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LITTON SYSTEMS INC MORRIS PLAINS NJ AIRTRON DIV
R F BELT ET AL. DEC 82 N00014-81-C-0656

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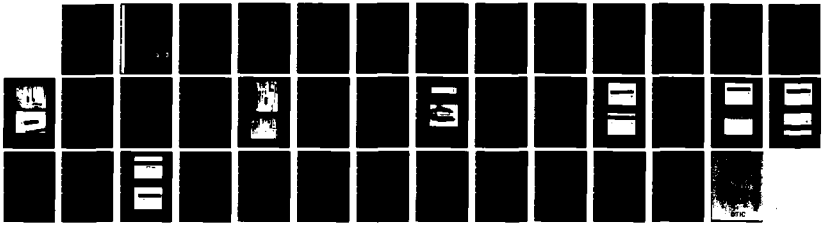

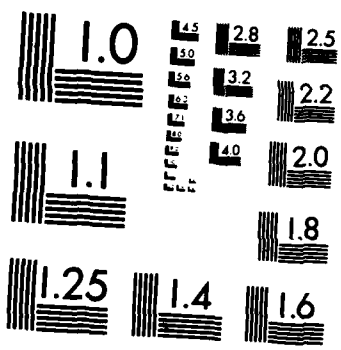

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PREPARATION OF RARE EARTH DOPED LASER MATERIALS

by

R.F. Belt and R. Uhrin

Final Report
October 1, 1981 - October 1, 1982
Contract No. N00014-81-C-0656

Airtron Division
Litton Industries, Inc.
200 East Hanover Avenue
Morris Plains, N.J. 07950

December 1982

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SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER Final
4. TITLE (and Subtitle) PREPARATION OF RARE EARTH DOPED LASER MATERIALS		5. TYPE OF REPORT & PERIOD COVERED Oct. 1, 1981-Oct.1,1982 6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) R.F. Belt and R. Uhrin		8. CONTRACT OR GRANT NUMBER(s) N00014-81-C-0656
9. PERFORMING ORGANIZATION NAME AND ADDRESS Airtron division Litton Systems, Inc. 200 East Hanover Avenue Morris Plains, N.J. 07950		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS NR 379-050 421
11. CONTROLLING OFFICE NAME AND ADDRESS Defense Contract Administration Services 240 Route 22 Springfield, New Jersey 07081		12. REPORT DATE December 1982 13. NUMBER OF PAGES v + 27
14. MONITORING AGENCY NAME & ADDRESS(if different from Controlling Office) Office of Naval Research Department of the Navy 800 North Quincy Street Arlington, Virginia 22217		15. SECURITY CLASS. (of this report) Unclassified 15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Laser crystals
Czochralski growth
$YLiF_4$
BaY_2F_8
YF_3
YAG

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

This report describes the single crystal growth of various laser materials which may be useful in generating new wavelengths. It also includes some compositions of materials which are not available commercially but are necessary for test lasers at the Naval Research Laboratory. The host crystals include yttrium aluminum garnet ($Y_3Al_5O_{12}$), yttrium lithium fluorides ($YLiF_4$), and barium yttrium

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fluoride (BaY_2F_8). These hosts were doped with single or multiple rare earth elements for lasing action at wavelengths in the near infrared (1-3μm). After the crystals were grown, samples were provided in the form of cm size polished rectangular parallelepipeds or 5-6 cm cylindrical rods with polished ends. All of the samples were tested or used at laser facilities of the Naval Research Laboratory. No active tests were performed at the crystal preparation source.

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FOREWORD

This Final Report describes the single crystal growth of rare earth doped laser crystals. The major items are directly concerned specifically with dopants of Dy, Er, Tm, Ho, Pr, or Ce, which may prove to be suitable for possible new lasing schemes in the 2 - 5μm range. Another portion of the effort was directed towards the growth and laser fabrication of special crystals. The report summarizes all efforts under Contract No. N00014-81-C-0656 for the period October 1, 1981 to October 1, 1982. The contract work was under the coordination of Dr. Van O. Nicolai of the Office of Naval Research.

All compositions, preparations, single crystal growth, laser rod fabrication, and coating were performed in the laboratories of Airtron Division of Litton Industries, Inc., 200 East Hanover Avenue, Morris Plains, New Jersey 07950. Dr. Roger F. Belt was the technical director of the project and Mr. Robert Uhrin was the project engineer. Mr. John Yorston was the senior growth technician. Mr. Steven Turner supervised all laser material fabrication and provided the coated optics. Active testing of delivered laser samples was conducted at the Naval Research Laboratory by Dr. Leon Esterowitz. The report was prepared by Dr. Roger F. Belt and released for publication in December 1982.

ABSTRACT

This report describes the single crystal growth of various laser materials which may be useful in generating new wavelengths. It also includes some compositions of materials which are not available commercially but are necessary for test lasers at the Naval Research Laboratory. The host crystals include yttrium aluminum garnet ($Y_2Al_4O_{12}$), yttrium lithium fluorides ($YLiF_4$), and barium yttrium fluoride (BaY_2F_8). These hosts were doped with single or multiple rare earth elements for lasing action at wavelengths in the near infrared (1-3μm). After the crystals were grown, samples were provided in the form of cm size polished rectangular parallelepipeds or 5-6 cm cylindrical rods with polished ends. All of the samples were tested or used at laser facilities of the Naval Research Laboratory. No active tests were performed at the crystal preparation source.

1.0 Introduction

For nearly 15 years the most prominent and widely used laser host has been yttrium aluminum garnet (YAG or $Y_3Al_5O_{12}$). This material has been doped singly or in combination form with all of the rare earth elements to give satisfactory laser action at many wavelengths. The best of these crystals has been Nd:YAG which emits at 1.06 μm and is available readily from several commercial sources. Much additional research has been performed on other attractive laser hosts but these have generally not been offered as commercial products.

One of the more interesting areas of laser host development has been connected with fluoride based materials. The fluoride hosts offer some attractive compounds which can replace oxides. In addition, the crystal fields surrounding an impurity ion are modified sufficiently to permit entirely new wavelengths of laser action. Other advantages may accrue from a change of fluorescent lifetime, modification of linewidths, or the phonon spectra. The most desirable fluoride host has been $LiYF_4$ since it has a scheelite type structure and all the rare earth trivalent ions can be incorporated to some degree. The crystal has excellent physical properties and can be fabricated into a laser rod comparable to oxides. One notable exception is the problem of antireflection coatings. The low refractive index dictates a multi-layer type coating in place of a single layer as used on higher index

oxides. The $LiYF_4$ crystal was grown in the late 1960's by several workers who employed either a Stockbarger,[1] top-seeded solution,[2] or Czochralski method.[3] Other workers have developed good procedures which provide high optical quality for laser application. These include growth in an HF atomosphere[4,5] and other modifications.[6-8] The $LiYF_4$ is currently under consideration for an eye safe laser, i.e. one operating at wavelengths beyond 1.5μm.

