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VAPOR PHASE GROWTH OF RUBY MONOCRYSTALS

Semiannual Technical Summary Report

15 July 1966 - 15 January 1967

Philip S. Schaffer - Principal Investigator

Contract No. Nonr-4574(00)-2
Authorization ARPA Order Number - 306

February 1967



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ABSTRACT

Ruby monocrystals were produced by vapor phase growth using chromium organometallic compounds as the chromium dopant ion source. The low decomposition temperature of these compounds prevented high dopant homogeneity and sufficiently long growth runs to be successfully conducted. Growth furnace modifications were made which should enable three inch long by $3/8$ - $1/2$ inch diameter crystals to be grown having their longitudinal axes perpendicular to the impinging reactant gas stream. Other modifications were incorporated into the growth system which should enable continuous runs up to 100 hours duration, elimination of some of the problems of non-uniform dopant homogeneity and secondary nucleation, and further improve crystalline purity.

TABLE OF CONTENTS

	Page No.
ABSTRACT.....	i
TABLE OF CONTENTS.....	iii
LIST OF FIGURES.....	iv
1.0 INTRODUCTION.....	1
2.0 EXPERIMENTAL PROCEDURE.....	3
2.1 Chromium Organometallic Compounds..	3
2.2 System Modification.....	3
3.0 RESULTS AND DISCUSSION.....	5
3.1 Chromium Organometallic Compounds..	5
3.2 Ruby Growth.....	5
3.3 Growth Furnace Chamber.....	8
3.4 Powder Removal.....	10
3.5 Crystalline Purity.....	10
3.6 Pumping System.....	11
4.0 SUMMARY.....	13
5.0 REFERENCES.....	14
ACKNOWLEDGMENTS.....	15
DOCUMENT CONTROL DATA - R&D - FORM DD-1473...	17
DISTRIBUTION LIST.....	19

LIST OF FIGURES

	Page No.
Figure 1: Evaporation Rate of Chromium Hexacarbonyl, $\text{Cr}(\text{CO})_6$, as a Function of Temperature.....	6
Figure 2: Evaporation Rate of Chromium Acetylacetonate, $\text{Cr}(\text{C}_5\text{H}_7\text{O}_2)_3$, as a Function of Temperature.....	6
Figure 3: Schematic Diagram of Modified Ruby Vapor Growth Apparatus.....	9

1.0 INTRODUCTION

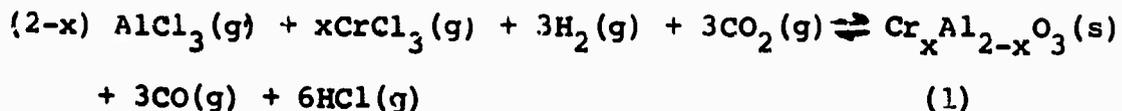
It is the overall objective of this program to develop the vapor phase growth technique to grow large, high perfection ruby monocrystals for laser applications. Under previous phases ¹⁻³ of the program, the feasibility of the technique was determined. Growth apparatus was continuously improved, enabling larger crystals to be grown and providing greater control of parameters during crystal growth. A laser rod was fabricated from an as-grown crystal weighing over 96 grams and optical and laser characteristics were determined.

Optical pumping behavior, light scattering and interferometric properties were measured of the as-grown vapor phase ruby crystal, 0.160 in. diameter x 1 in. long. When compared to Verneuil and Czochralski ruby crystals of the same dimensions, the vapor grown crystal was shown to be of high optical quality.

Although high chromium dopant homogeneity has been attained at low concentration (~100 ppm Cr_2O_3), high uniformity has not yet been achieved at concentration levels around 500 ppm Cr_2O_3 . The goals of the present phase of the program are to produce fabricated laser quality crystals, 3/8 - 1/2 in. diameter x 3 in. long, with improved homogeneity. The best way to achieve these goals appears to be by scaling up the present experimental apparatus and modifying to overcome some of the experimental problems which lead to secondary nucleation and dopant inhomogeneities. A four-inch inside diameter growth chamber will be used compared to a 1 7/8 inch diameter at present. This will enable crystals to be grown with their longitudinal axes perpendicular to the reactant gas stream rather than parallel as they have been grown.

