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Three main areas of activity are summarized in this report: (a) The creation of novel optical materials i.e. composites of gel-silica with materials such as pm.m.ma. (acrylic) so as to achieve invariance of the refractive index or optical path during thermal expansion -- a new class of optical materials for worldwide use. (b) The impregnation of porous gel-silica with anthracene, for example, to create a solid composite material capable of emitting blue light by electro-luminescence. This area of activity is aimed at the fabrication of solid state devices that can fill the gap not currently filled by semiconducting devices. Porous gel-silica anthracene complexes may provide at least an intermediate class of devices capable of providing reasonably pure blue light emission under electrical excitation. (c) The impregnation of porous gel-silica with imaging monomer to create thick volume optical elements for optical systems. The methodology is reasonably straightforward and leads to the formation of lenses or gratings in the volume of thick silica layers. Such materials are optically useful for beam steering, head up displays and other applications.

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A Report to the Air Force Office of Scientific Research (AFSC) Bolling Air Force Base, Washington DC Under Grant AFOSR-91-0300 DEF

First Innovation Report for the Year September 1991 - September 1992

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Summary

Four main areas of activity are summarised.

(a) The creation of novel optical materials i.e. composites of gel-silica with materials such as p.m.m.a. (acrylic) so as to achieve invariance of the refractive index or optical path during thermal expansion — a new class of optical materials for worldwide use.

(b) The impregnation of porous gel-silica with anthracene, for example, to create a solid composite material capable of emitting blue light by electro-luminescence. This area of activity is aimed at the fabrication of solid state devices that can fill the gap not currently filled by semiconducting devices. Porous gel-silica anthracene complexes may provide at least an intermediate class of devices capable of providing reasonably pure blue light emission under electrical excitation.

(c) The impregnation of porous gel-silica with imaging monomer to create thick volume optical elements for optical systems. By agreement with Du Pont in Wilmington Delaware, we have attempted the impregnation of porous gel-silica with several of the Du Pont variable index monomers. The methodology is reasonably straightforward and leads to the formation of lenses or gratings in the volume of thick silica layers. Such materials are optically useful for beam steering, head up displays and other applications. The formation of overcoated edge-illuminated holograms is a precursor to the main thrust of this section.

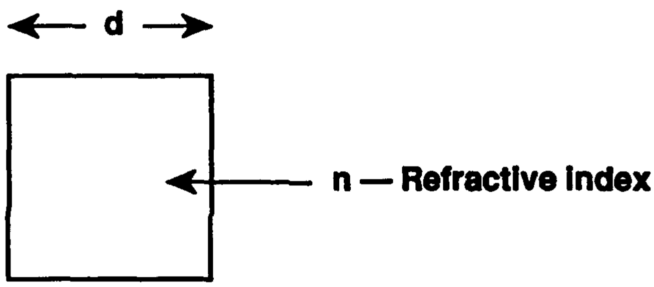
(d) The use of porous silica for the construction of novel explosive systems. This work is to be subject to discussion between ourselves, Professor Larry Hench of the University of Florida and Imperial Chemical Industries (UK and Canada).

(a) The thermal properties of gel-silica impregnated with materials such as p.m.m.a. — The qûest for a material whose refractive index is invariant with temperature

In a detailed study to the USAF (Upper Heyford Contract No. F6170890W0484) we point out that there is a possibility of compensating the change of refractive index of an optical solid such as silica by impregnation with a material such as p.m.m.a. (acrylic). Silica exhibits a positive coefficient of thermal dispersion i.e. its refractive index increases when its temperature increases. On the other hand p.m.m.a. behaves like a liquid. Its refractive index decreases when its temperature increases. We argue that an impregnated composite — p.m.m.a. in porous gel-silica — might be constructed to provide a balance between these properties and hence create a thermally invariant material with respect to the refractive index. We point out that actually, what is required is a material that exhibits no change of optical path as thermal changes occur. Again, we have outlined the demands of this problem and we make a strong case for further study on the grounds that we could be looking at a whole range of important new optical materials with uses worldwide as a replacement for simple thermally unstable optical materials.