Another fluoride host of great interest for tunable laser action is BaY_2F_8. This material was investigated first at Bell Laboratories for phonon terminated lasers involving Ho^{3+} as the activator.[9,10] The host crystal has never been available widely or commercially. BaY_2F_8 is a monoclinic crystal (close to tetragonal), apparently congruently melting near 975°C, and able to incorporate all rare earth ions. The phase diagram of the BaF_2- YF_3 system has been studied.[11,12] even though a crystal structure is not published. The crystal offers some potential for lasers which operate up to 3μm. BaY_2F_8 can be grown in the same apparatus as $LiYF_4$ using identical procedures. However each rare earth may present a slightly different problem because of size effects. The ionic radii of Dy, Er, Tm, and Ho are close to that of Y so that small dopant levels are attained easily. Furthermore the distribution coefficients are close to unity for those elements.

Other fluoride hosts are worthwhile for investigation in

an extended program. Possibilities are YF_3, LuF_3, GdF_3, LaF_3 and many other mixed systems such as $NaF-YF_3$, $KF-YF_3$, $LiF-BaF_2$, and KBiF of fluorite structure. Most of these have been grown under laboratory conditions and in small size. Few if any have ever been produced in laser rods and tested. Our program did not include these as deliverable items.

This research effort was undertaken to provide sp ..fic oxides and fluorides for possible laser action at new - lengths. The particular host crystals, rare earth dopants, and their quantitative levels were chosen from discussions with the laser group at the Naval Research Laboratory. A preferred list of materials was developed at the inception of the program and crystals were delivered at a rate of 1-2 rods/month. Usually the easiest crystals were grown first and then the work progressed to the more difficult.

2.0 Experimental

This program consisted of prefixed oxide or fluoride laser crystals. The exact compositions were specified by the contractor. Most systems were studied previously although the particular compositions were not routine. A systematic schedule was developed to perform the easiest growth task first. Crystals of the fluorides usually required a few trials because of growth variable changes such as pull or rotation rates. Even after these were optimized, the quality of a boule was impaired partially due to strain, bubbles, diameter excursions, or other

defects. In most growth runs some material or rods were obtained.

2.1 Oxides

The growth of large single crystals of doped $Y_3Al_5O_{12}$ (YAG) was performed on one of Airtron's production type Czochralski growth stations. A station is illustrated in Figure 1. Iridium crucibles of 2 or 2.5 inch diameter were used to contain the melt and to serve as a susceptor for the 450 KHz RF heating. The crucibles were contained within a glass bell jar in order to maintain a controlled atmosphere. A mixture of N_2-O_2 was used for oxidizing conditions to prevent a serious amount of oxide defects. All crystals were doped with various rare earths in a range of 0.05 to 1.0 atomic %. Appropriate segregation coefficients were applied for each doping element.[13] Total crystal lengths of 3-4 inches and diameters of 0.6 - 0.8 inch were grown. These boules were sufficient to provide up to a (6 x 60)mm laser rod plus additional pieces for sizeable polished rectangular blocks, cubes, or discs. The latter were used for many experiments in spectroscopy or as direct samples pumped by other lasers.

All crystals were grown along [111]. Seeds were composed of undoped YAG to prevent contamination of the melt with traces of other elements such as Nd. It was also necessary to clean the crucibles thoroughly between runs to avoid