The general chemical reaction still most desirable for

vapor phase ruby growth, although other chromium sources were investigated, is the simultaneously-controlled hydrolysis of aluminum and chromium chlorides, according to the following reaction:



Vapor deposition and epitaxial growth are achieved by flowing the reactant gases over an oriented, isothermally-heated substrate under controlled supersaturation, temperature and pressure. Control of process variables, growth rate, substrate nature and orientation, results in oriented, monocrystalline growth, high purity and low strain.

2.0 EXPERIMENTAL PROCEDURE

2.1 Chromium Organometallic Compounds

Two chromium-containing organometallic compounds, chromium hexacarbonyl ($\text{Cr}[\text{CO}]_6$) and chromium acetylacetonate ($\text{Cr}[\text{C}_5\text{H}_7\text{O}_2]_3$), were investigated as alternative sources of chromium vapor generation as the dopant ion in vapor growth of ruby. These two compounds appeared most desirable compared to other chromium organometallic compounds as far as known physical properties,⁵⁻⁶ purity, availability, and cost were concerned.

A well-stirred water and oil bath for the carbonyl and acetylacetonate, respectively, were used in conjunction with a precision temperature controller, based on the vapor pressure of various liquids, was capable of control to $\pm 0.02^\circ\text{C}$.

Evaporation rates as a function of temperature were determined for each compound using carbon monoxide as a carrier gas. Several different methods of introduction of the reactant vapor species and different injector configurations were utilized to separate and subsequently mix the reactant gases for desired crystal growth. Carbon monoxide, hydrogen and argon were each used as a carrier gas to determine the most appropriate one. Using 1/8 inch sapphire rod substrates, ruby crystals were overgrown to about 1/4 inch diameter.

2.2 System Modification

In the phase of growth system development and modification, a design was arrived at which would permit crystals to be grown with their longitudinal axes perpendicular to the reactant gas stream. These crystals should be grown up to three inches long with increased dopant homogeneity. In this same phase, all of the components subjected to gaseous

chlorine and aluminum chloride contact were coated with Teflon to improve corrosion resistance and to attain higher crystalline purity. An anodized aluminum and a recrystallized alumina injector system were both prepared for evaluation. A molybdenum furnace liner was evaluated which would greatly increase the life of the expensive, nonporous alumina (99.7% Al_2O_3) furnace tubes. Use of a calcium trap was investigated as a possible substitute for liquid nitrogen to prevent corrosive hydrogen chloride, one of the product gases of the vapor growth reaction, from attacking the internal mechanical vacuum pump components, reducing its life. Other methods of attaining reduced pressure and evacuating HCl gas such as a steam ejector and a water aspirator-blower combination were compared with a mechanical vacuum pump as to cost, efficiency and reliability.

In a phase of continued optical and laser evaluation, an appropriate annealing cycle was developed and a previously evaluated as-grown ruby was annealed at 1975°C.

3.0 RESULTS AND DISCUSSION

3.1 Chromium Organometallic Compounds

Chromium hexacarbonyl, $\text{Cr}(\text{CO})_6$, is a white crystalline solid having an extrapolated boiling point of approximately 146°C . Upon heating to elevated temperatures, it decomposes to chromium and carbon monoxide. Chromium acetylacetonate, $\text{Cr}(\text{C}_5\text{H}_7\text{O}_2)_3$, is a reddish-violet crystalline solid having a melting point of 214°C and a boiling point of 340°C . It boils without decomposition at 340°C , giving off a green vapor. It appeared that both of these compounds could be heated to relatively low temperatures to obtain sufficiently high vapor pressure to use as the chromium source in the ruby growth reaction. Thus, using a carrier gas, a controlled vapor pressure of the compound could be generated and introduced into the growth zone.

