

MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS-1963-A

AD-A143 565

INVESTIGATION OF OPTICAL FIBERS FOR NONLINEAR OPTICS

L.G. DeShazer, J.A. Harrington, A.C. Pastor, and S.C. Rand

Hughes Research Laboratories
3011 Malibu Canyon Road
Malibu, CA 90265

APRIL 1984

F49620-82-C-0030

Final Report

1 January 1983 through 31 December 1983

U.S. AIR FORCE OFFICE OF SCIENTIFIC RESEARCH
Bolling Air Force Base, DC 20332

DTIC
12-1

DTIC FILE COPY

84 07 25 149

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE

AD A 143 565

REPORT DOCUMENTATION PAGE

1a. REPORT SECURITY CLASSIFICATION UNCLASSIFIED		1b. RESTRICTIVE MARKINGS Proprietary	
2a. SECURITY CLASSIFICATION AUTHORITY		3. DISTRIBUTION/AVAILABILITY OF REPORT	
2b. DECLASSIFICATION/DOWNGRADING SCHEDULE		Approved for public release:	
4. PERFORMING ORGANIZATION REPORT NUMBER(S)		5. MONITORING ORGANIZATION REPORT NUMBER(S) AFOSR-TR. 04-0114	
6a. NAME OF PERFORMING ORGANIZATION Hughes Research Laboratories	6b. OFFICE SYMBOL (If applicable)	7a. NAME OF MONITORING ORGANIZATION AFSC, Bolling AFB, DC 20332 AFOSR	
6c. ADDRESS (City, State and ZIP Code) 3011 Malibu Canyon Road Malibu, CA 90265		7b. ADDRESS (City, State and ZIP Code)	
8a. NAME OF FUNDING/SPONSORING ORGANIZATION USAF, AFSC	8b. OFFICE SYMBOL (If applicable)	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER F49620-82-C-0030	
8c. ADDRESS (City, State and ZIP Code) Air Force Office of Scientific Research Bolling AFB, DC 20332		10. SOURCE OF FUNDING NOS.	
11. TITLE (Include Security Classification) Investigation of Optical Fibers for Nonlinear Optics		PROGRAM ELEMENT NO. 61102F	PROJECT NO. 2301
12. PERSONAL AUTHOR(S) Larry DeShazer, James Harrington, Antonio Pastor, Stephen Rand		TASK NO. A1	WORK UNIT NO.
13a. TYPE OF REPORT Final	13b. TIME COVERED - From 1 Jan 83 to 31 Dec 83	14. DATE OF REPORT (Yr., Mo., Day) 1984, April 17	15. PAGE COUNT 42
16. SUPPLEMENTARY NOTATION			
17. COSATI CODES		18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)	
FIELD	GROUP	SUB. GR.	Fiber optics, optical materials, single crystal fibers, evanescent wave coupling, hybrid fibers
19. ABSTRACT (Continue on reverse if necessary and identify by block number) The principal objective of this research program is to develop single crystal (SC) fibers for use in nonlinear optical devices. This encompasses measurement of physical and chemical properties of several candidate materials, fabrication of SC fibers, and investigation of nonlinear optical applications. This report describes the successful fabrication of more than a half dozen single crystal fibers using new growth techniques and progress with hybrid fibers which show great promise for nonlinear optical applications.			
20. DISTRIBUTION/AVAILABILITY OF ABSTRACT UNCLASSIFIED/UNLIMITED <input type="checkbox"/> SAME AS RPT <input checked="" type="checkbox"/> DTIC USERS <input type="checkbox"/>		21. ABSTRACT SECURITY CLASSIFICATION UNCLASSIFIED	
22a. NAME OF RESPONSIBLE INDIVIDUAL Larry DeSahzer		22b. TELEPHONE NUMBER (Include Area Code) 703/7769-4908	22c. OFFICE SYMBOL 711

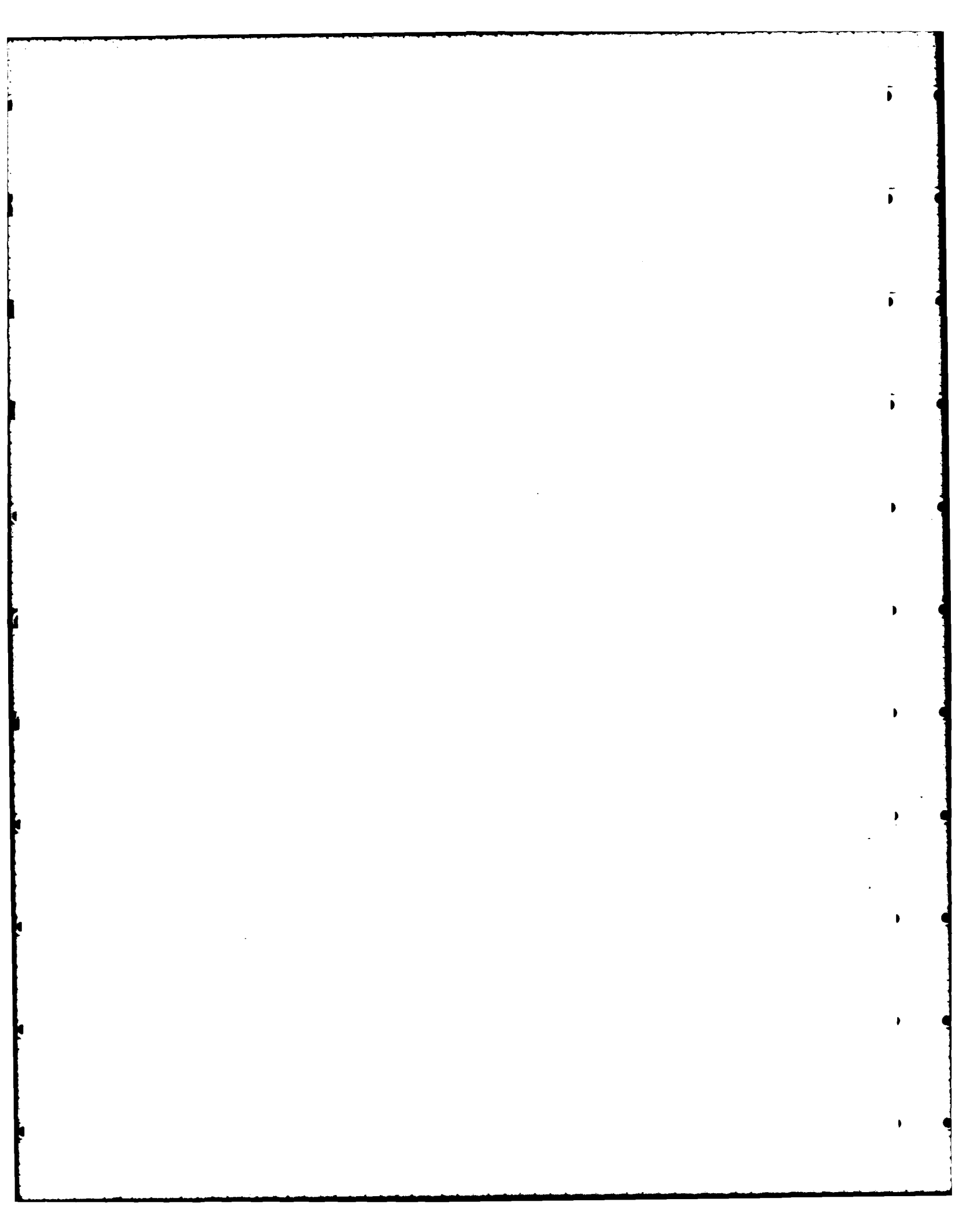
DD FORM 1473, 83 APR

EDITION OF 1 JAN 73 IS OBSOLETE

UNCLASSIFIED
SECURITY CLASSIFICATION OF THIS PAGE

LIST OF ILLUSTRATIONS

FIGURE		PAGE
1	(a) A schematic diagram of traveling zone apparatus.....	4
	(b) Traveling zone apparatus with horizontally mounted fiber.....	5
2	CuCl single crystal.....	8
3	Schematic diagram of the capillary-fed Czochralski apparatus for growth of SC fiber....	12
4	Schematic diagram of nucleation in the capillary-fed Czochralski method.....	13
5	Capillary-fed Czochralski apparatus.....	14
6	Methods of fabricating second order nonlinear fibers.....	16
7	The critical angle θ_c for fibers embedded in uniaxial crystals.....	18
8	Experimental geometry for demonstration of non-critically phase-matched 3-wave mixing in a hybrid ADP fiber.....	19
9	Laue back-reflection x-ray diffraction from end of NaNO_2 fiber showing single crystallinity.....	21
10	Laue back reflection photograph of ADP showing single crystallinity and direction of growth along the optic axis.....	22
11	NaNO_2 single crystal fiber viewed between crossed polarizers (a) as grown, (b) after methanol treatment for 4 minutes.....	24
12	SC fiber of ADP encased in quartz capillary (a) Transmission of unpolarized light (b) transmission between crossed polarizers.....	25
13	Fiber spectrometer.....	27
14	UV transmission spectrum of ADP fiber.....	28
15	IR transmission of bulk, single-crystal CuCl....	29
16	AgBr SC fiber with growth axis along (100).....	30
17	(110) Slip planes resulting from tensile stress applied along the axis of AgBr SC fiber.....	30



SECTION 1

RESEARCH OBJECTIVES

The main objective of this contract is to find methods of fabricating single crystal nonlinear optical fibers. Successful techniques are to be utilized to make crystal fibers that will be useful for nonlinear devices, particularly in the infrared. Device concepts and applications are also to be formulated. This work has been divided into three parts which are described below.

Task 1 entails the measurement of physical and chemical properties of linear and nonlinear materials relevant to the preparation of single crystal and nonlinear fibers. Differential thermal analysis (DTA), vapor pressure analysis and thermogravimetric methods (TGA) are employed for this purpose. The emphasis is on selection of fiber growth methods most appropriate for particular materials or classes of materials. The scope of methods investigated is intended to be broad in order to encompass at least three different candidate materials.

