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SBIR Final Report
Liquid Core Optical Scintillating Fibers

Department of Energy
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INTRODUCTION

This Phase I SBIR project focussed on developing flexible scintillating liquid core optical fibers, with potential uses in high energy calorimetry, tracking, preradiators, active targets or other fast detectors. We hereby summarize the progress on each of the Tasks in the project. The technical developments involve 3 technology components:

(1) Highly flexible capillaries or tubes of relatively low n (index of refraction) to serve as cladding and liquid core containment.
(2) Scintillator (and clear) fluids of relatively high n to serve as a core. These fluids must have a high light transmission, and for some applications, radiation hardness.
(3) Optical end plugs, plug insertion, and plug-cladding tube sealing technology to contain the core fluids in the tubes, and to transmit the light.

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Task 1: Liquid Scintillator and Core Liquid Acquisition and Properties

The best high index liquid scintillator produced was based on reagent grade 99.9% benzyl alcohol as the active solvent. A visible attenuation length was measured to be 1.3 m using a HeNe laser over a 80 cm distance 2 cm dia. x 30 cm pipe. A Si pin diode was immersed in the fluid and moved inside the fluid, with measurements every 5 mm. A 50-50 mix of toluene and pseudocumene were used to predissolve a concentrated mix of fluor solutes. Nile blue nitrate(~1% M) and PPD (~200 g/l) were the best solutes for benzyl alcohol. This mixture was then diluted 20:1 by volume with benzyl alcohol. This proved to have the best optical properties of any of the other solvents. A commercial refractometer measured the index n=1.54 at 540 nm. It had a light yield relative to to a liquid scintillator with 40% anthracene light output of about 50% as measured using both ^{241}Am or a beta source immersed in 3 mm of the LS in a glass dish, and viewed by a 2" PMT coupled directly to the liquid surface.

The isopropyl biphenyl we obtained proved to be too absorbing (L~50 cm using the HeNe) to be useful as a long fiber core material, although with PPO(1 g/liter)/bisMSB(0.1 g/l) it had a light yield nearly equal to the benzyl alcohol over the 3 mm distance of the Am source measurement.

The phenyl and methyl naphthalene samples obtained from 2 chemical houses and from Bicron appeared yellow in spectrometer flasks, and therefore were not used.

Figure 1: Photograph of some liquid scintillator preparations used for liquid core fiber studies.

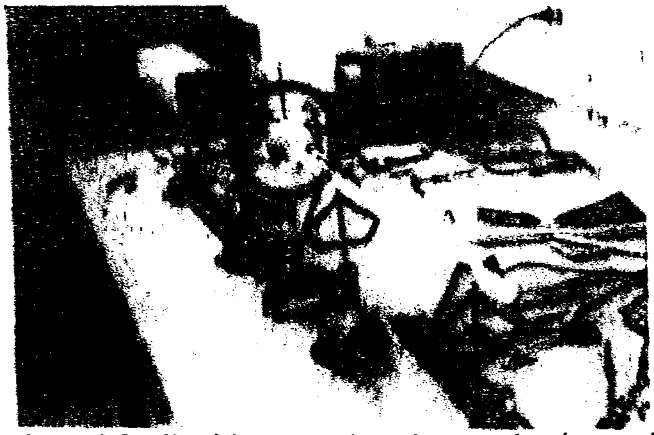


Figure 2: Optical bench used for liquid attenuation characterization and fiber transmission and NA measurements.

Task 2: Cladding Materials and their Characterization

Quartz capillaries with a diameter of 800 μm with 50 μm walls were obtained, with the results as shown in Fig. 3a. Polyimide capillaries (Fig.3b) in diameters $0.1<d<1$ mm were obtained in small lengths of 10-15 cm. - PEEK tubes with diameters 1 mm were also obtained in 3 mm diameters. They proved to be useless for transmission of light using the fluids proposed. Teflon and Tefzel capillaries in diameters of 3 and 4 mm and ID of 2 and 3 mm were obtained (see figs. 3 below).