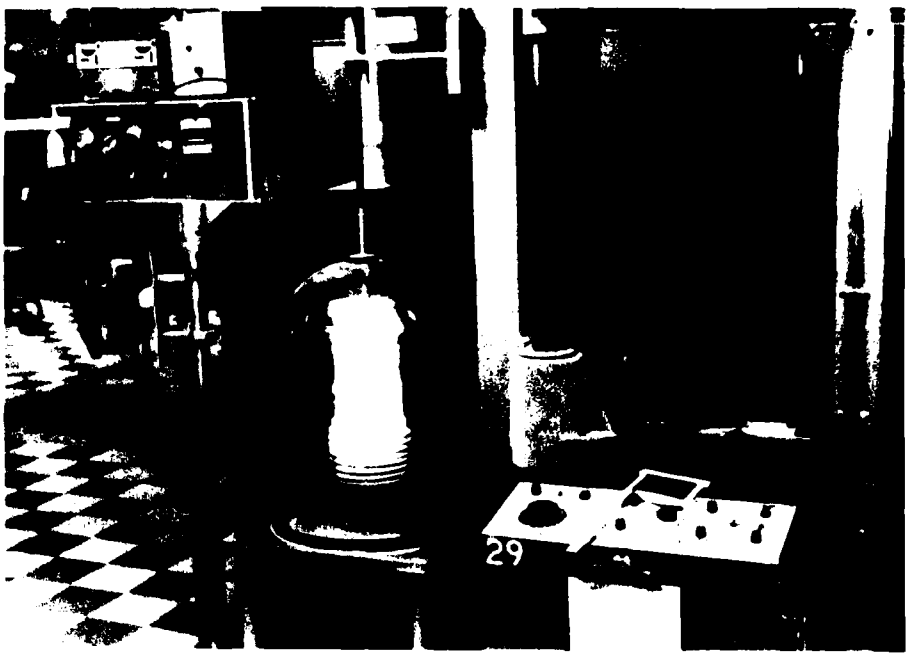

Figure 1 Czochralski Station for Oxide Growth

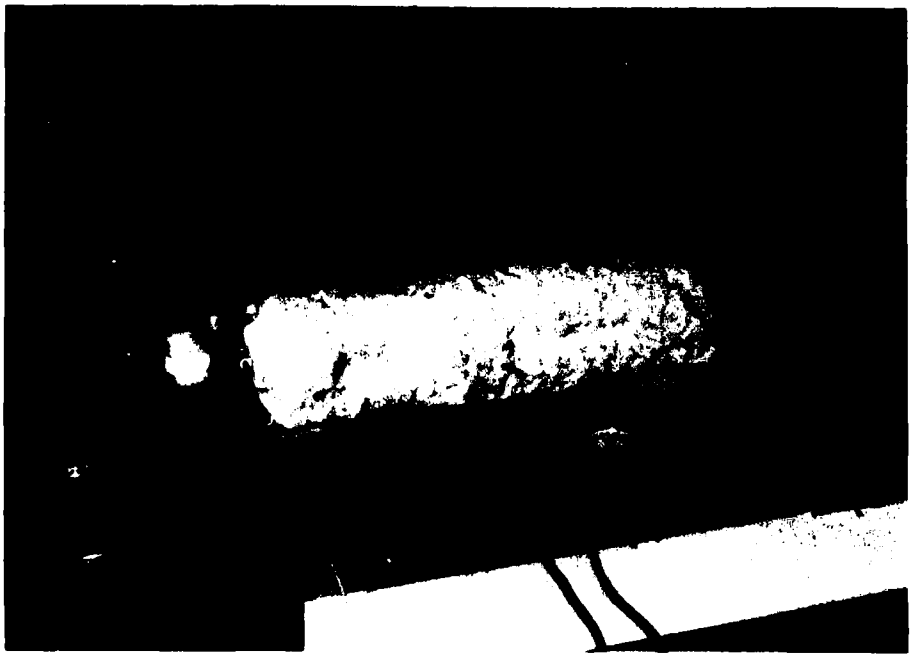

Figure 2 Hydrofluorination Apparatus

trace contamination from th. various dopants. Cleaning was performed by core drilling and removing most of the residual melt, flux treatment to dissolve material, and finally a regular acid cleaning. Crucibles are then preheated, checked for leaks, and repaired if necessary before the next crystal growth run.

Laser rods were fabricated in Airtron's production facility and presented no special problems. Several special end configurations such as Brewster angle, off orientation, and convex curvature were prepared in addition to the flat-flat type. Rectangular parallelepipeds also were fabricated where pumping was performed with another laser instead of a flashlamp. Most of the finished rods were prepared with a single layer AR coating for the chosen wavelength of testing. However some rods required special multilayer coatings for dual wavelengths in the 1.5 - 4 μm range. These rods were not coated at Airtron but were done at an outside vendor.

2.2 Fluorides

Preparations of doped $YLiF_4$ were synthesized from LiF and 99.99% purity oxides of Y_2O_3 and rare earths which were converted to fluorides. The oxides were obtained as polycrystalline powders from Research Chemical Corp. and the LiF was purchased as single crystal ultraviolet grade material from Harshaw Chemical Co. Analyses of all components were based on rare earth elements only. One deviation was made in

our current work wherein several growth runs were prepared from 5-9's starting oxides. The latter were the highest purity obtainable in any rare earth compound.

All compositions were prepared with individual mixtures of the component oxides. Our procedure utilized about 150 g of mixed oxides which were converted subsequently to fluorides by means of a high temperature platinum hydrofluorination apparatus. Final temperatures were raised to 1200°C for all the fluorides. This temperature is above the melting point of each of the component fluorides. As a result a very homogeneous mixed crystal of mm size particles is obtained. The polycrystalline mass can be stored with little chance of further hydrolysis. A picture of the hydrofluorination apparatus is given in Figure 2.

Single crystals of substituted $YLiF_4$ were grown by means of the top-seeded solution method.[14] A platinum growth furnace and crucible were utilized to maintain growth under a flowing atmosphere of 90% N_2 - 10% HF. A weight sensing diameter control system which performed a continuous weighing of the seed gave excellent results on the crystal shape. Starting compositions were 47 mole % rare earth fluorides and 53 mole % LiF. The crucible size was 2.0 inches diameter x 2.0 inches high. It was filled to 90% of its capacity at the growth temperature. All boules were [100] or a axis oriented along the growth direction. The

pull rate and rotation rate were varied to control the solid-liquid interface shape. The latter was flat in most cases. When several runs of one composition were made, the melt was replenished with no decrease of crystal quality. If a composition was changed the crucible was cleaned thoroughly between runs. The greatest asset to a clear defect-free single crystal was a proper heating and homogeneous solutions before growth was attempted. A high pull rate also tended to give small bubbles concentrated along the growth interface. Most of our crystals were grown at 0.6 mm/hr and 20 rpm. The boule were about 2cm in diameter x 5-6cm long. There appear to be no difficulties in going to larger sizes if these are needed for increased yields. From the 2 cm diameter boules at least two and sometimes four (4 x 50)mm laser rods could be core drilled in rough form.

Two different growth furnaces were used for fluorides. The first of these is pictured in Figure 3 and was similar to others used in the past at Airtron for fluoride growth. The maximum operating temperature for this furnace is about 900-950°C. Thus it is suitable for all compositions of $RLiF_4$ where R = rare earth. An alternate furnace was purchased from Astro Industries of Santa Barbara California. This furnace is pictured in Figure 4 and consists of a graphite resistance heated chamber capable of operation close to 1800°C. A weight control system was added to maintain constant diameter.