Under Task 2 a variety of methods for production of single crystal (SC) fibers are to be developed, recognizing that no one method can be applied even in principle to SC fiber growth of all materials of interest. Efforts will be directed toward, but not limited to, the study of:

- Traveling Zone Method - A method for conversion of polycrystalline fibers to SC fibers. This technique uses a small heater to recrystallize small sections of extruded polycrystalline fiber which pass through a hot zone on rollers. Long lengths are possible in principle for congruent materials.
- Vapor-Stabilized Bridgman Method - A Bridgman method of producing single crystal fibers in which the melt is vapor-pressure stabilized. This method will be applicable to nearly congruent melters like KDP. When operating parameters have been established its applicability to isomorphous compounds such as ADP and deuterated forms such as KDP will be assessed.

- Capillary-Fed Czochralski Method - A method for congruently melting materials. Capillary designs can overcome thermal steady state limitations of the conventional Czochralski technique applied to fiber growth. By reducing the ratio of free surface of the melt to growth interface, thermal problems can be minimized and surface tension may be useful in providing mechanical stability at the growth interface.
- Hybrid Single Crystal Fiber Method - Develop a hybrid single crystal fiber. In the presence of good optical contact between a glass fiber core and a bulk, nonlinear crystal, the evanescent portion of the guided wave can couple to nonlinear polarization in the crystal. This results in a nonlinear hybrid fiber with properties controlled by fiber diameter and orientation relative to crystal axes.

Task 3 is concerned with the measurement of optical properties of the fabricated fibers and development of device applications. Characterization of absorption and scattering losses as well as observation of nonlinear optical effects constitute the core of this part of the program. Measured properties are to be analyzed using existing theories of nonlinear processes and light scattering in fibers (particularly from surface imperfections). New theoretical approaches are to be developed only if necessary for analysis.

SECTION 2

STATUS OF THE RESEARCH EFFORT

A. INTRODUCTION

During the second year of the Optical Fibers for Nonlinear Optics program the most promising methods for single crystal growth have yielded half a dozen new fiber crystals. Single crystal (SC) fiber fabrication of AgCl, KDP, ADP, and AgBr achieved by the Traveling Zone and Bridgman methods is very encouraging. Results with these approaches are described in Subsections B and C, respectively, of this section. In Subsection D results with the Capillary-fed Czochralski approach are shown to hold considerable promise for congruently melting crystals. In Subsection E a new geometry for Hybrid SC Fibers is discussed which should provide a novel, versatile method of fabricating oriented single crystal fibers in which second order nonlinear optical mixing can be phase matched. Finally in Subsection F measurements of key properties of the new fibers are reported.

B. TRAVELING ZONE METHOD FOR SC FIBER GROWTH

The heart of this method is sketched in Figure 1. Two sets of synchronously driven wheels move extruded polycrystalline (PC) fiber through a small heater coil which is used to form a melt zone with a length between 0.1 and 4 fiber diameters. Since surface tension of the melted fiber is large, we find no sagging even if growth is horizontal. Our growth rate is approximately 1 cm/min. The fibers grown to date have all preserved the original PC fiber diameter (1:1), but we see no difficulty in making slight reductions in the SC fiber diameter by independently driving the two sets of wheels.

As an interesting aside, we note that it is also possible to surface or skin melt the PC fiber. We have done this for 620- μ m diameter AgBr fiber to a skin depth of about 20% of the

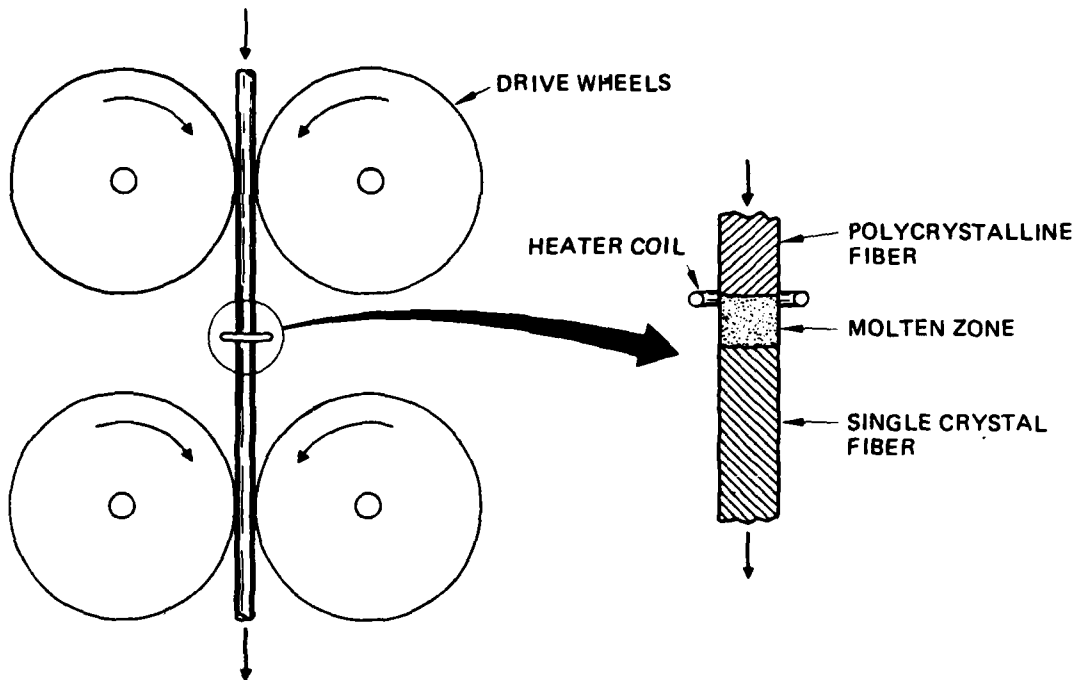


Figure 1(a). A schematic diagram of traveling zone apparatus.

M14868

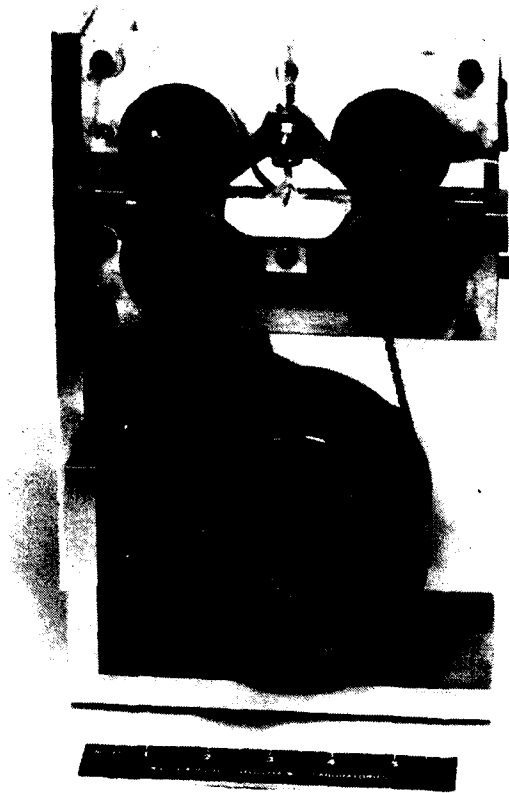


Figure 1(b). Traveling zone apparatus with horizontally mounted fiber.

fiber diameter. The resultant fiber has a single crystal outer layer and a large grain polycrystalline core. The advantage of only a skin melt would be the added strength afforded by a PC core and for the improvement of the surface quality of PC fibers.

The extruded PC fibers we have converted into SC fibers include Tl_2BrI (KRS-5), $TlBr$, $CuCl$, $AgCl$, and $AgBr$. The thallium salts pose a problem because the vapor pressure of these salts is high and the resultant SC fiber surface is dull due to thermal etching. For the first time, PC fibers of $CuCl$ of 1-m length and 500- μm diameter were extruded from a water-white single crystal grown at HRL from which SC fibers of $CuCl$ were produced. However, because the conversion of PC to SC fiber was conducted in air, the melt surface reacted with the atmospheric oxygen producing a mottled brownish layer on the fiber. A chamber surrounding the melt zone is currently being constructed to control both the high vapor pressure and reaction problems of these materials.

Initially our best results were obtained for the silver salts which have a very low vapor pressure and good resistance to atmospheric contamination at the melting point. As an example of our results, we chose $AgBr$. Extruded $AgBr$, 620- μm diameter, traveled through a melt zone one fiber diameter in length obtained with a single-turn heater power of 0.87 W. The solidus-liquidus interface remained quite stationary during growth and meter long lengths are possible without any adjustment of the initial growth parameters. The results of our optical absorption measurements at 10.6 μm are given in the section on characterization of fibers.

Extension of the traveling zone method to other infrared transmitting materials is being considered currently. In particular $CuCl$, which can be extruded transmits beyond 20 μm and is electro-optically active, looks promising. This compound is chemically stable in a dry non-oxidizing atmosphere from room

temperature through its melting point of 422°C, but will readily undergo oxidation in moist air to a cupric (Cu^{2+}) derivative, such as $\text{CuCl}(\text{OH})_x$, $\text{CuClO}_{y/2}$, or mixtures of these, where x and y are indices of the extent of oxidation and may have appreciable values at the interface between the vapor and the condensed states. The indices x and y are also measures of the cupric content of the anion-contaminated material. The cupric ion itself is an alien species in cuprous chloride, and must be regarded as a cationic contaminant. Thus cuprous chloride material that is claimed to have high purity by spectrophotometric standards may be inadequate for crystal growth. Therefore, it is a practical necessity that we develop procedures for the chemical preparation of cupric ion-free cuprous chloride powder for use as charge material in our investigation of single crystal fiber growth of this material.

In a separate IR&D project involving the growth of bulk single crystals of cuprous chloride we have already formulated an original procedure for the chemical preparation of cupric ion-free cuprous chloride that has the same purity relative to other cations as the raw materials (Figure 2). It is an aqueous method of preparation, with unknown anionic purity of the end material. But anionic purification of a material in a condensed state is precisely the type of problem for which reactive atmosphere processing (RAP) is the solution. The preparation of cupric ion-free cuprous chloride is not a routine problem in RAP, since RAP is generally based upon redox reactions, to which the cupric-to-cuprous ion ratio would be sensitive. In our in-house project we have already experimented with several RAP recipes and procedures in bulk single crystal growth of cuprous chloride. In the meantime we are redesigning our single crystal fiber drawing (SCFD) system to incorporate RAP in the SCFD of this material.

