GENERAL ELECTRIC

GENERAL ELECTRIC COMPANY
CORPORATE RESEARCH AND DEVELOPMENT
Schenectady, N.Y.

OXIDE CERAMIC LASER

SEMI-ANNUAL TECHNICAL REPORT (ITEM 0002-A002)

Program Code No.: 01D10K71
Contract No.: N00014-70-C-0360
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Contractor: General Electric Company
Effective Data: June 1, 1970
Expiration Date: May 31, 1972
Amount of Contract: $198,448.00

January 25, 1972

Scientific Officer: A. M. Diness, Code 471
Office of Naval Research
Arlington, Va. 22217

Sponsored by

Advanced Research Projects Agency
ARPA Order No. 1587. Amend. #1

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This research was supported by the Advanced Research Projects Agency of the Department of Defense and was monitored by ONR under Contract No. N00014-70-C-0360

R

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UNCLASSIFIED
Security Classification

DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author) General Electric Company Research and Development Center Post Office Box 8 Schenectady, New York 12301	2a. REPORT SECURITY CLASSIFICATION Unclassified 2b. GROUP

3. REPORT TITLE

OXIDE CERAMIC LASER

4. DESCRIPTIVE NOTES (Type of report and inclusive dates)
Semi-Annual Technical Report, June 1, 1971 through December 31, 1971

5. AUTHOR(S) (First name, middle initial, last name)

Charles D. Greskovich

6. REPORT DATE January 25, 1972	7a. TOTAL NO. OF PAGES ~~14~~ 19	7b. NO. OF REFS -
8a. CONTRACT OR GRANT NO. N00014-70-C-0360 b. PROJECT NO. ARPA Order #1587 Amend. #1 c. 01D10K71 d.	9a. ORIGINATOR'S REPORT NUMBER(S) SRD-72-016 9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)	

10. DISTRIBUTION STATEMENT

11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY Advanced Research Projects Agency 1400 Wilson Boulevard Arlington, Virginia 22209

13. ABSTRACT

The calcination temperature of Y-Th-Nd oxalate powder was increased to 650°C and resulted in fewer large pores of about 100μm in size in sintered material made from unmilled powder. Calcined powder particles are not single crystal in nature but are composed of small crystallites of the order of 200Å in size.

Grain growth of sulfur-containing Yttralox ceramics doped with neodymium approximately followed a (time)$^{1/3}$ behavior whereas sulfur-free material approximately followed a (time)$^{1/2}$ law at 2000°C in dry H_2. Throughout the time measurements of this study, however, the grain size of the slow growing material containing sulfur was always larger than that of the sulfur-free material. The mechanism by which sulfur controls grain growth is not now understood.

Columnar grain formation occurred in specimens fired above 2210°C in dry H_2 and caused specimen cracking. The cracking of Nd-doped Yttralox rods during thermal cycling below a soak temperature of 2210°C in dry H_2 has been solved by placing Nd-doped Yttralox granules between the rods and the tungsten setter.

The major short term processing goals are to understand the role of sulfur in the development of the grain and pore structure and to determine the origin of pore clusters in sintered material.

During the past six months Nd-doped Yttralox of high optical quality has been produced from Y-Th-Nd oxalate. By using the calcined oxalate in conjunction with dry milling in a rubber-lined ball mill, reproducible fabrication of ultraclear Nd-doped Yttralox rods has been achieved. A Nd-doped Yttralox rod was produced with a laser threshold value of 18 joules, this value being referenced to an Owens Illinois laser glass (ED-2) with a laser threshold value of 9.7 joules in the same laser resonant cavity. This laser threshold value was the lowest of all of the rods synthesized to date. The variation in refractive index within a typical Nd-doped Yttralox rod of laser quality was determined by a double-pass Twyman-Green type interferometer and calculated to be approximately 5×10^{-6}. Spectral transmittance of undoped-Yttralox, including surface reflection losses, was greater than 80% between 3/4 and 6μm for a 3 1/2 mm thick plate.

DD FORM 1 NOV 65 1473 REPLACES DD FORM 1473, 1 JAN 64, WHICH IS OBSOLETE FOR ARMY USE.

UNCLASSIFIED
Security Classification

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Nd-doped Yttralox lasers						
Sintering						
Pores						
Variation in refractive index						
Sulfur contamination						
Spectral transmittance						

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TECHNICAL REPORT SUMMARY

General Goals

The long range goal of the current research program is to develop an intermediate gain, solid laser material which has an emission line width between that of neodymium-doped glass and neodymium-doped yttrium aluminum garnet single crystals. Such material should be suitable for generating high average power combined with high peak power.