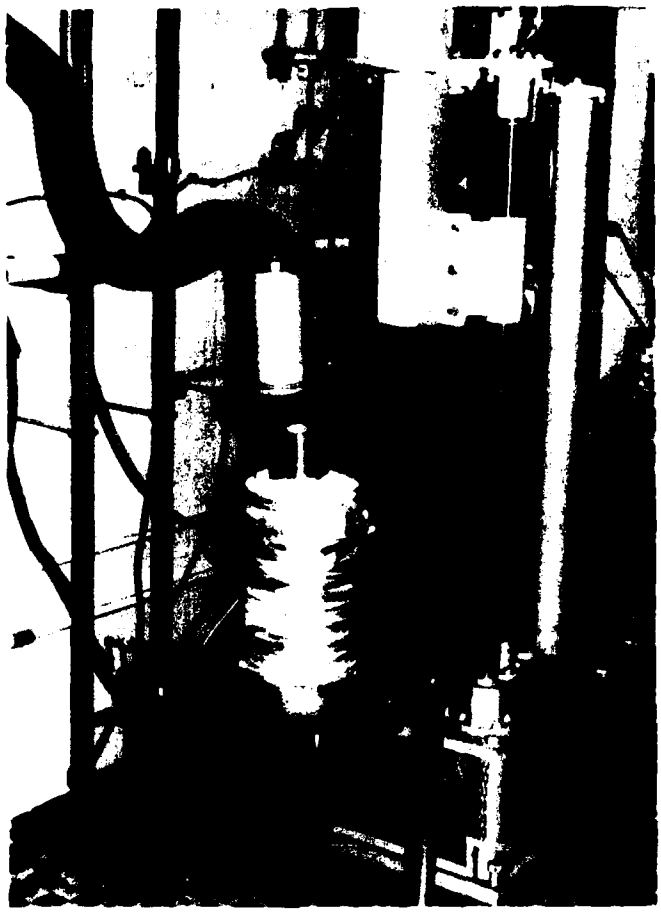

Figure 3 Growth Station for Low Melting Fluorides - $LiYF_4$

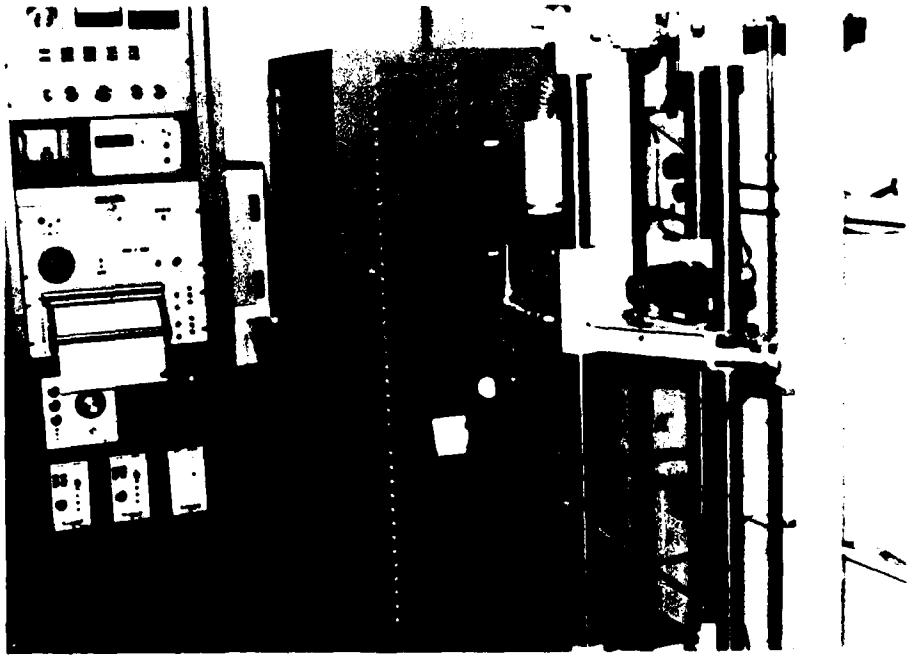

Figure 4 Growth Station for Use of Fluorides to 1500°C

This furnace was also capable of operating in an HF atmosphere at 1000-1500°C. The Astro furnace was used for high melting materials such as BaY_2F_8, YF_3, MgF_2, or LaR_3.

All fluoride materials were processed in a manner identical to Nd:YAG. The grown boules were inspected optically, ends were cut, rods were core drilled, and then fabricated according to specifications. After polishing all rods, they were used with no antireflection coating or a special multi-layer composition was designed for the intended test wavelength. No active testing was performed at Airtron. All delivered samples were composed of cylindrical rods with flat-flat ends or cm size rectangular parallelepipeds with one pair of laser polished faces.

3.0 Results of Preparations

In this section we describe briefly all of the materials grown under the contract. The purpose for each material is not stated explicitly. In most cases the scheme of intended laser action and wavelength output is known from previous literature. However the particular references are not recorded in this report because of their number. As a general reference text, one may consult the book by Kaminiski[15] which mentions most important materials and their physics.

3.1 Growth of YAG

Nearly all full year's work was devoted to various garnets in a previous program.[16] Therefore only a few more

compositions were attempted under the present effort. The first doped YAG was a triple doped system of Ho, Er and Tm designed for possible laser action at 2.06μm. This material was prepared in an identical composition to many $YLiF_4$ crystals grown for 2.06μm application. The composition was 61.6% Er, 32.6% Y, 3.7% Tm, and 2.1% Ho. A rather large crystal was grown (Figure 5) and (5 x 57)mm laser rods were extracted. The rods were all good quality and equivalent to commercial Nd:YAG. No problems arise in most of these runs even though the doping levels are high.

3.2 Growth of $YLiF_4$

The versatility of this host is well known and it can accept all the rare earths in a similar fashion to YAG. Many $YLiF_4$ crystals were grown under our program. The easiest of these were obtained when the rare earth ion consisted of the series Dy to Lu. All of these were grown first with the most popular being the Er, Tm, Ho system. Thus several trials were performed for this composition which gives a useful laser at 2.06μm. The crystals of $LYiF_4$ were always grown in the a-axis orientation of the tetragonal unit cell. This orientation is perpendicular to the c or optic axis. The a-axis crystals exhibit a two-fold symmetry during growth while the c-axis has a four-fold axis of symmetry. The crystals have no cores as long as the growth interface is nearly flat.

The first composition of $YLiF_4$ which was attempted

Figure 5 YAG:Er,Tm,Ho Boule NC-74

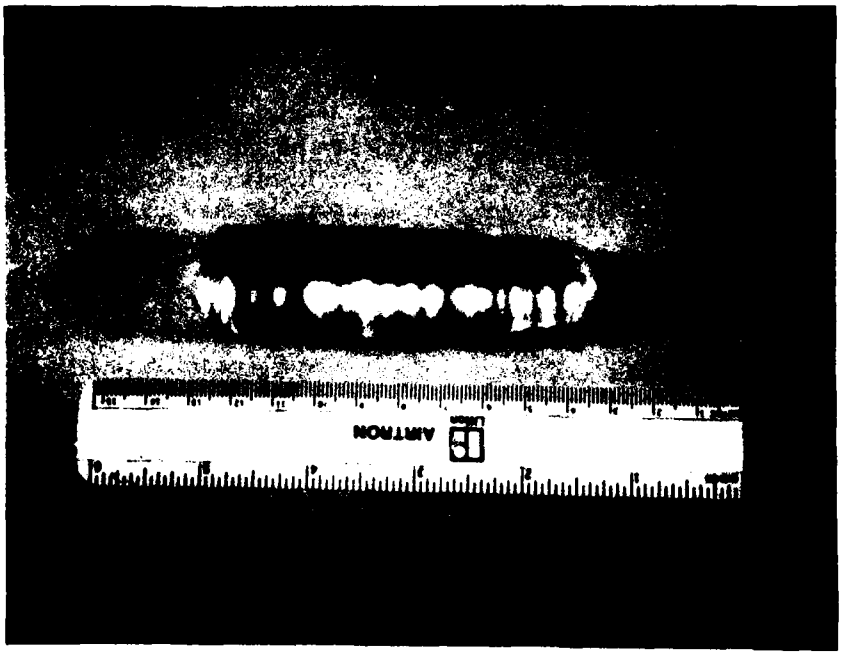

Figure 6 YLF:Er,Tm,Ho from YLF-78

under the program was the triple doped system of 35% Er, 10% Tm, and 2% Ho. This composition was desirable in the rod form of (5 x 50)mm size. An initial run from materials prepared by our hydrofluorination showed a fair amount of oxide or oxyfluoride contamination. Furthermore a new furnace was arranged and control systems were checked. In any circumstance, this run did not progress satisfactorily and only small pieces of single crystal were obtained from YLF-75.