(a) Porous silica impregnated with p.m.m.a. (acrylic)

A potential new material for general optical use — exhibiting invariance of refractive index with temperature



Example of solid optical material

The optical path nd changes with temperature T

$$\frac{\partial}{\partial T}(nd) = \underbrace{n\frac{\partial d}{\partial T}}_{\substack{\text{Thermal}\\ \text{expansion}}} + \underbrace{d\frac{\partial n}{\partial T}}_{\substack{\text{Thermal}\\ \text{dispersion}}}$$

We define coefficients thus

$$\varepsilon = \frac{1}{d}\frac{\partial d}{\partial T} \quad \text{and} \quad \gamma = \frac{1}{n}\frac{\partial n}{\partial T}$$

ε is *positive* for silica and p.m.m.a.

γ is *positive* for silica and *negative* for p.m.m.a.

We propose that silica - p.m.m.a./ composites might exhibit compensation of effects to produce either an invariant refractive index or an invariant optical path as temperature changes

Such materials would have general worldwide use in robust-lightweight optics in widely varying temperature conditions

Samples of a perfected form of impregnated silica are now under investigation

Samples are to include:

Silica + p.m.m.a.

and

Silica + titania + p.m.m.a.

The latter material is expected to be especially low in thermal expansion

Section (a) is considered in detail based on four years of work under:

Department of the Air Force R.A.F. Upper Heyford

Contract number F6170890W0484

(b) The search for a compact source of blue light using porous silica impregnated with anthracene

This work has been triggered off by the observation of important Israeli studies noting that a sub-wavelength source of blue light can be constructed by filling the bore of a micro pipette with anthracene. The idea is that if the base of the micro pipette is reduced in size by pulling so that its tip is in fact sub-wavelength in diameter, then an orifice smaller than the wavelength of visible (blue) light can be created. Anthracene can be excited by electronic means or by the action of ultra-violet light so that it fluoresces in the blue. However, the communication of energy through the bulk of single crystal anthracene is **excitonic** i.e. by quanta whose De Broglie wavelength is smaller than the wavelength of visible light (perhaps of the order of 10 Å). Such quanta can propagate down the bore of a micro pipette without the limitations imposed by diffraction on the propagation of quanta of much larger (visible) wavelengths.

In the cited Israeli work, the anthracene was excited by ultra-violet light fed to the large diameter end of the pipette and the emergence of blue fluorescent light from the tip was observed to come from a tip size below the wavelength of the emitted light e.g. a spot size of approximately 20 nm was quoted.

We conclude that a material of high structural strength exhibiting the fluorescent properties of anthracene pure can be fabricated by pore filling of gel-silica with anthracene. Such a material would have general use in the fabrication of sources of blue light competitive with and more simply expedited than semiconductor structures.

Fluorescence created by ultra-violet radiaton can be replaced by **electro-luminescence** created by direct electrical excitation thus leading to electrically driven devices.

(b) Impregnation of porous silica with anthracene

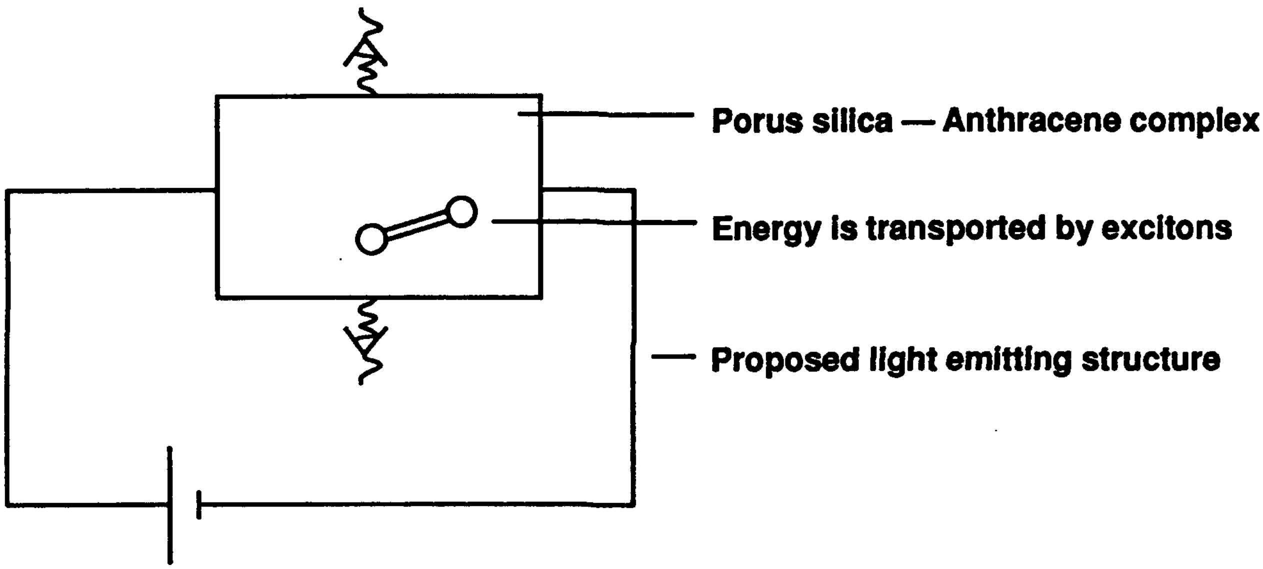
The search for a new form of blue light source

