditional glass production, and hence the energy used in heating is significantly less. Also note that the process does not require high purities or extensive handling, and therefore is a good candidate for mass production (other than the pressure requirement). Therefore it may be concluded that, with the exception of the supercritical drying step, there are no significant problems in mass producing aerogel. If the requirement for this step can be reduced or eliminated, the cost of producing aerogel will be determined by the scale of the mass production facility, leaving the material cost as the major expense.

Sealing Techniques

The surface of the aerogel may be sealed by laminating it between two glass plates during production, by coating the surface, or by densifying the surface. The lamination process has been demonstrated[12] and shown to produce stable glazings that have good adherence to the cover plates. The aerogel even shows a strengthening near the surface due to the cover. The edges may be coated with an epoxy, and partially evacuated or permeated with another gas to increase the thermal resistivity. This lamination process to produce a composite aerogel window solves most of the problems with the strength and environmental fragility of the aerogel. One of the few drawbacks is that somewhat more time is required for the supercritical drying process because the fluid must make its way out the sides of the package. Because the complete glazing incorporates two pieces of glass in addition to the aerogel, the first composite windows will be somewhat less transmissive than standard double glazings due to the added scattering in the aerogel. However, with development of the preparation process this degradation can very likely be decreased and should be minimal.

Other methods of sealing the aerogel include coating or spraying the surface with various transparent materials. A simple (but probably not cheap) method would be to vacuum evaporate silicon dioxide onto the surface. However, this would result in a very thin coating that may not have sufficient scratch resistance and may not contribute the necessary strength. Another possibility is to melt the aerogel at its surface. This could be done by using a CO_2 infrared laser tuned to the main lattice absorption bands. This process has the advantage of producing a controllable thickness surface layer that could be tailored to give the best surface characteristics and may provide a partial antireflection coating.

Optical Properties

Another important developmental area is the improvement of the optical properties of the aerogel by reducing the scattering. The cause of the scattering is still not well understood. It may be due primarily to the individual grains of silica acting as very small scattering centers as in Rayleigh scattering[1,20]. Alternatively, it may be caused by larger scale inhomogeneities in the effective index of refraction of the aerogel[21]. The mechanism is important to understand because if it were due to Rayleigh scattering, the method to reduce it would be to make the grains smaller. If it were caused by inhomogeneities, the scattering would be reduced by making more uniform gels. This is a primary area of investigation in the program studying porous optical materials at LBL.

C. Research Requirements for Bench Scale Tests

To investigate the use of aerogel as an insulating glazing, several tasks need to be accomplished. First, an aerogel preparation facility must be set up. Next, the three R&D areas discussed earlier, of improving the optical properties, developing sealing techniques, and investigating alternative production methods need to be pursued. An internal program was initiated at LBL to investigate some of these topics early in 1982, and the DOE Passive Solar Program recently provided funding for R&D in the aerogel material field.

Work has been initiated on an aerogel preparation facility at LBL to produce samples of the material. In a joint effort between the Advanced Micromaterials Program of the Solar Group and the Windows and Daylighting Group at LBL a supercritical drying facility is being constructed to make aerogels. For this purpose an autoclave with a five inch diameter was purchased to perform the supercritical drying of the gel. By mid 1983 we expect to be producing our first aerogels. Thus, work towards bench scale fabrication and testing of an aerogel window is already underway.

As part of the current program, optical measurements, including spectral transmission and angular scattering, will be used to determine the causes of the residual scattering before and after the super critical drying step, and to quantitatively evaluate attempts to reduce scattering . Preparation and drying conditions will be varied to reduce the scattering. If the work proceeds smoothly we will attempt, to produce a small composite aerogel window by forming it in the autoclave between two glass covers. A concerted attack on the sealing problem will probably not be undertaken until FY 1984.

Not included in the current program is a detailed investigation of alternative processes for producing microporous materials. A R&D effort to explore the full range of methods to produce microporous materials would significantly aid the process of bringing this technology to the private sector.

