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RUBY IMPROVEMENT FOR LASERS - TASK I

REPORT NO. 3

U. S. Army Signal Supply Agency; Contract No. DA 36-039-SC-89089
DA Project No. 3A-99-21-001 Task #1

THIRD QUARTERLY PROGRESS REPORT

1 NOVEMBER 1962 TO 31 JANUARY 1963
U. S. Army Signal Research & Development Laboratory
Fort Monmouth, New Jersey

LINDE CO.

DIVISION OF UNION CARBIDE CORPORATION

CRYSTAL PRODUCTS
EAST CHICAGO, INDIANA



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403 133

RUBY IMPROVEMENT FOR LASERS TASK I

REPORT NO. 3

SIGNAL CORPS CONTRACT DA 36-039 SC89089

SIGNAL CORPS TECHNICAL REQUIREMENT NO. SCL-2101N

DA PROJECT NO. 3A 99-21-001-TASK #1

THIRD QUARTERLY PROGRESS REPORT

1 NOV 1962 TO 31 JAN 1963

**OBJECT: To investigate the most important variables
in the Verneuil growth of ruby for laser
application.**

PREPARED BY: R. L. Hutcheson

**FOR: Union Carbide Corporation
Linde Company Division
Crystal Products Department
4120 Kennedy Avenue
East Chicago, Indiana**

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PURPOSE

The aim of this program is to produce ruby for laser application by the flame fusion (Verneuil)¹ crystal growth process superior to presently available ruby. The program to accomplish this end is outlined in Table 1 (Revised 1 Sept 62). The work is divided into producing ten lots of ruby boules², consisting of three to five boules each. Each lot studies one to three variables with each subsequent lot being grown with the one best growth condition from the previously tested parameters.

The growth parameters being investigated are as follows:

1. Thermal gradients across the crystal during growth (Lots I through III)
2. Crystal growth rates (Lots I through VI)
3. Annealing cycles (Lots IV through VI)
4. The addition of fluxing agents (Lot VII)
5. Crystal axis orientation related to the growth direction (Lots VIII and IX)

Lot X is to be grown and annealed under the best conditions as selected from Lots I through IX and serves to summarize the work on this project.

The growth techniques employed for this work were available at Linde's East Chicago facilities prior to the inception of this contract and no new techniques are being employed. To the knowledge of the Linde Company this work is the first major attempt to relate growth parameters and crystal quality to laser performance. Information disclosed on the crystals will be only that information necessary to define the variables listed above.

Concurrent with this program the Linde Company is working on improved powders for the growth of ruby crystals via the flame fusion technique. The results of this internal work will be made available to this program at the earliest possible date. In conjunction with Task I of this contract, Perkin-Elmer Corporation is working on crystal evaluation under Task II. In addition some evaluation work will be done at Fort Monmouth and the Linde Company.

¹ Verneuil - U.S. Patent No. 1004505.(1911)

² Boule is a term commonly used to define the crystal grown by the flame fusion process. It originates from Verneuil's original work.

TABLE 1

SUMMARY OF PROGRAM FOR "RUBY IMPROVEMENTS FOR LASERS"
(Revised 1 Sept 62)

Lot No.	No. of Boule Per Lot	Thermal Gradients From Cap To Seed	Seed Quality	Seed Orientation	Fluxing Agent	Annealing Cycles	Relative Growth Rate	Scheduled Shipping Date
1	3	300°C	See Note 2	90°	None	Normal	3	7-31-62
2	3	150°C	See Note 2	90°	None	Normal	3	7-31-62
3	3	Approx. 50°C	See Note 2	90°	None	Normal	3	8-31-62
4	5	50°C	See Note 2	90°	None	(1) Normal (Spec. Powder) (2) Normal (Conv. Powder) (3) In Place (Conv. Powder) (4) Fast Cooling (Conv. Powder) (5) Slow Cooling (Conv. Powder)	2	10-1-62
5	5	50°C	See Note 2	90°	None	Same as Lot 4	4	10-31-62
6	5	50°C	See Note 2	90°	None	Same as Lot 4	1	11-30-62
7	3	50°C	See Note 2	90°	0.1% Flux A 0.1% Flux B 0.1% Flux C	See Note 4	See Note 5	12-31-62
8	3	50°C	See Note 2	0° 30° 45° 60°	See Note 3	See Note 4	See Note 5	1-31-63
9	3	50°C	See Note 2		See Note 3	See Note 4	See Note 5	2-28-63
10	3	See Note 6	See Note 2	See Note 6	See Note 6	See Note 6	See Note 6	4-30-63

NOTES: 1. The thermal gradient used is to be the best as determined from results of tests on Lots 1, 2, and 3.

2. The seed quality is to be held constant throughout the growth of all boule lots and is to be fabricated from specially selected sapphire disc boule and used in sapphire rod holder.

