DEVELOPMENT, FABRICATION, AND DELIVERY OF NEODYMIUM DOPED YAG LASER RODS

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3rd Quarterly Report
Covering Period from September 18, 1965 to December 18, 1965

Contract No. NAS8-11865

National Aeronautics and Space Administration
George C. Marshall Space Flight Center
Huntsville, Alabama 35812

Contractor
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I. SUMMARY

One of the major problems initially associated with the growth of the larger size Nd:YAG crystals in the scaled-up stations has been satisfactorily overcome, namely, harmful diameter fluctuations can be avoided. Several of the crystals grown were of a size adequate for the fabrication of tube-free rods 1/4 inch in diameter by 2 to 3 inches long; however, persistent cracking in the bottom regions of the crystals has thus far prevented fabrication of 1/4-inch diameter rods longer than 1.5 - 1.8 inches. This cracking is thought to be initiated by surface irregularities produced in approximately the lower third of the crystals as a result of bubble attachment during growth. These bubbles have thus far usually appeared only in melts from which the largest diameter crystals were growing, and only after about 2 inches of growth had occurred. Experimental work is underway to determine how to suppress the formation of the bubbles.

Long-time (40 hr) annealing at 1850-1870°C caused no perceptible change in the appearance of the optical inhomogeneities associated with the tube region, both in Nd:YAG and undoped YAG rods. However, annealing a tube-free rod for only five

hours at 1850°C caused an apparent improvement of some 20% in passive beam divergence. Additional experimental work is planned in order to gain further knowledge about the reproducibility of the effect and to ascertain whether still further improvement in optical quality might be obtained via annealing.

II. EXPERIMENTAL RESULTS

A. Growth of Larger Size YAG Crystals

Major effort was concentrated on learning how best to operate the scaled-up growth stations for the consistent production of high quality Nd:YAG crystals large enough to yield laser rods 1/4-inch diameter by 1-1/2 inches long (or as long as possible). During the last quarter, operating personnel became sufficiently familiar with the new setups so that the diameter fluctuation described in the previous quarterly report was largely eliminated. As a result, a number of crystals were grown that were 3 to 3-1/2 inches long and 0.6 to 0.75 inch in diameter. During growth, as nearly as could be judged by eye under less-than-optimal viewing conditions, internal quality appeared excellent. However, as growth experience with this size range accumulated, a new problem was encountered with considerable regularity: these larger crystals seemed to be much more susceptible to cracking than were the smaller sizes. Figure 1 shows a typical example of the kind of failure that occurred. Normally, the cracks developed within about one minute after shutdown, although one crystal cracked during growth, shortly before it was scheduled to be pulled from the melt. When this cracking was first encountered, it was considered a thermal shock problem caused primarily by too rapid and uneven cooling of the crystal after it had been pulled from the melt. Therefore, the shutdown procedure was drastically revised so as to stretch out the initial cool-down over a much longer period of time. However, the cracking persisted. Closer examination of the crystals then revealed that the cracks always passed through, and probably originated at, a type of surface irregularity that was characteristic of this series of growth runs. These surface defects were roughly hemispherical depressions usually 1 to 3 mm in diameter, numbering perhaps two or three, and found only after 2 to 2.5 inches of growth. Their cause is known: after about 2 to 2.5 inches of growth had occurred, small bubbles were observed to begin accumulating in the melt. These were never seen floating free: most of them remained attached to the crucible wall, but occasionally one or two were seen attached to the crystal at the level of the growth interface. There they interfered with growth, resulting in the kind of depressions described above whose sharply irregular contours must surely make them prime suspects as crack initiators. The origin of these bubbles is a speculative matter at present; however, experimental work is in progress aimed at learning how to prevent their formation.