In order to achieve this goal, experimental and theoretical work has been done in the past 18 months to (1) develop a process to reproducibly fabricate Nd-doped Yttralox^{TM}, a polycrystalline oxide ceramic comprised of 89 mole % Y_2O_3, 10% ThO_2, and 1% Nd_2O_3 in solid solution, of improved optical quality, (2) understand the densification process with respect to the kinetics of porosity change and (3) evaluate Nd-doped Yttralox rods as a potential laser material by means of optical measurements.

Current Processing Status

The calcination temperature of Y-Th-Nd oxalate powder was increased to 850°C and resulted in fewer large pores of about 100µm in size in sintered material made from unmilled powder. Calcined powder particles are not single crystal in nature but are composed of small crystallites of the order of 200A in size.

Grain growth of sulfur-containing Yttralox ceramics doped with neodymium approximately followed a $(\text{time})^{1/3}$ behavior whereas sulfur-free material approximately followed a $(\text{time})^{1/2}$ law at 2000°C in dry H_2. Throughout the time measurements of this study, however, the grain size of the slow growing material containing sulfur was always larger than that of the sulfur-free material. The mechanism by which sulfur controls grain growth is not now understood.

Columnar grain formation occurred in specimens fired above 2210°C in dry H_2 and caused specimen cracking. The cracking of Nd-doped Yttralox rods during thermal cycling below a soak temperature of 2210°C in dry H_2 has been solved by placing Nd-doped Yttralox granules between the rods and the tungsten setter.

The major short term processing goals are to understand the role of sulfur in the development of the grain and pore structure and to determine the origin of pore clusters in sintered material.

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Optical Evaluation of Product

During the past six months Nd-doped Yttralox of high optical quality has been produced from Y-Th-Nd oxalate. By using the calcined oxalate in conjunction with dry milling in a rubber-lined ball mill, reproducible fabrication of ultra-clear Nd-doped Yttralox rods has been achieved. A Nd-doped Yttralox rod was produced with a laser threshold value of 18 joules, this value being referenced to an Owens Illinois laser glass (ED-2) with a laser threshold value of 9.7 joules in the same laser resonant cavity. This laser threshold value was the lowest of all of the rods synthesized to date. The variation in refractive index within a typical Nd-doped Yttralox rod of laser quality was determined by a double-pass Twyman-Green type interferometer and calculated to be approximately 5×10^{-6}. Spectral transmittance of undoped-Yttralox, including surface reflection losses, was greater than 80% between 3/4 and 6μm for a 3 1/2 mm thick plate.

January 25, 1972

Director
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I. INTRODUCTION

The last six months of the current research contract, "Oxide Ceramic Laser", was devoted to (1) improving and optimizing powder preparation and processing techniques, (2) understanding the influence of sulfur on the development of the Yttralox microstructure and (3) improving the optical perfection of Nd-doped Yttralox ceramic. One of the laser rods synthesized during this period had the lowest laser threshold of any produced. The presence of pore clusters inside the grains in sintered material that is currently made is the major obstacle in producing Nd-doped Yttralox with higher optical perfection. Experiments are under way to identify the source of these clusters and eliminate them.

II. POWDER PREPARATION AND PROCESSING

Large Pores and Orange Peel in Yttralox Prepared from Unmilled Powder

The oxalate process is the standard way of preparing Nd-doped Yttralox powder for the conventional technique of cold pressing and sintering. This process of powder preparation involves dripping Y-Th-Nd salt solution into an oxalic acid bath at a rate of 100 drops/min., washing the oxalate-hydrate precipitate in 55 ℓ. of deionized water, vacuum filtering and oven drying the precipitate at 110°C, calcining at 800°C for 4 hours in air, ball-milling for 6 hours, pressing into a rod or disk-shape at about 40 Kpsi and sintering in dry H_2 at 2170°C for various times.

Sintered Yttralox specimens prepared from calcined Y-Th-Nd oxalate-hydrate which has <u>not</u> been milled contain large pores and "orange peel", a point-to-point variation in refractive index. The origin of large pores may be the incomplete decomposition of the starting powder whereas orange peel may have its origin in compositional inhomogeneity in the starting powder.

In order to determine if the origin of large pores ($\approx$150-200μm in size) ara caused by incomplete decomposition of the starting powder at 800°C for 4 hours in air, the calcination temperature was increased to 850°C for 4 hours in air. Sintered specimens prepared from this powder contained fewer large pores greater than 100μm. Apparently a calcination temperature of 850°C more completely decomposed Y-Th-Nd oxalate-hydrate to the respective oxides and minimized the formation of large pores originating from gas evolution from calcined powder which was incompletely decomposed. A calcination temperature of 850°C for 4 hours in air will be used in future experiments because it more completely decomposed the starting material without causing excessive particle growth and agglomerate formation.