It is suggested that a new effort be initiated to study methods to produce microporous materials that are easier to implement in mass production and will result in more stable material. This work would complement the ongoing program by providing a broader technology base. Alternative processes to be investigated include methods to make microporous material with a closed cell structure. This may be accomplished by processes that evolve micro gas bubbles rather than a liquid during the formation phase of the material. Techniques based on the use of fumed silica as a starting material would also be investigated. One additional scientist would be brought into the program to study material fabrication processes and perform experimental work. The current year's effort would identify the most promising alternative processes and initiate this new experimental work. It would be anticipated that the work could produce new microporous materials during FY 1984.

4. RESOURCES AND DEVELOPMENT

A. Current Research in Porous Optical Materials

Much of the history and recent work was covered in chapters two and three of this report. According to the open literature, most of the present activities in porous optical materials development are confined to producing aerogels and studying their properties for application to Cherenkov detectors.

Professor van Dardel of the Fysiska Institutione at Lund was responsible for interesting several groups in the properties of aerogel. He stimulated a group at the University of Lund to begin production of aerogel. The facility at Lund had produced over 1200 liters of aerogel by mid-1980 for Cherenkov counter use. Van Dardel brought samples of this material to the US in the summer of 1981 and stimulated interest in both the conservation and solar groups at LBL.

Work was initiated on porous optical materials in the spring of 1982 by the Solar Group with internal LBL funding. In the summer of 1982, a program was initiated by the DOE Passive Solar Program to study a number of options for improving the energy performance of a building by controlling the energy fluxes through building apertures. Both the Solar and Conservation Groups at LBL became involved in this program. The Solar Group initiated a program to study the methods of production and the optical and thermal properties of porous optical materials. A broad generic study of glazing types was started by the LBL Conservation (Windows and Daylighting) Group. As part of this work they became convinced of the potential of aerogel and initiated a study of aerogels for insulating glazings. The two groups have been collaborating in setting up a laboratory to produce aerogel and study its properties.

During 1980 and 1981, William Schmitt, a chemical engineering graduate student at the University of Wisconsin, developed an improved technique to produce aerogel for glazing applications. He successfully laminated aerogel between two glass covers to demonstrate its use as a window. After completing his Master's degree work at Wisconsin he moved to MIT to continue his graduate work and at present is not actively involved in aerogel development.

In related work dealing with silica powders, several commercial products have been developed that are based on the gel approach to making silica. Several products, namely Cabosil, made by Cabot Corporation; Aerosil 180, by deGussa Corporation; and Santocell, by Manzanto Corporation have been on the market for several years. These materials are available as silica powders of very small diameters and are used for a variety of purposes. One important and wide spread use is as food fillers (often the so called "inert ingredients" referred to on the label). To the best of the author's knowledge these companies do not produce silica in the form of transparent bulk solids.

B. Private Sector Research

If a successful case can be made to glazing manufacturers that porous optical materials have commercial potential, it is likely that they will undertake a development program to manufacture and test new products. These manufacturers may undertake applied research aimed at improving and expanding their product lines. Because this work would be proprietary in nature it is not likely that much of the results of this work would be made public. Non-governmental research organizations interested in funding or performing further research in porous optical materials could include EPRI, GRI, and individual utilities. As yet none of these groups has been contacted by LBL. Some groups representing building manufacturers may find there is general interest in performing research on these materials, but in general these type of groups do not fund materials research.

5. FUTURE APPLICATIONS AND SPINOFFS

The field of porous optical materials is one of the most exciting recent developments in materials science. Aside from the window glazing applications, there are possibilities of tailoring materials to provide any desired density or index of refraction. By grading the index of refraction within a solid it is possible to make solid blocks of material act as lenses or concentrators. These blocks could be used for building construction, visually exciting architecture, or integral solar collectors. These materials may find a number of niches in solar energy applications. Their use as insulating glazings for flat plate solar collectors was suggested by Professor van Dardel. One of the key questions that arises in considering future applications is what other types of material besides silica can be made both porous and transparent. It may be possible to make materials with unique optical and insulating characteristics, such as virtually transparent walls or windows. From this brief list it can be seen that, as the material is better developed, the number of possibilities is certain to rise.

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