Evaporation rates of each compound were determined as a function of temperature using carbon monoxide as a carrier gas. These data are shown in Figs. 1 and 2. These results indicated that saturation of the carrier gas was not yet reached.

3.2 Ruby Growth

Various methods of introduction of the vapor species of each organometallic compound and different injector configurations were utilized to separate and subsequently mix the reactant gases prior to reaching the growth zone. Carbon monoxide, hydrogen and argon were each used as a carrier gas to introduce the carbonyl and acetylacetonate vapor.

Ruby monocrystals were vapor-grown up to approximately one-quarter inch in diameter, overgrown on one-eighth inch diameter sapphire rod substrates. These crystals showed identical visual fluorescence behavior as those grown using CrCl_3 vapor as the dopant source. This indicated that the dopant

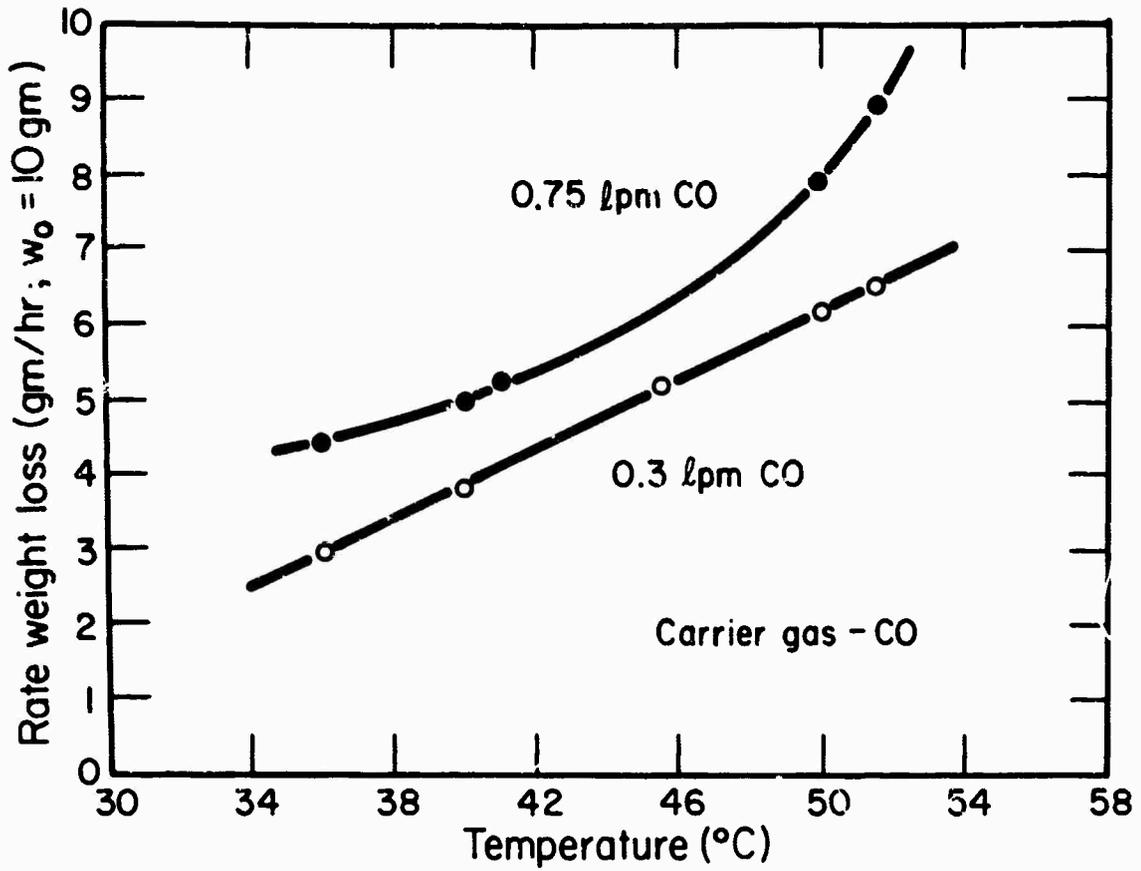


Figure 1: Evaporation Rate of Chromium Hexacarbonyl, $\text{Cr}(\text{CO})_6$, as a Function of Temperature