3. Based on the results of tests conducted on Lots 1 through 7, it will be determined if a fluxing agent is to be used and if so, which one.

4. Based on the results of tests conducted on Lots 1 through 6, the best annealing cycle is to be determined and used.

5. Based on the results of tests conducted on Lots 1 through 6, the best growth rate is to be determined and used.

6. The decision as to the factors associated with the growth of the boules in Lot 10 will be based on the results of tests performed on Lots 1 through 9 and will represent the optimum growth condition as determined from the proposed Development Program.

TABLE 1

ABSTRACT

The effects of growth rate and the addition of fluxing agents to starting powders on the growth of ruby are discussed. Growth data on two lots of ruby crystals are presented.

CONFERENCES

Subject: Future Contract Planning

Person
Attending: Fort Monmouth—Mr. Morris Katzman
Mr. Charles Kellington

Linde Company—Mr. B.N. Callihan
Mr. R.L. Hutcheson

Held: USASRDL
Fort Monmouth, New Jersey
27 November 1962

Object: To review status of the work performed to date on the contract and to make the necessary decisions related to the growth of crystals for Lots VII, VIII and IX.

- Conclusions:
1. As per the past meeting of 9 August 1962, lower thermal gradients as studied in Lots I, II and III are necessary for improved crystal quality. Thus, all growth should be on thermally stabilized (T.S.) equipment with an approximate 50° thermal gradient.
 2. From observations and evaluations of Lots III, IV, V and VI in which relative growth rates were studied on T.S. equipment, a growth rate of 2 produces crystals of the most uniform quality. Therefore, all additional crystal lots for this contract will be grown at a relative growth rate of 2. It will be necessary to grow lot VIII at a different rate because the crystal orientation is 0°.
 3. No conclusions as to the effects of different annealing cycles can be drawn from preliminary evaluations. If annealing procedures are related to optical maser crystal quality, it can only be proven from data obtained during actual laser operation. Therefore, all additional crystals as produced for this contract will be annealed in the normal manner unless indicated otherwise by the evaluations of other parties.
 4. Preliminary conclusions drawn from evaluation of crystals prepared for Lots IV, V and VI indicate that specially prepared growth powder improves overall crystal quality. Therefore, all additional crystal lots will be grown with specially prepared powders.
 5. Preliminary conclusions indicate that seed quality is related to crystal quality. Therefore, all additional crystals will be grown on seeds specially X-rayed and found to be of less than 10 min. misorientation.

DISCUSSION

Introduction:

The crystal growth process used to grow ruby for this contract is the flame fusion or Verneuil technique. The process is shown schematically in Figure One. Powder of sufficiently high purity and of proper dispensing characteristics is dropped into a high purity oxygen stream. The powder is carried by this stream into a furnace chamber heated by a post mixed oxy-hydrogen burner. The powder is dropped through this chamber to the molten cap of a seed crystal. A thermal gradient is established vertically down the seed crystal. This gradient allows solidification to take place on the seed in an orderly manner thereby establishing crystal growth. By proper control of the volume of the molten cap and the rate of solidification from this cap, a crystal of the desired size and shape can be grown.

The reports for the first and second quarters of this contract have presented growth data on crystal lots I through V (See Table I). Included in these lots are crystals for studies on thermal gradients measured vertically down the crystal during growth, growth rate, annealing cycles and effects of seed misorientations. Also some methods for crystal internal quality evaluation are discussed. The conclusions reached through the second quarter are:

1. A thermal gradient down the crystal of 50°C produces ruby of superior quality as compared to crystals grown at higher thermal gradients.
2. Crystals grown at a relative growth rate of 2 are of superior quality as compared to those crystals grown at relative growth rates of 3 and 4.
3. Different annealing cycles show no obvious effect on crystal quality.
4. Seeds used to grow improved ruby via the flame fusion process must be of superior quality.

Growth data on crystal lots VI and VII are presented in Table II. Lot VI completes the crystals necessary for growth rate and annealing cycle studies. Lot VII studies the addition of fluxing agents to the growth powder.

