CZOCHRALSKI RUBY

Annual Technical Summary Report

Period: January 1, 1966 - December 31, 1966

March 20, 1967

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I. INTRODUCTION

The object of this program has been to develop the Czochralski growth technique to yield large ruby crystals with optical quality suitable for use as solid state lasers.

The effort during this past period has been divided into two parts. The first part has been devoted towards a study of the material properties of the ruby with special reference to its behavior during active lasing. This aspect of the work is important as it gives a clearer understanding of the problems involved and allows a more definite effort to be made towards their solution through effective changes in the growth conditions.

The second part describes our efforts in scaling up the present growth process to produce large ruby crystals.

II. RESULTS

A. Material Properties of Ruby Laser Crystals

As part of a continuing program to improve the quality and performance of our ruby laser crystals, two main problems are currently receiving attention. These are: (1) chromium distribution in the crystalline alumina lattice and the inhomogeneities in optical quality associated with localized changes in composition, and (2) the identification and removal of particulate inclusions which are a limiting factor in the power obtainable from a Q-switched laser system. Current research effort in both these areas is described below.

(1) Chromium Distribution

(a) Average Compositional Variations

In this area we have confined our attention to variations in the chrome content along the length of the crystal as well as across the radius. Both of these factors depend on keeping the effective distribution coefficient as close to unity as possible throughout the complete growth cycle; this in turn is a function of interface shape and growth conditions.

In the past we have had considerable success in maintaining a coefficient close to unity; however, further experimentation has shown the sampling procedure used to be somewhat misleading and a systematic variation does occur in the larger diameter ruby boules. Figure 1 shows the percentage variation in chromium content as a function of crystal length for a number of ruby samples. This variation had gone undetected due to the practice of sampling as-grown crystals at the top and bottom only; as indicated in the previous report, these two values were identical and thus it had been assumed that no longitudinal variation existed. This variation must be due to changes in the thermal conditions during crystal growth as it is directly related to the power input from the RF generator.

To maintain the crystal diameter, a balance is required between the heat flow to the growth interface plus the latent heat of solidification, and the heat

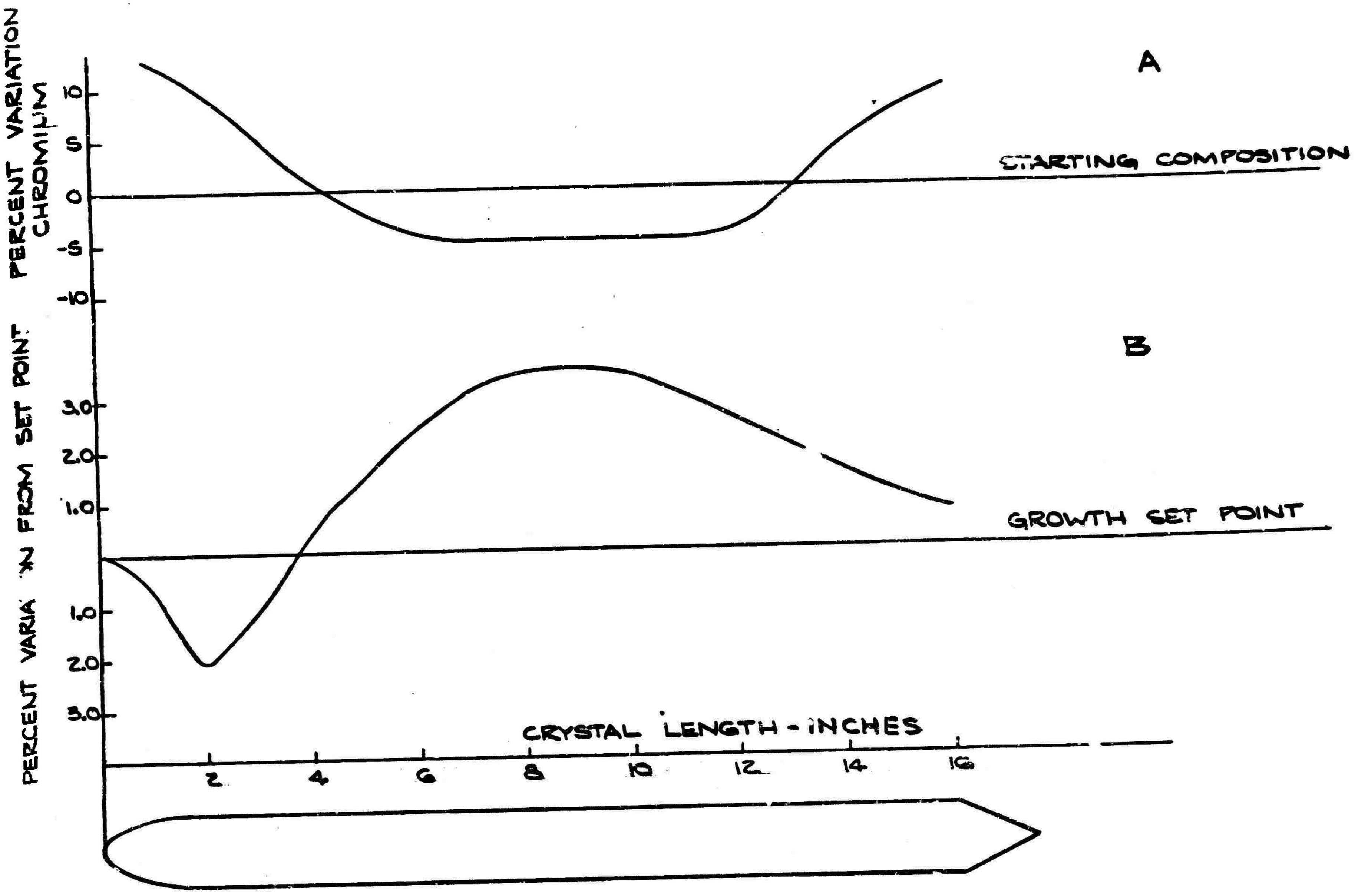

FIG. 1 (A) CHROMIUM CONCENTRATION AND (B) GENERATOR POWER PLOTTED AS A FUNCTION OF CRYSTAL LENGTH

removed by radiation through the solid. At the early stages of growth when the top of the crystal emerges from the furnace insulation, a continuous increase in power to the melt is required. At a critical length, which is a function of crucible and insulation geometry and crystal diameter, the power requirements level off and are subsequently reduced; this point occurs when a substantial amount of the melt has been used up and the rate of heat removal through the crystal is influenced by the absorption of radiant energy from the hot crucible walls.

An explanation for the observed relationship between the heat flow variations and chromium content in the solid has not been established. However, it is known that the effective distribution coefficient can be changed through variations in both the growth rate and thickness of the diffusion layer along the interface; both of these parameters are influenced by thermal conditions during growth.

Radial chromium variations exist to varying degrees depending on where along the length of the crystal the measurement is made. At the top and bottom where the effective distribution coefficient is significantly greater than one the center portion of the crystal has a lower chromium content than the edge. In the middle of the crystal where the effective distribution coefficient is very close to one, the radial chromium gradient is extremely low.

The above observations stress most clearly the need for an effective distribution coefficient of unity throughout the entire growth cycle to eliminate both longitudinal and radial chrome variations. This could best be achieved through manipulation of the appropriate growth parameters, and future experimental work will be carried out along these lines.