B. Annealing Studies

A setup was completed during the last quarter that has proved satisfactory for annealing specimens at temperatures as high as 1900 - 2000°C. The first thing studied was the effect of higher annealing temperatures on the tube region. Rods 1171Y and 1209Y were chosen for this purpose since they had already been fully evaluated as part of an earlier lower temperature (1380°C) annealing test (see 2nd Quarterly Report. There was no effect at this temperature). The rods were heated to 1840°C over a period of 5 hours, then held in a range 1850-1870°C (uncorrected pyrometer) for a total time of 40 hours. At the end of this time, the furnace was cooled 100°C/hr to 1400°C, then shut off. A nitrogen atmosphere was maintained in the furnace during the run. Visual observation of the cooled rods showed little change: no obvious color changes had occurred although the ground sides of the rods did seem smoother than before. Comparison of Twyman-Green photos taken before and after annealing showed no perceptible differences (see Table I). Next, a higher quality tube-free Nd:YAG rod (1493B; evaluation data presented in 2nd Quarterly Report) was annealed at 1850°C for 5 hours under a nitrogen atmosphere. The same cooling schedule was used as described above. The before and after annealing

Twyman-Green photographs and far field photographs appear identical (see Table I); however, the passive beam divergence seemed significantly improved (Figure 2). An attempt was made to reanneal this same rod at 1900°C; however, a temporary overshoot in temperature resulted in meltdown. Additional tests are planned as rods become available.

III. DISCUSSION OF RESULTS OBTAINED DURING THE QUARTER

Although cracks have occasionally occurred in Nd:YAG crystals during earlier periods of this program, they were usually caused by some kind of easily recognizable and fairly drastic change in some system parameter during the course of a run such as e.g., sudden furnace shut-down. The presently encountered crack problem has a more subtle origin and did not even become obvious until the largest size Nd:YAG crystals were being grown regularly and in an otherwise well-controlled fashion. However, the apparent correlation between the bubble attachment to the growing crystal and subsequent cracking seems quite reasonable at the present time, and steps are being taken to determine how best to eliminate the problem. The fact should be emphasized here that the bubble formation/cracking difficulties have not been encountered in the growth of smaller crystals, such as those required for the fabrication of 3 mm x 75 mm rods.

The work of this last quarter lays to rest any lingering hopes that the tubes might be eliminated from Nd:YAG rods by some kind of annealing step. However, in the case of the tube-free material, the improvement in passive beam divergence is considered sufficiently encouraging to warrant additional experimental work.

IV. PROGRESS RATE AND FUND EXPENDITURE

The progress rate and fund expenditures to date are shown in Figure 3. The broken lines project the anticipated rates through the final quarter to the end of the contract.