A new composition was prepared for Run YLF-76 and a crystal was seeded easily. About 20-30mm of a good single crystal was obtained. However this length did not yield the 50mm rods but only spectroscopic pieces and a few good seeds for additional runs. The burn-off at 30mm was caused by a faulty control or improper gradient in the furnace. Both of these items were checked thoroughly prior to the next run. The a-axis seed was also X-ray oriented. A new furnace element was constructed with proper thermal gradient.

Run YLF-77 was made under the new furnace conditions and gave a fairly good boule of about 60mm. Another recharge of material produced a boule of about 90mm length. A sufficient portion of this boule was free of any optical scattering sites, so an attempt was made to core drill the first laser rods. Four different rods were tried but each one fractured during the course of drilling. At this point the mechanical construction of the drills, runout at drilling, and other

factors were checked with the manufacturer.

The subsequent growth run YLF-78 yielded an excellent crystal about 20-25mm in diameter and 80mm in length (Figure 6). The optical quality was very good with only a few scattering sites near one side of the boule. The boule was cut on the ends and a good section was removed. Eight rods of (5 x 55)mm were attempted. Three of these broke during drilling. The remaining five rods survived all drilling, fabrication, and passive testing. Three of the best rods were delivered.

At this stage a new composition of $LiYF_4$ with 5% Dy was prepared. A fairly good single crystal was obtained as shown in Figure 7 for run YLF-82. The diameter control was a bit erratic but a good quality boule of 15mm diameter and 110mm length was grown. At the center some gaseous bubbles appeared but cm size spectroscopic samples and several 50mm laser rods were prepared. These samples were delivered after proper fabrication.

A new platinum crucible of a size 2 x 2.25 inches was introduced into our growth system. This enabled a larger charge of material to be added and possibly a larger final boule. Run 84 was then made with the Er, Tm, Ho dopant again. A 20 x 100mm boule was obtained with excellent results. A picture is given in Figure 8. An optical examination showed a small amount of scattering due to gaseous inclusions. These were located near the midlength of the boule and in the center

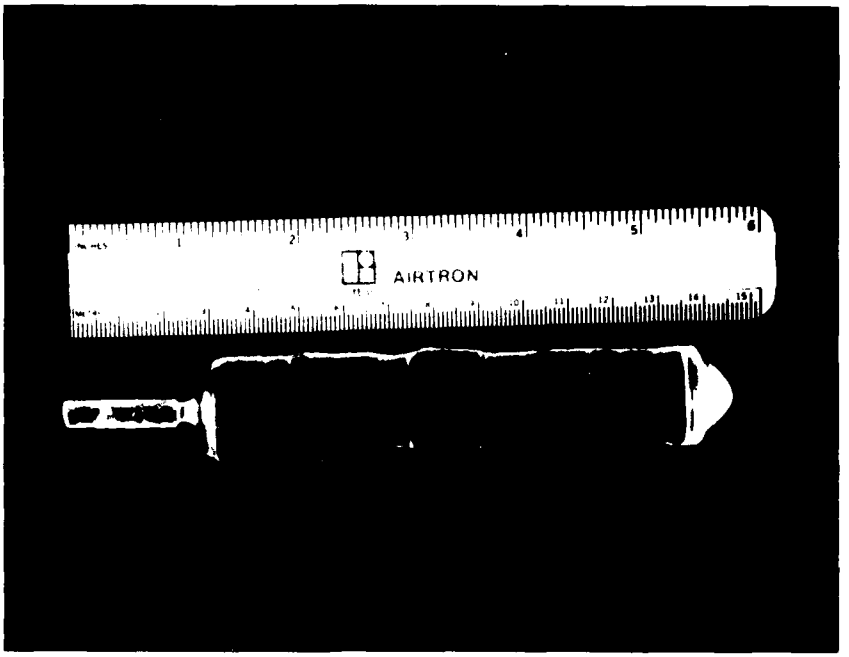

Figure 7 YLF:Dy from Run 82

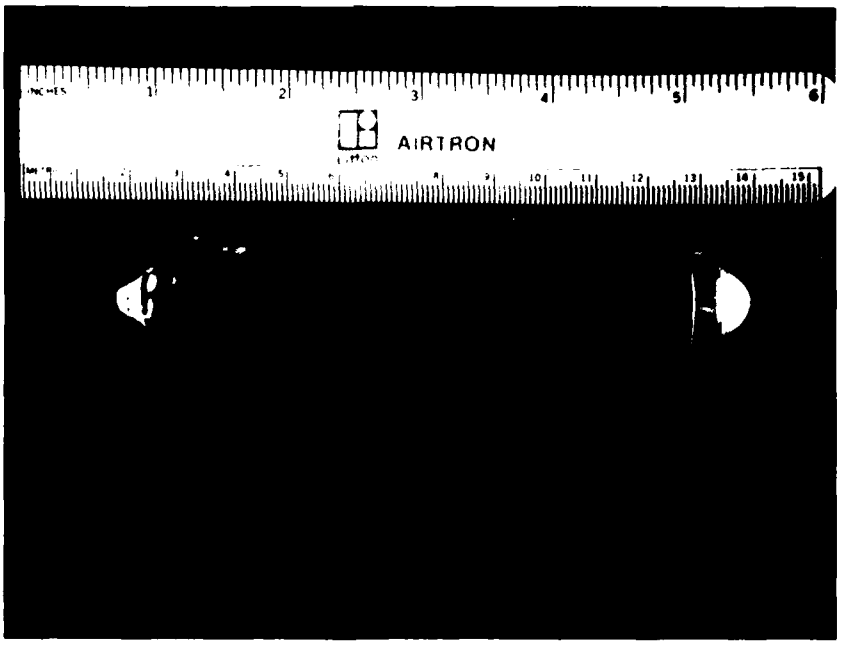

Figure 8 YLF:Er, Tm, Ho from Run 84

of the cross section. Fortunately 50mm long laser rods could be drilled outside of the defective area. Rectangular sections of several cm could also be obtained. Several rods and pieces were delivered.

