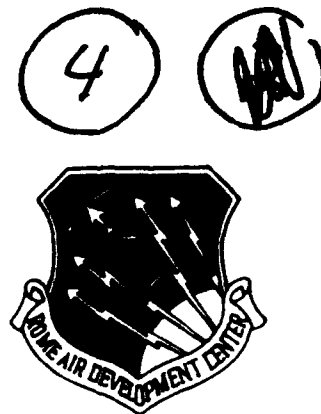


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September 1988



PREPARATION OF THE SUPERCONDUCTOR SUBSTRATE: STRONTIUM TITANATE

Kenneth P. Quinlan, Robert M. Hilton, Joseph A. Adamski

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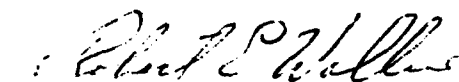
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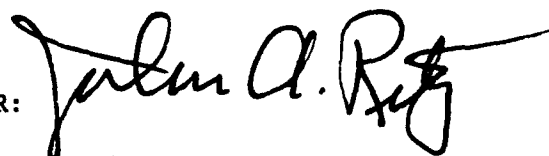
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Preface

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Preparation of the Superconductor Substrate: Strontium Titanate

1. INTRODUCTION

Ceramic superconductors with transition temperatures (T_c 's) above the boiling point of liquid nitrogen ($>77K$) have received an unprecedented amount of investigation since their recent discovery.^{1,2,3} These superconductive materials have been predicted to revolutionize transportation, electrical transmission, magnetic instrumentation and microcircuitry. The immediate application of these superconductors will most likely be realized in the area of microcircuitry such as computer interconnects, semiconductor-superconductor hybrids, Josephson junctions, etc.

The application of superconductors in microelectronics necessitates the development of techniques to prepare thin films of the superconductive materials. Various approaches for the preparation of the high T_c superconducting films are currently underway, such as electron beam evaporation, single target magnetron sputtering, magnetron sputtering from three metal targets, laser deposition, molecular beam deposition, etc. Thin film formation requires substrates that are

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1. Bednorz, J.G. and Muller, K.A. (1986) Possible high T_c superconductivity in the Ba-La-Cu-O system, *Z. Phys. B* **64**:189.
2. Wu, M.K., Ashburn, J.R., Torng, C.J., Hor, P.H., Meng, R.L., Gao, L., Huang, Z.J., Wang, Q., and Chu, C.W. (1987) Superconductivity at 93K in a new mixed phase Y-Ba-Cu-O compound system at ambient pressure, *Phys. Rev. Lett.* **58**:908.
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compatible with the superconductor being deposited. Among the properties that need to be compatible are (1) the same crystal structure with matched-lattice constants, (2) similar thermal expansion and (3) minimum interface reactions. Strontium titanate has been shown by various investigators to exhibit these characteristics required for their use as substrates for the superconductive films.

A program was initiated to investigate the synthesis of crystalline strontium titanate. Crystals of strontium titanate have been grown by various techniques: flame-fusion^{4,5,6,7}, flux-growth^{8,9,10,11,12}, floating zone¹³, Czochralski method¹⁴ and solution growth.¹⁵ Among the techniques, the flame-fusion method has the advantage of producing large size single crystals. Wafers of appreciable dimensions are required when used as substrates for the superconductive thin films. This report describes the synthesis of single crystals of strontium titanate with the flame-fusion technique and their fabrication to superconductor substrates.

2. EXPERIMENTAL

The flame-fusion techniques for the growth of single crystals of strontium titanate is derived from the original method developed by Verneuil.¹⁶ The general procedure for the growth of single crystals by the flame-fusion technique is depicted in Figure 1. A highly purified feed-powder of strontium titanate is tapped through a 40-mesh sieve into a stream of oxygen flowing in the center