(b) Localized Compositional Variations

The core represents an inhomogeneous structure extending throughout the length of the crystal which is associated with the formation of a facet on the interface.(1) This effect is not unique to ruby and is encountered in almost

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(1) F. R. Charvat, J. C. Smith, and O. H. Nestor, Characteristics of Large Ruby Crystals, Proceedings of an International Conference on Crystal Growth, Boston, U. S. A., ed. H. S. Peiser, 45-50, June 1966.

all melt-grown crystals, notable examples being doped Ge and Si (2)(3), and Nd^{3+} doped YAG.

There is no clearly defined reason for this phenomenon, but generally it can be said that a stable nucleus forms at the interface with an effective distribution coefficient which differs from the average value across the remainder of the interface. The orientation of this stable nucleus is normally a low angle crystallographic plane which in the case of ruby is $\{10\bar{1}1\}$ or $\{0001\}$.

There is a very definite relationship between both the size and distribution of the core and the growth orientation of the crystal. A series of experiments have been carried out with crystals containing a nominal 0.05 wt % Cr_2O_3 grown under identical conditions and using different seed orientation. The facets on the interface were examined closely and their size and shape related to schlieren photographs taken of polished windows cut from the top and bottom of the boule. The boules themselves were also examined and will subsequently be fabricated into laser rods to examine the effect of the core on active lasing.

Examination of crystals grown using seeds of 60° orientation showed that the facets on the interface were positioned corresponding to the "r" planes closest to the growth direction. One "r" plane normal approximately 27° away from the boule axis gave a facet at the tip, whereas as the other normal at approximately 67° to the growth interface produced two relatively large facets; one of these was close to the tip with the other midway along the interface. A schlieren photograph of a polished section of the boule adjacent to the interface showed three distinct cores corresponding to the facets. The core for the facet with normal closest to the growth direction was the most distinct; this core could be seen running through the length of the boule section after flats had been polished on its side. Both the

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(2) J. A. M. Dikhoff, Cross-sectional Resistivity Variations in Germanium Single Crystals, Solid State Electronics 1, 202-210 (1960).

(3) J. A. M. Dikhoff, Inhomogeneities in Doped Germanium and Silicon Crystals, Philips Technical Review, 25, No. 8, 195-206, 1963/64.

facets on the interface and corresponding schlieren photographs of the adjacent boule sample are shown in Figure 2. Cores corresponding to the facets lying almost tangential to growth interface were not so clearly defined and were unable to be detected from a schlieren taken along the boule length.

A white sapphire boule was grown with the same orientation, and identical facets were observed on the interface. A schlieren photograph of the adjacent boule section showed only a faint core corresponding to the low angle facet at the tip.

The schlieren technique allows a measure to be made of any change in refractive index across a localized region in the crystal. The index variations can arise from a number of different sources, the most important being solute concentration, misorientations, residual stress, and temperature changes(4). The core in ruby is more pronounced than in white sapphire for crystals grown with identical seed orientation; this indicates clearly the importance of changes in chromium concentration. With white sapphire it is possible that the refractive index changes are caused by residual impurities having a different effective distribution coefficient across the facet. This is consistent with work carried out on undoped InSb(5) where a core was visible with an impurity concentration of approximately 10^{14} atoms/cm^3.

To investigate further the core structure, four ruby boules were grown and are shown in Figure 3. The shape control was good with the average diameter constant down the length of the boule; slight rippling did occur which was attributable to incorrect controller settings. Inspection ends with a six micron diamond finish were fabricated on to the boules and schlieren photographs taken to reveal the shape and size of the core.

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(4) O. H. Nestor, Refractive Index Variations of Ruby; Beam Divergence and Interferometry, Speedway Research Laboratory Miscellaneous Note No. 37, June 1964.

(5) J. B. Mullin and K. F. Hulme, Orientation-Dependent Distribution Coefficients in Melt Grown InSb Crystals, J. Phys. Chem. Solids, 17, Nos. 1/2, 1-6 (1960).

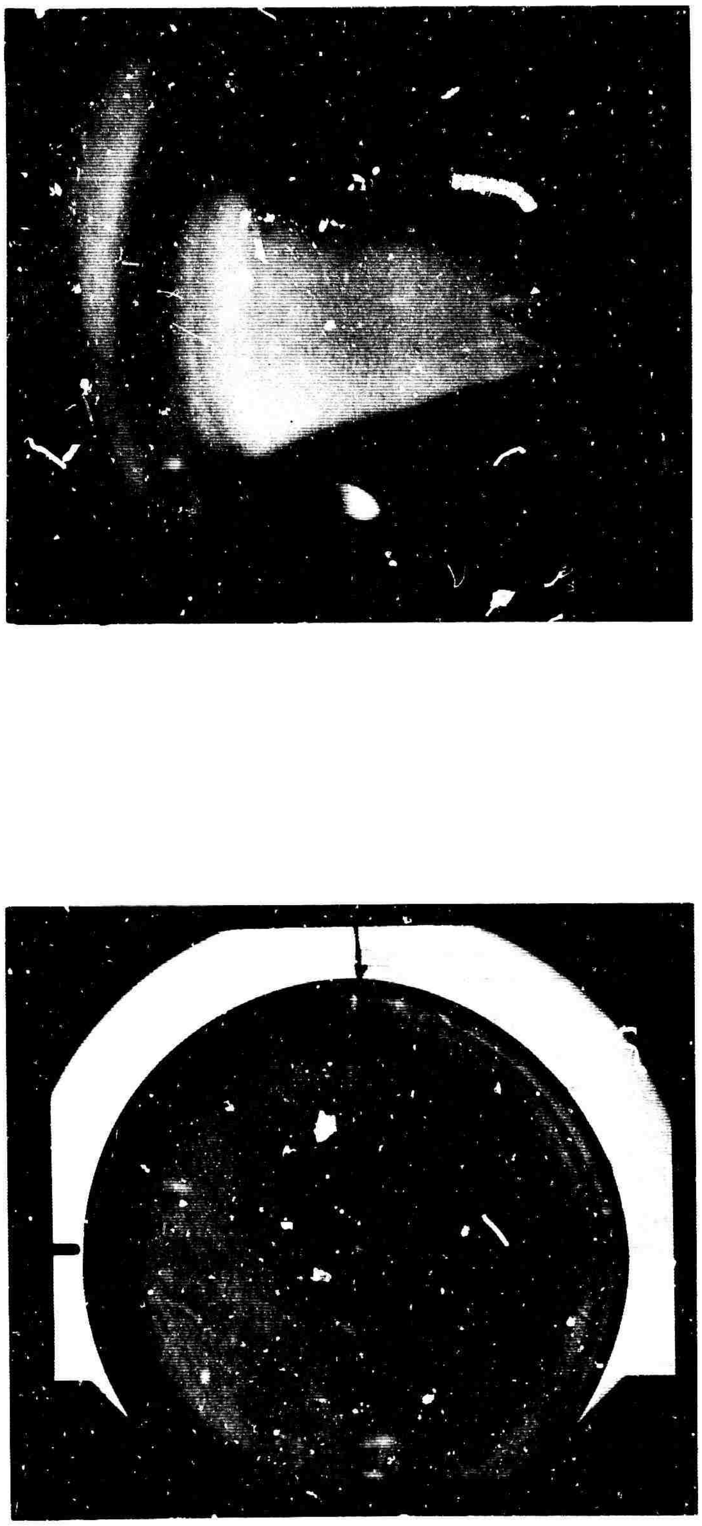


Figure 2. Showing correlation between the facet on the growth interface and the corresponding schlieren photograph.