Anthracene fluoresces *blue* under ultra-violet irradiation — it also electroluminesces *blue*

It must be single crystal in form

Energy communicates via *excitons* which have a de Broglie wavelength ≈ 10 Å i.e. much smaller than that of blue photons c 4500 Å

We are attempting impregnation of porous gel-silica with anthracene



We have used gel-silica with intercommunicating pores of size ≈ 40 Å - 75 Å

We hope to produce a competitor for normal semiconductor blue sources (notoriously unsuccessful to date)

(c) The impregnation of porous gel-silica with imaging monomer to create thick volume optical elements for optical systems

Holographic, diffractive and refractive optical elements can be manufactured by using variable index media such as photopolymers. Du Pont in Wilmington, Delaware have been researching and manufacturing such materials for a number of years since the early 1970s. Only in recent years have such materials been taken seriously and with special emphasis on military and non-military head up displays.

If holographic optical elements (complex diffraction gratings) are made very thick, they can become very selective of direction and wavelength of incident light. However, if we make a very thick layer of a non-rigid polymer (say 1 - 2 mm thick) then the integrity may not be sufficient to provide the necessary optical rigour of performance.

The concept of a very thick silica-polymer element is attractive since such an element could be non-fragile due to the ingression of polymer and can be very light i.e. about half the weight of silica pure.

In our work, we have impregnated porous gel-silica with Du Pont monomer and have then imaged into the impregnated monomer using coherent light. Thick diffraction gratings have been successfully recorded.

One of the basic difficulties of the method is that the impregnated monomer must have a finite mobility within the pores of silica so it usually contains solvents to control its viscosity. We have been able to cope with this situation by exposing a thin skin of the impregnated sample to short wavelength ultra-violet light thus creating something like a candy with a soft centre.

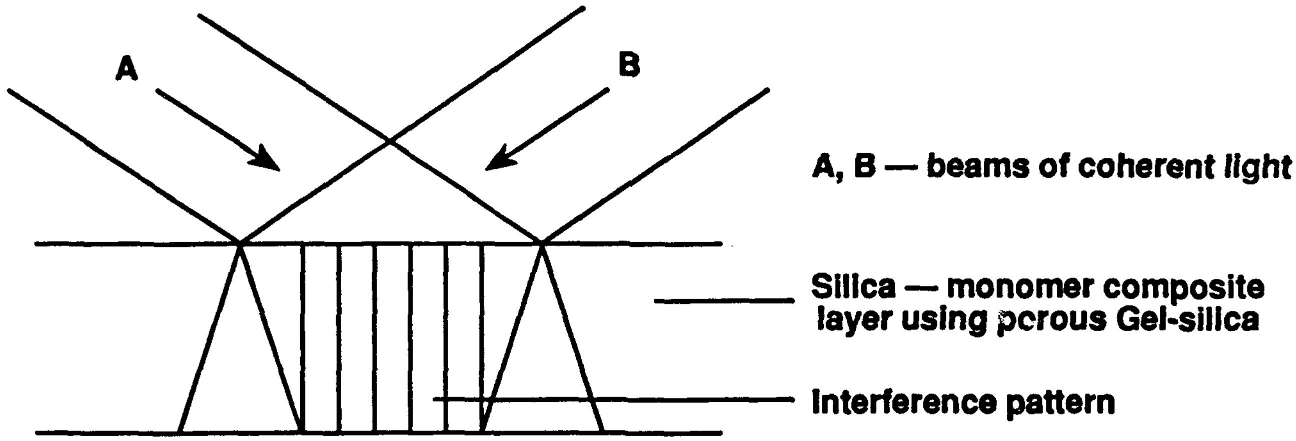
Novel refractive and diffractive elements are to be constructed in ensuing work.