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4. Bednorz, J.G. and Scheel, H.J. (1977) Flame-fusion growth of SrTiO_3 , *J. Crystal Growth* **41**:5.
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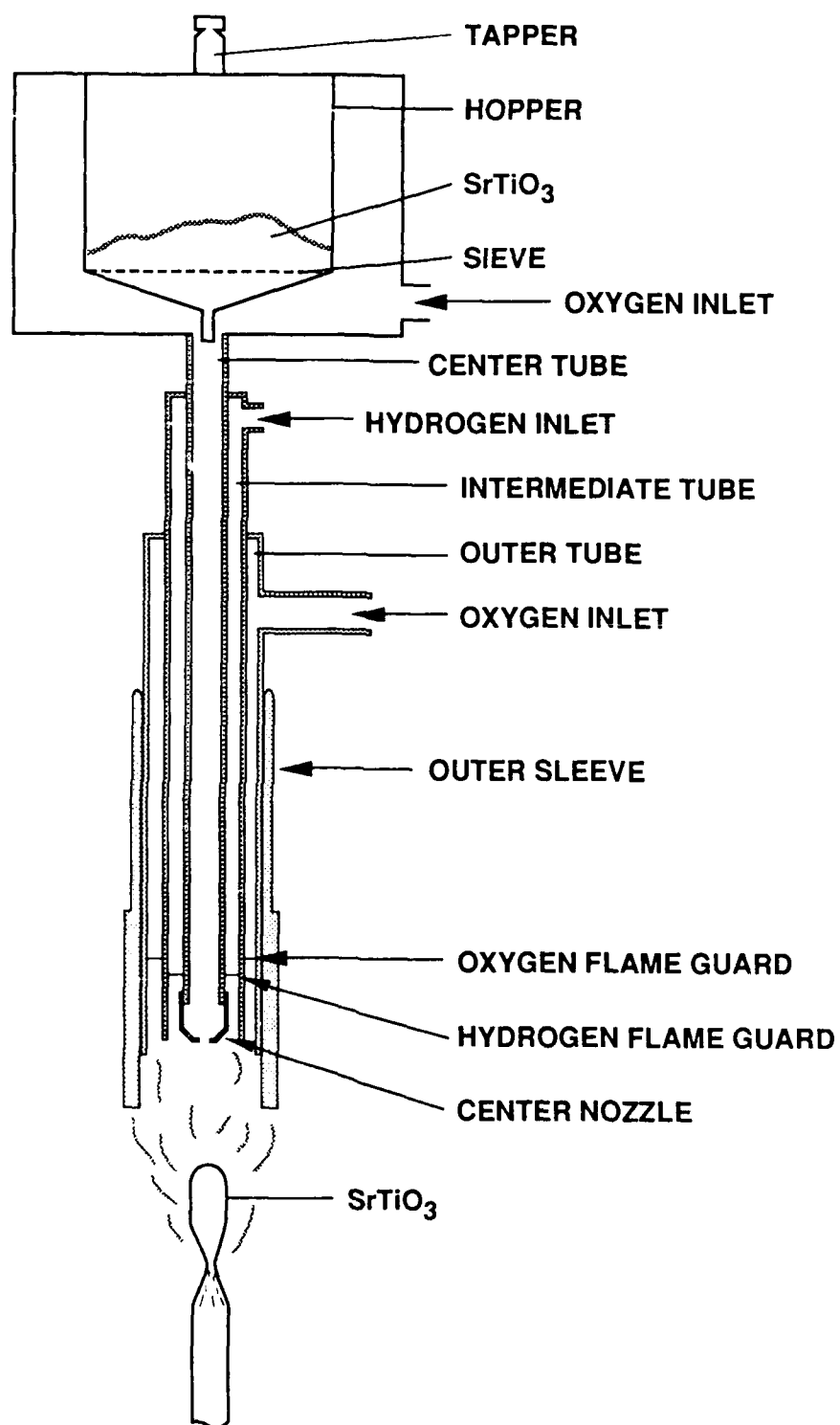


Figure 1. Schematic Diagram of the Flame-Fusion Apparatus

tube of an oxy-hydrogen burner. The burner is a three-tube postmix type with the center tube carrying oxygen; the intermediate tube, hydrogen; and the outer tube, oxygen. A detailed description of the burner is presented in the burner section of the report. The flame of the burner is directed into a 3.8 cm top-center hole of a refractory sleeve prepared from cast alumina. The refractory sleeve is 25.4 cm in diameter and 24.1 cm high. The sleeve contains a side-view circular window (diameter, 1.9 cm) whose center is located 7.3 cm from the top of the refractory sleeve. The center of the window is in line with that temperature of the flame where the SrTiO_3 crystal is grown. Bednorz et al.⁴ reported the growth of the single crystal of strontium titanate in the range of 2470-2480° C. In the initial segment of growth, the feed powder falls on a pressed-alumina pedestal whose position is at a temperature where a sintered cone of SrTiO_3 is formed. The top of the sintered cone is then positioned at that temperature where a small melt droplet of SrTiO_3 is formed. A neck of approximately 5 mm is formed by lowering the pedestal at a relatively high rate. The diameter of the crystal is then increased by lowering the pull-rate of the pedestal. Boules of strontium titanate 6 cm long and 1.4 cm in diameter have been achieved by this method. Single crystals may also be grown from seeds.