Figure 3. "As grown" ruby boules with inspection ends.

A set of photographs were taken with the normal orientation technique of lining up the light beam normal to the polished end. These are shown in Figure 4, 6 and 8 for $\vec{E}$ parallel and normal to the plane containing the C axis (in all photographs this plane is vertical). (Results are given for three boules only as the fourth gave no additional information.)

The same rods were then rotated about both the horizontal and vertical position in order to minimize the area of the core; the corresponding schlieren photographs are reproduced in Figures 5, 7, and 9 with $\vec{E}$ parallel to the plane containing the C axis in all cases. Figure 10 indicates the variation in core size and shape which can be obtained with small deliberate changes in the orientation along the horizontal and vertical direction away from that shown in Figure 9. The identification number and orientation of the boules are given in Table I.

TABLE I

Crystal No.	Seed No.	r_1	r_2	r_3	c	a
2500-12-6	103	7			57	46
2500-37-18	152	37-1/2	64	64	60-1/2	32-1/2
2500-10-5	102	27	65-1/2	75	61-1/2	27-1/2
2500-33-16	102	27	65-1/2	75	61-1/2	27-1/2

The above findings point to some important conclusions concerning the core in ruby. With the same seed and under identical growth conditions, it is possible to change the apparent shape and size of the core through slight misorientations in the polished boule face relative to the original growth direction. Consideration of a boule grown from a seed of sixty degree orientation shows the central core to be about 0.3 mm diameter after corrected alignment; this core corresponds to faceting on the "r" plane with normal 27° to the growth direction.

Previous schlieren photographs taken of a sample with flats polished on the side indicate that this core remains straight and of constant diameter down the length of the boule. The result is to be expected because of the low angle

$\vec{E}$ parallel to C.

$\vec{E}$ perpendicular to C.

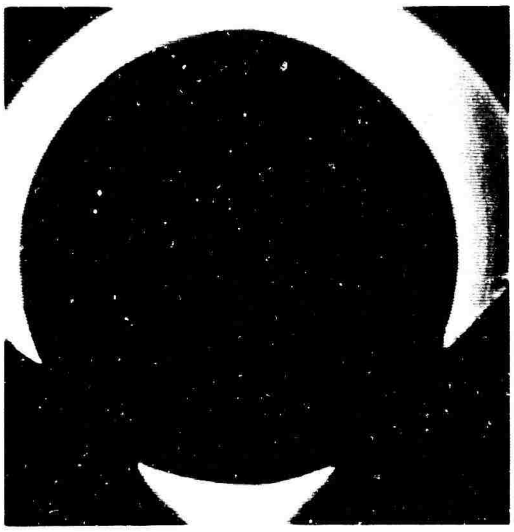
Figure 4. Boule No. 2500-12-6 - schlieren taken with light beam normal to inspection face.

Figure 5. Boule No. 2500-12-6 - orientation chosen to eliminate core area.

Figure 6. Boule No. 2500-33-16 - schlieren taken with light beam normal to inspection face.

Figure 7. Boule No. 2500-33-16 - orientation chosen to minimize core area.

$\vec{E}$ parallel to C.



$\vec{E}$ perpendicular to C.

Figure 8. Boule No. 2500-10-5 - schlieren taken with light beam normal to inspection face.

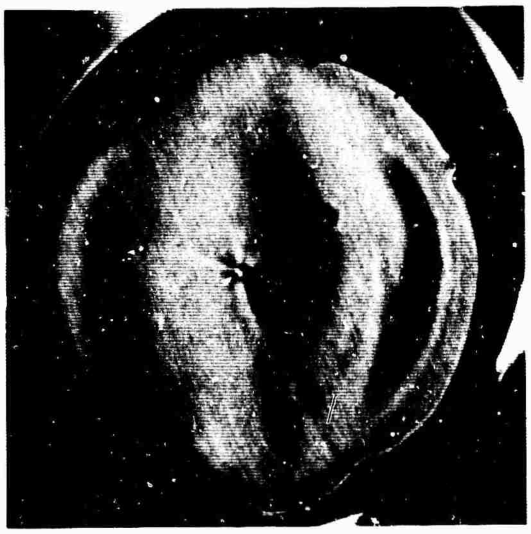


Figure 9. Boule No. 2500-10-5 - orientation chosen to minimize core area.

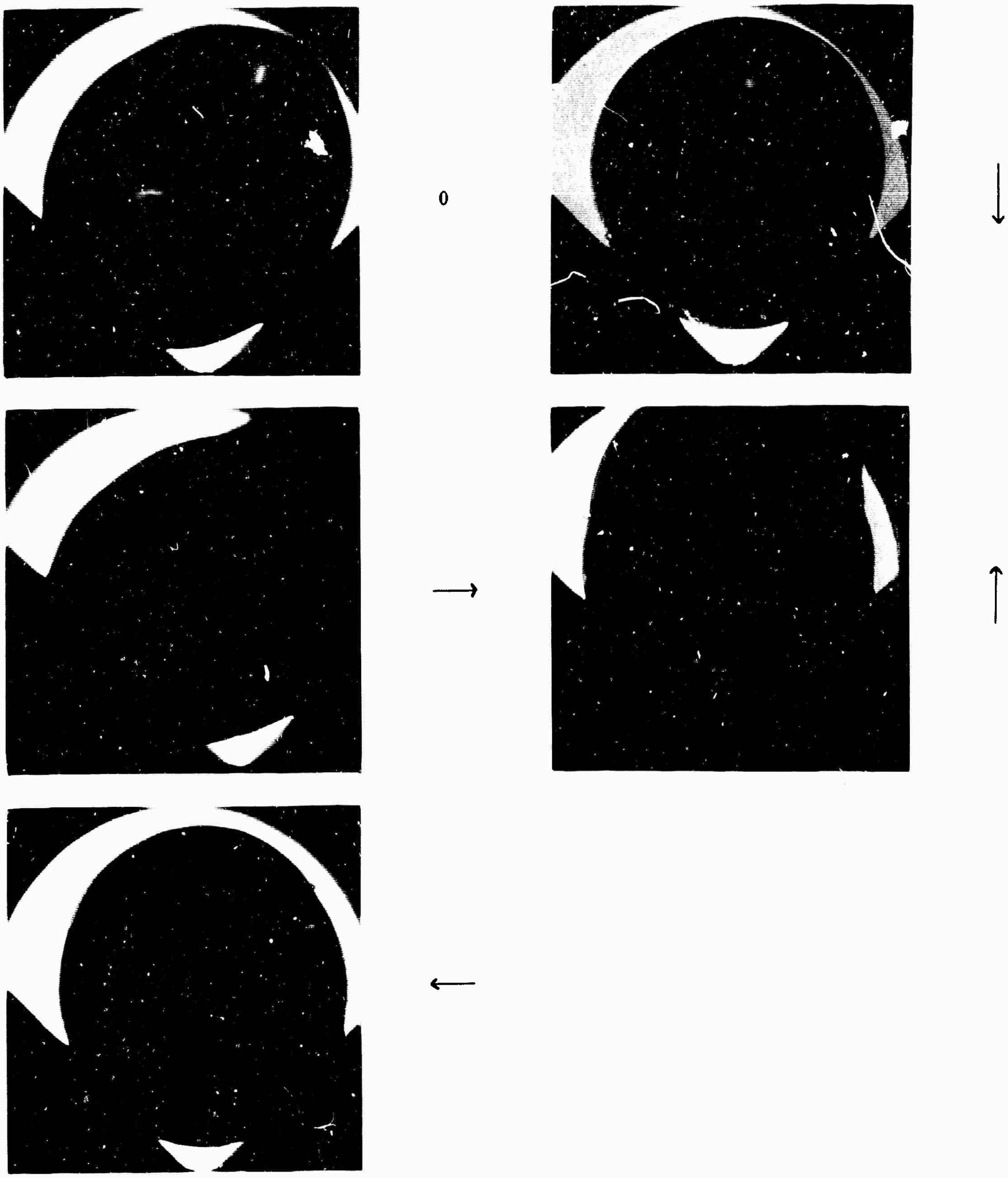


Figure 10. No. 2500-10-5 - Schlieren photograph showing the effect of horizontal and vertical movement of the boule on core shape and size.

conical interface found in ruby crystal, where all "r" plane facets with normals to the growth direction between approximately sixty degrees and zero are confined to the tip.

