

AD-A 024 442

RIA-76-U270



A024442

TECHNICAL
LIBRARY

Research and Development Technical Report

ECOM-74-0376-F

USADACS Technical Library



5 0712 01014401 1

LASER CARTRIDGE CONCEPT DEVELOPMENT STUDY

R. Wisnieff & D. Longo
United Technologies Corporation
Norden Division
Norwalk, Ct. 06856

April 1976

Final Report for Period 9 May 1974 - 15 November 1975

Distribution Statement

Approved for public release; distribution unlimited.

Prepared for

ECOM

US ARMY ELECTRONICS COMMAND FORT MONMOUTH, NEW JERSEY 07703

BEST AVAILABLE COPY

NOTICES

Disclaimers

The findings in this report are not to be construed as an official Department of the Army position, unless so designated by other authorized documents.

The citation of trade names and names of manufacturers in this report is not to be construed as official Government indorsement or approval of commercial products or services reference herein.

Disposition

Destroy this report when it is no longer needed.
Do not return it to the originator.

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER ECOM-74-0376-F	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) LASER CARTRIDGE CONCEPT DEVELOPMENT STUDY		5. TYPE OF REPORT & PERIOD COVERED FINAL 9 May 1974-15 Nov. 1975
		6. PERFORMING ORG. REPORT NUMBER 1249-R-0018
7. AUTHOR(s) R. Wisnieff D. Longo		8. CONTRACT OR GRANT NUMBER(s) DAAB07-74-C-0376
9. PERFORMING ORGANIZATION NAME AND ADDRESS United Technologies Corporation Norden Division Norwalk, CT 06856		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS 1S7-62703-DH93
11. CONTROLLING OFFICE NAME AND ADDRESS U.S. Army Electronics Command CS & TA Laboratory (DRSEL-CT-L) Fort Monmouth, N.J. 07703		12. REPORT DATE April 1976
		13. NUMBER OF PAGES 46
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		15. SECURITY CLASS. (of this report) Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE
16. DISTRIBUTION STATEMENT (of this Report) Approved for public release; distribution unlimited.		
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Laser Rangefinder Nd:YAG Laser Q-switch		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) A laser cartridge has been developed which incorporates a Nd:YAG laser rod, passive Q-switch and resonator mirror into an integral bonded assembly. The laser rod is 3 millimeters diameter and 15 or 16 millimeters long. Output energy is over 0.5 millijoule when pumped with 1 joule into a Xenon flash lamp.		

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE(When Data Entered)

Six cartridges have been built and delivered to the U.S. Army Electronics Command. Tests have been conducted over an operational temperature range, and life tests have been successfully run.

The key elements of the program were: development of a total process for manufacturing nickel-complex dyed polymethylmethacrylate (PMMA) with accurate control of optical density in a high purity damage resistant material; selection of an optical adhesive with necessary dimensional stability and strength while bonding materials with widely different temperature expansion coefficients; the development of alignment and assembly procedures for consistently obtaining good laser cartridges.

The program has demonstrated the feasibility and applicability of the ECOM laser cartridge concept.

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE(When Data Entered)

SUMMARY

This report describes the development of a Laser Cartridge accomplished by Norden Division of the United Technologies Corporation under U.S. Army Electronics Command Contract DAAB07-74-C-0376 (DA Project No. 1S7-62703-A-186-06-18), and through a parallel in-house effort by Norden and United Technologies Research Center (UTRC), both part of the United Technologies Corporation.

The laser cartridge successfully developed on the program consists of an integral bonded assembly of a Nd:YAG laser rod (3 mm diameter x 15 mm long), Q-switch, and a resonator mirror. The Q-switch is an organic nickel complex dye in a polymethylmethacrylate (PMMA) host. This unit has been tested and shown to satisfy the ECOM technical guidelines requirements. A beam divergence specification was not included in the technical guidelines because the beam divergence could not be determined a priori and it would have been beyond the scope of this program to attack such a problem. Complete static and limited operational tests were conducted over temperature, and a life test was run.

The key elements in the program were: the development of a total process for producing the PMMA/dye Q-switches with accurate control of optical density in a laser damage resistant high purity material; the test and selection of an optical quality adhesive with the necessary dimensional stability and strength which could permit the relative expansion and contraction of the joined materials without failure; the development of alignment and assembly techniques for consistently obtaining laser cartridges with predictable performance.