As a pre-cursor to the true volume imaging media studies, we are creating edge-illuminated holograms using Du Pont photopolymer overcoated on glass or silica substrates. This geometry is an essentially new regime of holography.

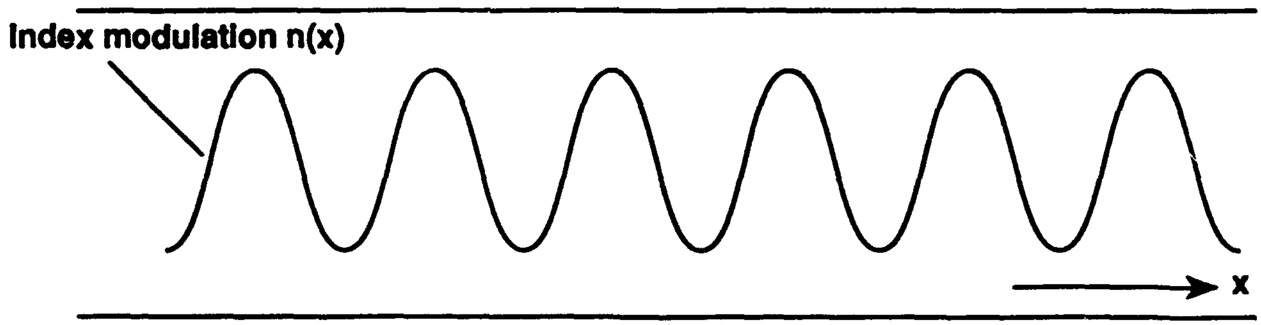
(c) Polymer-silica optical elements constructed from gel-silica impregnated with Du Pont imaging monomer