To separate the central cores on a schlieren photograph would require a relative movement between the top and bottom of the rod of about 0.5 mm; with a six-inch long rod, this represents an angle of approximately 10 minutes. It is suggested then that more consistency can be achieved over both the size and shape of the core if the laser rods are fabricated with faces normal to the growth direction to within an accuracy 10 minutes.

With our "sixty degree" orientation seed crystal, a second "r" plane occurs with a normal at 67° from the growth axis. Under present growth conditions, this plane is almost tangential to the growth interface and two facets are formed, one coming away from the tip and the other half way up the interface. The large angle cores are not so distinct as those confined to the center; however, because of their much larger relative size slight misorientations relative to the growth axis have a much more pronounced effect on the apparent core size and shape.

It has been shown that changes in the melt temperature can effectively change the conical angle of the growth interface. Because of the steep angle encountered with ruby and sapphire crystals, a relatively small change in temperature is sufficient to move the facet from the side to the tip of the interface when the angle between the "r" plane normal and the growth direction is around 65°. The temperature fluctuation is not enough to affect the quality of the boule but would most likely be reflected as a small change in the "as grown" diameter.

It can be shown that a change in seed orientation such as to move the angle between the "r_2" normal and the growth direction to a value greater than 70° will effectively remove the facet (and corresponding core) tangential to the growth interface. Using a seed of orientation such that growth direction is along the "r" plane normal would do this most effectively. A complication arises, however, due to the fact that there is a tendency for a crystal growth with this orientation to become elliptical and produce an elliptical shaped core; this can clearly be seen in Figure 3.

In addition to retaining a concentric boule throughout the growth cycle, it is also important that the crystal grows straight. The effect of not keeping the boule straight can be seen by examination of Figures 7 and 9. Boule No. 2500-33-16 was not perfectly straight and it was impossible to manipulate the orientation to produce one well defined core; this should be compared with Boule No. 2500-10-5 which has been grown from the same seed but was much straighter down its length.

This preliminary study has shown that the size and shape of the core in a fabricated laser rod is dependent on three factors:

(i) The growth orientation

(ii) The straightness and concentricity during growth

(iii) Misorientations during fabrication

The effect of growth orientations is becoming clear and work will proceed in order to reduce the core size. The use and better understanding of the shape monitoring device has in recent months improved both the straightness and concentricity of the as-grown boules. The improvement is such that contributions to the apparent core size and shape during the actual growth are relatively small compared to that introduced by small misorientations during fabrication. The effect of these misorientations could be minimized by lining up the rods, using the schlieren technique. to give minimum core size and fabricating the ends normal to the laser light beam.

In an attempt to determine the variation in chromium content across the core measurements have been made on polished windows using both the electron probe and spectrographic absorption techniques.

The electron probe work has not yet been completed and will be reported at a later date. Preliminary work using an optical absorption technique has indicated an increase in chromium content in the vicinity of the core. Figure 11 shows the result of scanning across the window using light polarized with $\vec{E}$ normal

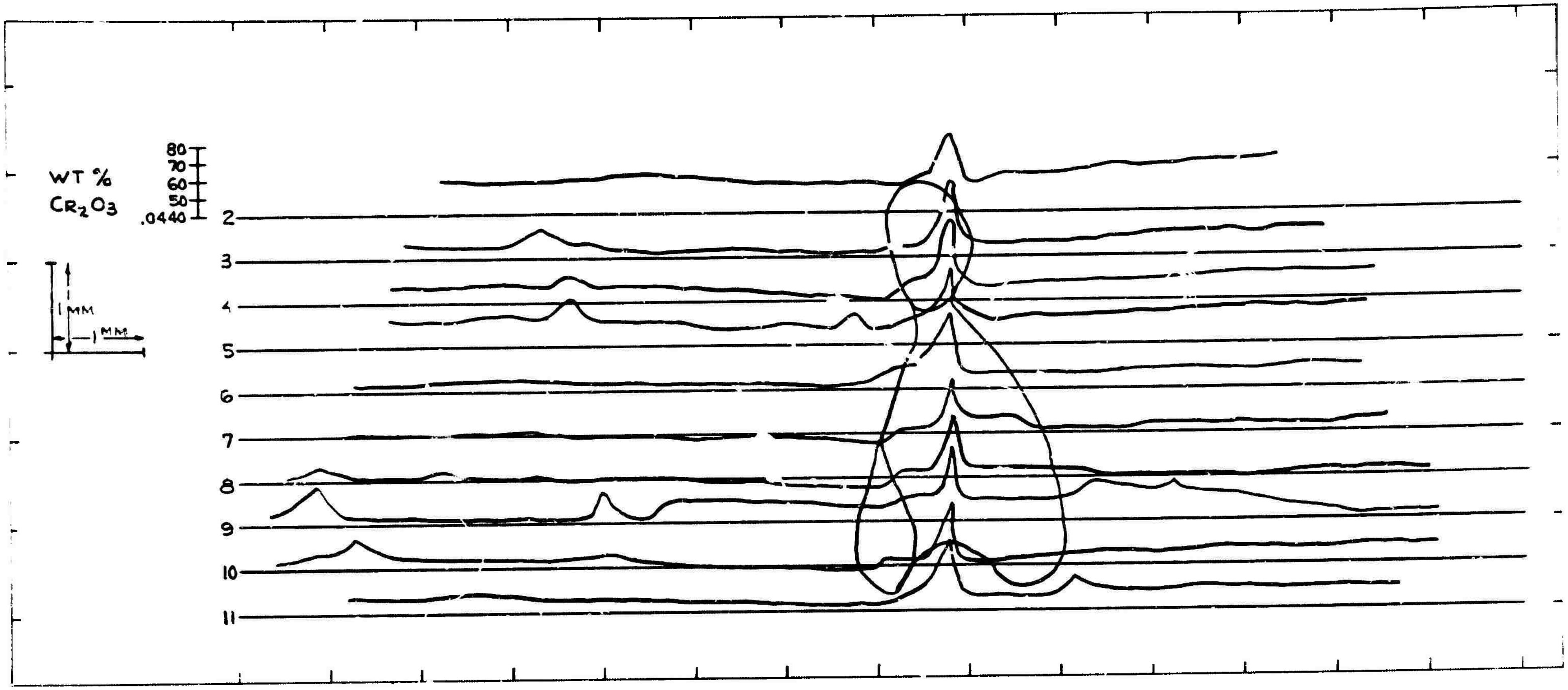


FIG. 11 OPTICAL ABSORPTION SCAN ACROSS A RUBY WINDOW TO DETERMINE THE CHROMIUM CONCENTRATION IN THE VICINITY OF THE CORE.

to the C axis. The scan was made along the direction shown on a schlieren photograph of the window in Figure 12. The base line for each scan shown in Figure 11 was taken as 0.0440 wt % Cr_2O_3, and the shape of the core shown on the diagram was copied directly from the schlieren photograph; the larger peaks are due to reference scratches on the surface.