Besides the chemical problem just described, a possible source of difficulty in crystal growth of cuprous chloride is

M14904

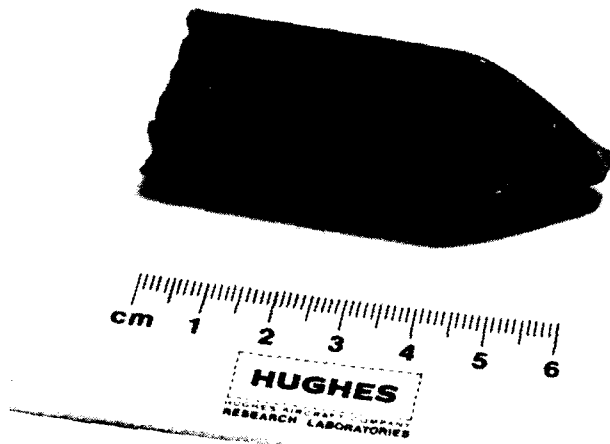


Figure 2. CuCl single crystal.

the wurzite-to-zincblende type of transition it undergoes at 407°C. If this transition is impurity conditioned, it may be circumvented by RAP, if it is truly intrinsic to the material, then there is still the possibility that the transition may not cause microstructural degradation in single crystal fiber.

C. VAPOR STABILIZED BRIDGMAN METHOD FOR SC FIBERS

In our work on KDP with water vapor overpressure (≤ 10 atm) in early 1983, we encountered the problem of chemical corrosion by molten KDP of vitreous silica from which the crucible liner and the capillary molds were made. The solution to the corrosion problem would be to line the surface of the vitreous silica with a film of material that is inert to the melt and vapor at working temperatures, or to replace the silica entirely with inert material. We chose the former alternative because it involved smaller changes in the parameters of operation of the crystal growth system. Therefore, the problem was reduced to a search for a good lining material for the silica glassware and a recipe for laying films of it on that substrate. The best crucible lining we have found so far is pyrolytic carbon.

In the meantime we have focused our attention on ADP, hoping that the lower working temperature of growth would reduce the corrosion problem. Several runs with unlined silica glassware yielded fibers with a nominal diameter of 0.6 mm and with lengths ranging from a few (3) to several (8) centimeters.

Some differential thermal analytical (DTA) studies were made on commercial specimens of deuterated cesium dihydrogen arsenate (CD*A), but the results we obtained were erratic and therefore inconclusive. This had led us to believe that our material sample was inhomogeneous although it was a single crystal, and that we would need to undertake an investigation of the

method of chemical preparation of this material before we can make a systematic study of its crystal growth behavior.

In fact all our previous work with the KDP family of materials indicate that chemical preparative studies should be made of each and every member of this family to insure stoichiometric accuracy compositional uniformity, and dryness of the charge materials for crystal growth. Commercially available KDP and ADP powders are prepared from aqueous solutions, and their occluded water content will vary from sample to sample even if these came from the same batch. The presence of occluded water can be the cause of two problems: (1) it will make the monitoring of water vapor overpressure during crystal growth difficult at best, and (2) it may bring about localized hydrolysis when the powder is heated, which may prove to be irreversible in a polymerizable material, as are phosphates in general.

Characterization of the properties of ADP single crystal fibers is discussed in Subsection 2F. The optical transmission spectrum and x-ray diffraction results are reported there.

D. CAPILLARY-FED CZOCHRALSKI METHOD FOR SC FIBERS

This method of single crystal fiber growth is in fact distinct from the Stepanov or the EFG method in that it has no melt vapor interface surrounding the growth interface, and therefore cannot be regarded as a derivative form of the Czochralski method. Neither is it a derivative form of the Bridgman method - because of the presence in it of a crystal-vapor interface. There is direct physical contact between crystal and shaper during growth, and the shaper truly functions as a die. This method therefore has no analog among the established methods of bulk single crystal growth and represents a significant development in its own right. It has been used successfully to make NaNO_2 fibers.

The method makes use of a crucible and die assembly such as that illustrated in cross section in Figure 3(a). For growth materials with melting points lower than 1000°C this assembly could be fabricated from vitreous silica. The die was made by drawing, i.e., stretching longitudinally while above softening temperature, a piece of heavy-walled capillary tubing and cutting from it a piece that had an internal diameter which tapered from 0.8 mm at one end to 0.3 mm at the other. A pair of ordinary microscope slide glasses served adequately as shutters, the function of which was to provide control of the axial thermal gradient in the die. The crystal pulling rod is hollow from end to end. It was originally made of borosilicate glass tubing with a nucleating tip of thin-walled capillary platinum tubing, as is shown in Figure 3(b). The platinum tubing had an inside diameter of 0.180 mm and an outside diameter of 0.250 mm. An alternative design of the pulling rod consisted of vitreous silica tubing that was drawn at one end to 0.25 mm diameter. Its nucleating tip was quite fragile, and required great care in alignment with the die orifice at the start of each fiber drawing run. If it broke in the orifice, the die had to be replaced, and a great portion of the start-up procedure had to be repeated.

To achieve steady-state growth a scan had to be made of pulling rates in order to determine the steady-state value of that parameter. If that value was exceeded, melt would periodically seep through past the growth interface and freeze onto the fiber. A fiber grown under such conditions would have an irregular, polycrystalline surface. If the fiber was pulled at less than the steady-state growth rate, the growth front would advance deeper into the die, the solid-to-solid contact surface, i.e., the crystal-die interface, would increase, and the resistance to pull would eventually exceed the tensile strength of the fiber.

The growth end of the fiber is held rigidly in place by the die as illustrated in Figures 4 and 5, thus obviating the need

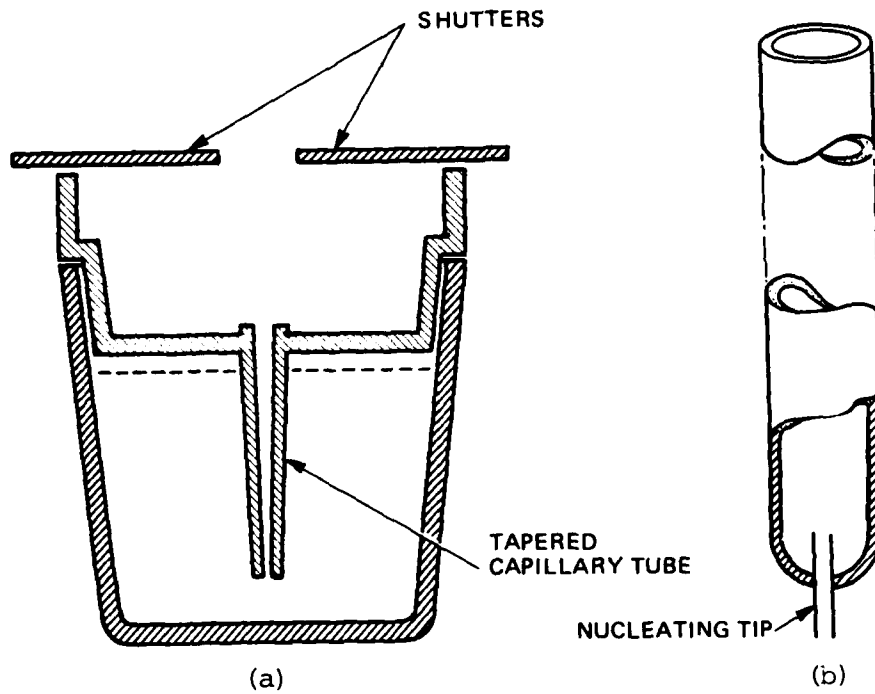


Figure 3. Schematic diagram of the capillary-fed Czochralski Apparatus for growth of SC fibers.

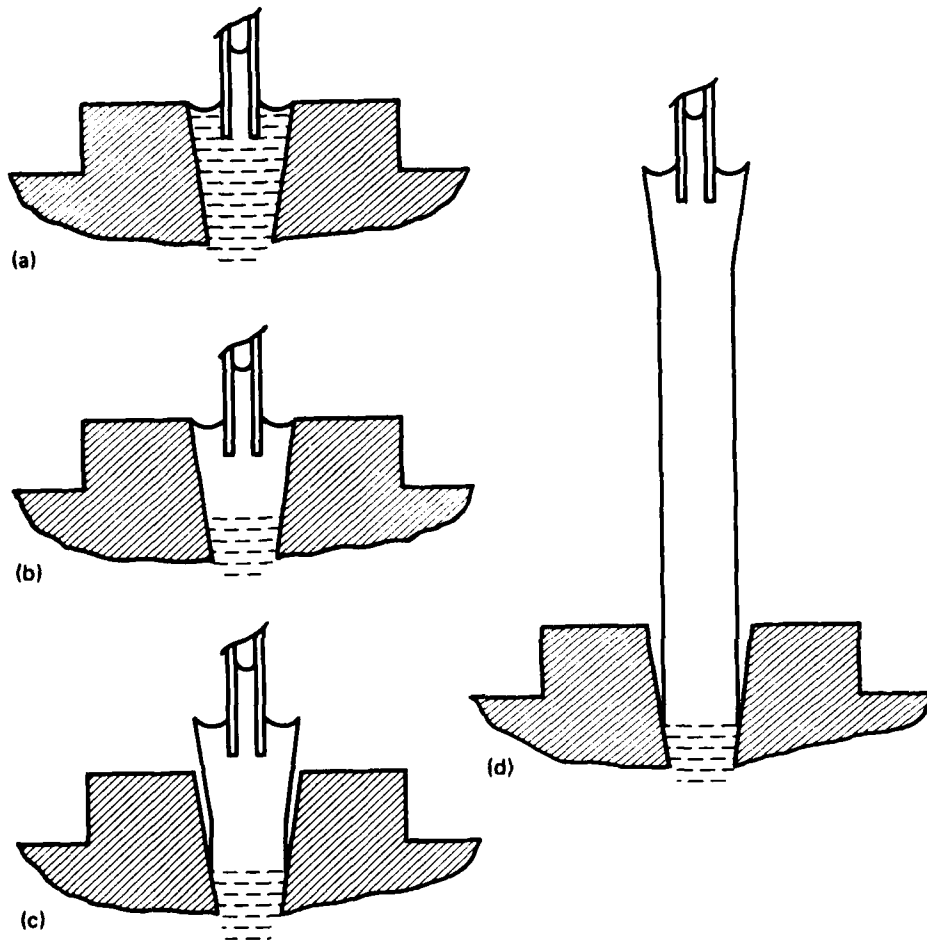
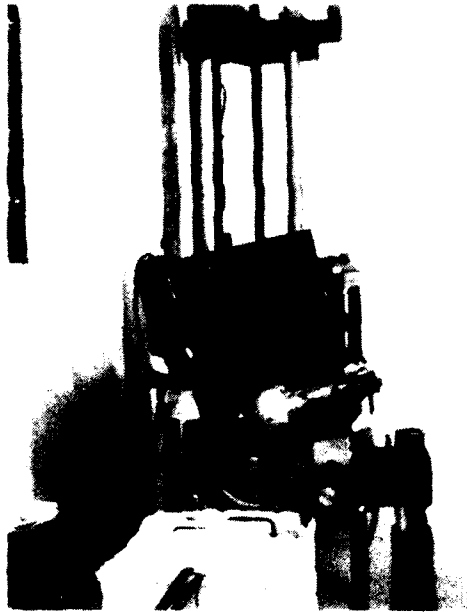