Two changes were made during powder preparation in order to determine if the amount of orange peel can be reduced in sintered material. One was to decrease the drip rate of Y-Th-Nd salt solution into the oxalic acid bath while magnetically stirring from 100 to 50 drops/min. whereas the second was to drip the oxalic acid bath into the Y-Th-Nd salt solution at a rate of 50 drops/min. Decreasing the drip rate from 100 to 50 drops/min. caused no noticeable change in the amount of orange peel in the sintered product. The reverse precipitation procedure gave rise to a "grinding" noise at the bottom of the glass beaker, caused by the movement of agglomerated powder particles. Sintered material prepared from this calcined starting powder always contained a solid second phase which was thoria-rich. Second phase particles as large as 75µm could be found in material sintered for 57 hours at 2170°C in dry H_2. The thoria-rich second phase must originate from the formation of thoria-rich agglomerates during the precipitation step. In addition, the sintered material contained a considerable amount of orange peel. It appears to be very difficult to control orange peel by changing the precipitation rate by dripping the Y-Th-Nd salt solution into the oxalic acid bath or by dripping the oxalic acid bath into the Y-Th-Nd salt solution. Therefore, the Y-Th-Nd salt solution will be dripped into the oxalic acid bath at a rate between 50 and 100 drops/min., and orange peel will be continued to be eliminated by a dry ball-milling operation unless another approach is found which will produce a more chemically homogeneous starting powder.

It was previously thought that calcined powder derived from the oxalate process contained particles which were essentially single crystals. A transmission electron micrograph (Fig. 1) clearly showed, however, that the calcined Nd-doped Yttralox particle contained small crystallites of the order of 200Å in size. An electron diffraction pattern of the same particle gave a well-defined ring pattern verifying that the particle was polycrystalline in nature. X-ray (Debye-Scherrer) photographs showed that Y-Th-Nd coprecipitate powder calcined at 800°C for 4 hours was composed of a body-centered-cubic solid solution (Yttralox) plus a trace of ThO_2 containing some Y_2O_3 in solid solution.

B. Sulfur

It has been established during the first year's contract that dry ball-milling the calcined oxalate powder in the presence of 1 wt. % stearic acid produced laser quality, Nd-doped Yttralox rods. The milled powder picked up sulfur from the rubber lining of the ball-mill, and this sulfur is thought to aid in the densification of the powder compacts. In order to determine if sulfur aids in the densification of the powder compacts, two mills which were supposedly sulfur-free were obtained from Norton Company. One was a neoprene-lined mill having a volume of 1 liter, and the second mill had a polyurethane lining and a volume of about 2 liters. A comparison of the optical quality of the sintered material prepared from powder milled in a rubber (containing sulfur) lined mill and that prepared from powder milled in sulfur-free mills would then show if sulfur was helpful in eliminating pores.

Sintered material prepared from powder that was milled in the neoprene-lined ball-mill always contained a grain boundary phase which was very similar to the sulfur-rich phase found at the grain boundaries in sintered Yttralox specimens prepared from powder milled in a rubber-lined ball-mill. A standard, high temperature combustion test of the neoprene material revealed that there was about 1.93 wt. % sulfur in it. The author questioned Norton Company about the neoprene liner being "sulfur-free", and a subsequent investigation by the Norton people into the matter led to their "discovery" that the neoprene liner, indeed, contained about 2 wt. % sulfur.

Powder milled in the polyurethane-lined mill was bluish-white and did not have the light yellowish tint characteristic of sulfur contaminated powders milled in a rubber-lined or a neoprene-lined mill. Sintered Yttralox specimens prepared from powder milled in this polyurethane-lined ball-mill showed no evidence of a grain boundary phase. There were strong indications that the polyurethane is sulfur-free although a sulfur analysis will be made. An examination of the pores in the sintered material showed that there was a tremendous number of large pores approximately 75μm in diameter, large tubular pores about 25μm in diameter and 140μm in length, and pore clusters of various sizes. This experiment tentatively shows that sintered material prepared from powder milled in a sulfur-free polyurethane-lined ball-mill contained more porosity than that prepared from powder milled in a sulfur-containing rubber-lined ball-mill, assuming that both mills comminuted and dispersed the powder equally and that impurities other than sulfur were not influencing pore development or disappearance. An experiment is currently in progress to clearly determine if sulfur is necessary or not for the production of highly dense Yttralox ceramics. In this experiment sulfur will be intentionally added in the form of $Y_2O_3 \cdot SO_3$ to the mill charge in the polyurethane-lined mill.