2.1 Three-Tube Postmix Burner

The development of the burner has been adequately described by J. Adamski.¹⁷ The burner consists of three stainless steel concentric tubes of different diameters. The center tube carrying the oxygen and feed powder is threaded at the bottom so that different inconel nozzles may be used. The intermediate tube, which carries the hydrogen, contains an inconel baffle with a series of holes permitting the concentric flow of hydrogen. The holes are equally spaced with diameters of 0.062 inch on a 0.531 inch diameter bolt circle. The center tube and the intermediate are held together by a specially constructed T made of brass. The outer tube carrying the oxygen is silver soldered to the intermediate tube. The outer tube contains an inconel baffle at its base. The baffle contains a series of holes (0.062 inch diameter, equally spaced on a 0.937 inch bolt circle) permitting the flow of oxygen. A water-cooled inconel sleeve is placed over the outer tube of the burner. This sleeve may be positioned along the outer tube until a desired flame pattern is obtained.

2.2 Preparation of Strontium Titanate Feed Powder

The preparation of the feed powder is critical. The powder must be relatively free of impurities and possess a certain particle size. Bednorz et al.⁴ and Linz et al.⁵ used particle sizes in the range of 0.05-0.125 mm and 0.05-0.74 mm, respectively, while Merker⁶ reported the use of particle sizes less than 0.149 mm. The method to prepare the feed powder in the present study was the oxalate precipitation procedure modified after Linz et al.⁵

Oxalic acid ($\text{C}_2\text{H}_4\text{O}_4 \cdot 2\text{H}_2\text{O}$, 177g) was dissolved in 370 ml of distilled water in a jacketed beaker. The solution was maintained at 70° C by means of a constant temperature bath with a circulating pump. A solution containing 40 ml of titanium tetrachloride in 121 ml of distilled water was then added to the oxalic acid solution. The titanium tetrachloride solution was prepared by adding TiCl_4

17. Adamski, J.A. (1956) New oxy-hydrogen burner for flame fusion, *J. Appl. Phys.* **36**:1784.

(99.9%) to distilled water in a beaker that was cooled by an ice bath. The oxalic acid solution containing the TiCl_4 was then equilibrated to 70°C . A solution containing 152.0g of $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$ in 590 ml of distilled water was added rapidly to the oxalic acid-titanium tetrachloride solution. Slow addition resulted in powder having a particle size greater than 0.131 mm. Upon the addition of the strontium chloride solution, a white precipitate formed. This precipitate was digested for two hours at 70°C . The precipitate, filtered by suction, was washed with 8 liters of distilled water. The precipitate was dried overnight by suction.

We added 2.1g SrCO_3 to the partially dried precipitate. Bednorz et al.⁴ reported that an excess of SrO (equivalent to 3.8 percent SrCO_3) is necessary to compensate for the SrO lost in the flame-fusion process. A deficiency in strontium oxide produces a melt enriched in titanium oxide which leads to an overflow of the molten cap on the boule. The amount of SrCO_3 added in the present procedure is equivalent to the final powder of SrTiO_3 containing 3.8 percent of the carbonate. This calculation is based on an 80 percent yield determined previously for this SrTiO_3 process. The strontium titanate precipitate was transferred to zirconia crucibles and dried in a drying oven. The precipitate was slowly dried to 175°C and then for two hours at 1000°C in a muffle furnace. Analysis calculated for SrTiO_3 : Sr, 47.7 percent; Ti, 26.1 percent. Found: Sr, 47.8 percent; Ti, 25.6 percent. Analyses for impurities by ICP-emission spectroscopy found: Ca, < 0.01 percent; Si, < 0.01 percent; Cr, 0.01 percent; Fe, 0.09 percent; and Al, 0.03 percent.