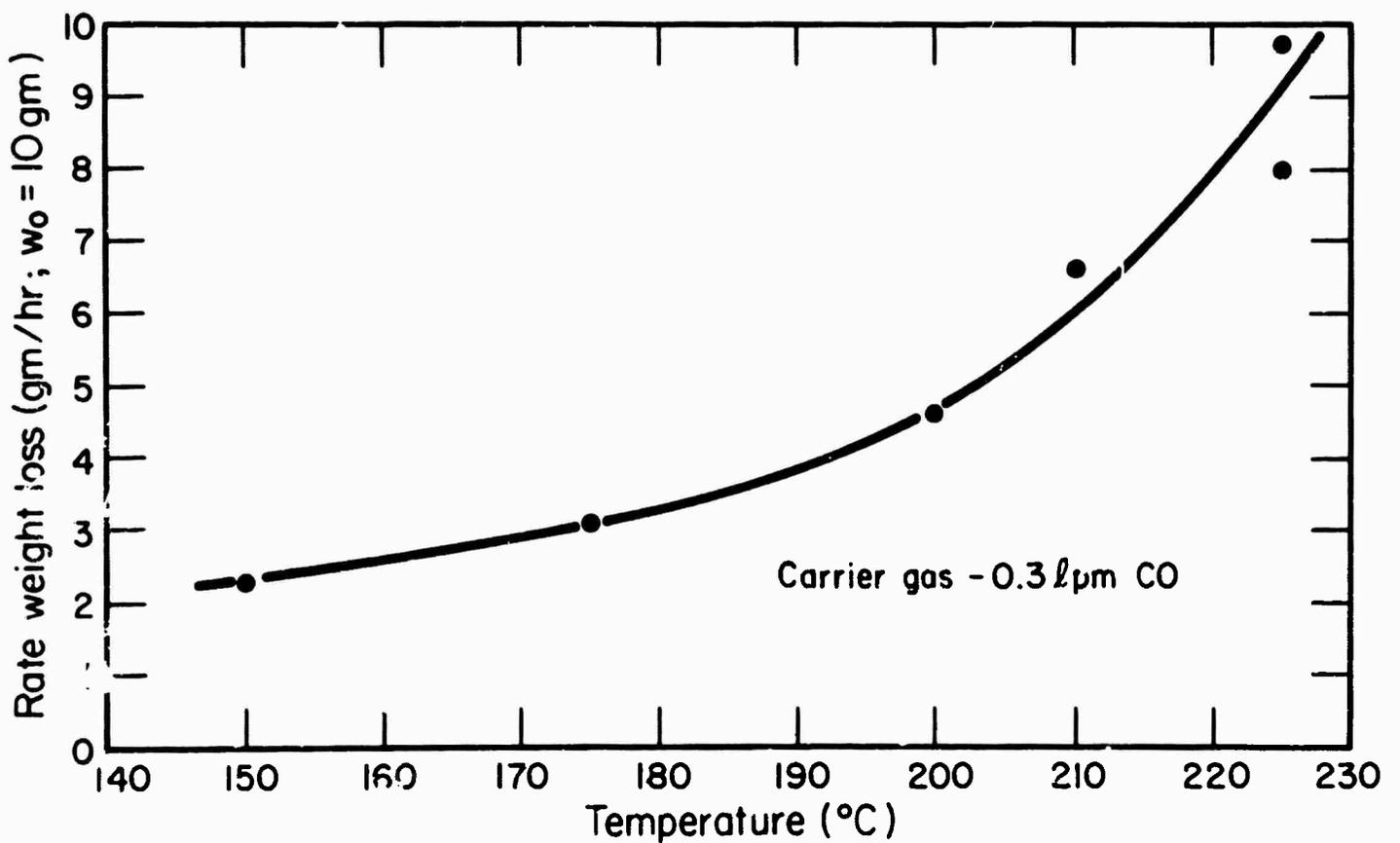


Figure 2: Evaporation Rate of Chromium Acetylacetonate, $\text{Cr}(\text{C}_5\text{H}_7\text{O}_2)_3$, as a Function of Temperature