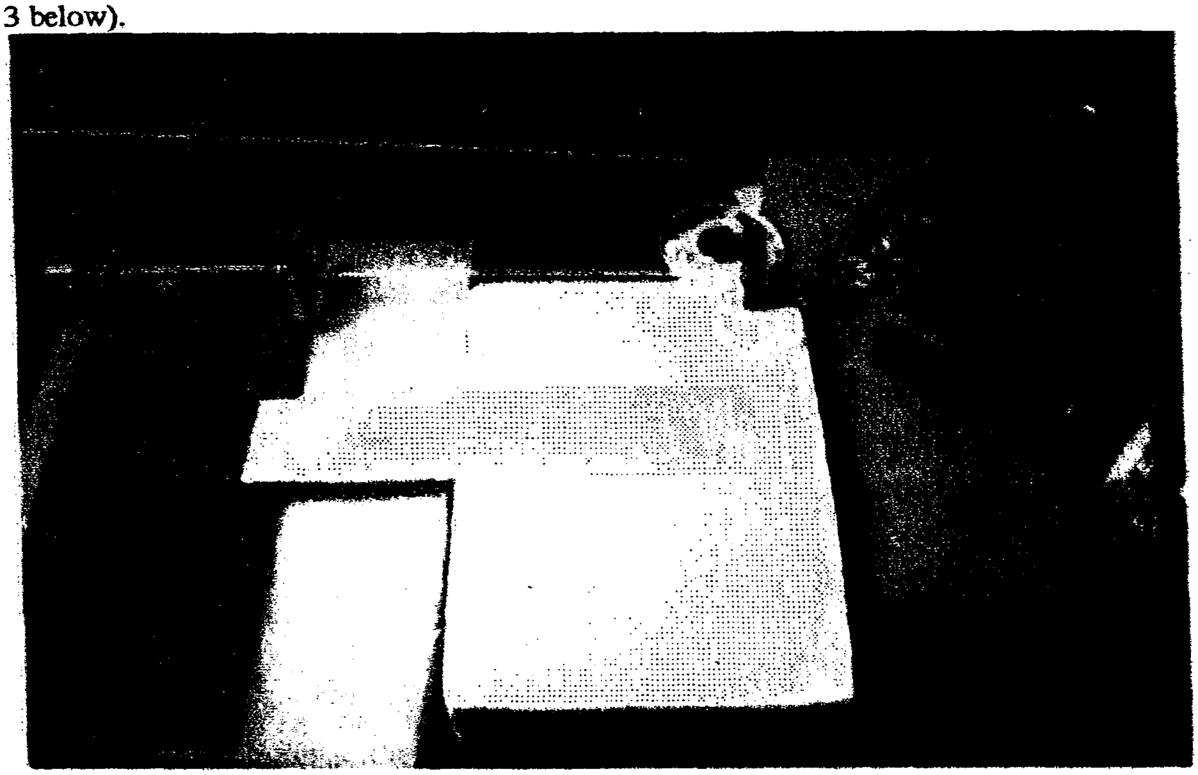


Fig. 3a: Flexible quartz capillary liquid scintillating fiber, shown in a measuring rig with photomultiplier tubes.

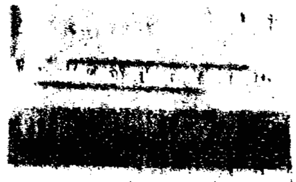


Fig 3b: Polyimide capillary tubes obtained for this project.

Task 3: Optical Fluid Plugs

Optical plugs were fabricated from compression fitted clear rods of plastic and glass matched to the fluorinated polymer tubing. The plugs were sized 10 μm over the ID of the tube for a good compression fit for tubing of 2 mm ID in size and 15 μm for the 3 mm ID tubing. Figure4a,b shows these plugs:

The polyimide and PEEK capillaries proved incapable of retaining fluid with an elastomer adhesives or optical epoxy plugs as obtained from materials from Epotek, Norland (particularly NOA 61, 65, 68) and Summers Labs (J-91). The plugs were fabricated by sacrificial casts of the material in tubing samples; the plugs were then cut out of the capillary "moulds", and then the diameter was slightly increased by a thin film of very low viscosity epoxy.Slight bending was enough to cause these plugs to fail. We speculate that the tubing material is not elastic enough to form a good compression fit.