Figure 4. Schematic diagram of nucleation in the capillary-fed Czochralski method.

13878-2



(a)

13878-1



(b)

Figure 5. Capillary-fed Czochralski apparatus.

for alignment devices that are essential to Stepanov or EFG fiber growth systems. Any instability due to transverse vibrations of the growing fiber, which will magnify as the grown fiber lengthens, will pose no problem in spite of the absence of such alignment apparatus.

This fiber growth apparatus was designed for operation in open air and could not be used with melts that were sensitive to moist air. Work is currently in progress to incorporate reactive atmosphere processing (RAP) into the design of the apparatus. RAP single crystal fiber growth will greatly broaden the spectrum of growth materials to which this method will be applicable.

Characterization of the properties of NaNO_2 single crystal fibers is discussed in Subsection 2F. X-ray diffraction and chemical etching results are reported there.

E. HYBRID SINGLE CRYSTAL FIBERS

The concept of hybrid fibers was developed at HRL in response to problems encountered or anticipated with early growth techniques of true single crystal fibers in various optical materials. The idea is sketched in Figure 6. Fibers in evanescent wave contact with nonlinear crystals were visualized as combining the desirable uniformity of glass fiber waveguides with the nonlinear optical properties of bulk, noncentrosymmetric crystals in a hybrid configuration. Operation as nonlinear mixers and amplifiers was postulated on the basis of previous experience in planar waveguides with coupling between evanescent waves in surface guides on nonlinear crystals. Also, two methods of producing hybrid fibers were demonstrated in late 1982.

More recently work has focused on the details of a particular hybrid combination which applies the "saturated solution press" method to ADP crystals. There are two possible approaches to the problem of obtaining guiding with strong evanescent coupling to nonlinear substrates. Table 1 gives values of refractive index for several glasses and KDP isomorphs. From the table it is clear that for extraordinary polarized light

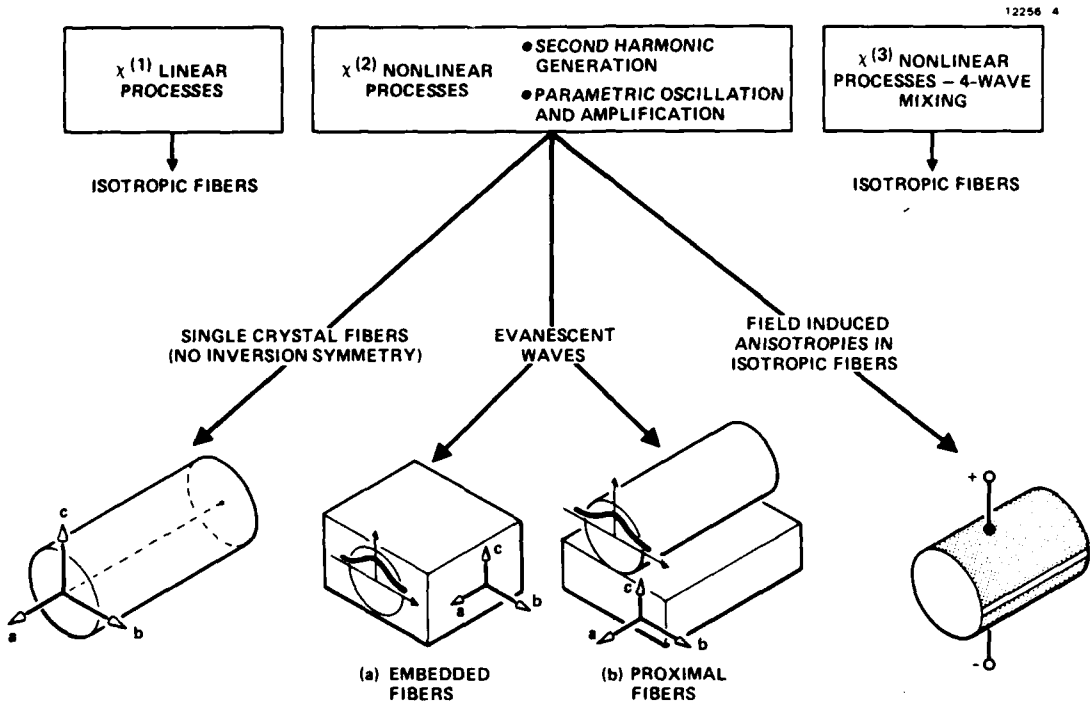


Figure 6. Methods of fabricating second order nonlinear fibers.

Table 1. Refractive Indices of Corning Glasses and KDP Isomorphs at $\lambda = 589.3$ nm ($\lambda = 546.1$ nm for AD*P)

Material	Refractive Index	
	n_o	n_e
Corning 7052 Glass	1.484	—
Corning 7760 Glass	1.473	—
Corning 7050 Glass	1.479	—
Corning 7070 Glass	1.469	—
9741 Glass	1.468	—
ADP	1.52419	1.47870
AD*P	1.5192 ⁺	1.4779 ⁺
KDP	1.5095	1.4684
KD*P	1.5057	1.4677

there can exist a "critical angle" corresponding to the condition $n_{\text{glass}} = n_{\text{crystal}}$, beyond which guiding is possible as the angle θ between fiber and optic axis (illustrated in Figure 7) is increased.* This is true provided the relation $n_o > n_{\text{glass}} > n_e$ obtains for negative uniaxial crystals like KDP. Inverse penetration depth γ of the evanescent wave depends on the difference between the refractive indices of fiber and crystal roughly as

$$\gamma \propto [n_1^2 - n_2^2]^{1/2} .$$

As a consequence, orientation of the glass fiber as it is pressed into the crystal is a critical factor in determining

* The condition for guiding is rigorously equivalent to that for total internal reflection only in slab waveguides, but serves as a useful guideline in fibers too.

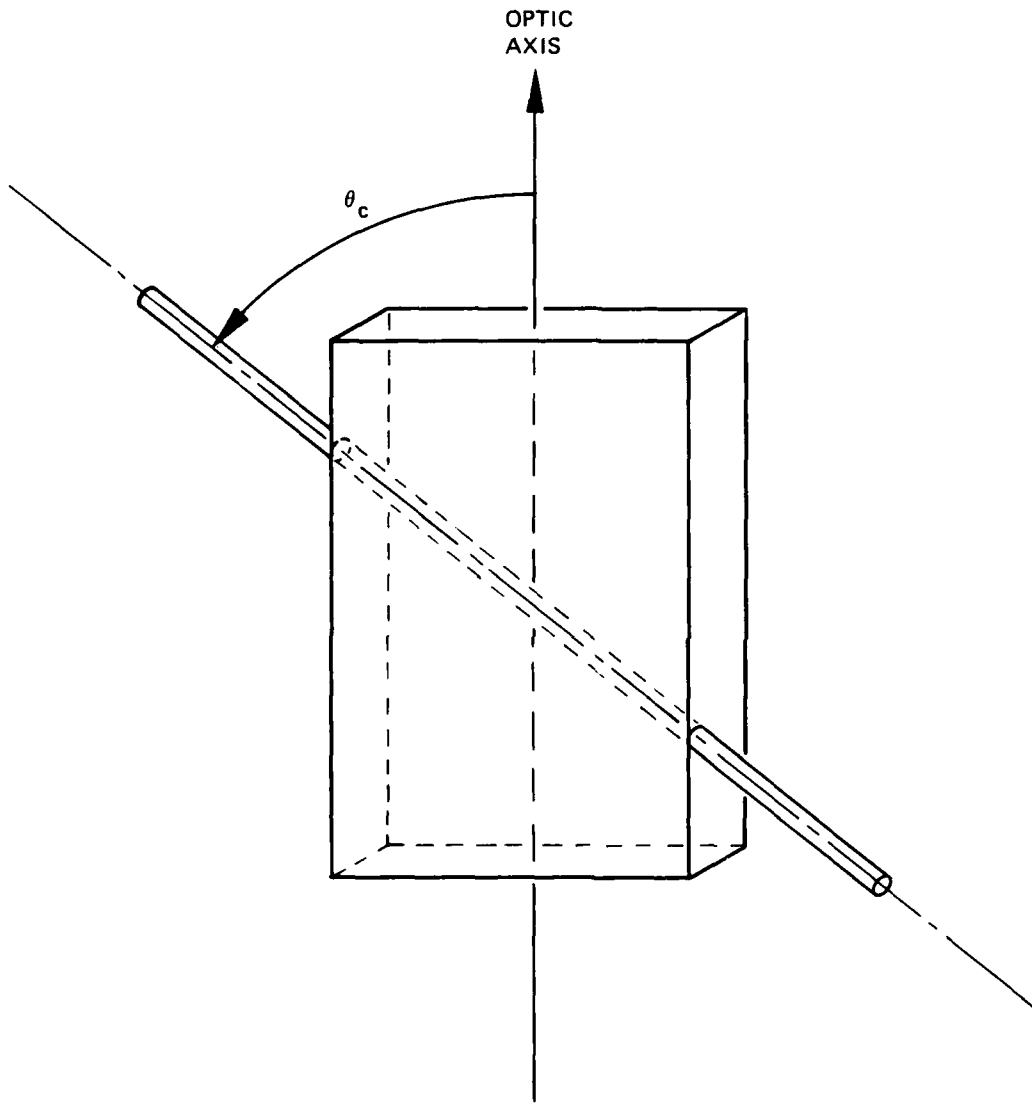


Figure 7. The critical angle θ_c for fibers embedded in uniaxial crystals.