C. Pore Clusters

In the past 4 months all sintered Yttralox specimens prepared from powder milled in a rubber-lined ball-mill contain a large number of pore clusters that are distributed throughout the bulk of the material. Prior to this time, sintered Yttralox specimens prepared by the same powder processing procedure contained very few pore clusters. A transmitted light photomicrograph of a typical pore cluster is shown in Fig. 2. The pores are approximately 5μm or less in diameter and vary in number within a pore cluster.

An investigation into the origin of pore cluster in sintered Nd-doped Yttralox specimens resulted in the following experimental findings. (1) Sintered material made from unmilled clacined powder contained very few pore clusters whereas that made from dry-milled, calcined powder always contained numerous pore clusters. (2) Changing the weight ratio of grinding media to charge in the rubber-lined mill from 24:1 to 20:1 had no major affect on the formation of pore clusters. (3) Dry milling the Yttralox powder with 1 wt. % stearic acid in rubber-lined, neoprene-lined, and polyurethane-lined mills yielded sintered material containing many pore clusters. (4) Dry milling Yttralox powder in the absence of stearic acid also yielded sintered material having pore clusters. (5) Pore clusters did not originate from contamination caused by the furnace atmosphere during the second calcination step used only for Yttralox specimens prepared from ball-milled powder. (6) Undoped Yttralox powder that was ball-milled produced sintered material having a large number of pore clusters. From these experimental findings it is concluded that pore clusters originate in sintered Yttralox specimens when the calcined powder is dry milled and that there is no influence of the weight ratio of grinding media to charge, different ball-mill linings, stearic acid, furnace atmosphere during the second calcination step, and neodymium on the elimination of pore clusters. A closer examination of the Yttralox balls used as the grinding media showed that there was excessive wear at various locations on the surface of several Yttralox balls. Therefore, it is presently thought that large particles are ground off of the Yttralox balls, mix into the powder batch, and act as nuclei for exaggerated grain growth during sintering. During the initial stage of exaggerated grain growth, pores are trapped inside of the fast growing (large) grains and could give rise to the kind of pore morphology illustrated in Fig. 2. The Yttralox balls will be fired at high temperature to smooth-out the surface of the balls and thereby reduce chipping during milling. Large particles of Yttralox may be intentionally added into the mill charge to determine any enhancement of pore cluster formation.

III. POWDER FORMING

The cold pressing of Yttralox disks in a double-acting die at 38 Kpsi occasionally produces laminations within a pressed disk having a thickness of 1/4 inch and diameter of 1 inch. After sintering such a disk at high temperature, a large number of pores can be seen in the plane of view when the specimen is observed under the optical microscope with transmitted white-light. These pores are concentrated near the circumference of the disk, and their sizes are larger near the circumference of the disk than far away from it. Such pores probably arise from the heating of cracks created by laminations in die-pressed disks. A double pressing procedure, whereby a disk-shaped specimen is die-pressed at a low forming pressure of about 3 to 5 Kpsi and subsequently isostatically pressed at 38 Kpsi, has been found successful in eliminating these pores.

IV. KINETICS OF GRAIN GROWTH AND SULFUR REMOVAL

A. Grain Growth Studies

The grain size in Nd-doped Yttralox compacts sintered at 2000 and 2170°C in dry H_2 for various times is given in Fig. 3. At constant time and temperature, sintered Yttralox prepared from powder milled in a rubber-lined ball-mill has a larger grain size than that prepared from the unmilled powder. Grain growth of sulfur-containing Yttralox specimens approximately follows a $(\text{time})^{1/3}$ behavior whereas sulfur-free (unmilled) material approximately follows a $(\text{time})^{1/2}$ law at 2000°C. Although the microstructure of sulfur-free Yttralox ceramic is characterized by a single solid phase at 2000°C, the microstructure of sulfur-containing Yttralox is characterized by a highly reflecting, solid second phase which is sulfur rich (Fig. 4). The detection of sulfur in the second phase particles was achieved with a scanning electron microscope equipped with a solid state x-ray detector. The mechanism of grain growth in sulfur-containing Yttralox at 2000°C is not exactly known but may be related to the inhibition of grain boundary movement by solid second phase particles. Grain growth in sulfur-free Yttralox follows square kinetics, as predicted by theory. It should be mentioned that the sulfur-rich second phase is solid at 2000°C and liquid at 2170°C in dry H_2.