The next composition was a $LiYF_4$ with a 2% Er. This crystal run was YLF-85 and gave excellent diameter control on the (20 x 110)mm size pictured in Figure 9. A small amount of scattering was found near the top and about 70mm down from top. The scattering started at the core and spread to the outer diameter. No diameter change, interface change, or growth artifact occurred at the initiation of these defects. This may suggest a complication of phase behavior after a certain fraction of melt removal. Rods were drilled from the boule and fabricated with little difficulty.

Attention was now given to the double doped system of 0.5% Pr and 1% Tm in $YLiF_4$. This crytal was grown satisfactorily but there may be some uncertainty of composition due to unmeasured value for the Pr segregation coefficient. A picture of the boule is given in Figure 10. About 40mm was clear of defects and several rectangular sections were cut from the boule and polished. Scattering was isolated at the top and bottom of the boule.

One last crystal of another 2% Er in $YLiF_4$ was grown. This boule was run 97 and is pictured in Figure 11. Again a uniform diameter crystal of (20 x 110)mm was obtained. The

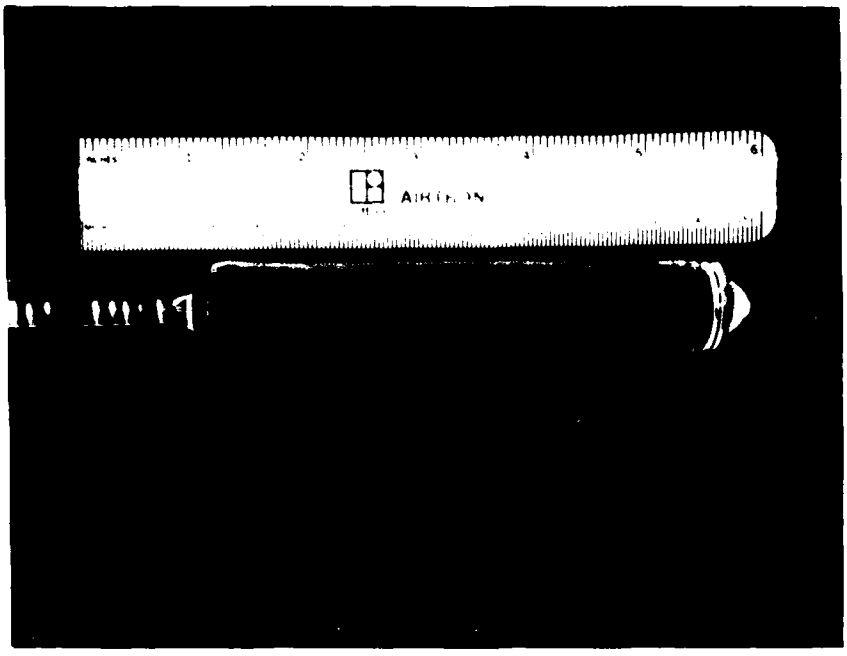

Figure 9 YLF:Er from Run 85

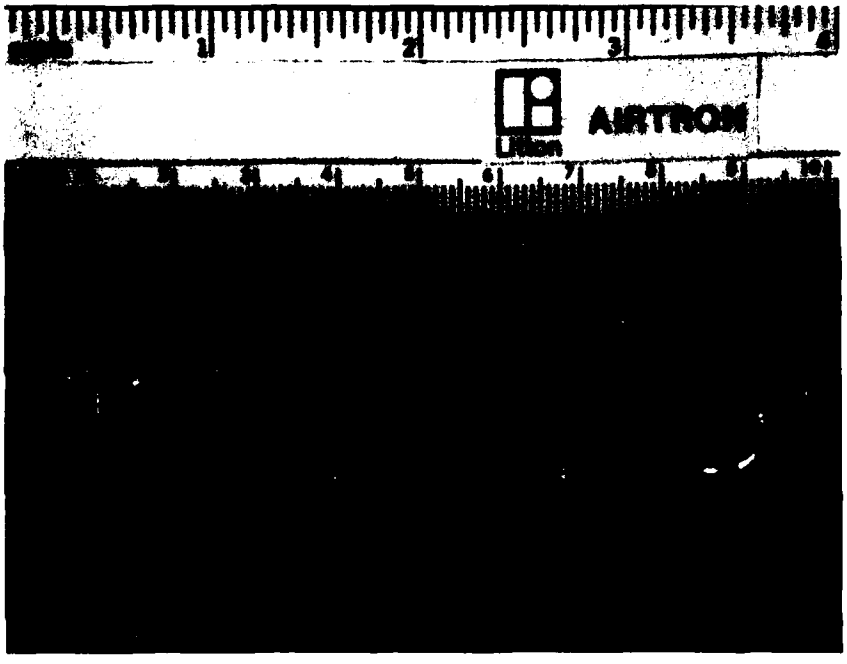

Figure 10 YLF:Pr, Tm from Run 86

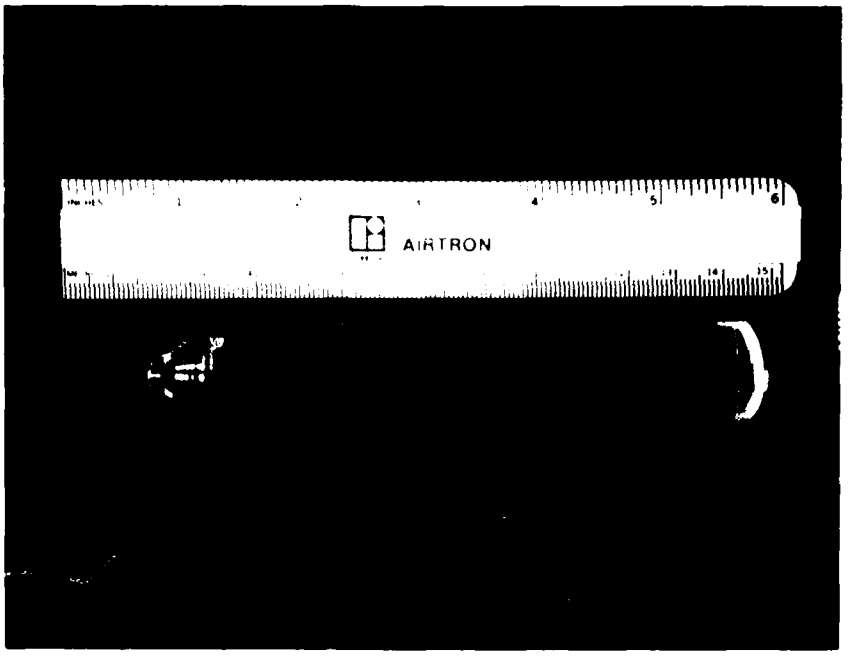

Figure 11 YLF:Er from Run 97

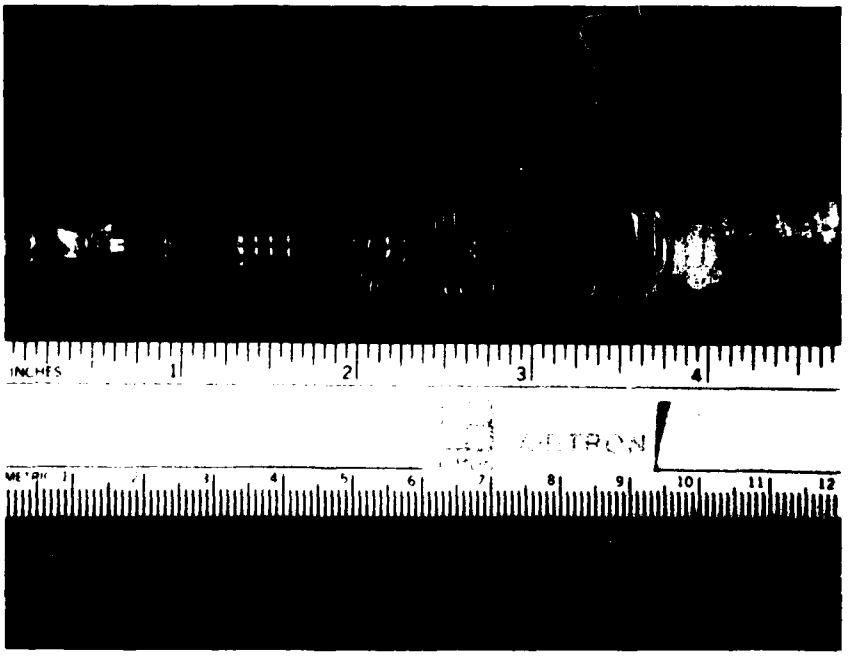

Figure 12 BaY_2F_8:Er from Run 87

crystal was clear optically and five rods of (4 x 55)mm size were core drilled successfully.

3.3 Growth of BaY_2F_8

Almost all of the $LiYF_4$ runs were made without major difficulties. The growth of BaY_2F_8 is more complicated from the standpoints of crystal structure, melting point, phase behavior, and general experience on the material. For our initial experiments seed crystals were provided by Dr. H. Guggenheim of Bell Laboratories. Seeds were also obtained by insertion of a platinum wire and growing a crystal from the seed on the wire. The small crystals were then cut and larger seeds were prepared. In all cases of Airtron preparations, the crystal phase and structure were compared by X-ray diffraction against the Bell Laboratory samples. A perfect match was obtained so further large crystals were grown and in the same axial orientation.