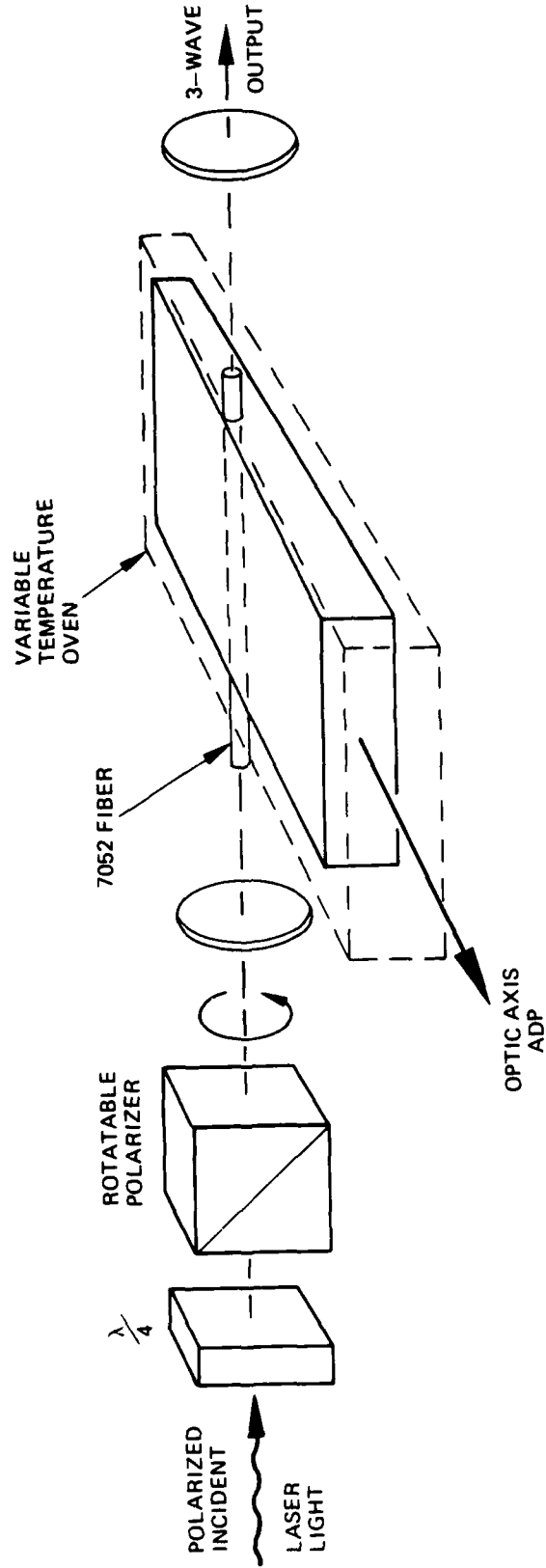


Figure 8. Experimental geometry for demonstration of non-critically phase-matched 3-wave mixing in a hybrid ADP fiber.

both the confinement and the degree of coupling between guided light and nonlinear polarization in the crystal in this method. A special stainless steel press with very fine angular resolution is necessary to fabricate such hybrids. While such a press has been designed and is under construction at HRL, an alternative approach is possible if other polarizations can be used.

In the second approach it is necessary (in the case of negative uniaxial crystals) that materials again be chosen such that $n_o > n_{\text{glass}} > n_e$. Then the fibers can always be oriented perpendicular to the optic axis and rotation of the incident light polarization used to vary the effective index of the crystal to achieve $n_{\text{eff}} \sim n_{\text{glass}}$. Notice from the data of Table 1 that for 7052 glass and ADP this match occurs for a very slight rotation of polarization away from the "o" direction. Since ADP does not exhibit birefringence for this orientation, no walk-off occurs in nonlinear coherent interactions. Hence this combination of materials is ideally suited to generation of non-critically phase-matched 3-wave mixing which is temperature tunable (see Figure 8).

Corning 7052 glass cane is readily available and has already been pulled in a drawing tower at HRL under automatic diameter control to yield long, unclad fibers of diameters 25, 50, 75, and 100 μm . Together with bulk ADP crystals and saturated solution on hand, this material constitutes the starting point for the assembly of the first ADP hybrid guides.

F. CHARACTERISTICS OF SINGLE CRYSTAL FIBERS

CuCl , AgBr , NaNO_2 , and ADP fibers prepared as part of this program have been studied by Laue x-ray and optical techniques to assess some of their key properties.

Reflection Laue photographs of NaNO_2 and ADP fibers are shown in Figures 9 and 10 respectively. The single crystal nature of these samples is evident from the existence of well-defined diffraction spots and the uniformity of optical

13471 2

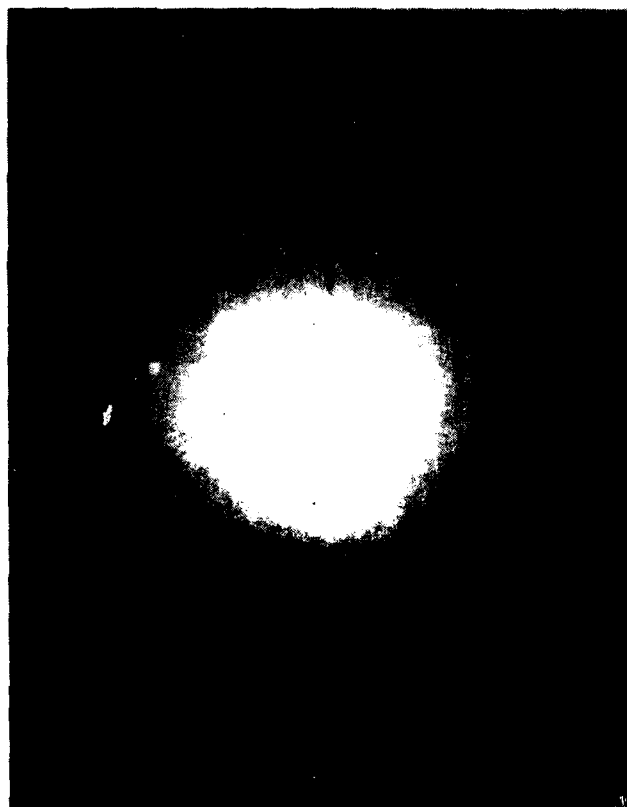


Figure 9. Laue back-reflection x-ray diffraction from end of NaNO_2 fiber showing single crystallinity.

13972-1

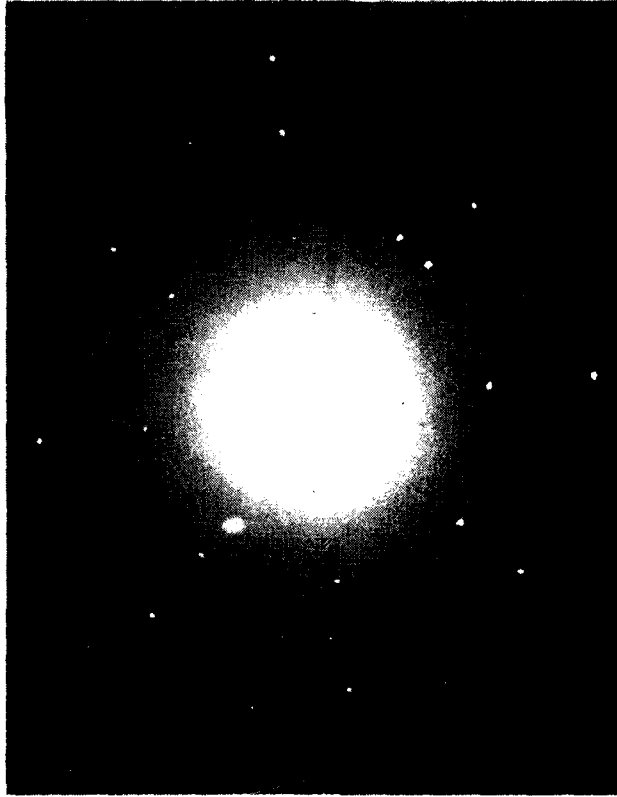
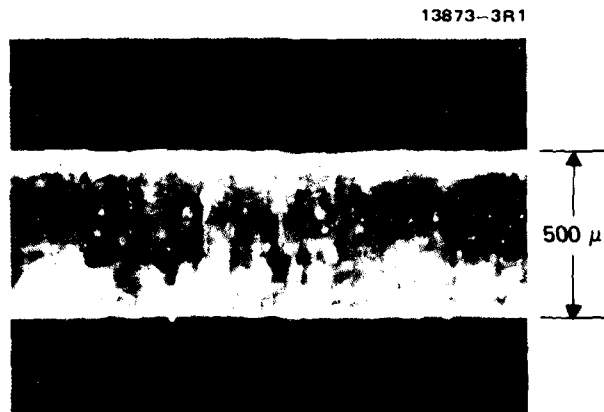


Figure 10. Laue back-reflection photograph of ADP showing single crystallinity and direction of growth along the optic axis.

transmission through these fibers between crossed polarizers (see Figures 11 and 12). The surface quality of the NaNO_2 fibers depended sensitively on the rate of pull from the melt however, and was not always good in the as grown condition. A simple procedure was found for improving the surfaces of these fibers and was used in particular on the fiber in Figure 11. This involved a solution etch in either methanol (fast etch) or acetone (slower etch) and yielded a uniform reduction in the fiber diameter from 500 to 100 μm , as shown in the figure. NaNO_2 , which is biaxial, showed no obvious symmetry in the Laue photo (Figure 9) and apparently grew with a considerable angle between the symmetry axis and the direction of pull. This is suggested in Figure 11 where cleavage planes at roughly forty-five degrees to the axis appear lightly decorated under polarized light following the methanol treatment. Also, when aged in air, the NaNO_2 fibers did cleave at this angle. The four-fold symmetry in the diffraction pattern of Figure 10 shows that ADP, which has 42 *m* tetragonal crystal symmetry, grew with its optic axis parallel to that of the capillary. Crystallinity along the fiber axis was checked by visually comparing transmission of unpolarized light (Figure 12(a)) with transmission between crossed polarizers (Figure 12(b)).