The dried SrTiO_3 powder was sieved using a nest of three sieves: 200, 270 and 325 mesh. The sieve analysis of the feed powder is depicted in Figure 2. The figure shows that 91.3 percent of the feed powder has a particle size of less than 0.044 mm. This size fraction was used in the preparation of the strontium titanate crystals.

2.3 Growth of Strontium Titanate Crystals

The operational parameters of a typical run for the growth of a SrTiO_3 bouie from a cone are given in Figure 3. The initial tap rate was 40 taps/min and was decreased to 24 taps/min after 15 minutes. The oxygen flow rates were constant at 15 l/min and 5 l/min for the outer and inner tubes, respectively. The hydrogen flow rate was increased from an initial value of 13.5 l/min to 21.0 l/min. The sintered cone was formed at the lower flow rate (lower temperature). Growth was carried out in an oxidizing flame where the ratio of H_2/O_2 was 1. This is in agreement with Gorina et al.¹⁸ who used an oxidizing flame to obtain clear transparent crystals. Bednorz et al.⁴ reported a slightly reducing flame where the H_2/O_2 ratio was 2. Merker⁶ used a strongly reducing flame with H_2/O_2 equal to 10. These results indicate that either a reducing or an oxidizing flame is capable of producing high quality single crystals of SrTiO_3 . A boule approximately 4 cm long and 1.5 cm in diameter was produced with these operational parameters. A large portion of the boule was single crystal.

The SrTiO_3 boules were annealed in zirconia crucibles. The boules were covered with SrTiO_3 feed powder and heated at 1000°C for 24 hours. The boules turned from black to colorless. Some of the

18. Gorina, Yu. I., Kashtanova, A.M., Maksimova, G.V., and Skanavi G.I. (1961) Preparation of monocrystals of strontium titanate and some data on their dielectric properties. *Kristallografiya* 6:473.