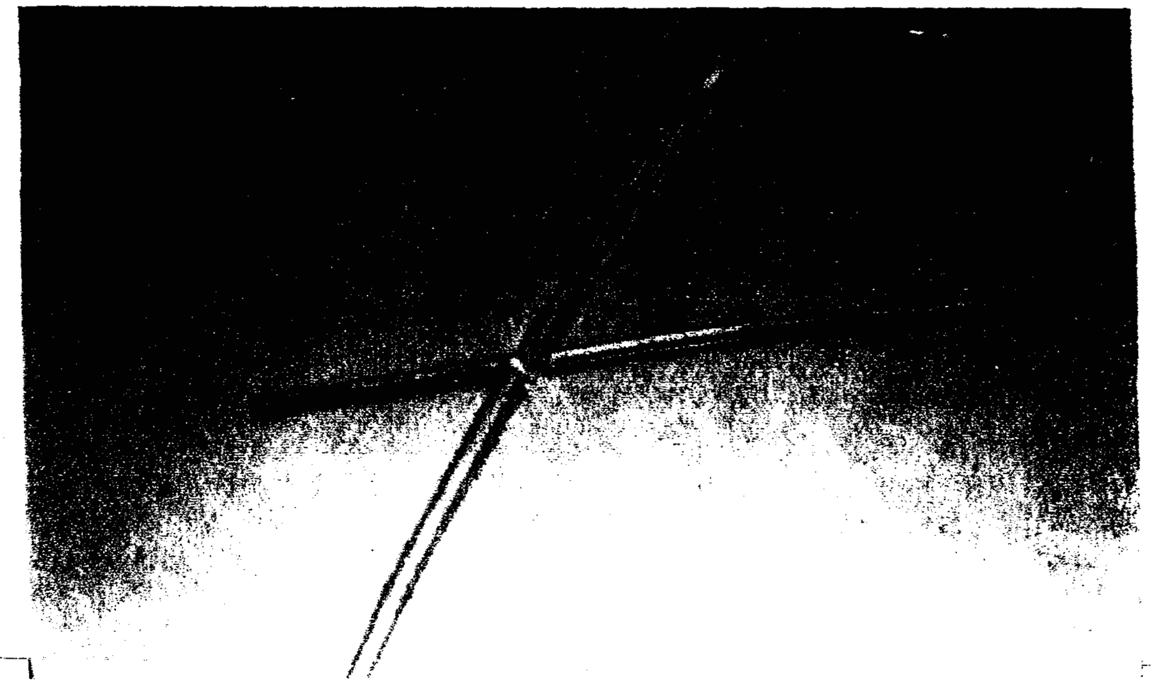


Fig. 4a: Tefzel (top, 2mm) liquid core fibers, shown with a quartz rod endplug.