ion was Cr^{3+} rather than another valence state of chromium. Cr_2O_3 dopant concentrations between approximately 100-500 ppm were attained, and from the aforementioned results, higher levels could have been readily achieved by increasing the vaporization temperature. During actual growth, both chromium organometallic compounds decomposed at relatively low temperatures. Some chromium metal and/or chromium sesquioxide (Cr_2O_3) always deposited either inside or at the exit end of an injector tube or on the walls of the furnace before reaching the substrate. This resulted in non-homogeneous dopant distribution in the crystals and injector constriction with increasing time. The problems of low decomposition temperature could not be suitably overcome and it was decided to concentrate on the chromium trichloride generation method, making further process improvements.

Reactant gas depletion in the growth chamber and non-uniformity of the distribution coefficient for Cr_2O_3 in Al_2O_3 did not permit sufficiently homogeneous Cr_2O_3 dopant concentration to be attained for distances greater than about one inch along the longitudinal axis of the grown crystals. In these growth runs, the longitudinal axis of the substrates was parallel to the impinging reactant gas stream. Several crystals were grown in which longitudinal axes of the substrates were placed perpendicular to the incoming reactant gas stream. Sufficient improvement was obtained in the dopant characteristics of these crystals to become very encouraged by this technique, although secondary nucleation along the forward crystalline faces frequently occurred. Therefore, modifications are presently being made in the growth system which should improve the quality of the grown crystals.

3.3 Growth Furnace Chamber

Desired system modifications are partially completed which should enable three-inch long ruby crystals of improved dopant uniformity to be grown. A schematic diagram of the overall system is shown in Figure 3. It is anticipated that continuous runs will be able to be conducted up to 100 hours duration. A two-zone (one for CrCl_3 generation) molybdenum heating element has been wound around a porous alumina tube inserted into a vacuum chamber.

A molybdenum insert in the form of a cylinder, about twenty inches long, $3 \frac{3}{4}$ inches in diameter by 0.010 inches thick, will be positioned inside the heating-element core. Since the resistance heating method requires that the furnace walls be heated to as high or higher temperature than the substrate, deposition occurs on the furnace walls as well as on the substrate at approximately the same rates. With increasing deposition on the furnace walls, three disadvantages with the previous technique were encountered. One was that the reactant gas velocity increased if temperature, flow rates and total pressure remained constant. The second was that the size of the growing crystal was limited by the inward growing walls and third was that the furnace tube had to be replaced after a given thickness of vapor growth was achieved which added considerably to the cost of the vapor growth process.

Preliminary experimentation with molybdenum liners resulted in slight oxidation and/or volatilization followed by subsequent deposition of molybdenum at the substrate-vapor growth interface, even when heated under a hydrogen atmosphere. Spectrochemical analysis of grown crystals; however, showed no indication of molybdenum. Molybdenum deposition at this