A total of 12 laser cartridges were fabricated during the program, 6 of which were delivered as required by the contract. Three of the delivered cartridges were specifically tailored to the ECOM mini-rangefinder program requirements, with the remaining three demonstrating significant stages in the development program.

Two alternate methods for cartridge manufacture were investigated. They were direct Q-switch dye sublimation on to the laser rod, and use of a Q-switch dyed adhesive. An adhesive to accept the dye was not found. The direct dye sublimation technique showed promise, but required substantially more research and development effort than was possible under the contract, and was therefore terminated.

The program has demonstrated that the ECOM laser cartridge concept is technically feasible. Working cartridges satisfying the technical guidelines have been manufactured which meet ECOM performance requirements in all areas except divergence. Recommendations for continued development are in the areas of performance optimization and investigation of techniques for reducing beam divergence without the need for collimating optics.

This program was conducted with continuing close liaison with the CS & TA Laboratory at ECOM. In particular, the technical guidance provided by Mr. Roland Wright, and the helpful suggestions from Dr. Rudolph Buser are acknowledged.

TABLE OF CONTENTS

<u>Section</u>	<u>Page</u>
1. INTRODUCTION	1
1.1 Program Technical Guidelines	1
1.2 Requirements for Cartridge Operation	1
1.3 Measurements	2
1.4 Report Content	3
2. LASER CARTRIDGE DEVELOPMENT	5
2.1 Basic Assembly	5
2.2 Q-Switch Host Selection	5
2.3 Q-Switch Preparation	6
2.4 Resonator Mirrors	10
2.5 Adhesive Selection	11
2.6 Cartridge Assembly	12
2.7 Performance Evaluation	15
3. ALTERNATES INVESTIGATED	21
3.1 Direct Dye Sublimation	21
3.2 Adhesive Host Q-Switch	21
4. CONCLUSIONS AND RECOMMENDATIONS	23
4.1 Conclusions	23
4.2 Recommendations	24
 <u>APPENDIX</u>	
A LASER ROD REQUIREMENTS	25
B VAPOR DEPOSITION OF Q-SWITCH LASER DYE	
C PHYSICAL DATA ON ADHESIVES STUDIED	39

LIST OF ILLUSTRATIONS

<u>Figure</u>		<u>Page</u>
1	Basic Laser Cartridge Configuration	5
2	Optical Density and Percent Transmission vs Dye Density	9
3.	Mirror/Q-Switch Bonding Fixture	13
4.	Cartridge Fabrication Alignment Fixture	14
5.	Typical Beam Pattern and Pulse Duration	16

SECTION 1 INTRODUCTION

1.1 Program Technical Guidelines

The basic goal of the program is outlined in the technical guidelines prepared by C.S. and T.A. Laboratory of ECOM dated September 1973. The objective is the development of an integrated, prealigned Nd:YAG resonator assembly containing the laser rod, passive Q-switch and resonator mirrors. The particulars of the laser performance are:

- a. laser output energy 0.5 to 3.0 millijoules with 1 to 2 joules into flash lamp
- b. Nd:YAG rod size - 3 millimeters in diameter by 15 millimeters long
- c. operating life 10,000 shots when pulsed at 1 cycle per second

The cartridge assembly is operated in an essentially elliptical cavity made of polymethylmethacrylate (PMMA) with the cartridge and flash lamp in conjugate positions. The PMMA cavity is overcoated with high reflectance material to assure maximum internal reflection of the flash lamp energy and optimum coupling between the flash lamp and the Nd:YAG rod. The Xenon flash lamp selected by ECOM has an arc length of 15 mm and an inside diameter of 1 mm and an outside diameter of 2 mm. The operating temperature range is -50°F to +160°F.

The contract deliverable items are:

- a. 6 laser cartridges representative of the contract development work
- b. monthly progress, interim and final technical reports

1.2 Requirements for Cartridge Operation

Operation of the laser cartridge requires peripheral assemblies such as the cavity, flash lamp energy storage and trigger circuits. The basic performance of the laser depends upon the operation of each of these elements as well as the laser rod itself. For example, the energy storage element may not deliver its full energy to the flash lamp. A typical

electrolytic capacitor storage system delivers approximately 2/3 of its input energy to the flash lamp. The standard ECOM mini-rangefinder storage network employs a 13 microfarad capacitor charged to a maximum of 500 volts and will deliver a maximum flashlamp input energy of

$$\begin{aligned} E &= 1/2 \eta CV^2 \\ &= 1/2 \times 2/3 \times 13 \times 10^{-6} \times (500)^2 \\ &= 1.08 \text{ joules} \end{aligned}$$

where: E = energy output in joules
η = capacitor efficiency
C = capacity in farads
V = voltage in volts

This value is at the lower end of the specified input energy range. Since this will be the energy storage element used by ECOM to excite cartridges fabricated on the program, the principal effort was concentrated on elements with power input (and hence power output) in the lower end of the technical guidelines range.