Growth Rate:

Crystal growth rate via the flame fusion process is related to the maximum allowable growth speed of any given crystal orientation, as to the per cent boil out of high vapor pressure impurities, and as to process reliability. Relating crystal quality parameters to growth variables, the following general comments can be made:

TABLE 11

CRYSTALS SHIPPED UNDER CONTRACT DURING THRID QUARTER

Lot No.	Crystal No.	Specification				Powder	Thermal Gradient °C	Anneal Cycle	Growth Rate Relative	Evaluation			
		Length Inches	Dia. Inches	Wt. Grams	(1) Orientation Degrees	(2) % Cr ₂ O ₃ Final				(3) Bubbles	(4) Lineage	(5) Smoke	(6) Schlierin
VI	CP-134-16	3 1/8	1/2	138	90	.034	Conv.	Fast	1.0	Light	Medium	Medium	Medium
	CP-134-24	3 1/2	3/8	103	90	.029	Conv.	Slow	1.0	Light	Light	Medium	Light
	CP-134-28	3 1/2	11/16	136	90	.030	Conv.	Normal	1.0	Light	Light	Heavy	Medium
	CP-134-48	3	5/8	132	90	.032	Lab	Normal	1.0	Light	Heavy	Heavy	Medium
	CP-134-52	3	1/2	111	90	.032	Lab	In Place	1.0	Light	Medium	Heavy	Medium
VIIA	1716-14	3 7/16	5/8	164	90	.045	Lab	Normal	2.0	Light	Medium	Heavy	Light
B	1716-22	3 1/2	5/8	142	90	.040	Lab	Normal	1.3	Light	Medium	Medium	Heavy
C	1716-42	3	1/2	102	90	.046	Lab	In Place	1.3	Light	Heavy	Medium	Heavy

1. Orientation angle is defined as the angle between the growth axis and the c-axis of the crystal.
2. % Cr₂O₃ is measured by comparing the optical density at 5600Å to the corrected white sapphire transmission at 5600Å.
3. Bubble content is graded by comparison to a standard boule in the area of the highest bubble concentration at 10X. The right angle scatter photographs in Figures Two and Three give a relative comparison.
4. Lineage is graded by comparison of the boule with standard boules between crossed polaroids. For examination of the boule, windows are polished perpendicular to the c-axis. The photographs shown in Figures Two and Three indicate the patterns obtained although contrast is lacking because of the black and white reproduction.
5. Smoke is graded by comparison of the Tyndall effect in the boule being compared to that same effect in a standard boule. The effect can be observed in the right angle scatter photographs shown in Figures Two and Three. End reflection must be neglected in the comparison of these boules.
6. Schlierin comparisons are explained in text of Second Quarterly Report of this contract.

TABLE 11

1. The bubble content is related to the entrapped impurities which will boil out when given sufficient time in the melt.

2. The average total lineage for a lot of several crystals should be related to process reliability. As crystal growth times increase, process reliability decreases; as for example, short term changes in powder feed rate and hence instantaneous growth rate.

3. The minimum lineage measurement for a lot of crystals should be related to time induced distortions of the crystal lattice. Other factors such as impurities have an influence on lineage formation. Therefore, because of statistical reliability factors, in a lot of several crystals grown under near identical conditions the minimum lineage value should approach the lowest lineage possible under the growth conditions employed.

4. The degree of chromia banding is related to the process reliability for the particular growth process being used. The severity of chromia banding caused by an equivalent growth condition fluctuation is increased significantly as the thermal gradients are decreased. To the knowledge of the author, the growth facilities used to produce crystal lots III through VII are the most reliable available in the field to date.

Figure Four is a plot of the various measured crystal quality parameters versus relative growth rate. The crystals used to make these measurements were grown under essentially the same growth conditions with the exception of growth rate and annealing procedure. The annealing procedure can be eliminated as a variable in these quality parameter measurements as annealing has been proven under separate programs to have little or no effect on the items measured. Based on items one through four above, the data indicates the following:

1. In Figure Four (a), the bubble count drops as the growth rate is decreased from 4 to 2, at which point the bubble count becomes constant. This indicates that crystals grown at a relative growth rate of 2 have been grown slow enough for the majority of bubble forming impurities to vaporize.

2. In Figure Four (b), a plot of the average lineage value for each crystal lot studied, shows increasing average lineage values with decreasing growth rate. The data indicates the significance of increased growth times.

3. In Figure Four (b), the minimum lineage value decreases as growth rate decreases. For this data to be conclusive, it would be desirable to have a significantly larger number of crystal samples than were grown for this contract. In addition, it would be desirable to have crystals grown at rates slower than 1 and faster than 4. This curve does, however, follow an expected pattern as per note three above.

4. In Figure Four (c), the Tyndall scatter is lower in crystals grown at a relative growth rate of 2. The cause of this scatter in ruby is not well known and further work in this area is justified in order to understand the cause and effect of this important quality parameter.

5. In Figure Four (d), the chromia banding increases with decreasing growth rate. This curve is the shape expected based on past experience.

Assuming crystal quality can be graded by the summation of the relative values used to plot the curves in Figure Four, the evaluation of crystal lots III through VI is as follows:

<u>Lot No.</u>	<u>Relative Growth Rate</u>	<u>Evaluation No.</u>
IV	2	5
VI	1	7
III	3	7.3
V	4	8

Using this same system of judging crystal quality, a "perfect" crystal would have an evaluation number of zero and a "poor" quality crystal would have a value of 45.