Optical transmission studies of the fibers were begun using a spectrometer specifically designed for handling fibers and illustrated in Figure 13. A silica fiber of diameter 100 μm was used to couple light into sample fibers mounted at the entrance of a low resolution grating spectrometer. A good end polish was achieved on the ADP fibers (as grown, in glass capillaries) using a series of emery cloths wetted with anhydrous methanol and culminating with a lens tissue polish, again using a methanol carrier. The UV transmission spectrum of an ADP fiber is given in Figure 14. The infrared spectrum was not obtained due to limitations of available germanium photodetectors and gratings. The transmission curve of bulk, single crystal CuCl



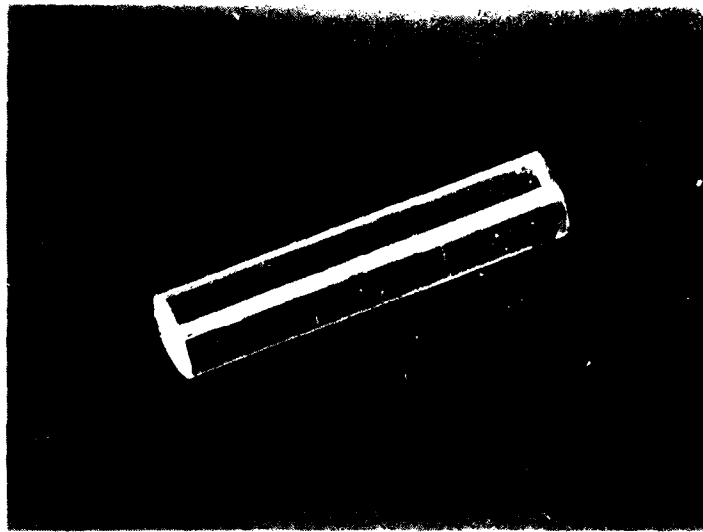
(a)



(b)

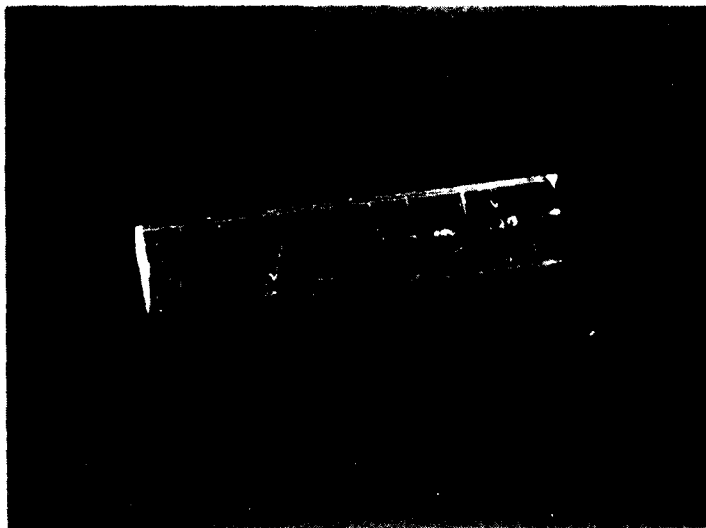
Figure 11. NaNO_2 single crystal fiber viewed between crossed polarizers (a) as grown (b) after methanol treatment for 4 minutes.

M15903



(a)

M15904



(b)

Figure 12. SC fiber of ADP encased in quartz capillary.
(a) Transmission of unpolarized light
(b) transmission between crossed polarizers

which was grown at HRL and used in making our first CuCl fibers is given in Figure 15. The range of this curve was restricted by instrumental limitations but illustrates the potential of CuCl in the far infrared.

The orientation of AgBr single crystal fibers was most simply obtained by using the preferential-etch method of Bridges et al.^{1,2} In Figure 16 we show a section of fiber surface etched in 30% sodium thiosulphate for two minutes. The (110) direction is matte and the (100) direction is shiny. This indicates that the growth axis is along the (100) direction. By sampling pieces along the length of the fiber, we determined that the entire fiber grows in this orientation. These results are also confirmed by slightly stressing the fiber. Figure 17 shows the (110) slip planes which result from tensile stress applied along the fiber axis.

The results of optical absorption measurements in AgBr fibers at 10.6 μm are given in Table 2.

Table 2. AgBr Fiber Loss at 10.6 μm

PC Fiber		SC Fiber	
Length, cm	Loss, dB/m	Length, cm	Loss, dB/m
94	2.6	80	6.6
49	8.5	28	8.3

We note for the longer fiber that the total attenuation is less for the starting PC fiber than for the converted SC fiber. This, plus the fact that the lowest loss of 6.6 dB/m is still rather high, is due to some irregularities in the fiber surface quality and a few bubbles included in the fiber core. We expect these losses to be reduced by better control of the fiber drive, by stabilization of the temperature, and by using an inert ambient environment around the melt zone.

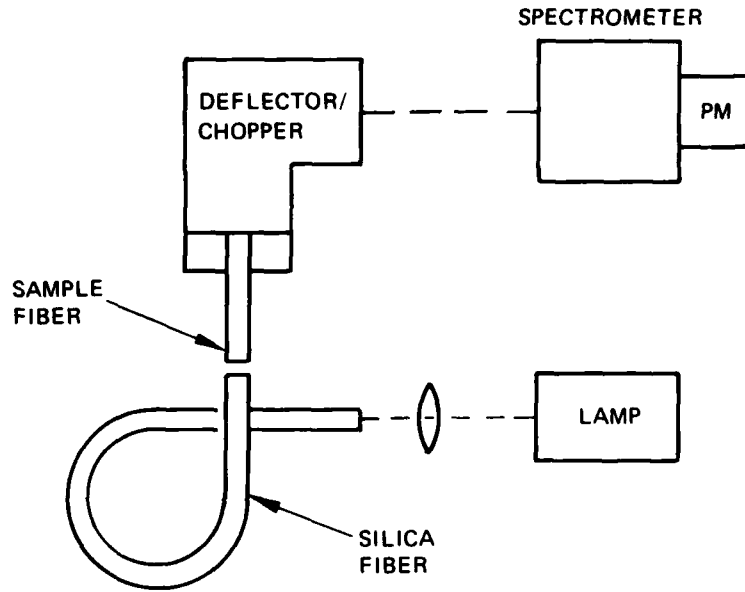


Figure 13. Fiber spectrometer.

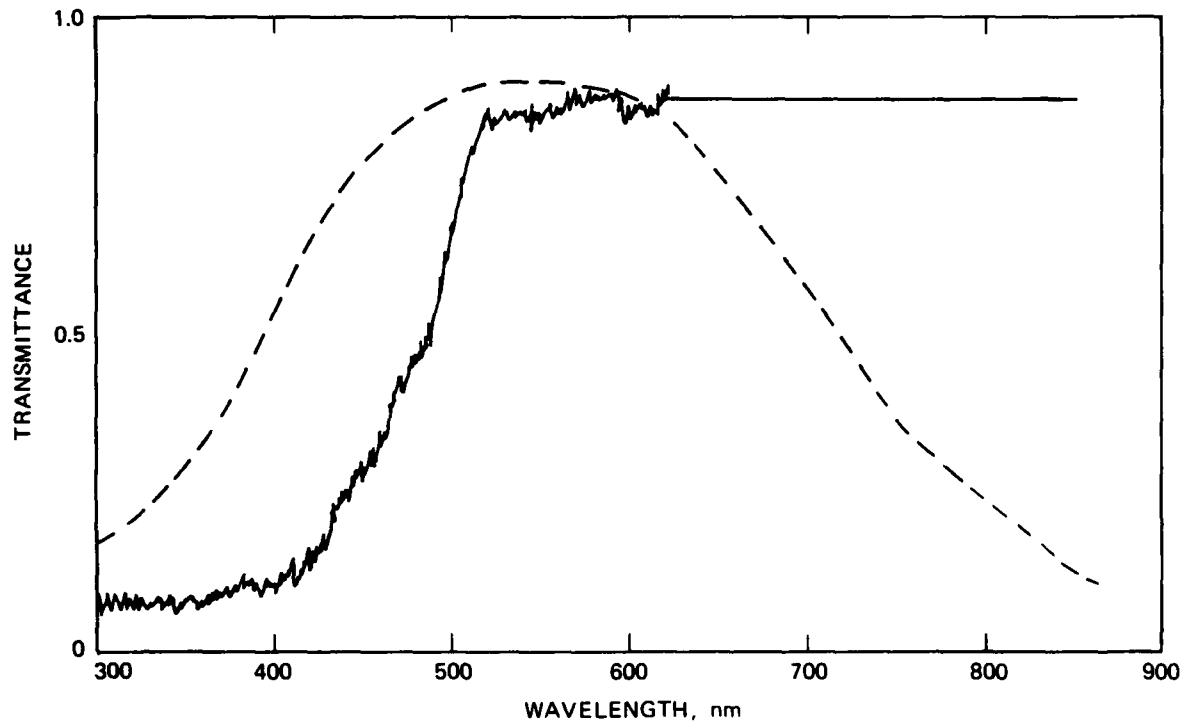


Figure 14. UV transmission spectrum of ADP fiber.