In the final stages of sintering, the microstructure of Nd-doped Yttralox exhibits grains which are uniform and equiaxed at temperatures as high as 2210°C in dry H_2. The sintering of Yttralox materials doped with neodymium above 2220°C produces a microstructure characterized by columnar grain formation, shown in Fig. 5 and by specimen fracture. These columnar grains are of the order of millimeters in length, and their presence may be the cause of specimen fracture. Columnar grain formation is not caused by sulfur concentrations greater than 50 ppm because a sintered Yttralox specimen containing 50 ppm was fired in dry H_2 at 2220°C for 40 hours and exhibited columnar grains in the microstructure. X-ray diffraction studies of Yttralox specimens fired at 2170°C and 2220°C showed that both specimens had a body-centered-cubic lattice which is characteristic of the c-type rare earth oxide structure (Tl_2O_3 prototype). It may be that columnar grain formation is not caused by a change in crystal symmetry or crystal structure but is probably caused by the presence of a finite amount of liquid phase which develops as the temperature exceeds the solidus temperature for the given composition. From these experiments, the maximum sintering temperature that can be used for the production of Yttralox specimens containing microstructures having equiaxed grains is about 2210°C.

B. Sulfur Removal During Sintering

The distribution of the solid second phase in sulfur-containing Yttralox changes with sintering time at 2000°C. If an Yttralox disk is sintered for 1/2 hour at 2000°C, there is a zone near the outer surface which is free of the solid second phase. This zone moves into the interior of the specimen with longer sintering times. By measuring the thickness (X) of this zone as a function of time (t), an effective diffusion coefficient (D) can be estimated for the rate of disappearance of the sulfur-rich phase by using the equation:

$$X^2 \approx 2Dt \qquad (1)$$

The effective diffusion coefficient is approximately 10^{-7} cm^2/sec at 2000°C in dry H_2 and is a measure of the diffusion for sulfur in Yttralox specimens doped with 1 mole % Nd_2O_3.

V. CRACKING OF YTTRALOX RODS BELOW 2210°C

The cracking of Yttralox rods had been a major problem during the first 15 months of the current research program. In many cases Yttralox rods having a length ~9 cm and a diameter ≈3/4 cm contained longitudinal and transverse cracks after cooling from the sintering temperature. It was observed that many of the cracks originated at the regions of contact between the rod and the tungsten setter in the sintering furnace. By placing Yttralox granules between the rods and the tungsten setter, the cracking problem has been cured and there has been a valuable saving of sintered material.

VI. OPTICAL MEASUREMENTS

A. Variations in Refractive Index

A quantitative definition of "orange peel" (a point-to-point variation in refractive index) has not been previously determined in Nd-doped Yttralox rods of laser quality. Recently we have assembled a double-pass Twyman-Green type interferometer operated with a He-Ne laser source (λ = 6328Å). This type of interferometer quickly reveals small variations in refractive index. An Yttralox rod, having ends flat to $\lambda/10$ using sodium light with λ = 5898Å and ends parallel to ±2 arc seconds, was placed in the interferometer and had an average variation in refractive index of 5×10^{-6}. This change in refractive index is characteristic of sintered Yttralox material prepared from powder milled in a rubber-lined mill. We plan to use the Twyman-Green type interferometer to quantitatively determine small variations in refractive index in all Yttralox laser rods and relate these values to laser threshold values.

B. Total Scattering Losses and Laser Thresholds

Total scattering losses were measured on each lasing quality rod at 6328Å relative to a Nd-doped glass rod (Owens Illinois, ND-11 laser glass) in an integrating sphere photometer. Values of total scattering loss and laser threshold for Nd-doped Yttralox rods about 7.6 cm in length and 0.4 cm in diameter are given in Table I.

Table I - Total Scattering Loss and Laser Threshold Values for Nd-doped Yttralox Rods Relative to Nd-doped Glass

Specimen	Loss	Laser Threshold (joules)
OI-ND-11 Glass	0	15
Yttralox 11-3	4.5	60
Yttralox 12-1	3.0	28
Yttralox 12-2	3.4	32
Yttralox 12-4	4.3	50
Yttralox 13-4	1.4	*
Yttralox 13-5	2.0	**
Yttralox 13-9	1.5	**
Yttralox 13-10	1.7	**

* Laser threshold was determined in a different optical cavity.

** These rods were sent to A. Diness without performing the laser thresholds.

The total scattering losses quoted in Table I were the average of three measurements. Specimens 11-3, 12-1, 12-2 and 12-4 were synthesized during the first year's contract whereas specimens 13-4, 13-5, 13-9 and 13-10 were synthesized during the beginning of the second year's contract. Scattering losses are caused by any change in refractive index within the Yttralox rod, such as pososity, solid second phase and "orange peel".

Table I shows that the higher is the total scattering loss, the higher is the laser threshold. Yttralox rod 13-4 has the lowest total scattering loss and, as will be shown later, this specimen has the lowest threshold value of all of the Yttralox rods tested. The measurement of total scattering loss is, therefore, a reliably quick method of qualitatively differentiating between Yttralox specimens of good and poor optical quality.