The basic aim of the program was to develop the cartridge fabrication techniques. Therefore, effort was not expended in optimizing the other elements of the laser system. Over-all laser efficiency might be improved in many areas. For example, in the experimental system the flash lamp energy passed through five major changes in index of refraction and corresponding reflection losses at each interface. If an index matching medium were introduced between the flash lamp and the cavity and between the cavity and the Nd:YAG rod the number of major index changes could be reduced to one (arc to flash lamp housing) with improved overall laser efficiency. Investigations of this nature were deferred to future contracts to permit concentration on the cartridge fabrication procedures.

1.3 Measurements

The technical guidelines outline a series of cartridge performance tests including output energy, beam divergence, pulse duration and operation in the military environment. All measurements on cartridge efficiency were made relating energy output from the cartridge assembly to energy input to the storage capacitor. The first cartridges assembled were measured using a cavity overcoated with vapor deposited silver. At the suggestion of R. Wright of ECOM, a cavity was then made with the outer surface coated with a highly diffuse white paint which resulted in an observable increase

(10-20 percent) in conversion efficiency, and this cavity was used for all measurements thereafter. All cartridges delivered on the program were measured with the diffuse cavity. However, because of the input energy limitations outlined above, the conversion efficiencies are relative rather than the absolute maximum achievable with the given cartridge.

Divergence measurements were made with a silicon target vidicon viewing the far field laser beam pattern. The vidicon target surface acts as an image integrator and accurately stores the nanosecond duration radiation pattern. The pattern is then read out by the electron beam scan and stored in a video tape recorder. The signal is later played back from the recorder for detailed pattern analysis.

The primary factor in performance in military environment is operation over the temperature range. The range of -50 to +160°F is more than can currently be achieved with available electrolytic high voltage storage capacitors. Shock and vibration performance are largely dependent upon the way in which the cartridge is mounted into the cavity and into the remainder of the transmitter assembly. Total environmental evaluation of the cartridge, therefore, depends on the specific implementation in the using system.

1.4 Report Content

This report describes the basic laser cartridge assembly technique and the materials used for the Q-switch and bonding. A detailed description of the processes developed to fabricate the dyed PMMA Q-switches, and to align and assemble the cartridges is included. Results of performance measurements are given.

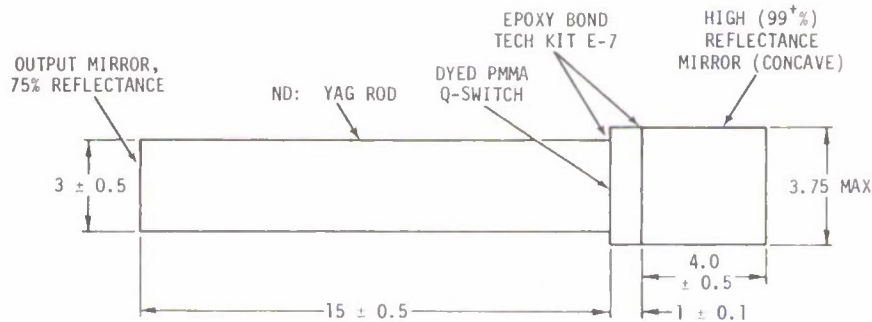
Several alternate cartridge manufacturing methods were investigated and found unsatisfactory for various reasons. These are briefly described in the body of the text. An appendix covers the details of a United Technologies Research Center (UTRC) investigation of vapor deposition of Q-switch laser dye.

Finally, recommendations are presented for future development effort considered necessary.

SECTION 2 LASER CARTRIDGE DEVELOPMENT

2.1 Basic Assembly

After a preliminary investigation of several alternate techniques (as outlined in Section 3) an assembly consisting of the Nd:YAG rod, a passive Q-switch of the nickel complex dye in a plastic disk and a high reflectance mirror was selected. All components are bonded together with an adhesive highly transparent in the 1.06 micron region. The adhesive is sufficiently rigid to maintain the high degree of alignment required and yet have enough flexibility to permit the differential thermal expansion between the various components. Figure 1 is an outline of the selected assembly.