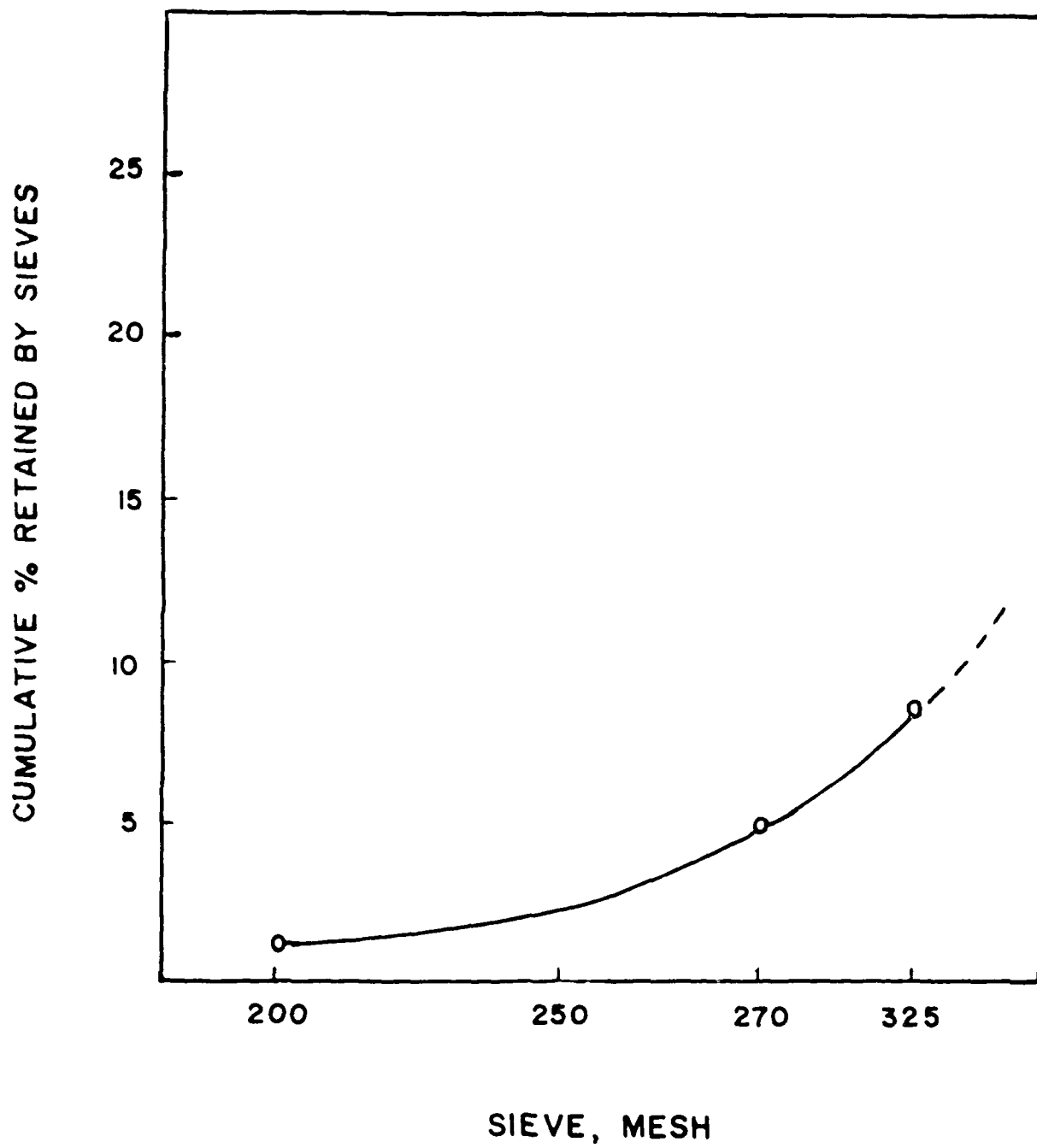


Figure 2. Sieve Analysis of the Strontium Titanate Feed Powder

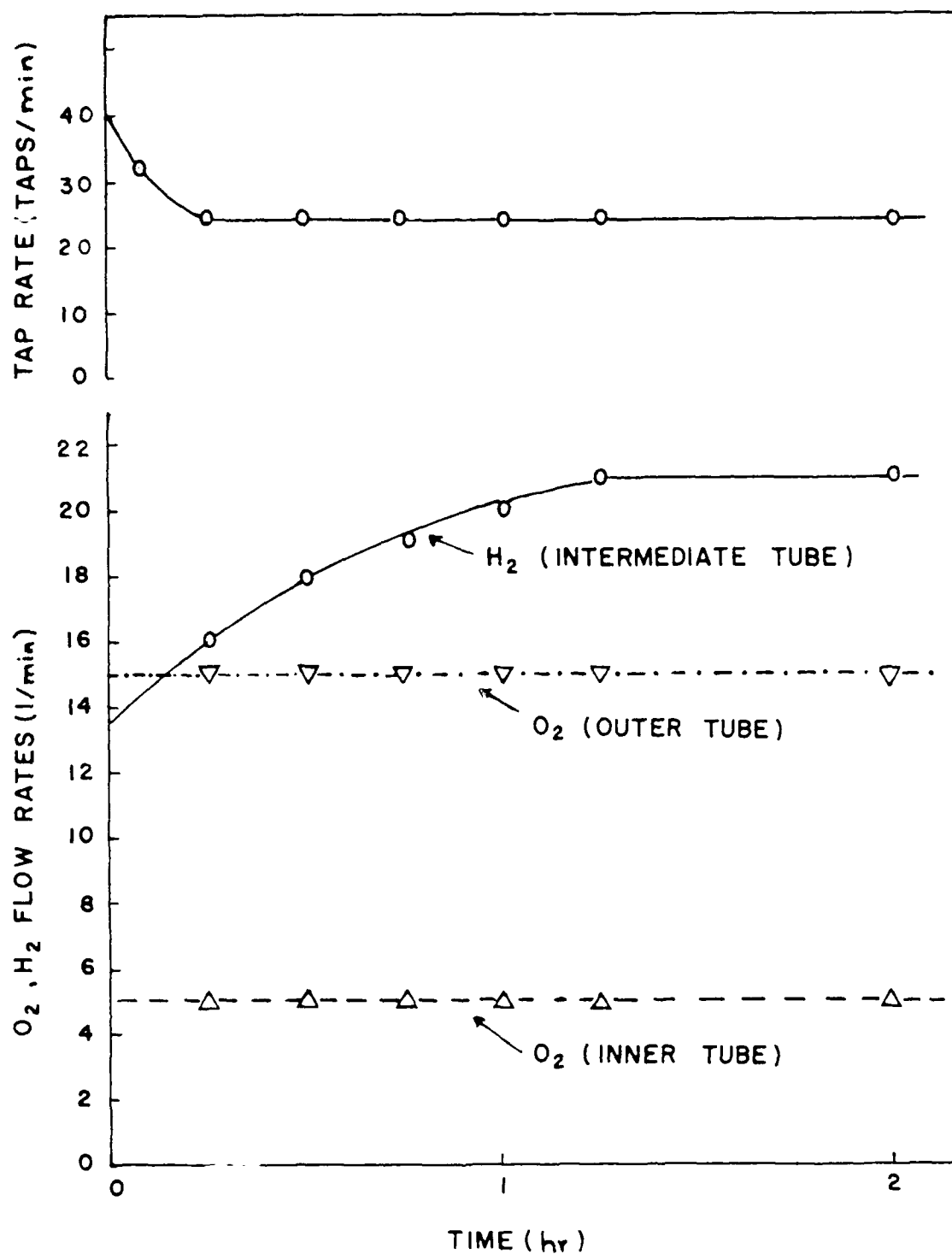


Figure 3. Operational Parameters Used for the Growth of SrTiO₃ Crystals by the Flame-Fusion Technique

boules possessed a yellowish tint. The black coloration may be due to a deficiency of oxygen in the crystals.

2.4 Preparation of The Substrates

Wafers of strontium titanate have been shown to be ideal substrates for the thin films of the superconductive ceramics.^{19,20,21} Therefore the boules must be sliced and polished. The SrTiO_3 boules were sliced using a diamond-coated metal blade. The diamond was 220 grit at a concentration of 75 percent at a 1/8 inch depth. Water was the coolant in the slicing procedure.

The polishing consisted of a rough grind with 320 grit SiC paper or SiC on glass plate to obtain a smooth planar surface. The slices were further polished with successive treatments of 15, 9, and 6 micron microgrit (Al_2O_3) with oil on a glass plate. This same treatment was repeated on a cast iron wheel on a bowl polisher. The final polishing was accomplished with 1 micron colloidal SiO_2 paste on nylon cloth or on politex supreme pads.

3. RESULTS AND DISCUSSION

Figure 4 shows a polished segment of the strontium titanate crystal obtained by the flame-fusion technique. The photograph demonstrates the excellent quality of the crystal. This quality is further depicted in the SEM micrograph of the surface of the SrTiO_3 crystal. These results show that crack- and inclusion-free crystals can routinely be obtained with the flame-fusion technique. The lattice constant (a) determined from x-ray diffraction studies was found to be $3.9056 \pm 0.0006 \text{ \AA}$. This is in excellent agreement with the value of $3.9050 \text{ \AA}$ reported by Swanson et al.