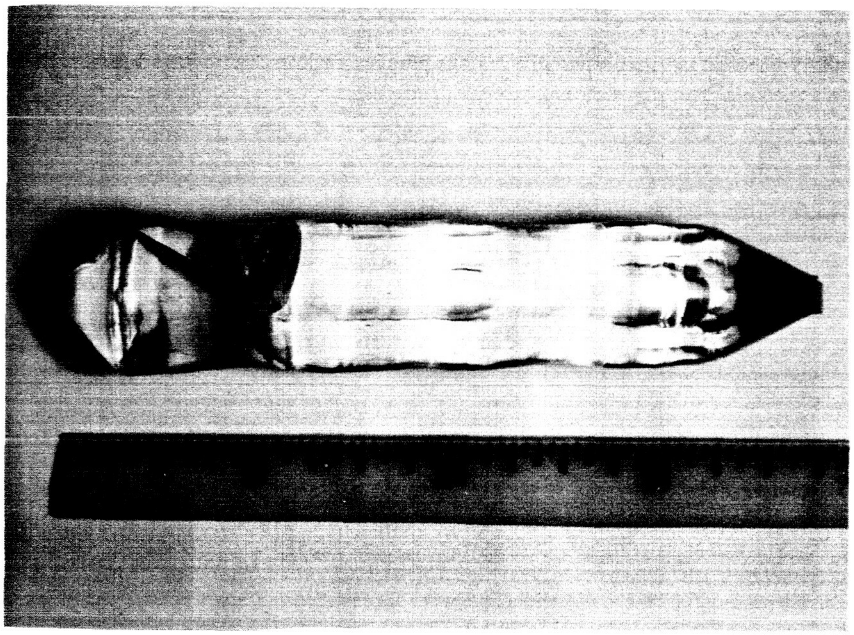


FIGURE 1 ILLUSTRATION OF CRACK PROBLEM IN LARGEST SIZE Nd:YAG CRYSTALS

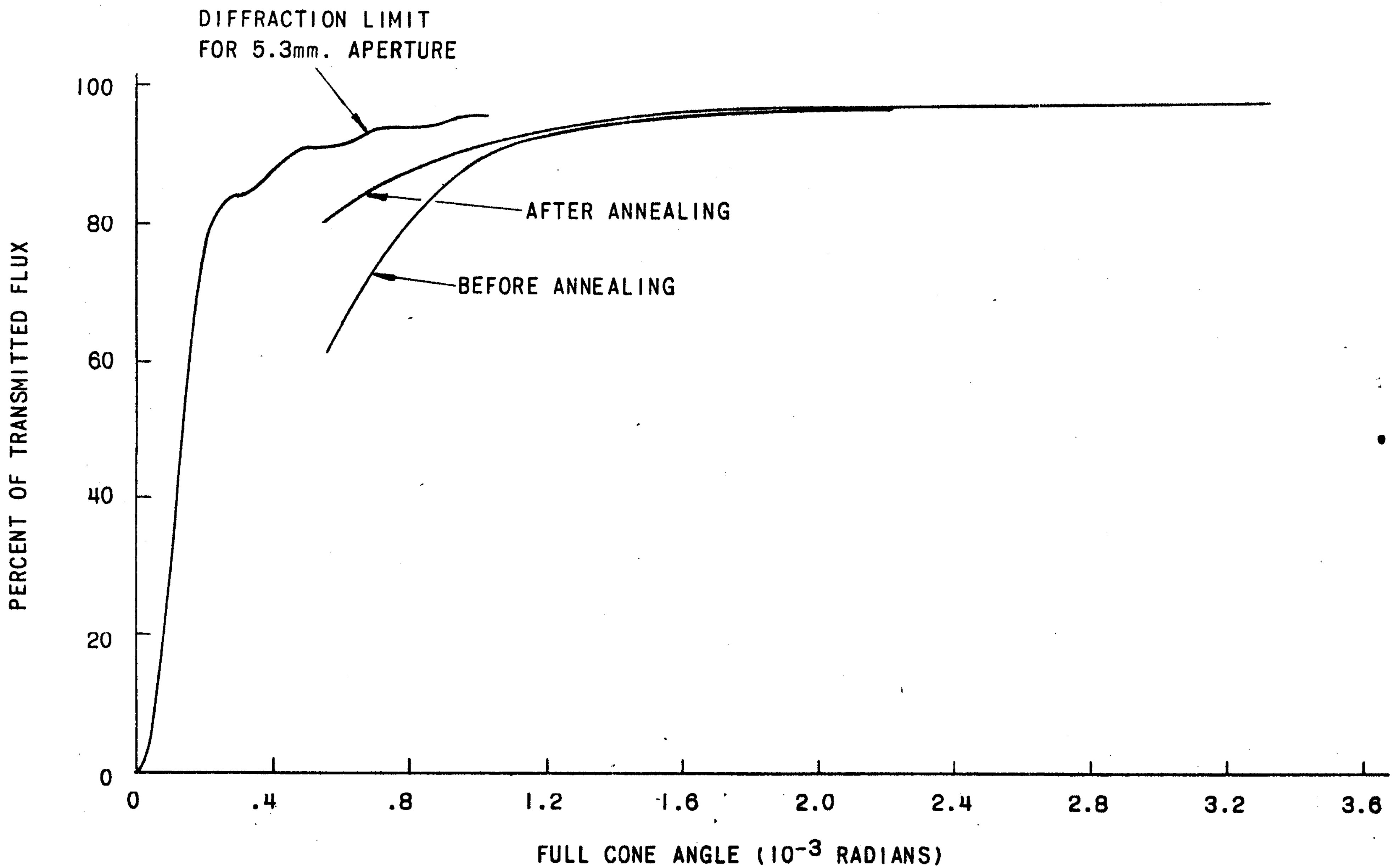
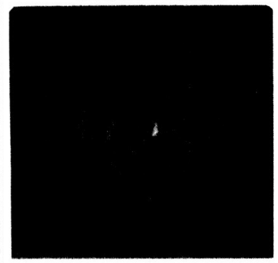
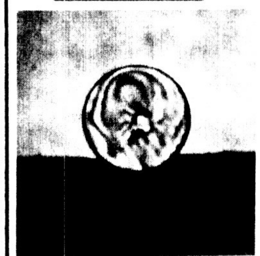