Fluxing Agents

The general purpose of fluxing agents is to remove impurities, increase the solubility of impurity ions in the solid state, and/or change the growth characteristics of the crystal to improve the resultant internal quality. Based on past experience, three fluxing agents denoted A, B and C, of significantly different characteristics were selected and added to the starting powders in equivalent weight per cent to the chromia. The crystals were grown under conditions similar to the conditions used to produce lot IV crystals. The thermal gradient across the crystal during growth was approximately 50°C and the relative growth rate was between 1 and 2 as required by the fluxing agent used. The powders used to grow the lot VII crystals were prepared by the Linde Company Speedway Laboratories under a separate powder program.

Crystal quality parameters versus fluxing agents are plotted in Figure Five. The average values for lot IV crystals are plotted for comparison. The following general statements concerning the data plotted in Figure Five can be made.

1. Fluxing agents A and B reduced the relative bubble count, whereas agent C caused an apparent increase.

2. Crystals grown with fluxing agents A and C have higher apparent lineage compared to the lot IV average, whereas agent B has

lineage of the same value as the lot IV average. It is unreasonable to draw a final conclusion from these data because of the small number of samples.

3. The Tyndall scatter in all crystals grown with the flux addition is higher than the lot IV average.

4. The chromia banding is decreased in the crystal grown with flux A additive. The chromia banding is severe, however, in crystals grown with fluxes B and C. The reasons for the decrease in chromia banding with the flux A addition is not known and justifies some further consideration.

Rating of the crystals of lot VII in the same manner as used to compare lot III through lot VI, the following values apply:

<u>Lot No.</u>	<u>Flux</u>	<u>Evaluation No.</u>
IV	None	5
VII	A	5.5
VII	B	6.7
VII	C	8.5

Special Powders:

Crystals grown with powder prepared under a separate program at the Linde Company Speedway Laboratories have been included in each of lot IV, V and VI. In addition all powder lots used for lot VII were of this type. The decision to include this special powder as a part of this program is based on the results of related efforts whereby lineage has been shown to be significantly reduced in crystals grown with this powder as compared to crystals grown with conventional powder. Conventional powder will be used for crystal lots VIII and IX (orientation studies) because it is necessary to compare the properties of these crystal lots to lots IV, V and VI in which only four crystals of 25 were grown with this special powder.