ALL DIMENSIONS IN MILLIMETERS

Figure 1. Basic Laser Cartridge Configuration

2.2 Q-Switch Host Selection

Two solid plastic materials have been successfully used as a host for the nickel complex dye. These are polymethylmethacrylate and cellulose acetate. Table 1 summarizes the relevant physical properties of the two materials. From the table it can be seen that both PMMA and cellulose acetate have the same maximum service temperature of 170°F but PMMA has a lower coefficient of thermal expansion, a higher flexure strength, and lower water absorption. The PMMA is also harder (and hence more rigid) than cellulose acetate, being measured on the M hardness scale rather than the R scale which is used for rubber like materials. The polymerized PMMA is subject to strain when included in a bonded assembly while the amorphous cellulose acetate is not. This is a disadvantage for PMMA. However, the superior mechanical properties of the

PMMA indicate the selection of this material for a bonded cartridge assembly in which the Q-switch host must act as a mechanical coupling element. Also the technology for bonding PMMA to optical components has been well developed while cellulose acetate requires the development of bonding techniques. It was, therefore, decided to concentrate on the PMMA host Q-switch configuration.

Table 1. Relevant Physical Properties of PMMA and Cellulose Acetate

<u>Property</u>	<u>PMMA</u>	<u>Cellulose Acetate</u>	<u>Units</u>
Coefficient of Thermal Expansion	8×10^{-5}	16×10^{-5}	/°C
Flexure Strength	14×10^3	9×10^3	PSI
Water Absorption	0.3	2	%/24 hrs.
Maximum Service Temperature	170	170	°F
Rockwell Hardness	M100	R100	-
Structure	Polymerized	Amorphous	-

2.3 Q-Switch Preparation

The PMMA host Q-switch is the heart of the laser cartridge. The preparation of the Q-switch material with controlled optical density and free of impurities represented a major portion of the laser cartridge development effort and is, therefore, presented in detail. The preparation required thorough solution of the nickel complex dye in solvent, dehydration of the PMMA pellets, combining the pellets with the dye solution to form a uniform mixture, complete removal of solvent from the PMMA dye solution, and the formation of the Q-switch disks from the PMMA/dye material. Each of these steps warrants detailed discussion, which follows.

2.3.1 Preparation of Dye/PMMA Solution

Pure bis-(4-dimethylaminodithiobenzil) nickel [nickel complex] dye is prepared by recrystallization of commercially available (Kodak) dye. The recrystallized dye is dissolved in chromatographic quality methylene chloride. The solute solvent ratio is typically 1 milligram per 100 milliliters. The solution is agitated periodically over a 16-18 hour period to ensure that the dye is completely dissolved.

The PMMA used as the host is optical quality PMMA pellets (such as Rohm and Haas V-811). The commercial pellets are dehydrated by baking in an air circulating oven at 110°C for a period of at least 5 hours. The dehydrated pellets are then dissolved in methylene chloride by adding the required amount of filtered dye methylene chloride solution to a measured amount of the dehydrated PMMA pellets and then adding additional methylene chloride to obtain a PMMA to solvent ratio of 1 gram to 10 milliliters. The filter consists of a 1.5 microns Millipore pre-filter and a 0.2 micron final filter. The dissolved PMMA dye solution is again passed through the filter to remove any impurities present in the PMMA. The filtered solution is placed in a wide-mouthed scrupulously cleaned ceramic crucible and covered to prevent contamination or formation of a skinned surface by rapid solvent evaporation.

2.3.2 Solvent Removal and Q-Switch Disk Formation

The covered crucible containing the dye PMMA solution is placed in an air circulating oven at 40°C for approximately 24 hours. During this period the bulk of the solvent is removed and the material solidifies in the crucible. The crucible is then transferred to a vacuum oven and the covers are removed. The oven pressure is reduced to 100 microns Hg with the sample temperature held at 40°C. The oven temperature is slowly increased while maintaining a minimum vacuum of 100 microns until a temperature of 130°C is reached. The temperature is then maintained at 130°C until the pressure falls to 60 microns. At this point at least 99.9 percent of the solvent has been removed and the PMMA/dye mixture is ready for pressing into Q-switch disks.