²² Laue back-reflection photographs of the polished surface showed that the growth direction was 5° off the [211]. This result differs from Linz et al.⁵ who suggested that a preferred growth direction is observed along the [110]. Bednorz et al.⁴ studied the growth directions of 20 crystals and indicated no preferred growth orientation. Spot splitting in the present study indicated the presence of low angle grain boundaries.

Some crystals possess a slight yellowish tint, possibly due to the presence of a trace amount of iron. An ICP-emission spectroscopy analysis showed the presence of the following impurities: Ca, 0.03 percent; Si, <0.01 percent; Fe, 0.03 percent; Al, 0.06 percent; Mo, 0.17 percent; and Co, 0.04 percent. The presence of molybdenum and cobalt in the crystal is probably due to contamination by the burner material since these elements are not found in the feed powder. Bednorz et al.⁴ reported

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19. Dijkkamp, D., Venkatesant, T., Wu, X.D., Shaheen, S.A., Jisrawi, N., Min-Lee, Y.H., Mclean, W.I., and Croft, M. (1987) Preparation of Y-Ba-Cu-oxide superconductor thin film using pulsed laser evaporation from high T_c bulk material, *Appl. Phys. Lett.* **51**:619.
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 22. Swanson and Fuyst (1954) *National Bur. Std. Circ.* 539, **3**:44.