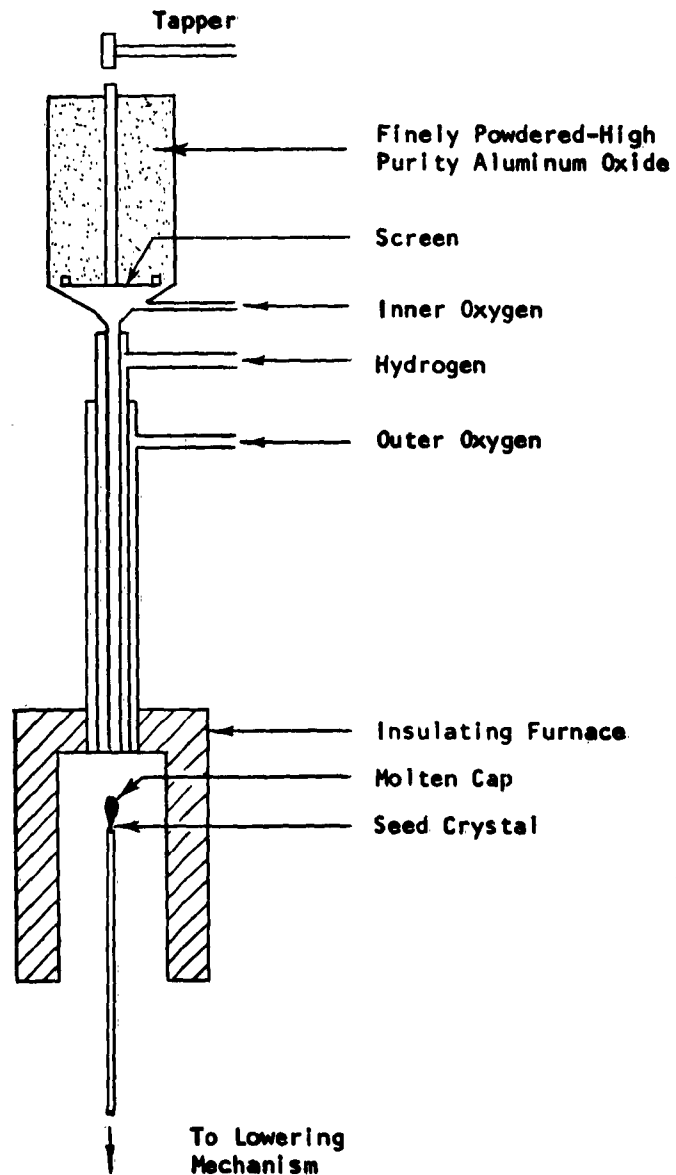


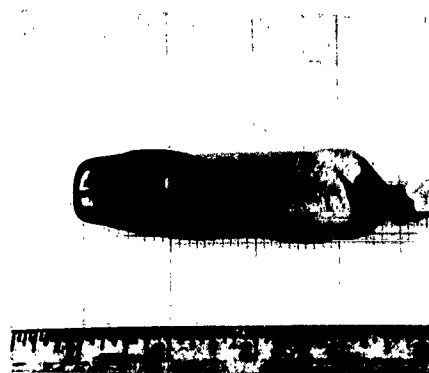
Fig. 1 Growing Sapphire by the Verneuil Technique

PHOTOGRAPHS OF CRYSTALS SHIPPED FOR LOT VI

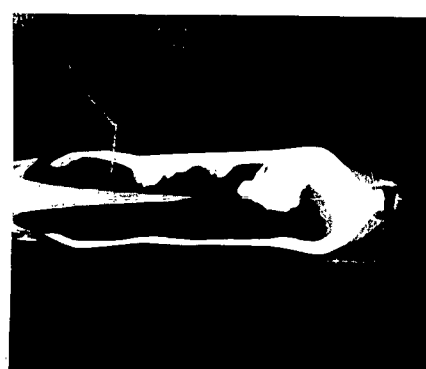
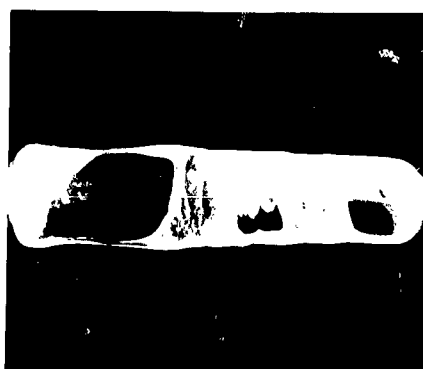
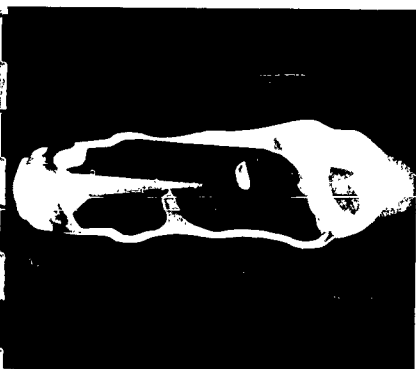
Crystal CP-134-16

Crystal CP-134-24

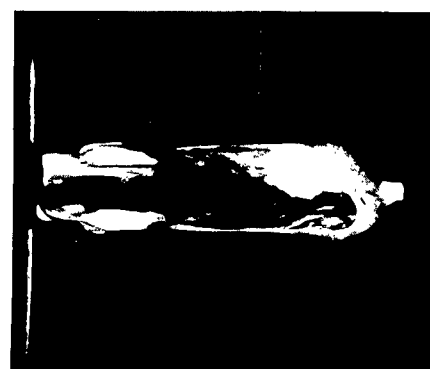
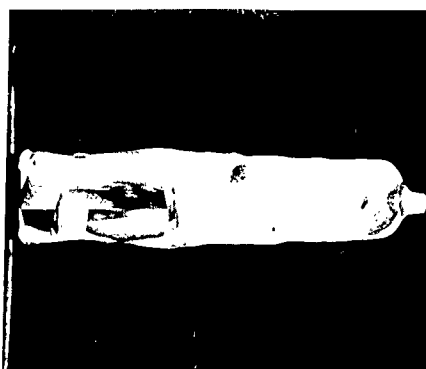
Crystal CP-134-28



A. As Grown Crystal



B. Tyndall

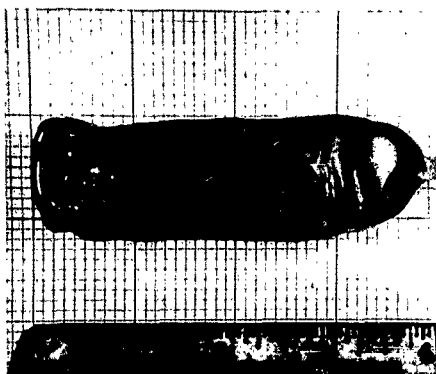


C. Crossed Polaroid

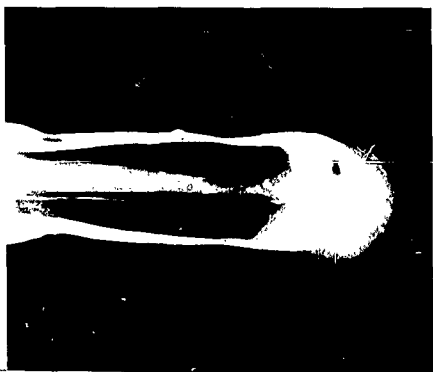
FIGURE TWO (A)

PHOTOGRAPHS OF CRYSTALS SHIPPED FOR LOT VI (Con't.)