The first attempt on BaY_2F_8 involved a 2% Er doping. An inch long crystal was obtained almost free of any scattering even though the growth rate was 2.5mm/hr. A new seed was prepared and a second run was made from the same melt. A fairly good crystal was pulled even though the diameter control was a bit erratic. The crystal from run 87 is shown in Figure 12. It was about 10mm x 60mm long and free of all scattering. A grain boundary or slight misoriented section was found near the top. Rods were core drilled and one (4 x 55)mm rod was fabri-

cated and delivered.

A fresh preparation of a Ho doped run was then synthesized and several crystal growth attempts were made. This material from Run 88 consistently gave a polycrystalline boule in spite of the use of a good single crystal seed. It appears that the melt was contaminated with impurities of oxyfluoride or oxide. The run was abandoned and a fresh charge was made for a future trial.

The next charge for a composition consisted of a 1.5% Nd doping in BaY_2F_8. This run was No. 93 and gave a fair initiation of growth from the seed. However several sections of the crystal necked in to a few mm from the 15-20 mm of normal growth. No laser rods were obtained but several single crystal sections of cm size were cut. The quality appeared to be good.

The following run 94 was a new preparation of 0.5% Pr. This crystal seeded very well, then necked in, and finally stabilized thermally. A crystal about (15 x 60)mm was grown and is pictured in Figure 13. One (4 x 54)mm laser rod was obtained from the boule and is also shown in the picture.

The final run 95 was a composition of 5% Dy in BaY_2F_8. A little difficulty was experienced in seeding but a single crystal was grown. The size was about (12 x 80)mm. Several cracks in the crystal precluded the drilling of a laser rod but several cm size section were cut and polished. The quality was