Du Pont manufacture variable monomer-polymer systems

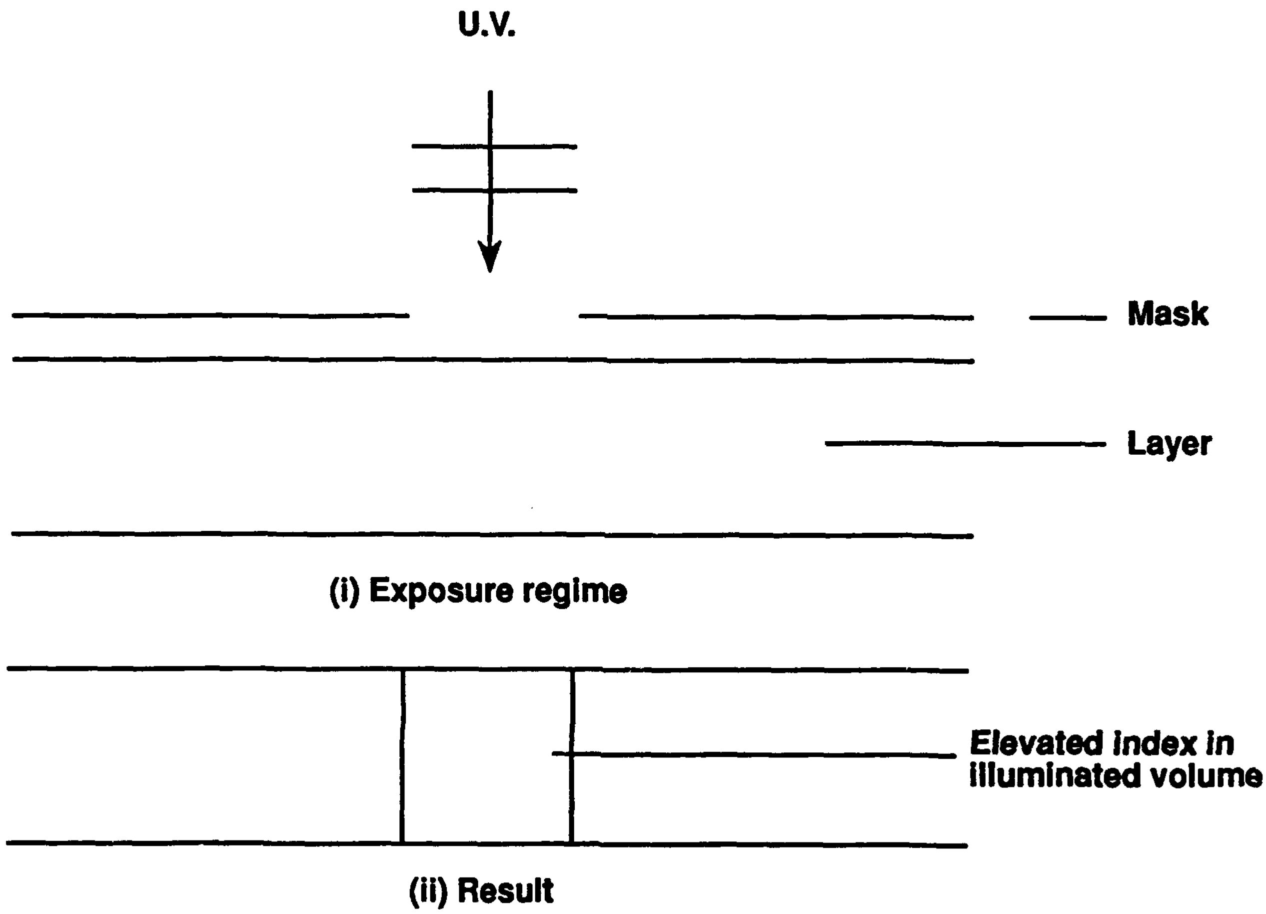
These work as follows:



Monomer diffuses towards antinodes of pattern thus creating refractive index variation in the layer

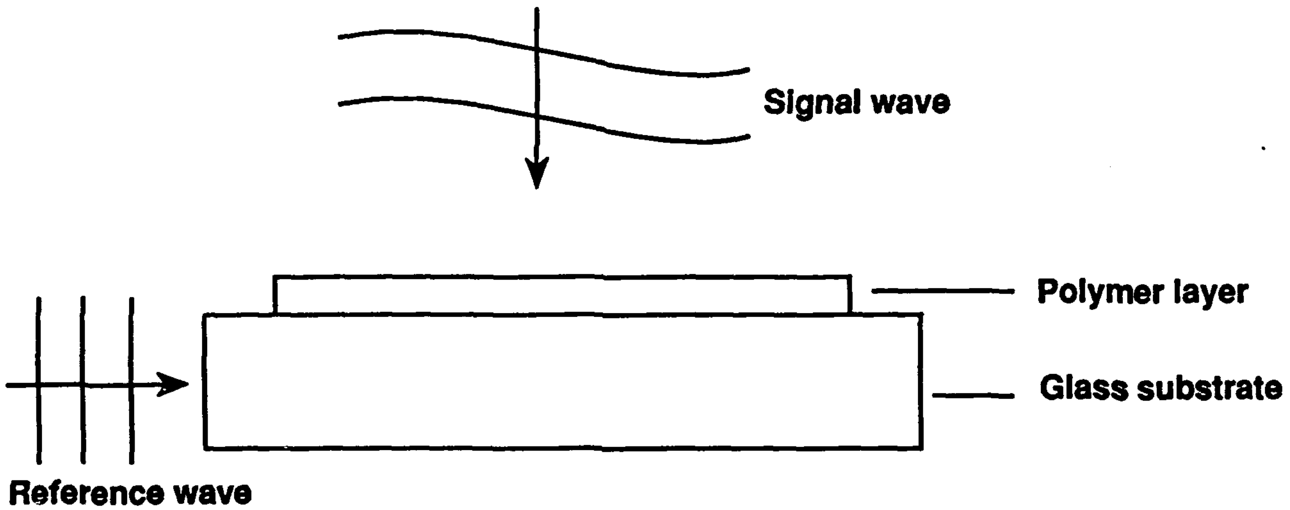


Optical elements have been created in thick gel-silica by interference as above or by lithography as below