It is possible to see a correlation between the core and a corresponding localized rise in chromium concentration. The increase is only about 3% and care has to be taken in proper alignment of the instrument as well as obtaining the optimum window thickness and surface finish. It is hoped that further refinement of the technique will lead to more conclusive results from future work.

In addition to finding out information concerning the origin and physical characteristics of the core, it is also important to know what is its influence on the active lasing properties of the ruby rod. Preliminary examination of the problem has shown that a possible association can exist between the core and an uneven energy distribution in the near field lasing pattern; it is also possible to correlate the schlieren patterns obtained under both active and passive testing.

To further this effort, a series of laser rods are presently being fabricated for testing in the laboratory's recently acquired Korad K2 Laser Head. These contain a number of different core structures obtained by changing the seed orientation and the corresponding facet formation at the interface; a comparison will be made between these rods and two others which have been selectively cut from a larger diameter ruby to be core free.

Figure 12. Schlieren of window used in optical absorption work; scan was made horizontally along the lines shown.

(2) Internal Laser Damage

The peak power obtainable from Q-switched laser systems is limited at this time chiefly by damage attributable to particulate inclusions. In the case of glass, it has been recognized for some time that platinum inclusions introduced during the melting process are at fault. It has been demonstrated that crucible material (iridium) inclusions led to damage in Czochralski rubies grown in the present program.(6) We have examined some of the Verneuil rubies damaged in laser studies by Avizonis and Farrington(7) and there too found evidence presented below that damage was related to inclusions.

The problem with platinum in glass and iridium in ruby is assumed to be that these inclusions absorb pump and laser radiation and thereby cause damage to the matrix by several possible routes ranging from expansion to fusion and perhaps vaporization of the inclusion, or perhaps by thermal stressing of the matrix. We have made calculations on various aspects of the heating of inclusions - specifically iridium - in an effort to clarify the damage mechanism, to point up more precisely the type of control against inclusions needed in the growth process and to indicate what experimental observations in damage testing may be fruitful. These calculations are summarized in Section (b) below, following a presentation of damage threshold data, including Czochralski ruby results, in Section (a).

(a) Damage Threshold Data

The damage threshold data of Avizonis and Farrington(7) are represented in Figure 13 by the circled points showing experimental scatter. The

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(6) "Czochralski Ruby", Report No. SRCR-66-2 by F. R. Charvat, O. H. Nestor, and J. C. Smith, Union Carbide Corporation, Linde Division, Speedway Laboratories, January 26, 1966. (Semi-annual Technical Summary Report, Contract NONr-4132(00).

(7) P. V. Avizonis and T. Farrington, Internal Self-Damage of Ruby and Nd-Glass Lasers, Appl. Phys. Letters 7 (8), 205 (15 October 1965).

curves drawn through these points are not those of Avizonis and Farrington. Rather, the curves drawn here - particularly that for ruby - point up a new summary of the results, viz. that the damage threshold is energy-limited at the shortest pulse lengths, but power-limited at longer (> 50 nanoseconds). How far the power limit may extend is clearly not defined. It presumably does not extend into the millisecond pulse length regime with the same power limits indicated in Figure 13 (300 megawatts/cm^2 for ruby and 120 megawatts/cm^2 for glass) for then the damage thresholds at 1 milliseconds would be 300 Kilojoules/cm^2 and 120 Kilojoules/cm^2, respectively, for ruby and glass. The latter is ca. 600x greater than the value reported by Avizonis and Farrington.

Figure 13 shows two other points - one for a Czochralski ruby designated by an open square and the other for a Verneuil ruby denoted by a full square - representing the results of other, more recent tests at Kirtland AFB$^{(8)}$ conducted to update the Czochralski-Verneuil comparison. Like the other points, these represent the onset of damage at the output end of amplifier rods. The pulse length assignment is nominal. The discrepancy between the new Verneuil rod and the others defining the full curve is not resolvable except in speculation. The new Verneuil rod and the Czochralski rod were remarkably similar in damage behavior. Each was damaged also at its input end in a manner similar to that shown in Figure 1 of Reference (6). That type of damage has been assumed to be a focusing - rather than an inclusion - induced effect, because the damage location is highly reproducible and the form of damage rather unique.

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(8) We gratefully acknowledge the cooperation of the Effects Branch of the AF Weapons Laboratory, Kirtland AFB, New Mexico, in testing rods for damage and in particular the helpful efforts of Dr. P. V. Avizonis, Capt. K. C. Jungling, and Sgt. W. Willoughby.