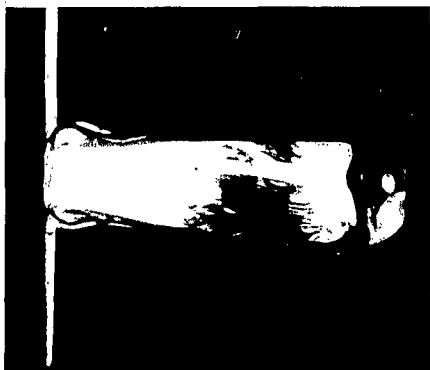
Crystal CP-134-48



A. As Grown Crystal

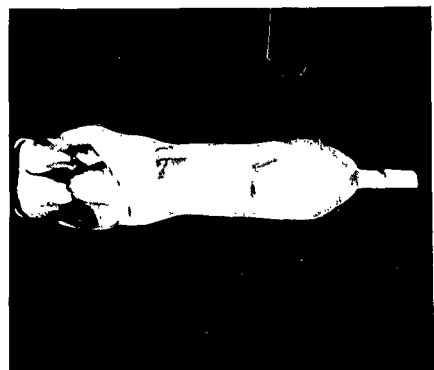
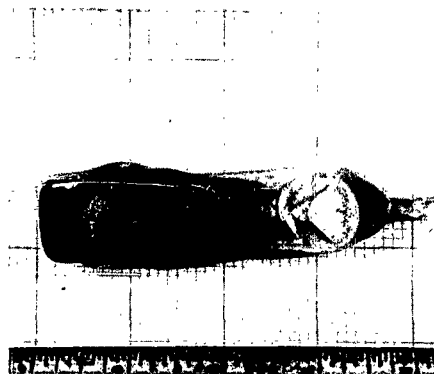


B. Tyndall



C. Crossed Polaroid

Crystal CP-134-52

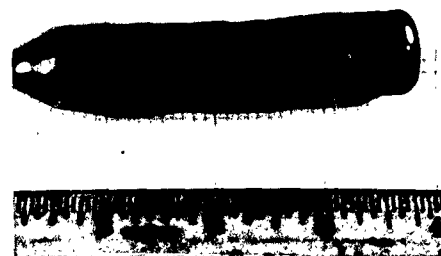
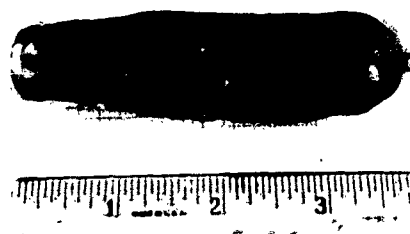
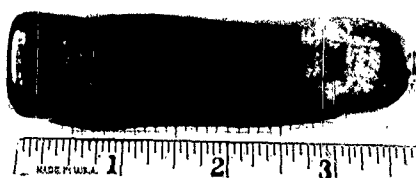