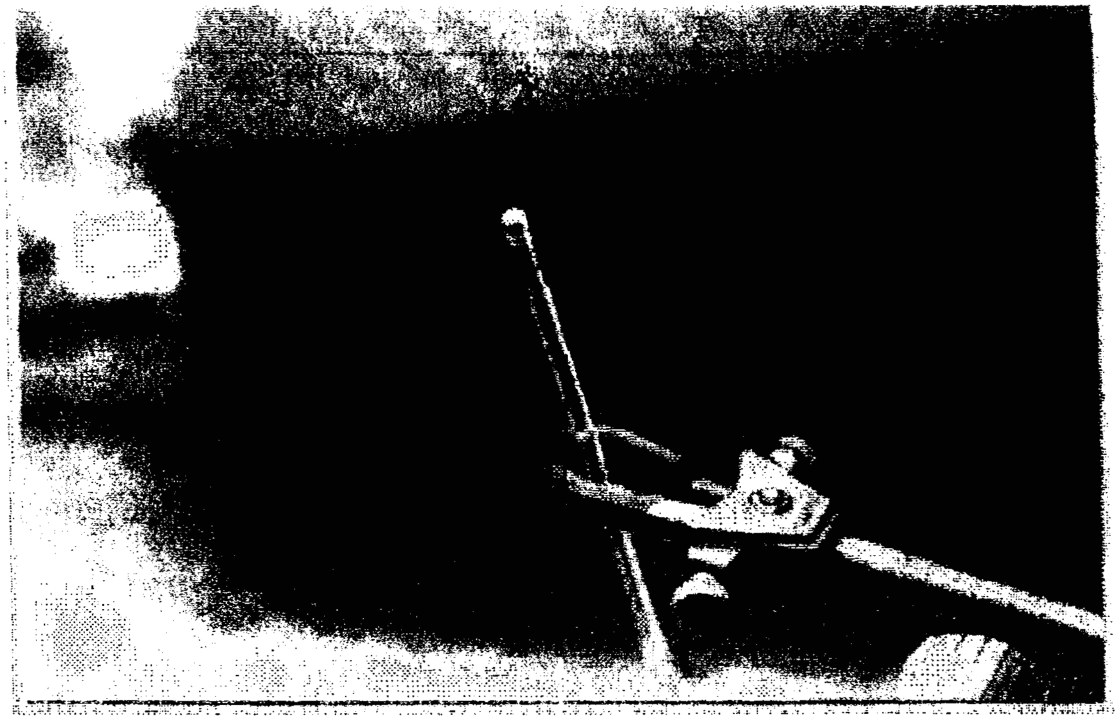


Fig. 4a: Teflon (bottom 3 mm) liquid core fibers, shown with a quartz rod endplug.

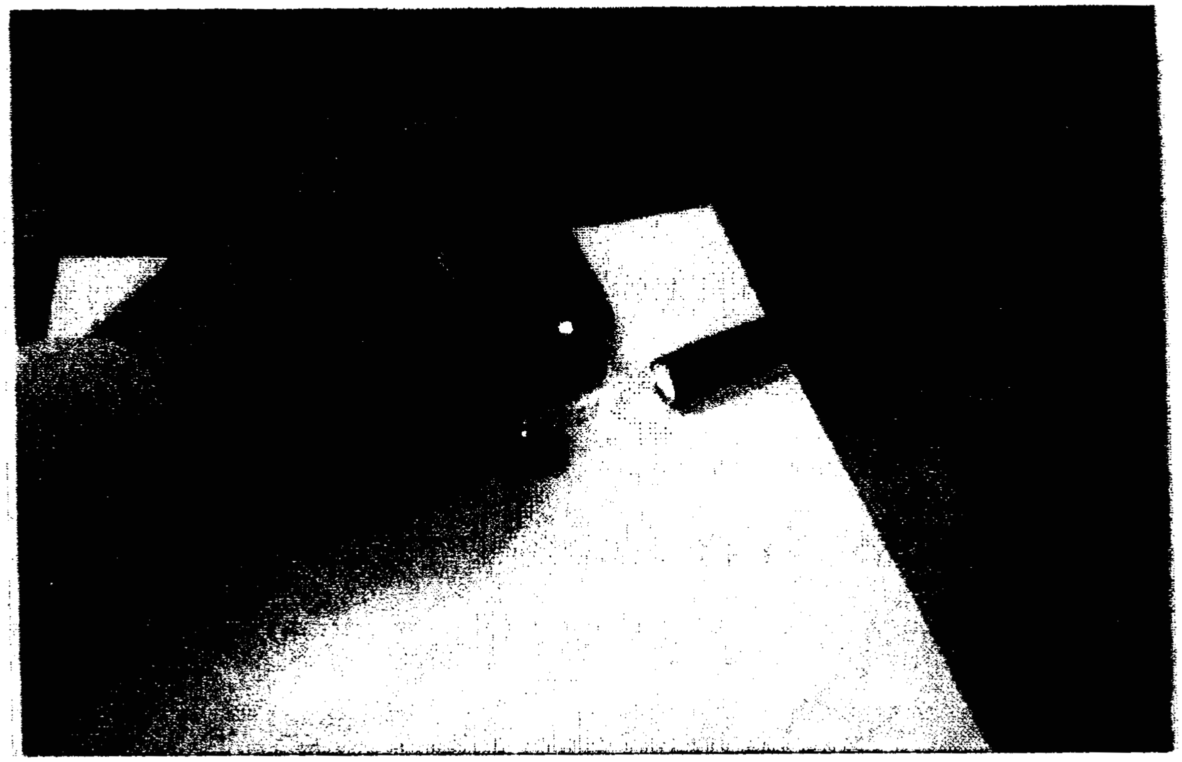
Task 4: Fluid Fill and Seal Techniques.