It has been previously stated thet in the last 4 months nearly all sintered Yttralox rods prepared from powder milled in a rubber-lined ball-mill contained porosity which was primarily in the form of pore clusters. Individual pores in a cluster were genarally 5μm or less in size. A Nd-doped Yttralox rod (16-3) that was representative of these rods was selected for the determination of its lasing behavior. The laser threshold for specimen 13-4 which contained relatively few pore clusters was also measured. Laser threshold values for 16-3 and 13-4 are shown in Table II along with two other Yttralox rods previously synthesized during the first year's contract.

Table II - Laser Threshold Values for Nd-doped Yttralox Rods Referenced to Nd-doped Glass

Specimen	Laser Threshold (joules)
Laser glass (OI, ED-2)	9.7
11-3	49
12-1	20
13-4	18
16-3	36

These threshold values were referenced to a Nd-doped glass laser (Owens Illinois, ED-2) and were determined in a different laser resonant cavity than that used for the determination of laser threshold listed in Table I. Yttralox rod 13-4 had a laser threshold of 18 joules and had the lowest laser threshold of all the Nd-doped Yttralox rods produced to date. The relatively high laser threshold found for specimen 16-3 will be correlated with its pore size and cluster size distributions. Similarly, the pore size distribution in specimen 13-4 will be measured and correlated with scattering losses and laser threshold.

C. Spectral Transmittance of Undoped Yttralox

The spectral transmittance of undoped Yttralox ceramic, including surface reflection losses, is given in Fig. 6. For a specimen of 3 1/2 mm in thickness, the short wavelength cut-on and long wavelength cut-off are approximately 1/4 and 9μm, respectively. At about 1μm, two surfaces will reflect about 18% of the incident radiation. Transmission is greater than 80% between 3/4 and 6μm. The fact that there is high transmittance from the visible to the middle infrared region of the spectrum makes Yttralox suitable for visually aligning long wavelength detectors behind infrared-transmitting windows, promising as a laser host material for the neodymium wavelength region of 1.07μm, and desirable for a variety of optical applications at high temperatures because of its high melting point (>2220°C).

Figure 1. Transmission electron micrograph of a particle of Nd-doped Yttralox powder calcined at 800°C for 4 hours in air. X20,000

Figure 2. Pore cluster in sintered Nd-doped Yttralox ceramic. Transmitted white light. X550