The PMMA/dye material is removed from the crucible by thermal shock and transferred to a 1-1/4 inch diameter mold press with polished stainless steel cylinder and pistons. The press is heated to 130°C and the pressure increased to 2000 pounds per square inch. The temperature is increased to 170°C and maintained for 20 minutes. The press and disk are permitted to cool to room temperature while the pressure decays. The 1-1/4" wafer, approximately 1 millimeter thick, is removed from the press by thermal shock and annealed in an air circulating oven at 85°C for eight hours and permitted to slowly return to room temperature over an additional eight hour period.

The wafer is then inspected microscopically to select areas free of defects from which the 3.75 millimeter diameter Q-switch disks can be cut. Four or 5 such areas are found in a typical wafer. These areas are tested for Q-switch action in a 5 x 30 millimeter Nd:YAG laser producing outputs in the order of 10 millijoules and examined for damage. If found damage free (≈ 95 percent for material prepared as outlined above) the 3.75 millimeter disks are cut from the selected areas with a water cooled diamond core-cutter. These disks are examined for mechanical damage and if acceptable are used for cartridge assembly.

The procedure outlined above evolved as the result of preparing 108 Q-switch sample wafers. In the source of the technique development many problems encountered were solved by changes in processing. Table 2 is a summary of this development. The inclusions were the most serious problem since any particle, even as small as one wave length, provided a site for damage. Absolute cleanliness is a prime requisite for Q-switch fabrication.

2.3.3 Optical Density

Optical Density of the final Q-switch material is a function of the amount of nickel complex dye per square centimeter of switch cross section. Figure 2 relates the optical density and percent transmission (at 1.06 microns) to the dye density in micrograms per square centimeter. The optical density is linearly related to the dye density.

Table 2. Q-Switch Development

<u>Problem</u>	<u>Solution</u>
Inclusions	Purity of ingredients, filtration, laminar flow bench processing
Surface Cracking	Pressing technique changes, annealing, cleaning techniques
Veiling (cloudy inclusions)	Solvent purity, pressure/temp.
Air Bubbles/Surface Quality	Solvent removal technique, pressing
Discoloration (depolymerization)	Limit maximum temperature of PMMA processing