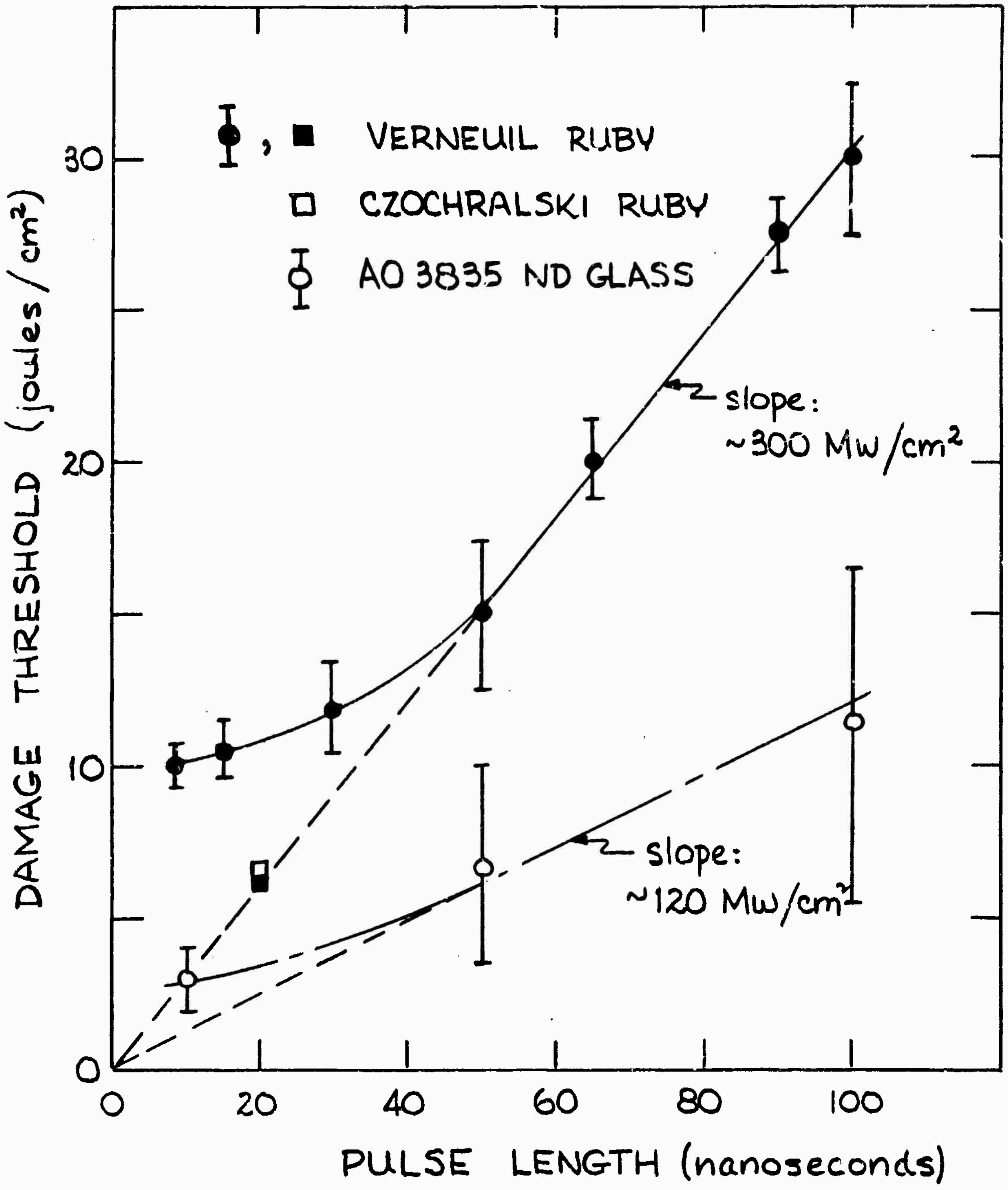

Figure 13. Energy Threshold for Laser Internal Damage vs Pulse Length

Avizonis and Farrington have noted that there was no damage observable due to pump light (u-v filtered out) even with pump energies higher than normally used. (7)

We have examined some of the Verneuil rubies damaged in their work. We succeeded in splitting open one of the rubies at and along a damage center "plane". X-ray studies identified this as an r-plane (10 · 1) and an electron microprobe survey of the damage zone disclosed a multiplicity of particles, each containing nickel, iron and chromium and ranging in size up to about 40 microns. (9) These were comparable in size to opaque inclusions spotted in other damage zones in the same crystal. The stainless steel "debris" indicated by the electron probe studies is assumed to have been in metallic form inasmuch as it appeared to be related to inclusions that are opaque and since it was entrapped during growth under reducing conditions.

(b) Heating of Inclusions

The data of Figure 13 invite interpretation as follows:

1. The energy threshold limit as pulse length approaches zero reflects what is needed to produce damage when inclusions do not lose thermal energy to their environment. The energy threshold for Czochralski ruby is shown below to be sufficient to bring iridium inclusions, as identified thus far, to melting, if re-radiation from the inclusion and conduction cooling are neglected. Re-radiation is in fact entirely negligible. It is assumed that conduction cooling is negligible in the short times involved.

2. The energy threshold increases with pulse length as a result of energy exchange from inclusion to the matrix. The exchange mechanism,

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(9) We are indebted to Mr. A. M. Hawley of our Laboratories for the X-ray work and to Dr. W. D. Forgeng and his staff members. Mrs. Gloria M. Faulring, and Mr. E. S. Malizie, of the Union Carbide Mining and Metals Division, Technology Department, Niagara Falls, New York, for the electron probe work.

if thermal, must be conduction. On this assumption, it is shown that temperatures of ca. 6000°K may be generated at the inclusion site during a laser pulse. But it also follows that the thermal gradients calculated are so excessive as to make the classical treatment of conduction suspect.

The purpose of this section is to describe the calculations supporting the above comments. The calculations are made with specific reference to iridium inclusions, since something of their size, shape and orientation is known. Iridium inclusions are taken to be platelets, as reported in Reference (6). Only laser radiation is included as a heat source, in light of the Avizonis and Farrington finding that the pump source produces no damage.

Symbols to be used are defined as follows:

s = platelet thickness, cm
ℓ = platelet breadth, cm
a = broad area of platelet cm^2
A = total surface area of platelet, cm^2
θ = angle between platelet normal and laser beam
ρ = density, gm/cm^3
C = specific heat, joules/gm-°K
e_λ = spectral emissivity at laser
e_T = total emissivity
T = temperature, °K
E = laser pulse energy "density", $joules/cm^2$
τ = laser pulse length, seconds
σ = Stefan-Boltzman constant

$= 5.67 \times 10^{-12}$ $watts/cm^2\text{-}deg^4$

t = time, seconds

The subscript "$_o$" will be used to denote the matrix.

(i) Energy Threshold Limit

It will be shown that the damage threshold given in Figure 13 for Czochralski ruby is of the order needed to heat iridium inclusions to melting. The heat energy needed to melt iridium is 1.2 x 10^4 joules per cm^3 including the heat of fusion (see Table 2 below). Then the energy needed for a platelet of broad area $\mathcal{a}$ and thickness s is $1.2 \times 10^4 \mathcal{a}$ s. The energy absorbed by the platelet from a beam of energy flux density E is $e_\lambda \mathcal{a} \cos\theta$ E. The flux density E_m needed to produce melting is then given by

$$e_\lambda \mathcal{a} \cos\theta \, E_m = 1.2 \times 10^4 \, \mathcal{a} \, s$$

or

$$E_m = \frac{1.2 \times 10^4 \, s}{e_\lambda \cos\theta} \tag{1}$$

For iridium, $e_\lambda = 0.3$ at the Cr R-line in ruby.(10) The platelets are presumed to have been pulled into the melt by the convection currents therein. It is assumed that the platelet then adheres to the growth interface and hence is oriented in the ruby according to the geometry of the interface. The latter is typically conical with a cone angle of 45° included. Thus, $\theta \sim 67°$ and $\cos\theta \sim 0.4$. Then

$$E_m = 10^5 s. \tag{2}$$

In Reference (6), it was noted that iridium inclusions were of the order of 10 microns (= 10^{-3} cm) across, on the average, and that the breadth to width ratio was roughly $\ell/s \sim 20$. Hence, $s \sim .5$ microns = 5×10^{-5} cm and

$$E_m \sim 5 \text{ joules/cm}^2$$

This is comparable to the damage threshold value for Czochralski ruby given in Figure 13.

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(10) AIP Handbook, Table 6k-5, 2nd Ed. (McGraw-Hill Book Company, Inc.).

It should be clear that too many factors - such as laser beam intensity distribution, inclusion sizes, and the like - are not well enough defined to allow pinpointing the damage criterion to the actual melting of iridium inclusions or to heating them to the melting point of sapphire or any other specific point at this time. But it does appear that significant heating of inclusions is necessary to produce damage.

It is assumed that a similar result applies rather generally, i.e., to Verneuil ruby and its inclusions and to platinum in glass, in the range of laser operating conditions considered here. It is of interest to compare the various metallic inclusions as to the energy needed to bring an inclusion of given volume to melting. This is done in Table 2 where the energy needed to heat an inclusion to the melting point of sapphire (2310°K is also listed).