PHOTOGRAPHS OF CRYSTALS SHIPPED FOR LOT VII

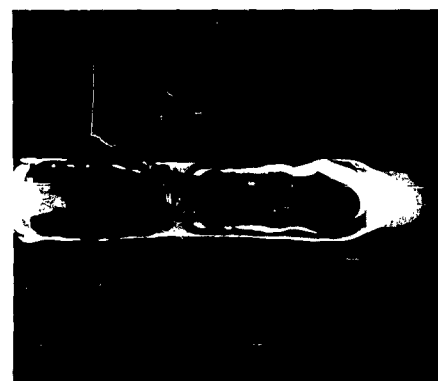
Crystal 1716-14

Crystal 1716-22

Crystal 1716-42



A. As Grown Crystal



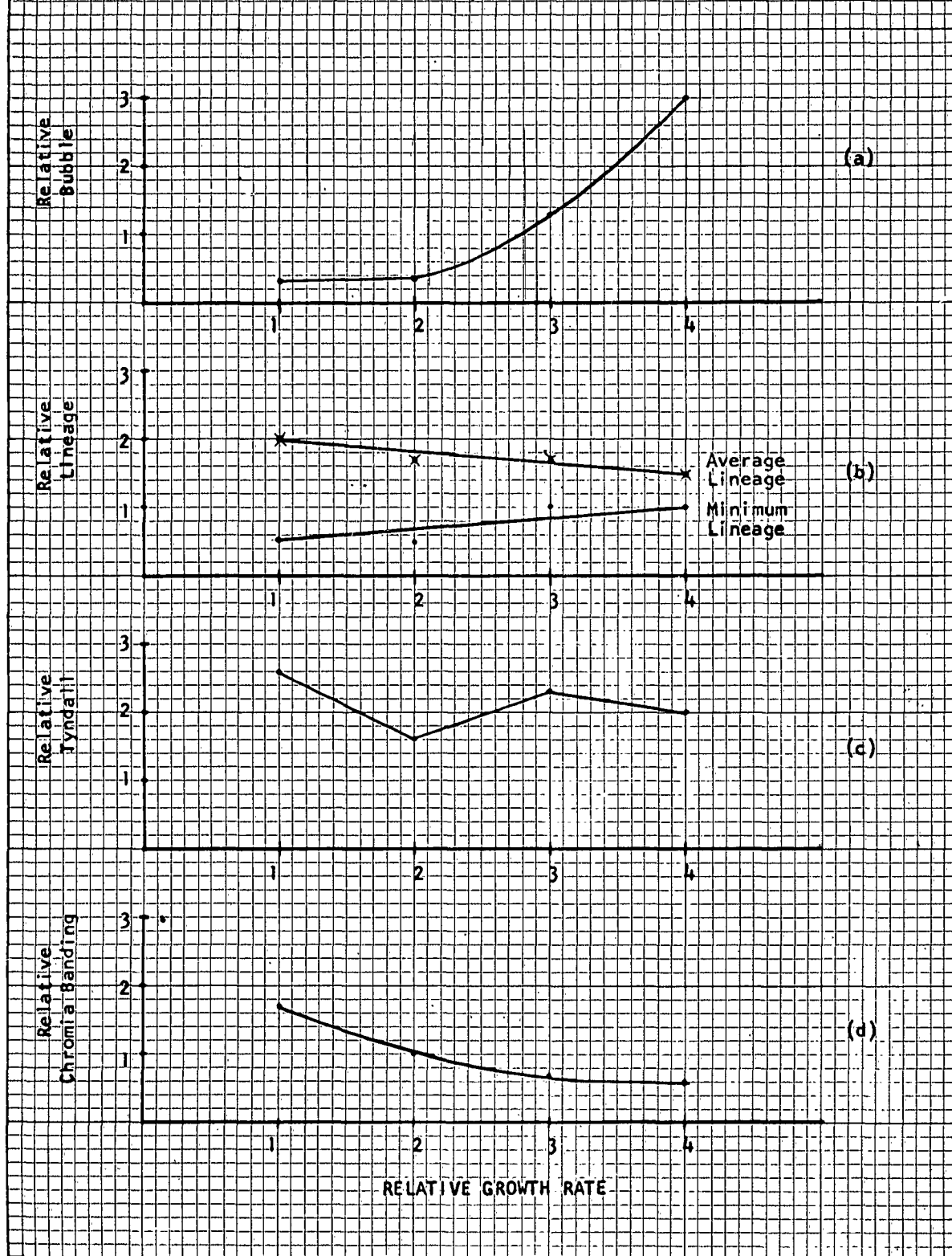
B. Tyndall



C. Crossed Polaroid

CRYSTAL QUALITY PARAMETERS VS. GROWTH RATE

Material: Lots III, IV, V and VI
Grown per U.S. Army Contract #DA-36-039-SCB9089



CRYSTAL QUALITY AS EFFECTED BY FLUXING AGENTS

Material: Lot VII and Lot IV
Grown per U.S. Army Contract #DA-36-039-SC-89089

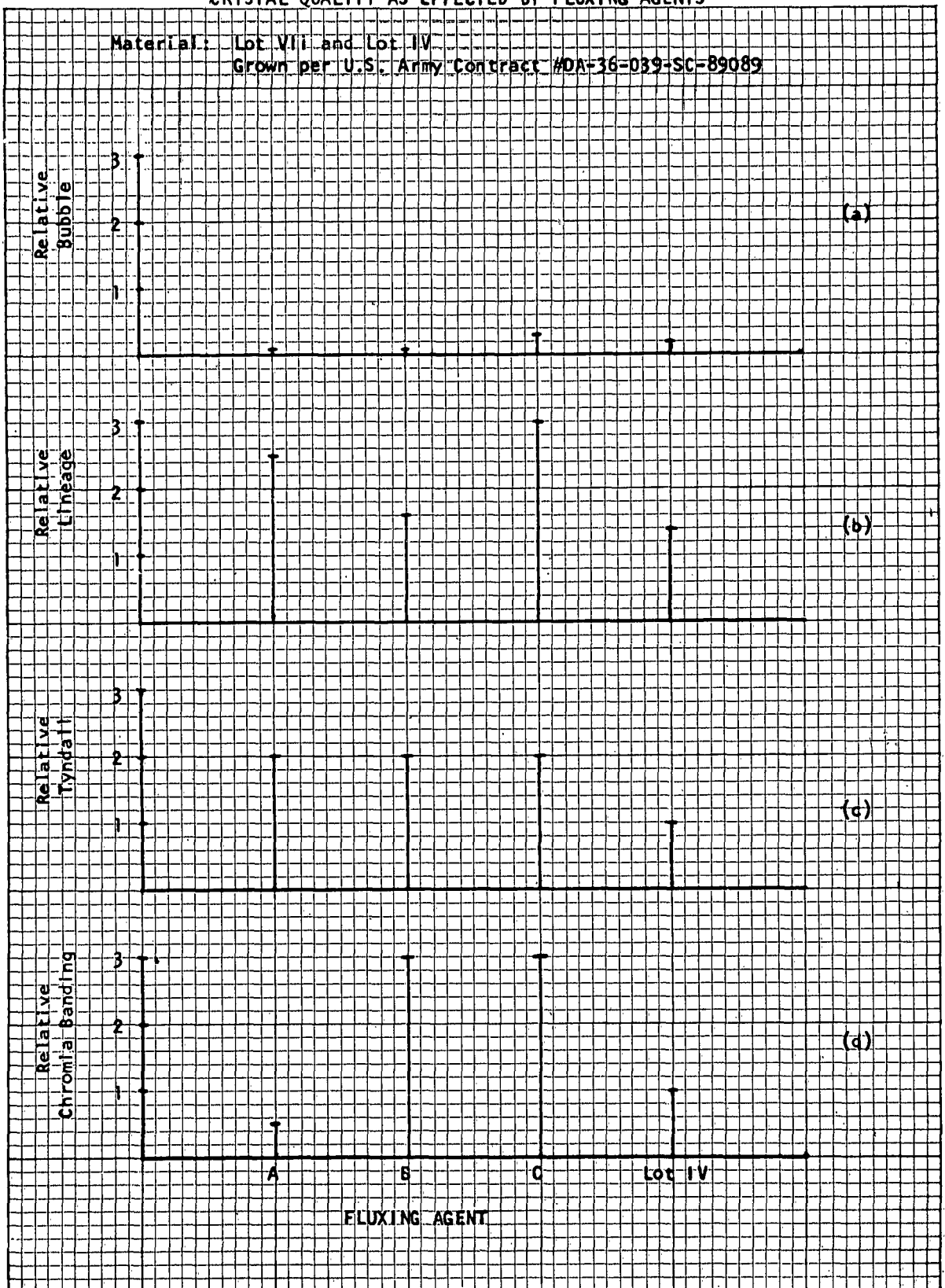


FIGURE FIVE

CONCLUSIONS

The following conclusions can be made as to the crystals shipped under this contract to date.

1. Crystals grown with the 50°C (lot III) thermal gradient have superior internal quality to the crystals grown with thermal gradients of 150°C (lot II) and 300°C (lot I).

2. Crystals grown with a relative growth rate of 2 (lot IV) have superior internal quality compared to crystals grown with relative growth rates of 1 (lot VI), 3 (lot IV) and 4 (lot V).

3. No evaluation of annealing cycles can be made until laser evaluation data is available.

4. Seeds used to grow crystals of high internal quality must exhibit no lineage boundaries as shown by x-ray mapping.