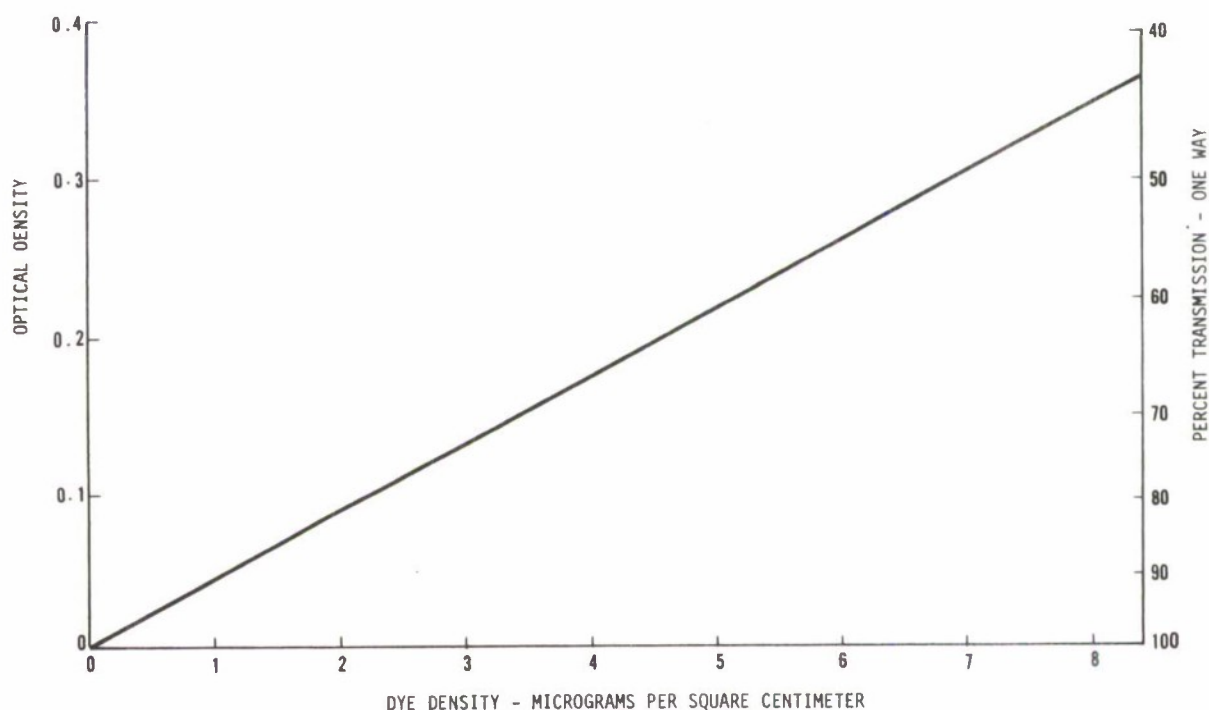


Figure 2. Optical Density and Percent Transmission vs Dye Density

2.4 Resonator Mirrors

A computer analysis of resonator performance developed by United Technologies Research Center was used to determine optimum resonator parameters for the 3 x 15 millimeter Nd:YAG laser. The result of this analysis indicated that for best laser efficiency the laser output mirror should have a transmission of approximately 75 percent. Accordingly, the laser rod was specified (Appendix A) with the output mirror coated on one end of the laser rod. Since the other end of the rod (index ≈ 1.8) is bonded with an adhesive with an index of refraction of approximately 1.5 and the standing wave within the resonator approaches the surface at normal incidence, the reflection loss at the bond surface for an uncoated rod is

$$r = \left(\frac{n_2 - n_1}{n_2 + n_1} \right)^2 = \left(\frac{1.8 - 1.5}{1.8 + 1.5} \right)^2 = \left(\frac{.3}{3.3} \right)^2 \approx 8 \times 10^{-3}$$

Therefore, because of the low reflection loss, no coating is applied to the bonded end of the YAG rod.

The second mirror of the resonator is the high reflectance mirror bonded to the rear of the Q-switch assembly. To minimize the number of interfaces in the optical path and provide protection for the mirror surface, the bond is made directly to the mirror surface. This decision was based on results of discussions with mirror vendors (e.g. Laser Optics Inc.) that indicated that dielectric mirror coatings produced by electron beam deposition are sufficiently well bonded to the glass substrate to allow use as the bonding interface. In addition, tests at Norden confirmed the suitability of the mirror coating as the bond interface. The mirror coating was deposited to provide a reflection in excess of 99.2 percent at 1.06 microns when bonded with the adhesive with an index of 1.5.

The mirror has an outside diameter of 3.75 millimeters and a minimum useful mirror diameter 3.25 millimeters. These dimensions allow normal optical fabrication techniques (bevel etc.) around the mirror edge and still provide a minimum of 0.25 millimeter for alignment with the 3-millimeter rod. The length of the mirror substrate is 4 millimeters. This provides an adequate length to permit holding the mirror with standard optical fixtures for cartridge alignment. For a production cartridge the use of special fixtures would permit the use of a much shorter substrate. This would appreciably reduce the mass cantilevered from the YAG rod and

reduce the stress on the bonds in a military shock and vibration environment. The mirror substrate is borosilicate - crown glass (BK-7) which has a thermal expansion coefficient of approximately 4×10^{-6} per degree Fahrenheit which closely matches the YAG rod. Thus, a bond satisfactory for the differential thermal expansion between the YAG and the PMMA will also apply to the mirror substrate to PMMA interface.

2.5 Adhesive Selection

Many representative optical adhesives were evaluated for bonding the Nd:YAG to the PMMA. Table 3 lists the commercial names of the selected adhesives and their generic types. Physical data for these adhesives are given in Appendix C. The basic requirement for the adhesive is the ability to maintain the alignment required for laser action while permitting the differential thermal expansion between the YAG and PMMA. The expansion coefficient of the PMMA is approximately ten times that of the YAG. Test bonds were made by first bonding commercial PMMA to standard glasses such as BK-7 which have an expansion coefficient similar to the YAG. The samples were then cycled over the temperature limit. Bond integrity and stability were evaluated. Mechanical strength of samples was determined by static loading tests. Optical alignment and stability were checked by observation of Newton's rings. The hard two-part epoxies and the cyanoacrylic formed excellent bonds but when cycled over the temperature range the thermal stress resulted in failure of bonded material in the vicinity of the bond. The elastomer bonds (such as RTV) withstood the temperature cycle but gave bonds of questionable stability. Two adhesives provided good stability and temperature performance. These were Hysol experimental adhesive 9419 and Techkits E-7. After preliminary tests with the 9419 it was learned that it had been discontinued and would not be available for production. No further testing was performed with this adhesive. The Techkits E-7 is a flexible two part epoxy in which the flexibility is controlled by the ratio of resin to activator. It was determined that a mixture of 40 parts (by weight) of resin to 60 parts activator resulted in the best cartridge bond. The adhesive mixture is first outgassed under vacuum for one hour at approximately 34°C. The bond is cured for 4 hours at 55°C under a pressure of 2100 grams per square centimeter (≈ 30 psi). Experimental bonds were subjected to a sequence of temperature cycles and maintained both bond integrity and optical alignment. The results of tests with two sample batches of Techkits E-7, produced about six months apart, were the same, indicating vendor batch-to-batch uniformity. The Techkits E-7 adhesive was, therefore, used for the cartridge assembly.