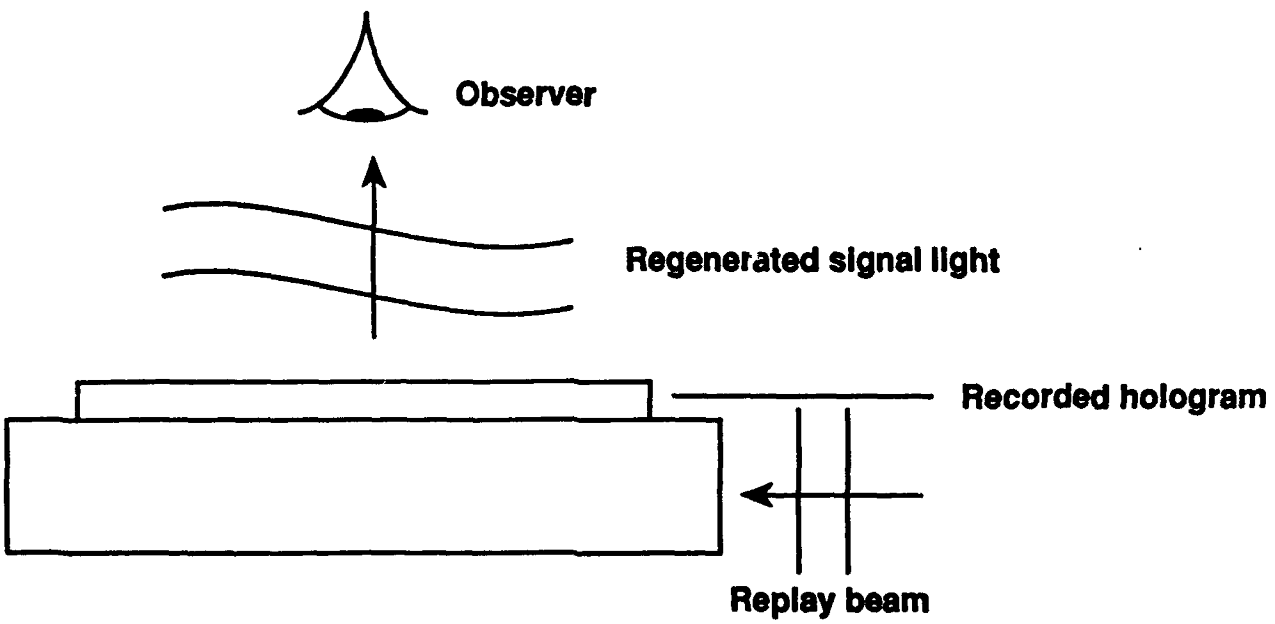


Such elements can be very selective of wavelength and angle of light and are robust because polymer-silica composite is non-fragile unlike pure silica