5. Fluxing agents of the types tested do not improve crystal quality as compared to crystals grown for lot IV.

PROGRAM FOR NEXT INTERVAL

The program for the fourth quarter (1 Feb 63 through 30 Apr 63) includes the growth of boule lots VIII and IX to study the effect of orientations other than 90° and the growth of boule lot X under the best concluded parameters to complete the program.

PERSONNEL

B.N. Callihan 25 hours

R.L. Hutcheson 126 hours

AD Linde Co. Div. Union Carbide Corp., East Chicago, Indiana RUBY IMPROVEMENT FOR LASERS-TASK I by R.L. Hutcheson, Report #3 for 1 Nov 62 to 31 Jan 63, 18p incl.illus. tables (Rept. Nr. 3-3rd quarterly report) (Contract DA 36-039 SC 89089) Unclassified Report The effects of growth rate and the addition of fluxing agents to starting powders on the growth of ruby are discussed. Growth data on two lots of ruby crystals are presented.	UNCLASSIFIED 1. Synthetic Ruby 2. Corundum 3. Aluminum Oxide 4. Sapphire-Synthesis I. Title: Lasers II. Hutcheson, R.L. III. Army Research and Development Ft. Monmouth, N.J. IV. Contract DA-36- 039-SC 89089 UNCLASSIFIED
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Union Carbide

3rd Quarterly Report
1 Nov 62 - 31 Jan 63

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