Table 3. Optical Adhesives Investigated

<u>TRADE NAME</u>	<u>FORMULATION</u>
Hysol 0151	Two-part epoxy
TRA-CON BB-2114 } TRA-CON BB-2129 } TRA-CON BB-2130 }	Two-part epoxy
Kodak HE-10	Modified two-part epoxy
Techkits E-7	Flexible two-part epoxy
Summers M-62	Two-part synthetic polyester
GE RTV-602	Two-part silicon
Dow Corning R-63-489	Two-part silicone elastomer
Hysol EA-9419	Two-part - epoxy/urethane and liquid amine
Loctite 04-E	Single component ethyl cyanoacrylic
Opticon UV-57	Single component photosensitive resin
Summers UV-71	Single component photosensitive resin

2.6 Cartridge Assembly

2.6.1 Mirror Q-Switch Subassembly

The cartridge is assembled by bonding the components previously described (rod, Q-switch and mirror) into an integrated unit. This bonding is accomplished in two steps. In the first step the 3.75 millimeter diameter Q-switch disk is bonded to the high reflectance mirror. The assembly is made in a laminar flow bench to maintain cleanliness. The mirror and the Q-switch are placed in precision cups of an alignment fixture and the adhesive is applied to the exposed surface of the Q-switch. Surface tension causes the adhesive to form a spherical surface such that when the Q-switch and mirror are brought into contact air is excluded from the bond. Figure 3 is a sketch of the fixture. The cups containing the Q-switch and mirror are mounted in chucks which are constrained to a common axis by the fixture. Thus, the Q-switch with adhesive is pressed along the fixture axis to the mirror surface.