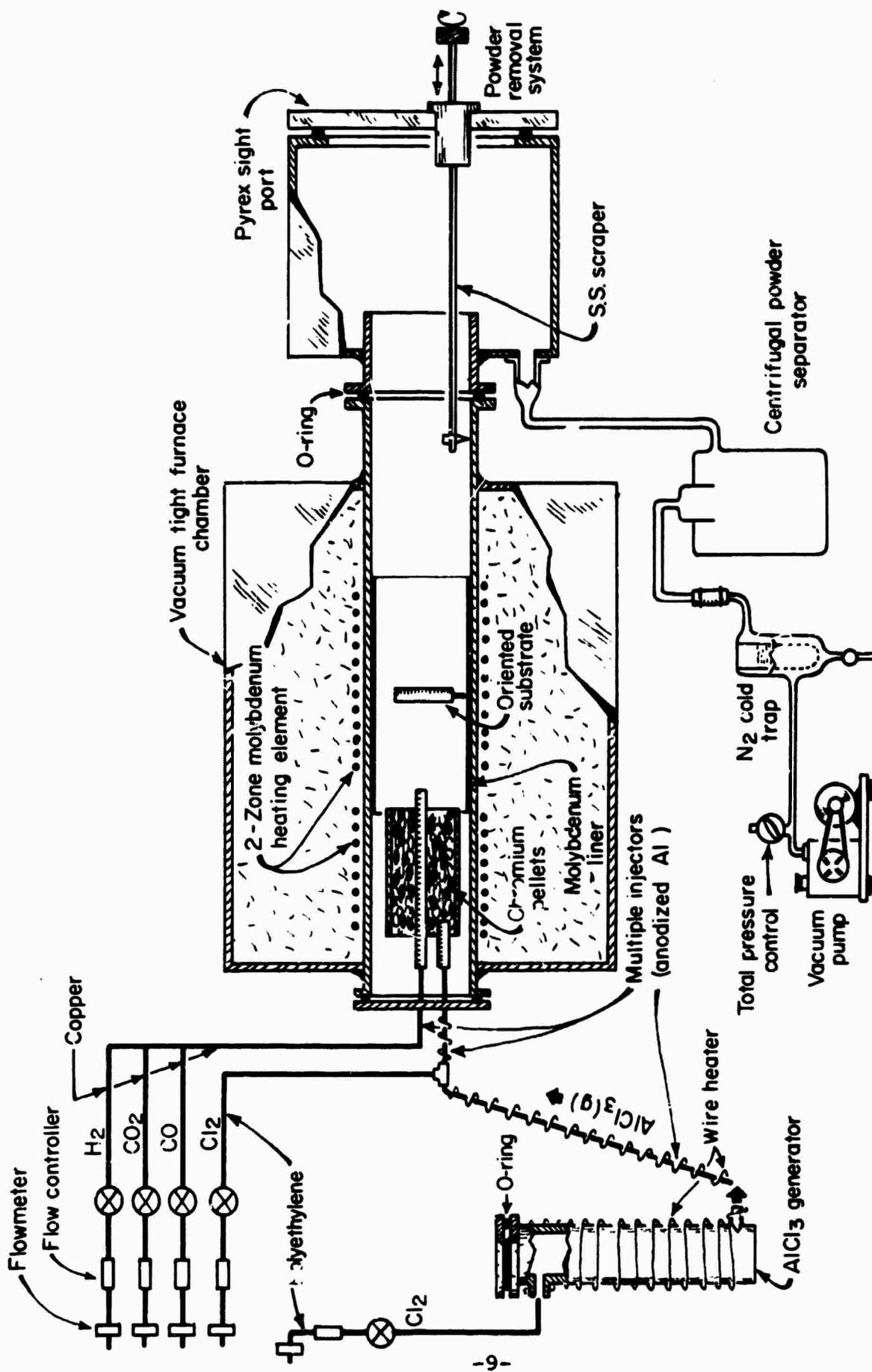


Figure 3: Schematic Diagram of Modified Ruby Vapor Growth Apparatus

interface is not a detriment as long as the original substrate is removed from the grown, fabricated crystals. If absolutely necessary, the molybdenum liners could be vapor-deposited with sapphire or flame-sprayed with alumina prior to insertion into the growth furnace. Smaller cylinders of molybdenum (8 inches long) were successfully flame-sprayed and vapor deposited; however, twenty-inch lengths would likely be difficult to successfully coat.

3.4 Powder Removal

During vapor growth, powder of identical or similar composition as the growing crystalline material forms at regions of high supersaturation in the growth furnace and deposits downstream from the growing crystal. These regions of high supersaturation are at the ends of the resistance heating elements where lower temperature prevails. Substantial amounts of powder deposits with increasing time, in the exhaust end of the growth furnace tube which leads to constriction of the tube. This in turn results in observation loss of the growing crystal and possible variations in gas velocity.