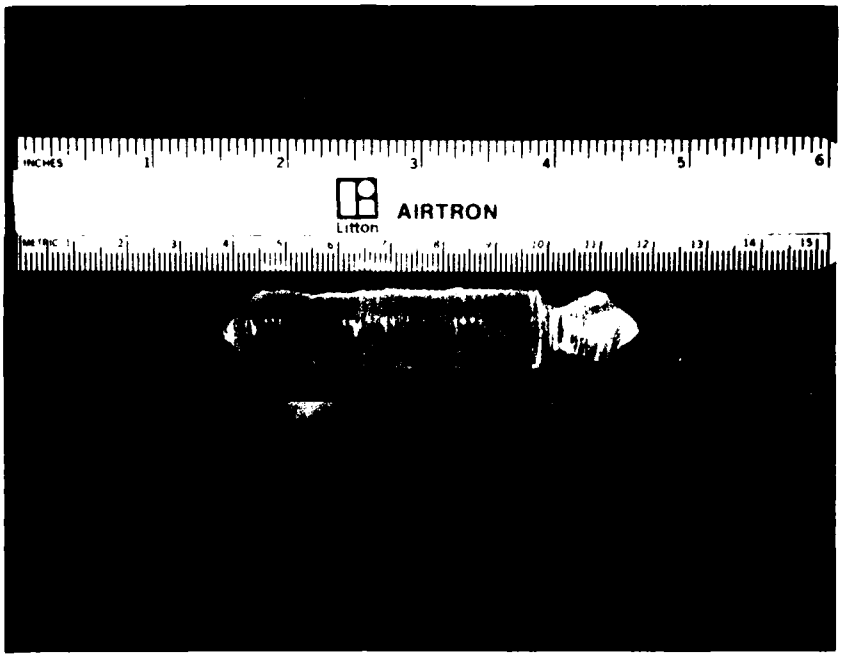

Figure 13 BaY_2F_8:Pr From Run 94

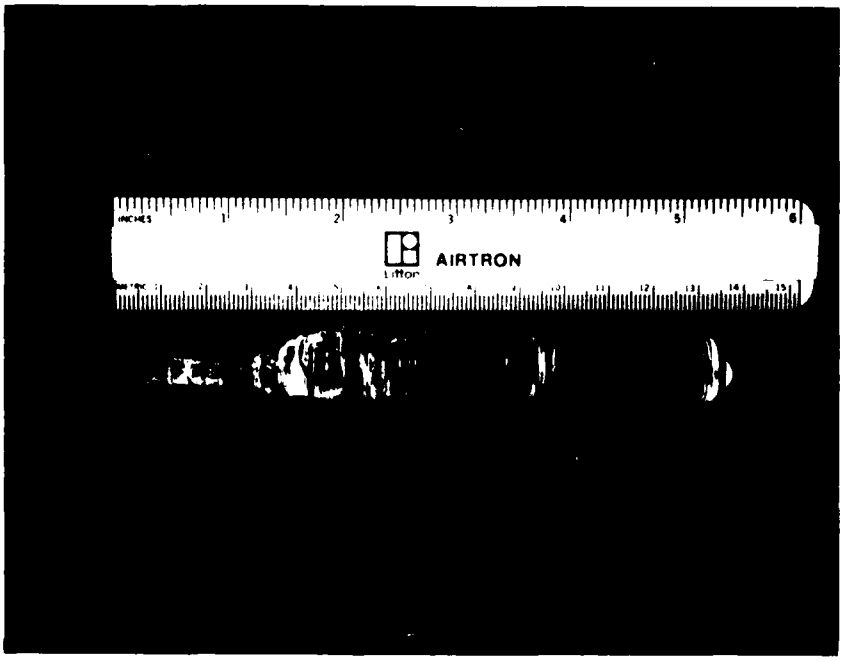

Figure 14 BaY_2F_8:Dy from Run 95

reasonably good and free of scattering centers (Figure 14).

3.4 Growth of YF_3

Single crystals of YF_3 were most difficult to obtain because of lack of seeds, high melting point, and phase behavior. YF_3 melts at about 1150°C. In its high temperature behavior, there is an analogy to other rare earth trifluorides. A phase transition probably occurs near the melting point; a hexagonal to orthorhombic transition may be as narrow as 25-50°C. The preparation of YF_3 must be free of YOF and Y_2O_3 to get good crystals. Seeding was attempted by using a platinum wire. All growth experiments were performed in the furnace of Figure 4.

The first attempt on YF_3 was run 83. The doping level was 0.1% Pr. The crystal preparation was not pure YF_3 but a mixture of YF_3 and LiF. The proportion of LiF was about 5.5 weight per cent. The melting point of this mixture was estimated from the phase diagram to be low enough to crystallize only the low temperature orthorhombic phase. After the material was melted in a crucible seeding was attempted several times. No good single crystal was started. During the run some graphite from the heaters and insulation flaked off and was blown into the crucible by gas turbulence. The impurities lead to a polycrystalline nucleation and no crystal was obtained. Later in the program more trials were made but it is clear that more time must be spent to get high quality crystals. The YF_3:Pr

was made before and a few crystals of cm size were obtained. Thus until the program is extended, little progress can be made in a few short growth runs.

3.5 Summary of Growth Runs

As an adjunct to the above descriptive data, a tabular summary of all fluoride materials is given in Table I. These entries give the run number, host crystal, dopant concentration, pull rate, rotation rate, and additional comments on each run. For the most part single crystals were obtained and either laser rods or rectangular blocks could be fabricated. This was possible because most of the crystals were grown at Airtron under previous research programs.

4.0 Conclusions

Single crystals of $YLiF_4$ and BaY_2F_8 were grown with a variety of rare earth ions as activators. The $YLiF_4$ compositions were the easiest to grow and large crystals were obtained in nearly all cases. Laser rods of a size (4 x55)mm were extracted from the boules. In a few growth runs where poor diameter control occurred or the crystal cracked, rectangular solids were fabricated for laser testing. The compositions of BaY_2F_8 were more difficult to grow because of a general lack of experience. However it was also expected that the larger rare earth activators had different phase diagrams which were not known thoroughly. The most difficult crystal was YF_3. Attempts were made to grow this crystal from LiF solution in

Table I

Summary of Crystal Growth Runs

Run No.	Host Crystal	Dopant Conc. (Atom%)	Pull Rate (mm/hr)	Rotation Rate (rpm)	Remarks
74	$Y_3Al_5O_{12}$	35 Er, 10 Tm, 2 Ho	0.6	30	2 - 4 x 57mm rods 2 - 5 x 57mm rods
75	$YLiF_4$	35 Er, 10 Tm, 2 Ho	1.27	25	Oxyfluoride contamination
76	$YLiF_4$	35 Er, 10 Tm, 2 Ho	1.27	25	Growth problems; no yield
77	$YLiF_4$	35 Er, 10 Tm, 2 Ho	1.27	25	Fair growth; rods broken in core drilling
78	$YLiF_4$	35 Er, 10 Tm, 2 Ho	1.27	25	3 - 5 x 50mm rods shipped
82	$YLiF_4$	5 Dy	0.76	20	1 - 4 x 55mm rod 2 - Rectangular solids
83	YF_3	0.1 Pr	0.76	22.5	No crystal
84	$YLiF_4$	35 Er, 10 Tm, 2 Ho	0.76	20	3 - 4 x 50mm rods
85	$YLiF_4$	2 Er	0.76	20	1 - 4 x 55mm rod 1 - Rectangular solid
86	$YLiF_4$	0.5 Pr, 1 Tm	0.76	20	2 - Rectangular solids
87	BaY_2F_8	2 Er	2.54	23	1 - 4 x 55mm rod

Table I (Continued)

Run No.	Host Crystal	Dopant Conc. (Atom%)	Pull Rate (mm/hr)	Rotation Rate (rpm)	Remarks
88	BaY_2F_8	2 Ho	2.54 1.27 1.27	23 23 11.25	Oxyfluoride contamination
89	YF_3	1 Pr	0.76	23	Polycrystalline growth; no crystal
93	BaY_2F_8	1.5 Nd	1.3	11	
94	BaY_2F_8	0.5 Pr	1.3	11	
95	BaY_2F_8	5 Dy	1.3	11	
97	$YLiF_4$	2 Er	0.76	20	5 - 4 x 55mm rods

order to obtain the low temperature orthorhombic phase. No success was achieved in a few runs.

All crystals were fabricated and tested passively using similar procedures developed for Nd:YAG. Samples of the materials were delivered to the Naval Research Laboratory for active testing.

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