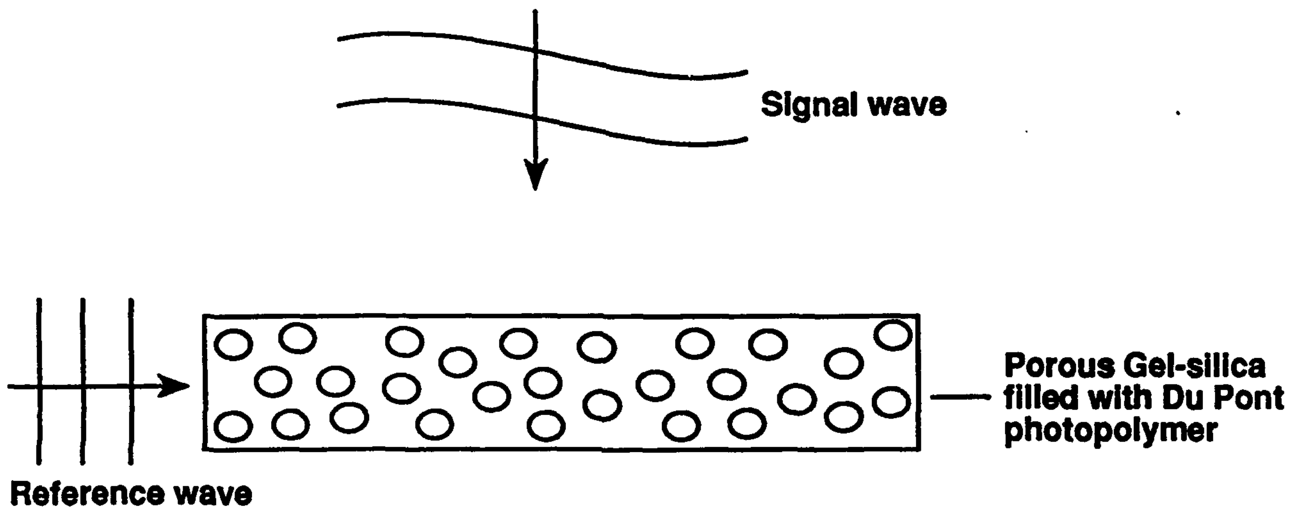
(c) As a pre-cursor to the manufacture of thick silica polymer gratings, we have successfully made edge-illuminated holograms using a novel Du Pont photopolymer coated on to the surface of a glass substrate

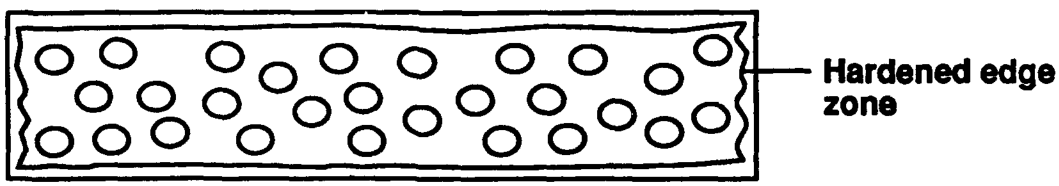


Such holograms replay only when edge-illuminated and typically recreate images of 3D objects when replayed as below



Our final aim is to create a solid silica-polymer composite hologram





Encapsulation of soft monomer is achieved using short wavelength u.v. exposure — Structure is like a candy with a soft centre

Studies are under completion

(d) The use of porous silica for the construction of novel explosive systems.

In this latter category, we remind the reader that the primary invention of Dynamite by Alfred Nobel involved the impregnation of a natural siliceous earth (Kieselguhr) with nitroglycerine. The rôle of the 'earth' was to act as a porous host of high specific surface in which the nitroglycerine would remain stable and free from spontaneous detonation. It is our belief that specialised shaped charges could be fabricated and that because of development by U.F. in the area of monodispersed pores, considerable control over explosive effects could be exerted.

This idea was first suggested by N.J. Phillips in the early part of this year's activity but has awaited the arrival of Professor Hench for his sabbatical year in the U.K. in order to expedite the discussion. It is not intended that fabrication of systems be undertaken at Loughborough University since we are not equipped with the necessary chambers for explosive study. A case can be made however, for certain general patent applications in the area.

(d) Construction of novel explosive systems

Alfred Nobel invented Dynamite (late 19th century)

His innovation was to impregnate unstable nitroglycerine into Kieselguhr, a naturally occurring siliceous (fossilised) earth

This earth has an unusually high specific surface

The analogy with porous gel-silica is very strong

Much interest centres on controlling explosive reactions by molecular size and concentration

Recent developments at U.F. under Professor Larry Hench in the area of monodispersed pore sizes open the door to incorporating explosives into porous gel-silica

Shaped charges of a novel kind with controlled reaction inhomogeneity should be possible

An impregnation of nitroglycerine into 'Kieselguhr'

This work to be progressed during Professor Hench's U.K. sabbatical year beginning October 1992 with Imperial Chemical Industries (U.K. and Canada)