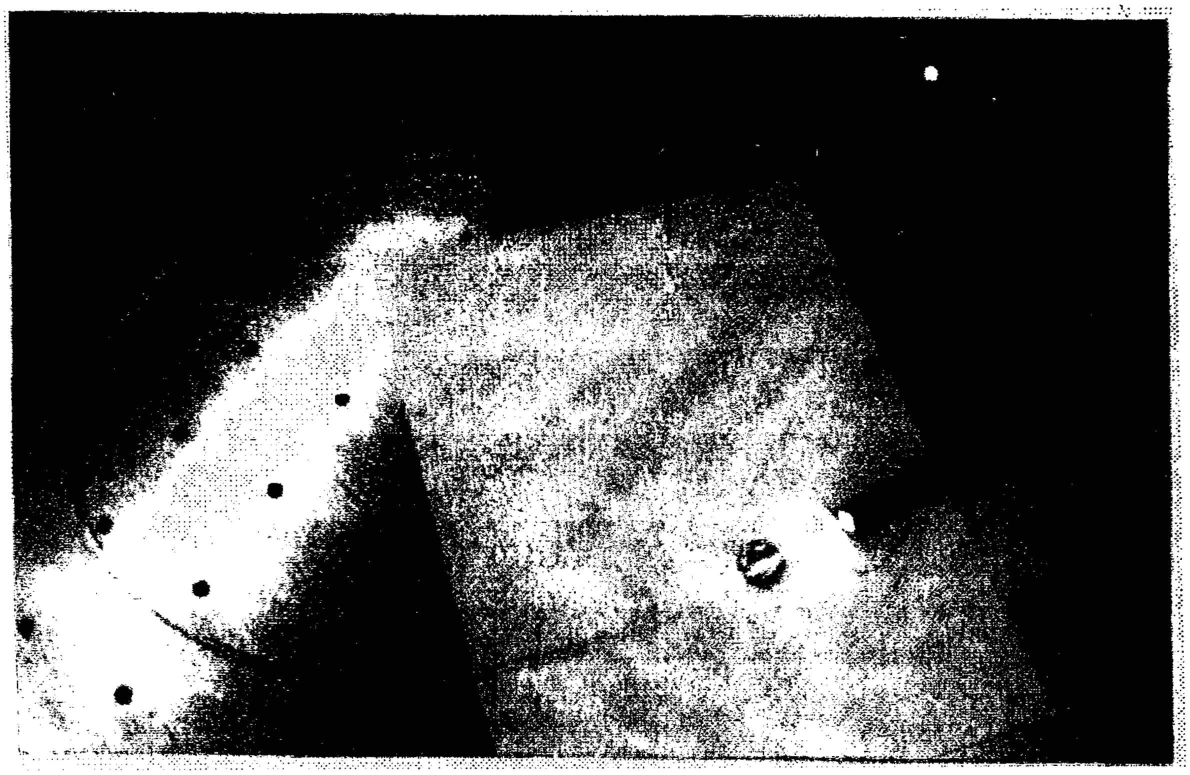
Plug seals on the teflon tubes were produced by a total immersion process, where the tubes were plugged by hand in a basin of scintillator. (See above figures 4). A post-plug mechanical aid by heat-shrink teflon films proved disastrous, causing warping of the capillary walls.