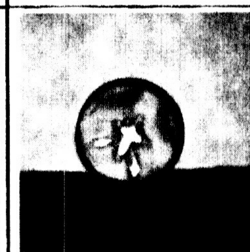
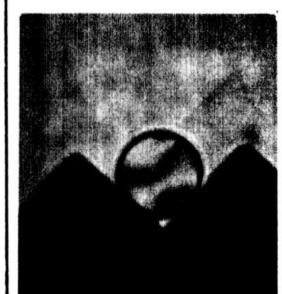
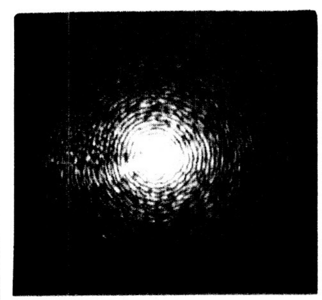
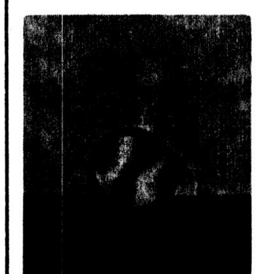
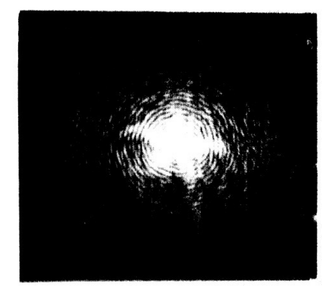
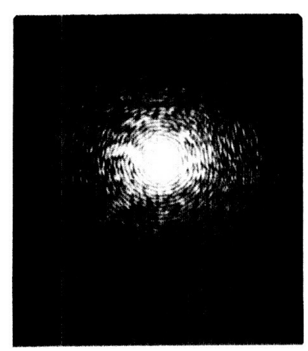