Figure 4. Polished Segment of a Strontium Titanate
Crystal Grown by the Flame-Fusion Technique



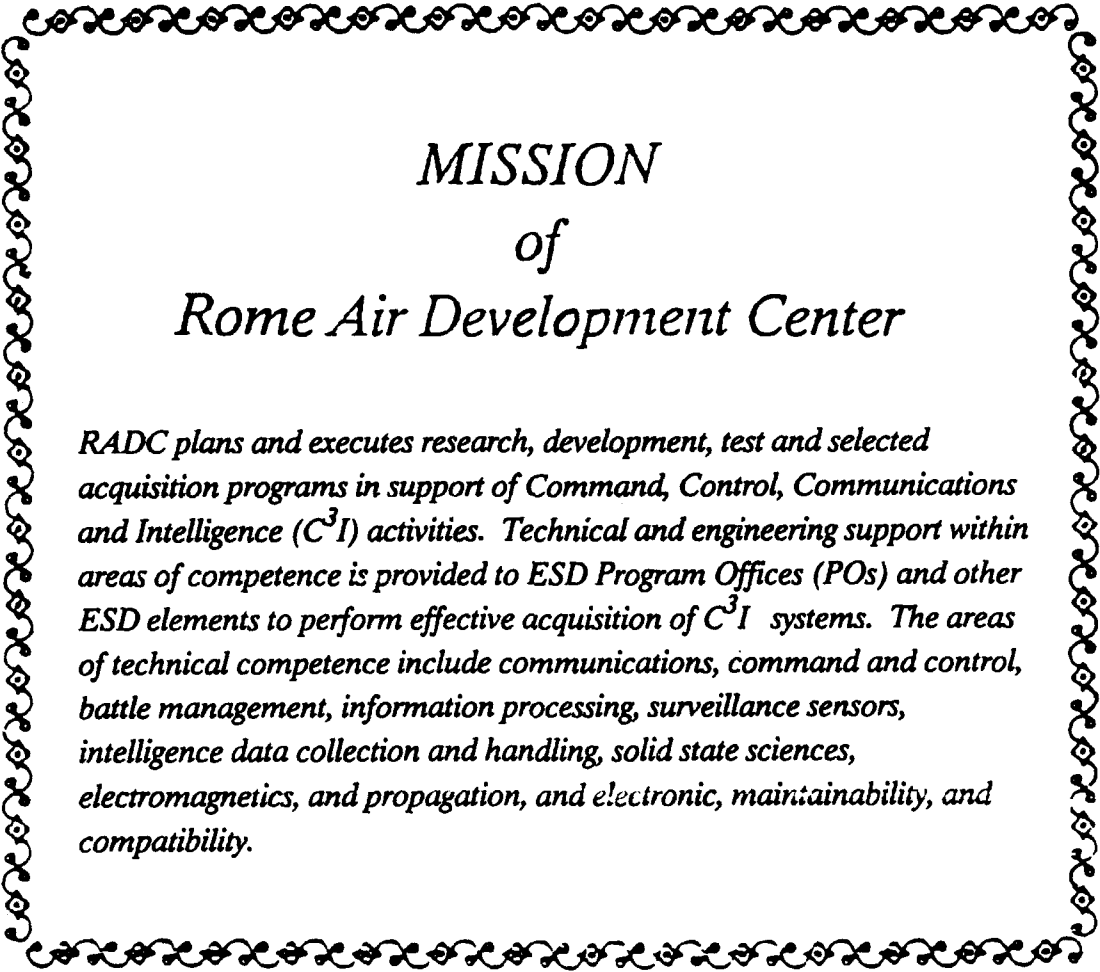
Figure 5. SEM Micrograph of the Polished Surface of a Strontium Titanate Crystal (4000X)

their strontium titanate crystals contained 0.005 percent Fe^{+3} and Cr^{+3} , but indicated that crystals prepared from high-purity strontium titanate feed powder contained only a few ppm of Fe^{+3} (~ 0.0003 percent). The crystals prepared by Gorina et al.¹⁸ contained the following impurities: Mg, 0.006 percent; Si, 0.006 percent; Al, 0.01 percent; Fe, 0.003 percent. The differences in percentages found for the impurities in the different studies may possibly be due to the sensitivity of the various methods of analyses such as EPR, spectral analysis and ICP-emission spectroscopy. Mo (VI) does not exhibit an electron spin resonance spectrum.

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RADC plans and executes research, development, test and selected acquisition programs in support of Command, Control, Communications and Intelligence (C³I) activities. Technical and engineering support within areas of competence is provided to ESD Program Offices (POs) and other ESD elements to perform effective acquisition of C³I systems. The areas of technical competence include communications, command and control, battle management, information processing, surveillance sensors, intelligence data collection and handling, solid state sciences, electromagnetics, and propagation, and electronic, maintainability, and compatibility.