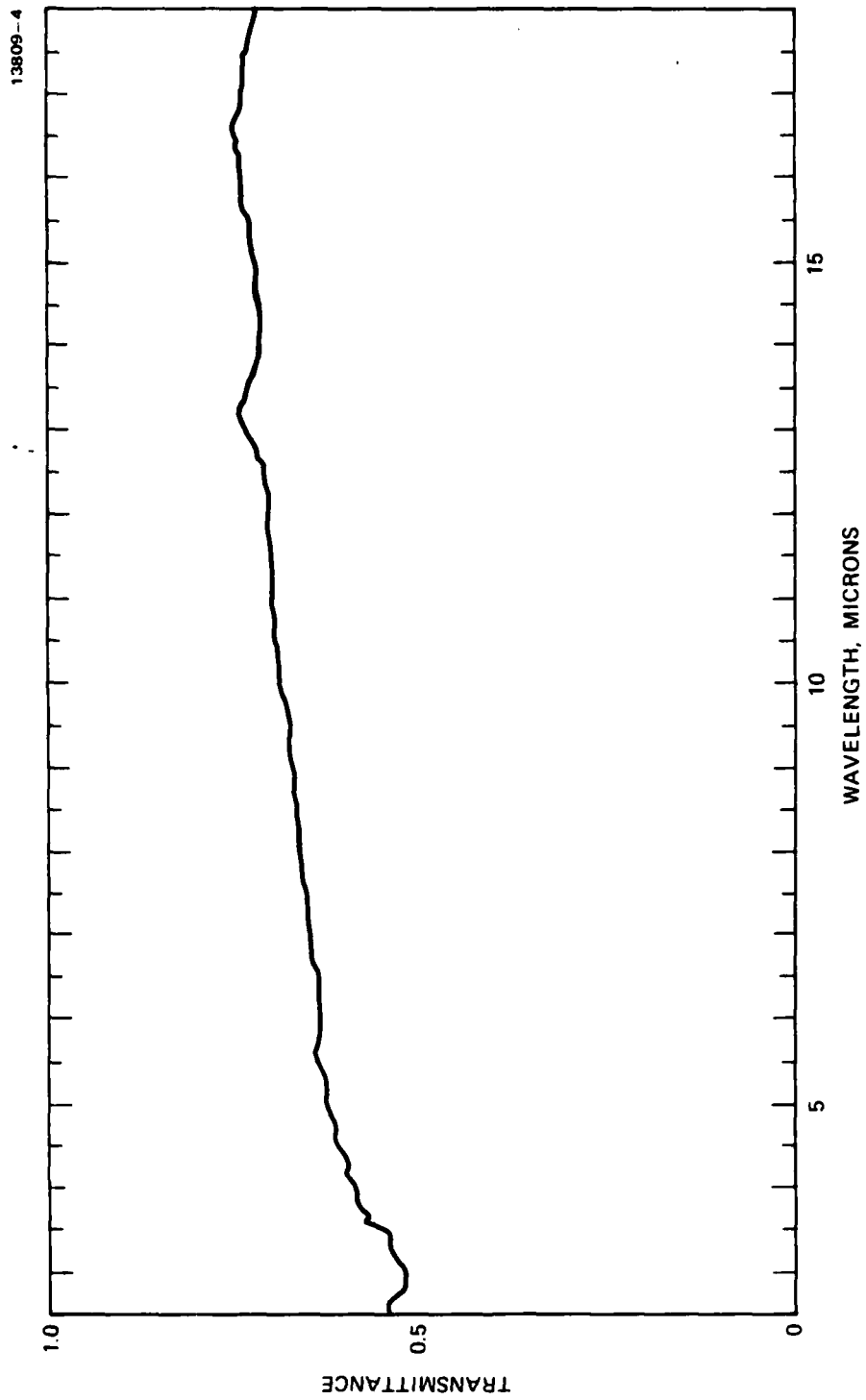


Figure 15. IR transmission of bulk, single-crystal CuCl.

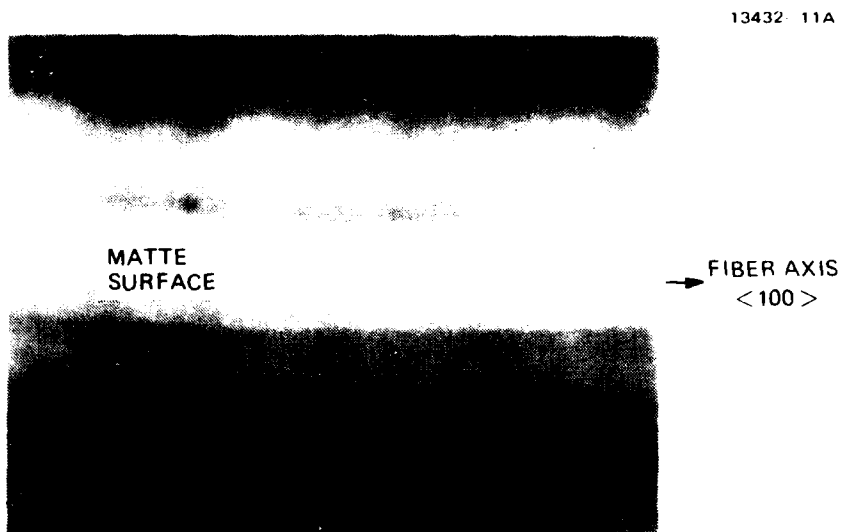


Figure 16. AgBr SC fiber with growth axis along (100).

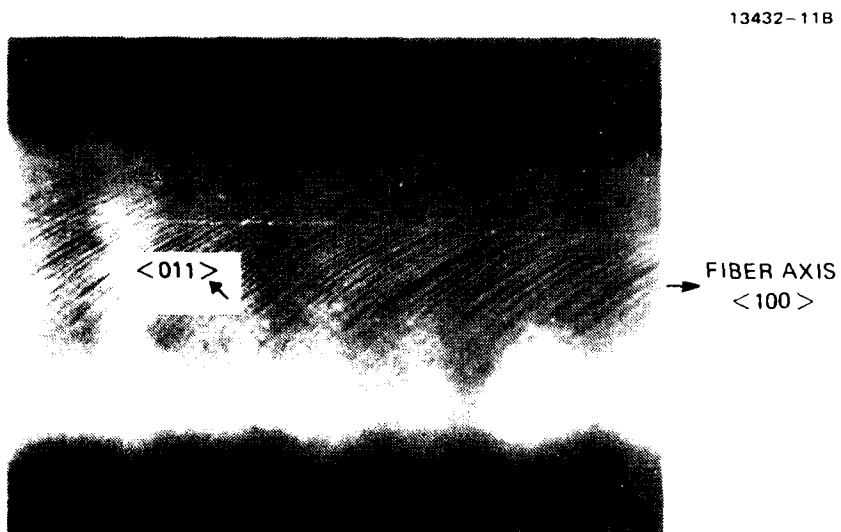


Figure 17. (110) Slip planes resulting from tensile stress applied along the axis of a AgBr SC fiber.

SECTION 3

BIOGRAPHIES OF KEY PERSONNEL

The biographies of the personnel who are contributing to the program are presented in the following pages. L.G. DeShazer is the Principal Investigator of this research program.

LARRY G. DeSHAZER, Head, Laser Optical Materials Section, Hughes Research Laboratories.

Education B.S. (Physics), University of Maryland, 1956;
Ph.D. (Physics), The Johns Hopkins University, 1963.

Experience De. DeShazer has 23 years of professional experience in the areas of solid-state lasers, nonlinear optics, laser damage physics and atomic spectroscopy. From 1963 to 1966, he was at Hughes Aircraft Company, Culver City, involved in research on energy transfer in solid-state laser materials and studies of laser mode selection techniques.

In 1966 he joined the Physics and Engineering Faculty at the University of Southern California as Associate Professor. He was mainly involved in spectroscopy of rare-earth ions in laser materials and pioneered in the measurement of self-focusing parameters by the optical pulse shaping technique and laser damage to thin dielectric films.

In 1973 De. DeShazer was appointed the first Director of the Center for Laser Studies at USC, an applied research organization serving as an interface between industry and university. While on leave of absence from USC, from 1975 to 1976, he was Liaison Scientist with the U.S. Office of Naval Research, London. He left USC in 1978 to return to Hughes at the Research Laboratories with responsibility in solid-state laser and nonlinear optics research programs.

Honors Fellow of Optical Society of America, Gilman Fellow at Johns Hopkins University (4 years), Phi Kappa Phi, Phi Eta Sigma, Sigma Pi Sigma, Gamma Alpha.

Professional American Physical Society, Optical Society of America, The Royal Institution of Great Britain, Sigma Xi, IEEE Group of Electron Devices, SPIE.

Publications Dr. DeShazer is the author of 82 technical papers in the fields of solid-state lasers, nonlinear optics and spectroscopy. He has 3 patents in solid-state lasers, and was the dissertation chairman for twenty Ph.Ds.

JAMES A. HARRINGTON, Senior Staff Physicist, Optical Physics Department, Hughes Research Laboratories.

Education B.S. (Physics), Grinnell College, 1964;
M.S. (Physics), Northwestern University, 1966;
Ph.D. (Physics), Northwestern University, 1970.

Experience Dr. Harrington has 13 years of research experience in the area of optical properties of solids. Since joining Hughes Research Laboratories in 1977 he has been involved in the study of low-loss materials and coatings for use as high-power infrared laser components. Recently, he has been appointed manager, infrared fiber optics responsible for coordinating all IR fiber programs. His studies of IR fiber materials include measurement of ultimate absorptive and scattering losses in transparent materials. Prior to joining Hughes, he was assistant professor of physics at the University of Alabama in Huntsville from 1973 to 1976. At the University of Alabama, he conducted fundamental investigations into the nature of optical absorption in highly transparent solids using infrared spectroscopic and laser calorimetric techniques. During the course of these investigations, he developed many refinements in state-of-the-art techniques to allow for the study of surface and bulk absorption. Before joining the University of Alabama, he was a research physicist at the Naval Research Laboratory from 1972 to 1973, where he conducted similar studies on the optical properties of ionic solids. These included the first high-temperature measurements made on alkali halides using CO₂ laser calorimetry.

At the University of Stuttgart (1970 to 1972) and Northwestern University (1964 to 1970), Dr. Harrington was involved in the study of the optical properties of pure and doped alkaline earth fluorides. These studies spanned the infrared, far infrared, and visible regions using the techniques of infrared spectroscopy, far infrared Michelson interferometry, Raman scattering, and low-temperature thermal conductivity.