TABLE 2

Heat Content ΔH above 273°K(11)

		ΔH (joules/cm^3)		
Element	M. P. (°K)	To Melting	Melted	To 2310°K
Nickel	1730	7250	9900	13100
Iron	1800	7850	9900	12300
Chromium	2160	9070	11300	12200
Platinum	2030	5860	8300	>8300
Iridium	2450	9200	12100 *	7320
Tungsten	3640	----	-----	6100

It is apparent that iridium is more or less preferred over transition metal inclusions depending on whether melting of the inclusions is more important than approaching the melting point of the matrix. In either case the factor of difference would be no more than 2x. Table 2 also shows platinum at a disadvantage by virtue of low heat requirement; and, if the lower melting point of glass as opposed to sapphire makes itself felt, then it is clear that damage thresholds should be lower for Pt in glass.

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(11) Calculated from data compiled by K. K. Kelley. Bulletin 371, Bureau of Mines, Dept. of Commerce, and from AIP Handbook data where denoted by * .

All these attempts to compare different materials are based on equal sizes of inclusions. In an experimental comparison the size, shape, and orientation of inclusions must be taken into account. Inasmuch as these factors are generally unknown for laser materials and in particular those of Figure 13, close correlation of threshold data with the kind of calculation given above cannot be made at this time.

Eq. (1) points up the fact that the thickness of inclusions is the important size parameter determining damage threshold (similarly, for spherical inclusions, $E_m \sim r$, the inclusion radius.) The first damage would occur to the thinnest (smallest) inclusions and as laser radiation increases in intensity progressively larger inclusions are damaged. This has several implications:

(1) Growth procedures that simply reduce the size of inclusions are ill-founded.

(2) Damage threshold differences between individual rods are a matter not only of the number density of inclusions but also of the size range in the manner noted above.

An apparent increase in the size of damage sites with increased laser output may prove on closer inspection to be the formation of new, larger damage sites.

(ii) Heat Losses from Inclusions

In the above discussion heat losses from the inclusion have been neglected. We will now consider these for the case of iridium in ruby.

The rate at which an inclusion re-radiates energy is given by $C_T \sigma A(T^4 - T_o^4)$. The ratio of this to the average heating rate $e_\lambda\, a \cos\theta\, E/\tau$ is:

$$R = \frac{A}{a \cos\theta} \cdot \frac{e_T}{e_\lambda} \cdot \frac{\sigma(T^4 - T_o^4)}{E/\tau} \qquad (3)$$

(For spherical inclusions the ratio $A/a \cos\theta$ is replaced by $A/a = 2$.) For platelets, $A/a \sim 2$, and, in the case of iridium in Czochralski ruby, $\cos\theta \sim 0.4$, so that $A/a \cos\theta \sim 5$. With $e_\lambda \sim 0.3$, $E/\tau = 300\ \mathrm{Mw/cm^2} = 3 \times 10^8\ \mathrm{watts/cm^2}$ (Figure 13) and neglecting T_o^4 relative to T^4:

$$R \sim 5 \times 10^{-8}\, e_T \sigma T^4$$

R equals unity for a black body ($e_T = 1$) at T = 42000°K! Hence, $R \ll 1$ typically.

At a nominal value of $E/\tau = 10^4\ \mathrm{watts/cm^2}$ and with $e_T = 0.3$ (as is the case for a variety of metals at $T \sim 2500$°K),

$$R \sim 3 \times 10^{-15} T^4$$

At the melting point of iridium ($T \sim 2.7 \times 10^3$ °K), $R \sim 1/6$. Thus even in this low laser power case, radiation cooling is quite negligible.

The only thermal cooling mechanism that can be of any consequence is conduction in the ruby material. What we have considered here is a simplified problem in which the inclusion is neglected except as it serves the intermediary function of making the laser energy it absorbs available to the ruby. Specifically, the energy is absorbed at rate

$$I = e_\lambda\, a \cos\theta\, E/\tau \qquad (4)$$

and is delivered to the matrix over area a. Considering a one-dimensional heat flow treatment to be justified by the short pulse times encountered, the temperature at distance X from the inclusion plane is given by (12):

$$T(x, t) = \frac{I}{a} \frac{\sqrt{a_o t}}{\mathcal{K}_o} \left[\frac{e^{-u^2}}{\sqrt{\pi}} - u \operatorname{erfc} u \right] \qquad (5)$$

where $u = X/(2\sqrt{a_o t})$, a_o = thermal diffusivity of sapphire = $\mathcal{K}_o/(\rho_o c_o)$, and $\mathcal{K}_o$, ρ_o, c_o are the thermal conductivity, density and specific heat of sapphire, respectively. This solution assumes temperature independent thermal properties of the matrix, which is not valid, but here it is used for simplicity and will be compensated for in part by assuming average values. Using now Eq. (4) and $e_\lambda = 0.3$, $\cos \Theta = 0.4$, $\rho_o = 4$, the temperature at $x = 0$ (the "surface", or plane of iridium - sapphire contact) is

$$T(o, t) = 0.034 \frac{E}{\tau} \sqrt{\frac{t}{c_o \mathcal{K}_o}}$$

The median value of the product $c_o \mathcal{K}_o$ over the range 273-2000°K is about 0.2 cgs; this will be used below. The "surface" temperature at the end of the pulse time $t = \tau$ for average laser power of $E/T = 3 \times 10^8$ watt/cm² is then

$$T(o, \tau) \sim 2 \times 10^7 \sqrt{\tau}$$

For = 70 nanosec = 7×10^{-8} sec, in the middle of the range covered in Figure 13,

$$T(o, \tau) \sim 6000°K$$

in excess of the boiling point of sapphire. This result is not taken literally, but again is indicative of the need for generating high temperatures to produce damage.

The above treatment of the conduction problem gives some cause for concern inasmuch as the implied temperature gradient at the "surface" is approximately 10^8 °K/cm, or 10°K per unit hexagonal cell (6 Al_2O_3 molecules) of sapphire. This is so large as to virtually violate the temperature concept, making the classical heat conduction treatment high tenuous.

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(12) H. S. Carslaw and J. C. Jaeger "Conduction of Heat in Solids", Clarendon Press, Oxford (1959), 2nd Ed., p. 262-3.

In summary, the above calculations support the assumption that inclusion heating via laser beam absorption is in fact involved in the ruby damage mechanism, and they suggest that heating to temperatures as high as, or higher than, the melting point of the inclusion, or of sapphire, is characteristic. They further point up the fact that damage sets in sooner - i.e. thresholds are lowered - as inclusion size decreases.

Recent experimental data (13) has further indicated the importance of the particle size on laser damage. A 1/4-inch diameter by 3-inch long ruby laser rod evaluated to have a considerable number of inclusions was lased and examined for damage after successive testing at an input energy of 125 joules.

A correlation was attempted between the observed damage sites and inclusions in the crystal which had been carefully mapped out prior to testing. Similar to what had been reported previously(6), the number of damage sites far exceeded the number of visible inclusions; no damage, however, was observed at any of the previously spotted inclusions.

Examination of polished windows cut from ruby boules under dark and bright field illumination on a Zeiss Universal Microscope has shown a large variation in particle size. A small number of inclusions are in the size range between 10-30 microns with the majority less than 10 microns. In the lasing experiment described above, the crystal was examined under low power in order to be able to scan over the entire length. Under these circumstances, only the larger inclusions would be visible; the majority of inclusions which were smaller and more likely to be damaged remained undetected during the initial examination.

No correlation was possible between theory and experiment as no measurements were made on the actual particle size; it is also possible that some particles below the level of detection for the microscopic technique used were still too large to contribute to laser damage at these input energy levels.

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(13) We are indebted to L. R Rothrock of our Laboratory for carrying out these lasing experiments.