A mechanism has been incorporated into the exhaust chamber, which by means of a push-pull, rotating scraper rod through a vacuum seal, permits manual removal of the deposited powder into a collection zone during a run. This system greatly increases the duration of growth runs without powder formation becoming a detriment.

3.5 Crystalline Purity

Although vapor-grown ruby crystals have been produced with typical cation impurity levels of 10-30 ppm and as low as <3 ppm (determined by spectrochemical analysis), it is still desired to improve the purity to further enhance optical properties. A major source of impurities has been

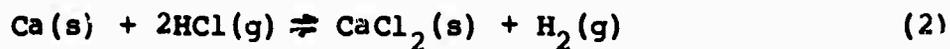
the chlorine and aluminum chloride transfer lines and flow system, which consist mainly of 304 and 316 stainless steel. It has been observed that these components show signs of corrosion and oxidation with increasing exposure times. Therefore, polyethylene transfer lines and polypropylene compression fittings have been installed in the chlorine transfer system. Anodized aluminum and high purity impervious alumina have both been tested with successful results and are now used in the injector system and aluminum chloride vapor transfer line.

All internal components of the chlorine flowmeters and flow controllers were coated with FEP Teflon.^{7,8} This coating is a mixture of high molecular weight copolymers of tetrafluoroethylene and hexafluoropropylene which should provide long life at room temperature by preventing any chemical reaction between chlorine gas and these components.

3.6 Pumping System

The present method for attaining a desired constant reduced pressure during crystal growth is by means of a mechanical vacuum pump (25 CFM), liquid nitrogen cold trap to remove condensable product gases (primarily hydrogen chloride) and a nitrogen gas bleed into the pump to control its capacity. It was determined that using a liquid trap, approximately 80 per cent of the condensable gases were removed by the trap. Although the relatively high freezing point of hydrogen chloride (-114°C) compared to the boiling point of liquid nitrogen (-196°C) suggests that all the HCl gas may be condensed, its odor is strongly present in the pump oil following a growth run. The oil is always changed after a run exceeding a few hours, which prolongs the life of the pump by reducing corrosion between the HCl and the internal pump components coming into contact with this gas. The major disadvantage of using liquid nitrogen is its cost, approximately \$0.60 per hour.

Calcium turnings were investigated as a means of removal of HCl by chemical reaction. All the product gases were flowed through a trap containing calcium at 700°C. The following reaction:



is thermodynamically favorable, $\Delta F^\circ_{700^\circ\text{C}} = -108$ Kcal/mole. However, difficulty arose in deriving a system which would expose sufficient surface area of calcium for complete reaction and would not change the cross-sectional area of the exhaust line exposed to the vacuum pump and thus cause total pressure variations.

Other possible vacuum systems were considered, primarily based on cost considerations, such as a steam ejector, a water aspirator - Rootes blower, and a mechanical pump without any trap, disposing of the pump after its useful life. Based on cost estimates, the aspirator-blower system would be the least costly to operate (as low as \$0.35/hour) and at least as effective as the present system; however, the present method will be utilized until the vapor growth technique modifications are thoroughly evaluated.

4.0 SUMMARY

1. Ruby crystals were vapor-grown using chromium organometallic compounds as the chromium dopant ion source. These ruby crystals showed identical visual fluorescence behavior as those grown using chromium chloride as the dopant source. However, the low decomposition temperatures of the compounds prevented high dopant homogeneity and sufficiently long growth runs to be achieved because the injector system clogged.

2. The growth furnace chamber was modified permitting crystals to be grown with the longitudinal axes of the substrates to be perpendicular to the incoming reactant gas stream. Preliminary growth using this technique showed improved homogeneity.

3. Molybdenum cylindrical inserts positioned inside the impervious, alumina furnace tubes were successful in extending the useful life of the furnace tubes. No molybdenum contamination was detected in the growing crystals once growth was achieved beyond the substrate-vapor growth interface.

4. Modifications were incorporated into the growth system which should allow continuous use up to 100 hours duration, eliminate problems of inhomogeneous dopant distribution and nucleation, and improve crystalline purity.

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