JAMES A. HARRINGTON (Continued)

Honors

Special Achievement Award, Naval Research Laboratory (1973); Publication Award, Naval Research Laboratory (1974).

Professional Societies

American Physical Society, Optical Society of America.

Publications

Dr. Harrington has authored over 25 publications in theories of laser calorimetry, lattice dynamics, and optical properties of solids.

ANTONIO C. PASTOR, Member of the Technical Staff, Optical Physics Department, Hughes Research Laboratories.

Education B.S. (Chemical Engineering), University of Santo Tomas (Manila, Phillipines), 1950; M.S. (Physics), University of Chicago, 1958.

Experience Mr. Pastor joined Hughes in 1968 and has been working on the growth of refractory laser and electro-optic crystals, as well as the design of growth equipments for use in the Hughes Reactive atmosphere process for highly transparent alkali halide and alkaline earth fluoride window material. More recently he has been involved in similar work on monocrystalline fiber growth of potassium and thallium halides and in the deposition of thin films of inorganic compounds using metallo-organic photoresists. From 1961 to 1968 he was employed at Korad, working particularly on improving the Verneuil method of crystal growth and the development of high temperature crystal preparation methods and the related ceramics and instrumentation techniques. In earlier years, he was an instructor at the University of Santo Tomas and at the Montana School of Mines, and also practiced chemical engineering and operated a commercial laboratory for chemical analysis. More recently, he has been teaching courses in materials science at Santa Monica college and West Coast University.

Professional Societies American Association of Crystal Growers, American Association for the Advancement of Science.

Publications Mr. Pastor is the author of several publications in the Journal of Chemical Physics, the Materials Research Bulletin, etc. He has numerous patents and patent disclosures in the field of crystal growth.

STEPHEN C. RAND, Member of the Technical Staff, Optical Physics Department, Hughes Research Laboratories.

Education B.Sc. (Physics), McMaster University, 1972;
M.Sc. (Physics), University of Toronto, 1974;
Ph.D. (Physics), University of Toronto, 1978.

Experience Prior to joining Hughes, Dr. Rand was engaged in light scattering experiments at the University of Toronto, reporting Brillouin measurements in all the rare gas solids and the family of deuterated methane compounds. He spent two years as a World Trade Fellow at IBM Research in San Jose, California. There his research was in the area of optical coherent transients and spectral hole-burning in rare-earth doped crystals. It culminated in the discovery of "magic-angle line narrowing" on an optical transition of Pr ions in LaF₃. Subsequently he spent two years as a research associate in the Stanford University Department of Physics. During this period he worked on radiative pair emission and absorption processes in rare earth materials.

Dr. Rand joined Hughes Research Laboratories in June 1982 and is currently involved in experimental physics research in fiber optics, nonlinear optics, color center lasers, stimulated pair processes, and optical coherent transients.

Professional Societies American Physical Society; Optical Society of America.

Publications Dr. Rand's publications in the above areas include more than 20 papers in Physical Review Letters, Physical Review, Optics Communications, Solid State Communications, Physics Letters and the Canadian Journal of Physics. He has also contributed to several books: Laser Spectroscopy IV (1979), Laser Spectroscopy V (1981), Light Scattering in Solids (1979) and Lasers and Applications (1981).

8/83

SECTION 4
INVENTION DISCLOSURES

A. SINGLE CRYSTAL FIBER DRAWING

- Inventor
A.C. Pastor
- Summary of the Invention

A new method of growth from the melt single crystal fibers of congruently melting materials is reported (See Appendix).

B. NONLINEAR OPTICAL DEVICES USING EMBEDDED OPTICAL FIBERS

- Inventor
S.C. Rand
- Summary of the Invention

This invention combines a number of high quality optical fibers with a nonlinear optical material by embedding the fibers in the material in such a way that their cores are in evanescent contact with one another and the nonlinear medium.

The purpose of this invention is to perform second order nonlinear optical signal processing in amorphous glass fibers. Devices for low power lasers such as frequency doublers, sum and difference frequency generators and "in-line," all-optical signal amplifiers operating in a parametric fashion can be realized using this invention.

This invention also addresses a major problem in fiber optical communication schemes. Currently, most integrated optical data processors and nonlinear optical processors are fabricated on planar structures. There exist major practical (i.e., mechanical) and fundamental (mode matching) problems in

achieving low loss coupling of radiation in fibers with these planar devices. With this invention embedded fibers act as the guiding structures, thereby minimizing coupling losses.

C. IMAGE CONVERTER WITH VARIABLE GAIN FOR INFRARED IMAGING

- Inventor
S.C. Rand
- Summary of the Invention

This invention provides a new image upconverter with adjustable gain and upconversion at one (variable) frequency in the infrared. The principle of the upconversion does not rely on phosphors or a microchannel plate but rather on sum mixing upconversion in an embedded fiber array.

The chief advantage of this invention is that it circumvents the need for high efficiency infrared phosphors. Instead of converting infrared light to visible light (for easy detection) in two steps the present invention accomplishes this conversion directly by a sum mixing process which does not rely on phase matching in bulk crystals. Intensity of the image is improved by virtue of the superiority of visible detectors over infrared detectors in the usual way. Room temperature operation and the inherent high speed performance make this device attractive for sensitive detection in laser rangefinding.

REFERENCES

1. A.C. Pastor, "Single Crystal Fiber Drawing," submitted to American Conferences on Crystal Growth VI, 1984.
2. J.A. Harrington, A.G. Standlee, A.C. Pastor, and L.G. DeShazer, "Single Crystal Infrared Fibers Fabricated by Traveling-Zone Melting," submitted to S.P.I.E.



Abstract of
New
Technology

FROM THE AIR FORCE SYSTEMS COMMAND

PROVIDES INFORMATION ON
THE PRODUCTS OF AFSC
RESEARCH, DEVELOPMENT,
AND TESTING PROGRAMS.



SINGLE CRYSTAL FIBER GROWTH SYSTEM WITH CAPILLARITY-FED DIE

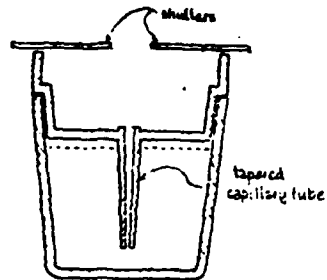


Fig. 1 - Crucible and Die Assembly

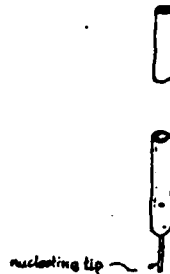


Fig. 2 - Pullrod

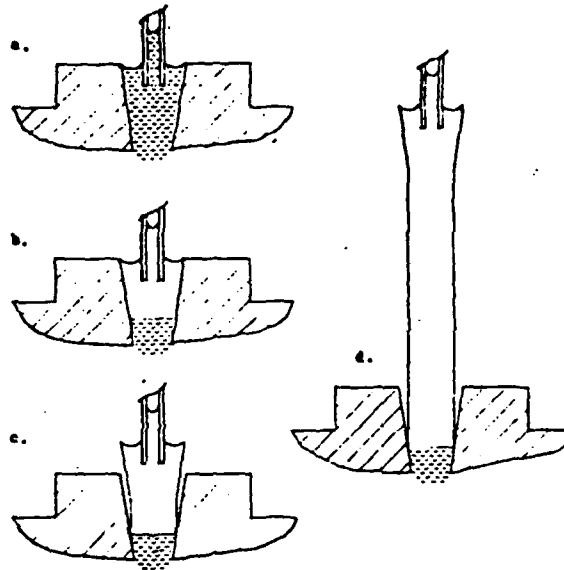


Fig. 3

3. Description: A new method of growing single crystal fibers of congruently melting materials and the system that was used in its implementation are described herein. Crystal growth is effected through a die that is constantly supplied with melt by the mechanism of capillarity.

A schematic cross section of the crucible and die assembly is shown in Figure 1. The feasibility of the process has been proved with sodium nitrate as the test material. For this purpose the assembly was made of vitreous silica, and a pair of standard microscope slide glasses were used as shutters. The heater that was used (not shown) was of the conventional Kanthal-wound clam-shell type. It is important to note that the hollow of the capillary tube is tapered, from 1 mm ID at the top to 1/4 mm ID at the bottom end. The natural tendency of the melt is to fill this tube to the top through capillary action.

This document was prepared under the sponsorship of the Air Force. Neither the United States Government nor any person acting on behalf of the United States Government assumes any liability resulting from the use of the information contained in this document, or warrants that such use be free from privately owned rights.

A schematic diagram of the crystal pulling rod is shown in Figure 2. The tip is a segment of platinum capillary tubing with dimensions of 0.25 mm OD x 0.18 mm ID x 30 mm length, and the entire pullrod is hollow from end to end so that, upon contact with the melt, the platinum tube is partly filled with material before nucleation of the crystal occurs.

Figure 3 shows in sequence the method of operation of this system. Only the cross section of the system in the neighborhood of the growth interface is shown in each of the diagrams in the sequence. It is implicit in these diagrams that the charged crucible and die assembly has been so positioned in the heater that the entire charge is at its melting point from the bottom of the crucible to the top of the capillary tube, with the shutters closed. Figure 3a depicts the system at the moment of contact of the tip of the pullrod with the melt, during which the shutters are in the closed position. Figure 3b shows the same system moments after the shutters have been opened, Figure 3c moments after pulling has been started, and Figure 3d much later, when steady-state growth is in progress.

It is important to note that the melt has no free surface, i.e., there is no interface between melt and vapor, during growth. It is this feature that distinguishes this method from the edge-defined film-fed growth (EFG) method. The advantage due to it are (1) that fiber diameter is a controlled parameter and (2) that the growth interface is stable against transverse mechanical vibrations of the grown fiber since the growth end is held in place by the die itself.

4. Source: Antonio C. Pastor, Hughes Research Laboratories, Malibu, CA 90265
Air Force Contract #F49620-82-C-0030
5. Publication: There is no publication on this method of single crystal fiber growth at this time of writing.

END

FILMED

8-84

DTIC