(3) Large Ruby Growth

A furnace has been constructed to house a 5,000 cc iridium crucible. The basic design of the furnace is similar to that used in the growth of smaller diameter boules with necessary modifications to allow for the larger heat capacity of the system and the increased weight of the crucible plus charge load. Considerable plastic flow occurs in iridium at operating temperatures, and the problem of providing the crucible with adequate support to prevent sagging of the sides and bottom had to be investigated closely. Also requiring attention was an investigation into correct combination of coil design and furnace insulation in order to come up with the most efficient heating conditions; this was done by coupling directly into the empty crucible and measuring the temperature as a function of coil diameter, number of turns, and thickness of furnace insulation. This work was necessary because the power level of the available generator was slightly low and care had to be taken to avoid drawing plate current in excess of the rated maximum. This information having been accumulated, a melt was established and a series of experiments carried out to establish the conditions for forming a suitable temperature distribution from which a crystal could be seeded.

The first growth was carried out under conditions which were far from ideal and the crystalline quality was poor. However, the size was correct and a considerable amount of valuable experience was gained from the exercise; the crystal is shown in Figure 14.

The main problem associated with this initial growth was in temperature control where matching was not achieved between the heat capacity of the system to the correct controller setting. The response of this system was much slower than normally encountered, and growth rate fluctuations arising from excessive temperature cycling of the melt temperature resulted in poor crystalline quality.

After modification of the control system, a second crystal was grown which was much improved both in shape and quality of the resultant boule. However, the improvement in quality created problems in the power requirements for growth. Because of the improved quality, more radiant heat was removed along the length of the crystal and more power was required from the generator in order to maintain growth. The combined effect of running the generator closer to its maximum rated power and an arcing problem at one of the input leads resulted in a shutdown towards the end of the growth cycle. Cracking occurred in the crystal due to the necessity of having to remove it too rapidly from the solidifying melt.

Modification of the RF generator was carried out to raise its operational power level and a third crystal grown. For this experiment, additional shielding was incorporated above the crucible and the crystal cooled over a very much longer period of time to eliminate thermal stressing. Also at this point, enough confidence was felt in both our ability and technique to employ the method routinely used on smaller diameter crystals for the reduction of iridium inclusions(6).

Figure 15 shows the "as grown" boule containing 0.05 wt % Cr_2C_3. The quality is good throughout except where a diameter change led to a drop in growth rate with a resulting nucleation of bubbles at the interface.

An examination of the boules, using the standard technique of shining a well collimated beam of light into the crystal and viewing the scattered radiation, failed to detect any inclusions. It is possible that some inclusions are present and remained undetected because the light intensity was lower than required for so large a boule; it is not expected, however, that the number exceeds that specified as being acceptable for present day ruby laser crystal requirements.

To adequately pump a ruby rod of dimensions 2 inches in diameter and 12 inches long, the chromium concentration must not exceed 0.013 weight percent Cr_2O_3(14); thus the latest large diameter crystal produced

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(14) V. O. Nicolai, private communication.

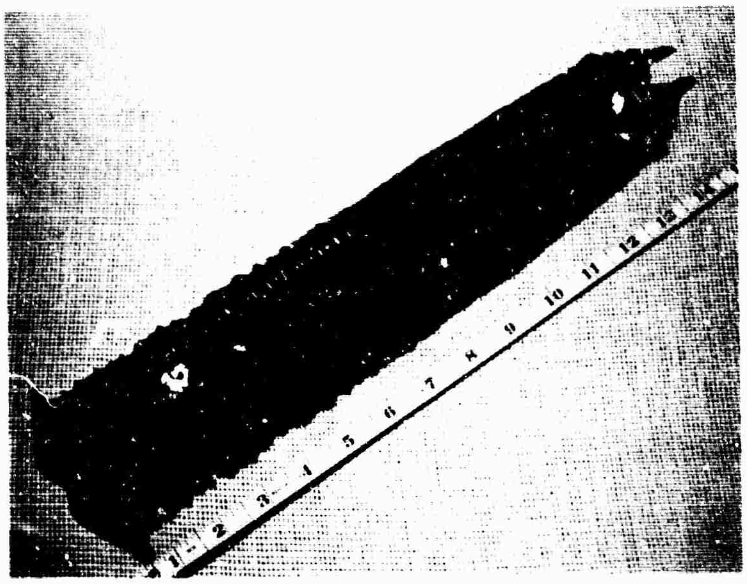

Figure 14. Large diameter ruby boule containing 0.05 wt % Cr_2O_3 - first growth.

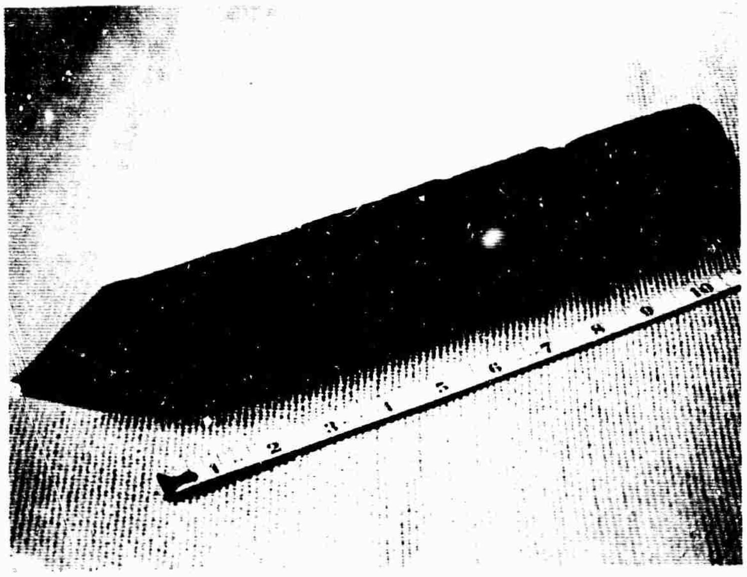

Figure 15. Large diameter ruby boule containing 0.05 wt % Cr_2O_3 - third growth.

under the present contract has been grown specifically with this concentration. This crystal will be fabricated with inspection ends to examine its optical quality.

Having overcome the technical problems of growing so large a single crystal of good quality, future work will be devoted to improving further the optical quality; this in turn will require modification of our present optical evaluation techniques which have been set up to accommodate crystals of somewhat smaller diameter.

III. PLANS FOR NEXT PERIOD

Future plans are for essentially a continuation of our present effort.

The effect of chromium distribution on active lasing will be investigated in more detail. This work will be complemented by an equivalent study of the core origin and how its effect can be minimized or eliminated by suitable refinement of our existing growth process.

Results indicate that the size of the inclusions is an important factor. Both the effect of modifying the growth technique and purification of the starting raw material on the size and distribution of inclusions will be investigated; a parallel study will again be carried out on the effect of these changes on laser damage during active testing.

The optical quality of the large diameter crystal will be evaluated and the necessary modifications made to the growth process in order to achieve the same high standard which is at present obtained with smaller diameter boules.

19 AUG 1966

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13. ABSTRACT

The Czochralski growth technique has ~~been~~ developed to yield large ruby crystals with optical quality suitable for use as solid state lasers. This has involved a scale-up of the present growth process to produce crystals 2 inches in diameter and 12 inches long. The material properties of the ruby have been studied with special attention given to its behavior during active lasing. The properties receiving attention have been chromium distribution and the identification and removal of inclusions causing laser damage during Q-switched operation.

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