FIGURE 2. EFFECT OF ANNEALING AT 1850°C ON PASSIVE BEAM DIVERGENCE OF ROD 1493B.

TABLE I

EFFECTS OF ANNEALING YAG AT 1850°C-1870°C

<table>
<tr><th rowspan="3">ROD NO.</th><th rowspan="3">SIZE</th><th colspan="2">DESCRIPTION</th><th rowspan="3">TUBES</th><th colspan="4">RESULTS OF PASSIVE OPTICAL TESTS</th></tr>
<tr><th rowspan="2">DOPANT</th><th rowspan="2">ORIENTATION</th><th colspan="2">BEFORE ANNEALING</th><th colspan="2">AFTER ANNEALING</th></tr>
<tr><th>TWYMAN-GREEN PATTERNS</th><th>FAR FIELD PATTERNS</th><th>TWYMAN-GREEN PATTERNS</th><th>FAR FIELD PATTERNS</th></tr>
<tr><td>1171Y</td><td>7x42 mm.</td><td>1.3at% Nd</td><td>(111)</td><td>YES</td><td></td><td></td><td></td><td></td></tr>
<tr><td>1209Y</td><td>7x22 mm.</td><td>UNDOPED</td><td>(110)</td><td>YES</td><td></td><td></td><td></td><td></td></tr>
<tr><td>1493B</td><td>5x36.5 mm.</td><td>1.3at% Nd</td><td>(111)</td><td>NO</td><td></td><td></td><td></td><td></td></tr>
<tr><td colspan="5">BLANK FAR FIELD PATTERN (NO SAMPLE IN LIGHT PATH).</td><td></td><td></td><td></td><td></td></tr>
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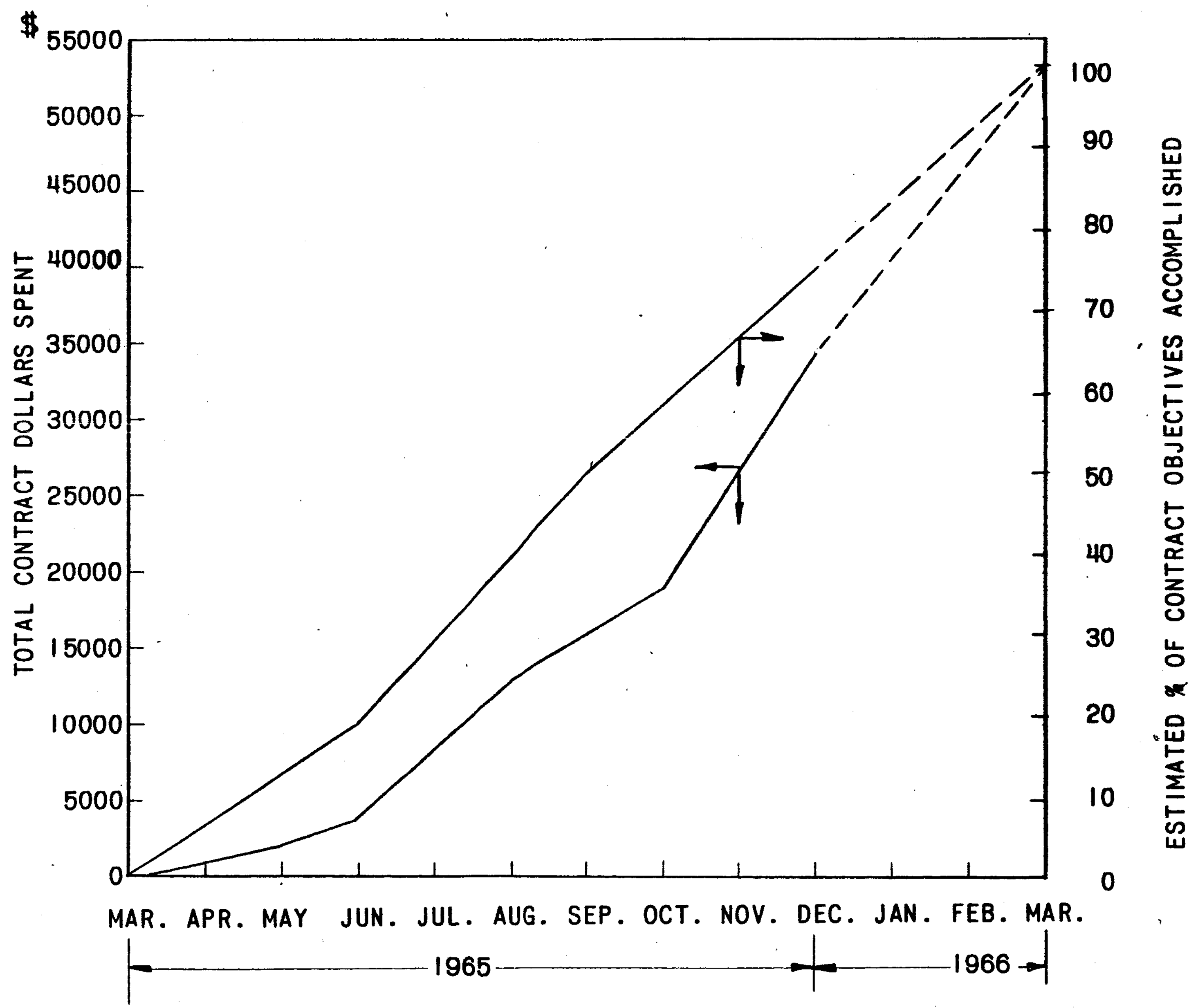

FIGURE 3 PROGRESS RATE AND FUND EXPENDITURES