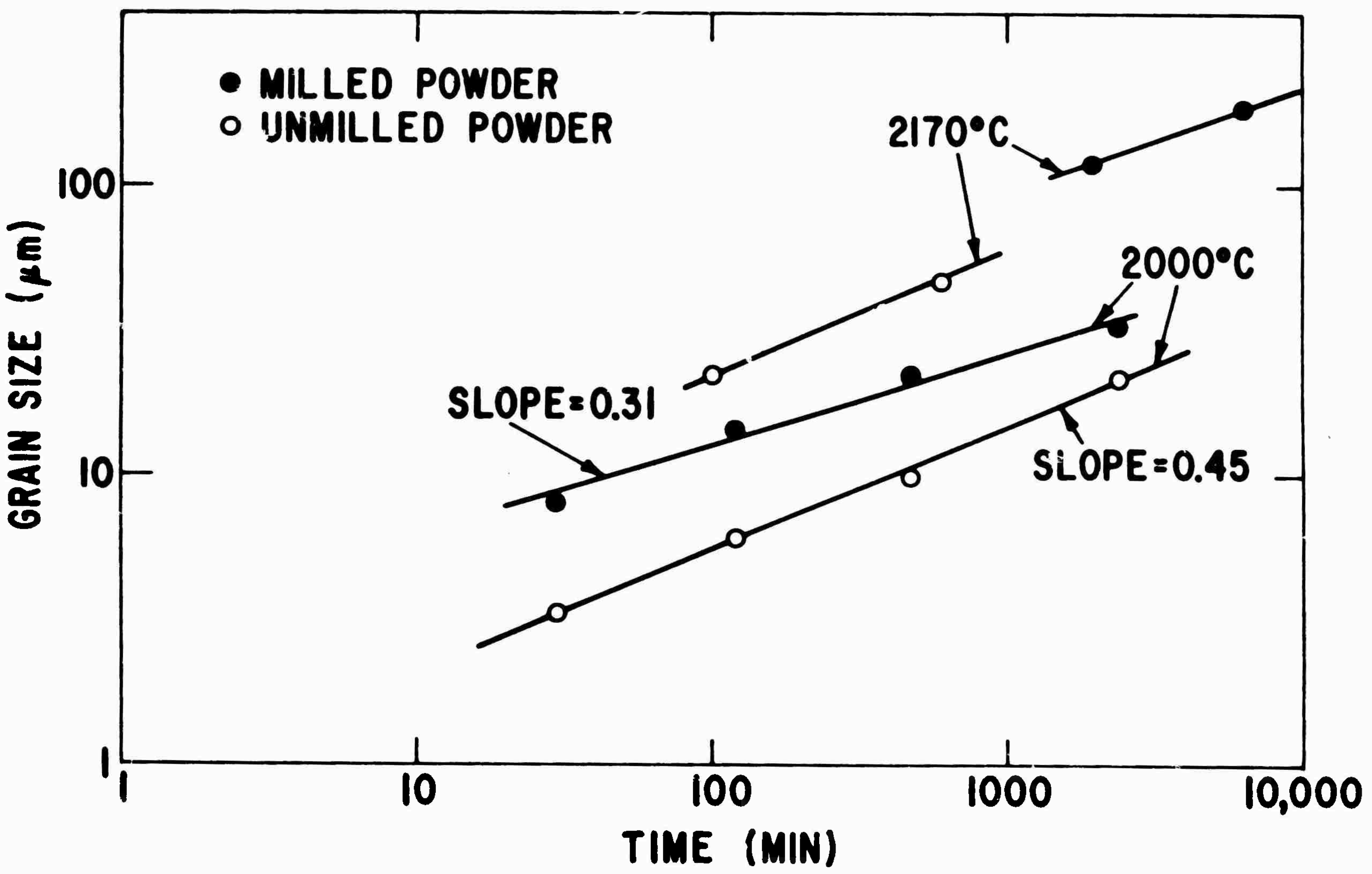

Figure 3. Grain size as a function of time and temperature for sintered Nd-doped Yttralox ceramic prepared from milled and unmilled powder.

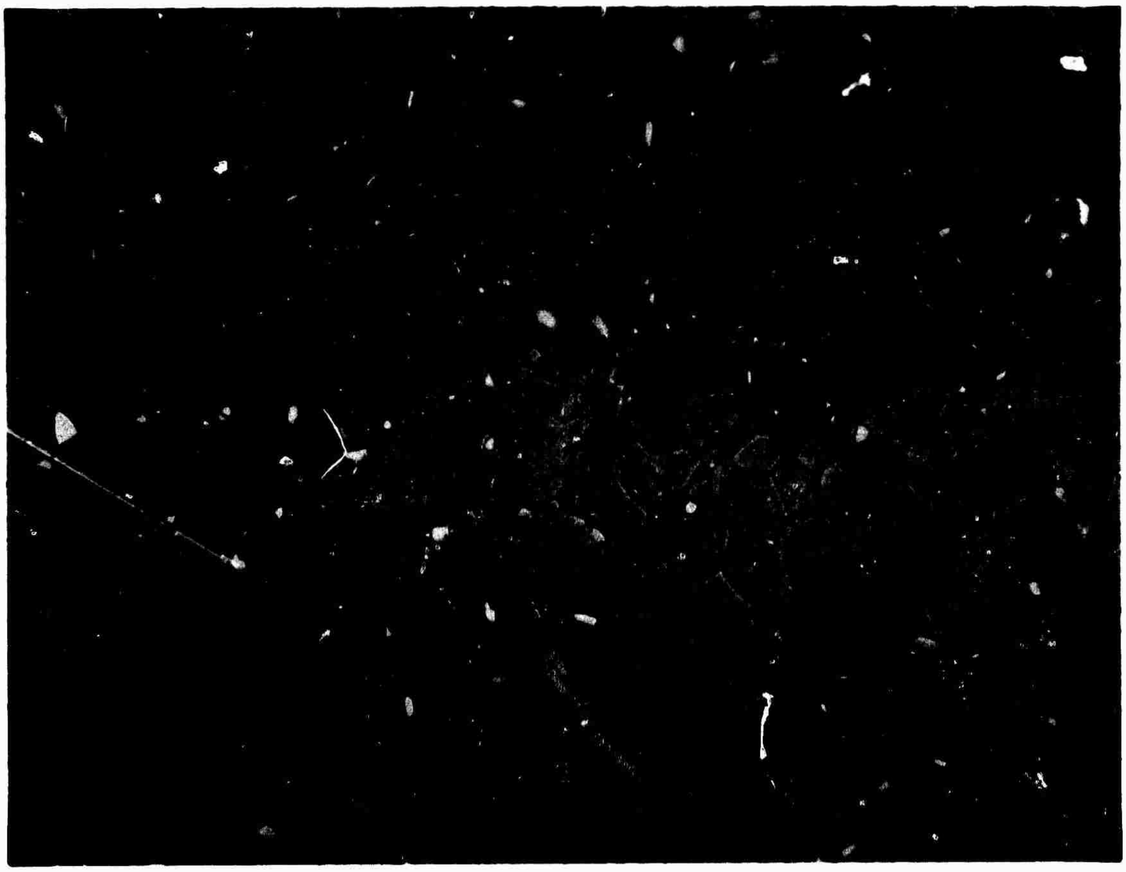

Figure 4. Sulfur-rich second phase in Nd-doped Yttralox ceramic sintered for 2 hours at 2000°C in dry H_2. Bright field illumination, X500.