A suction vacuum method was able to successfully fill and plug a teflon capillary. A liquid vessel consisting of a 1 m long vertically oriented teflon tube, 1/2" OD was used to fill the capillaries. The vessel was stoppered on the bottom with a threaded blind plug. The top is capped by threaded cross, to form a t-fitting on the top. Each of the 2 side-branches was valved. with one side connected to a small roughing pump, and the other to a supply of liquid scintillator with a gravity feed (which was seldom used). The top end of the cross (vertical) was fitted with a standard o-ring seal vacuum fitting for passing a 2 mm lucite rod, which was the plastic plug material (lucite). The end of this plug rod to be inserted into the capillary to plug it was scored with axially oriented grooves (like the rifling of a gun by without any twist) at most 2 mm long and 0.3 mm deep with a metal scribe, in order to let the air out of the capillary with the rod inserted just 0.5-1 mm into the capillary (in order to "start" the plug externally). An 80 cm long x 3mm OD teflon capillary tube with a plugged end was prepared, with a 2[nd] plug-rod inserted and withdrawn for prestretching to fit. This prepared capillary was then inserted into a very close-fitting SS tube, about 1 mm shorter than the teflon capillary, for stability and structural strength. The thin wall SS structural support tube (2 mil walls) was supported by slotted centering plugs every ~2-3" along the tube, placed inside the 1/2" teflon liquid "vessel". The scintillator fluid was injected into the teflon capillary by a syringe before the cross was threaded in place, to a predetermined level, 3 mm from the top of the liquid fiber tube. The cross with the 2 mm diameter plug rod in the o-ring seal was then threaded in place, with the plug-roid with its scored end pre-inserted ~1 mm into the capillary, and the vacuum returned to a low pressure (<0.1 Torr). The sealing o-ring fitting around the plug rod was then carefully loosened and the plug rod inserted another 5 mm into the teflon capillary. The vacuum was then let off and the capillary and plug rod assembly removed. The plug rod was then trimmed back to length, nearly flush with end of the capillary, and hand polished with a fine alumina grit followed by toothpaste. Figure 3 shows the results.

Task 5: Fiber Measurements

The selected liquid core fibers using teflon, tefzel and silica cladding capillaries were taken to an optical bench (Fig. 1) and measured for light transmission vs length, in the blue, green and red to determine light attenuation, and the NA=sin θ_m calculated on the basis of the injected and transmitted light pattern. We measured the output light cone angle by the diameter of the light cone at a fixed distance, and calibrating it by using 2 commercial clear fibers of known NA (0.22, and 0.46). The calibration showed the measurement to be only about 10% accurate. Figures 5 gives a visual impression using a flashlight on tefzel(3mm) and teflon(2mm) capillaries. The scintillation light yield was inferred from relative measurements of a well-characterized plastic scintillating fiber and plastic scintillating rod when exposed to a highly collimated beta source and to an 80KV endpoint x-ray source (Fig. 6). A high gain photomultiplier tube (R329) was be coupled to the fibers using plastic "cookies" (clear UVT lucite cylinders)with center coaxial holes just larger than the fiber diameters to accept insertion of the fibers (Figure 3a). The charge in the pulses was integrated by a digital oscilloscope or the x-ray induced current by a pAmmeter and compared with a standard blue scintillating plastic fiber of similar length from Bicron Inc. Figure 7 shows pulses obtained with a 3 mm teflon liquid scintillator fiber, showing them quantized – about 4-5 p.e. level.





Figures 5: transmission of light through liquid fibers produced by this project
(Top: 2mm tefzel, Bottome: 3 mm teflon)

Figure 6: X-ray generator rig in preparation for exposing scintillating liquid core optical fibers. A quartz fiber can be barely seen mounted on the glass slide.

Fiber	*Liq. Core Dia*	*Attenuation*	*NA*	*Light/Plastic*†
quartz	0.8 mm	81±6 cm	0.26±0.3	0.27±0.09
teflon	1.7 mm	98±7 cm	0.41±.05	0.96±0.11

†Light/Plastic is the ratio of detected light from the liquid fiber to that of a high quality 1mm plastic fiber from Bicron, of similar length, under identical conditions.

Figure 7: Pulse from a collimated triggered beta source through a teflon fiber. Superimposed are noise pulses for comparison.

Task 6: Final Report – herein.