Figure 5. Columnar grain formation in Nd-doped Yttralox ceramic sintered at 2220°C for 40 hours in dry H_2. Bright field illumination, X250.

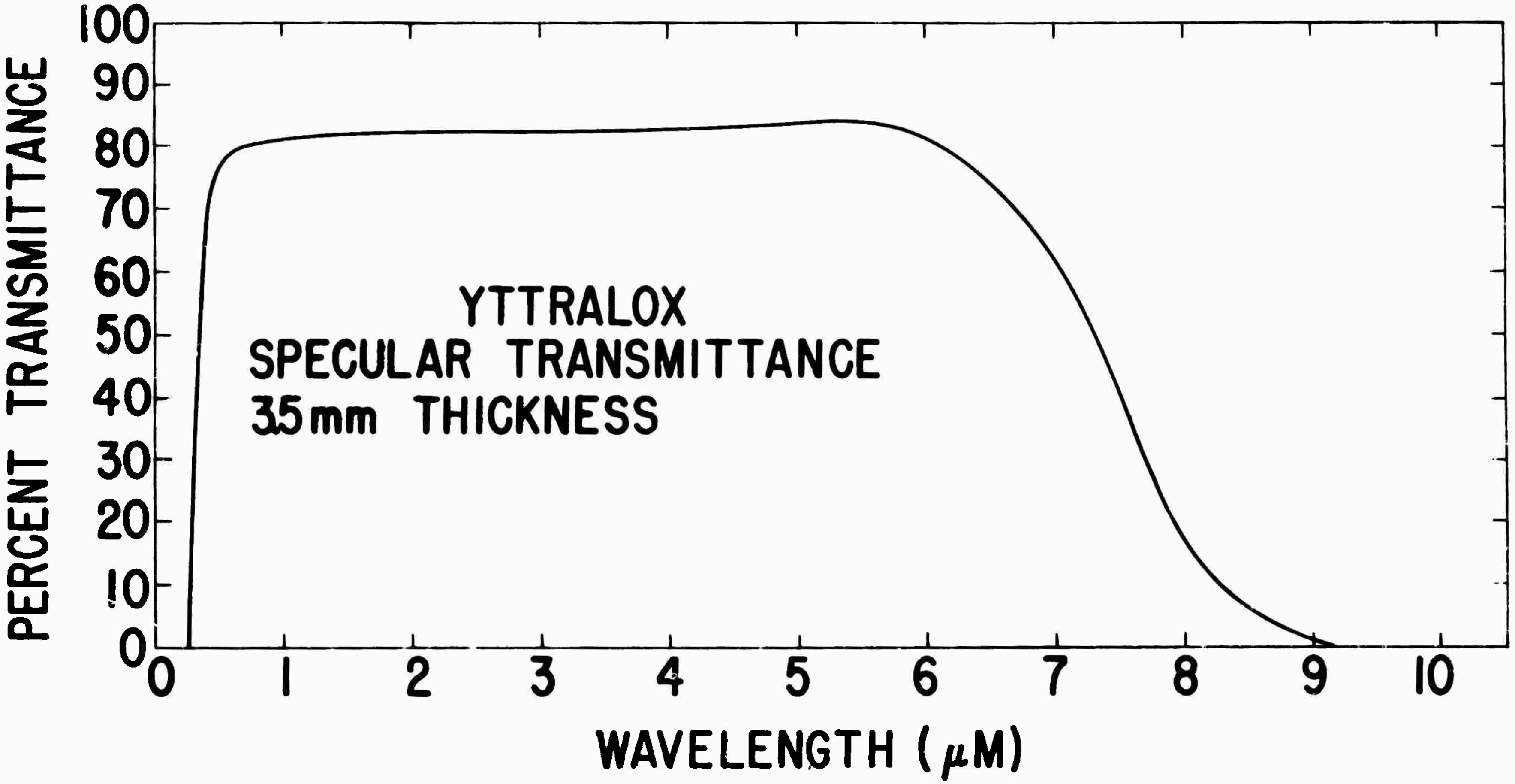

Figure 6. Specular transmittance of Yttralox ceramic (uncoated).