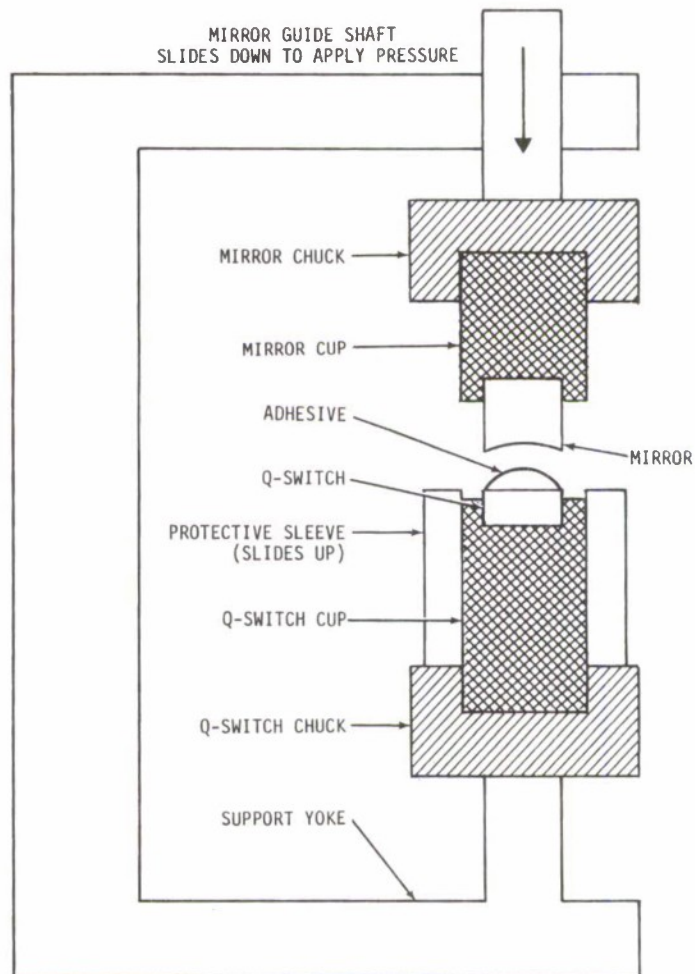


Figure 3. Mirror/Q-Switch Bonding Fixture

The mass of the fixture is such that with the fixture axis vertical the proper pressure is applied to the bond. A protective sleeve over the cups prevents any impurity from entering the bond area. The fixture is then placed in an air circulating oven and the adhesive cured as previously described. After curing, the bonded subassembly is removed from the fixture and inspected. It should be noted that precision optical alignment of the Q-switch with the mirror axis is not required since the Q-switch is index matched by the adhesive to both the reflecting mirror and the Nd:YAG rod.

2.6.2 Cartridge Assembly and Alignment

The final step in cartridge fabrication is the precision optical alignment and bonding of the laser rod to the mirror/Q-switch subassembly. This is accomplished on an optical table schematically shown in Figure 4. The basic technique uses a collimated beam from the He-Ne laser to align the output and high reflectance mirrors of the resonator. The beam expander reduces the approximate 1 milliradian divergence from the laser by a factor of 10 to produce a more nearly collimated incident beam. The beam-splitter permits examination of the resonator interference pattern on the viewing screen. The eight power beam expander enlarges the beam on the rear projection viewing screen. The viewing screen is placed adjacent to the mirror/Q-switch support base to permit the operator to adjust the two-axis vernier controlling the axis of the mirror/Q-switch to achieve resonator alignment. The high reflectance mirror is spherical so that concentricity of the interference fringes between the reflection from the output and high reflectance mirrors will indicate resonator alignment. The number of fringes in the pattern is limited by the 3 millimeter diameter of the output mirror on the rod and the radius of curvature of the high reflectance mirror. For example, mirrors of 1, 2, and 5 meters radius were used in fabricating experimental cartridges with the corresponding interference pattern containing 4, 2 and less than 1 fringe respectively. Accordingly the 1 and 2 meter mirrors were relatively easy to align while the 5 meter radius mirror was extremely difficult. The 2 meter mirror represented a compromise between ease of alignment and maximizing mode volume for increased output energy. Measurements of beam divergence showed little difference between cartridges fabricated with the 2 and 5 meter radius mirrors.

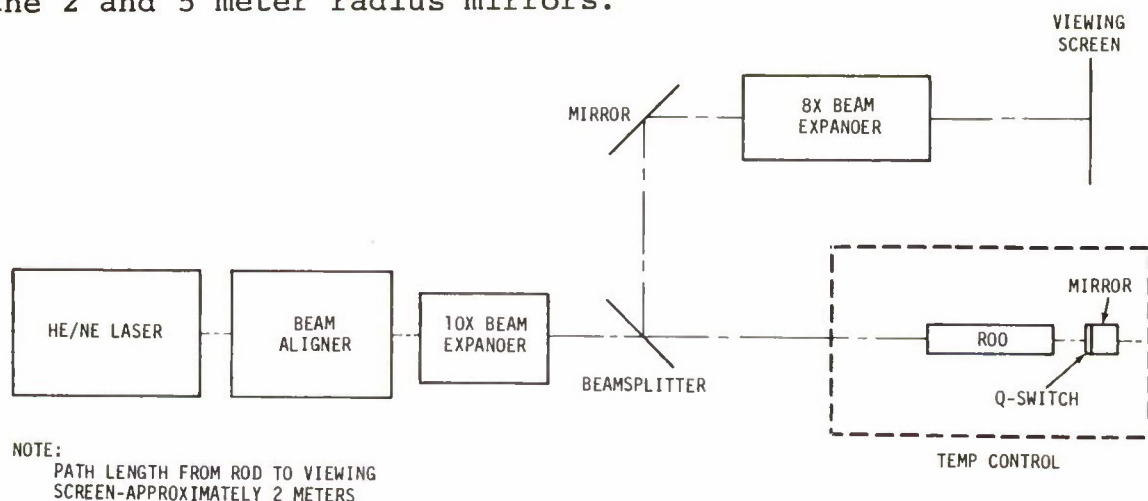


Figure 4. Cartridge Fabrication Alignment Fixture

The holder for the mirror/Q-switch subassembly is mounted on a precision slide capable of translation parallel to the optical (laser) axis. Therefore the output mirror on the Nd:YAG rod is first aligned normal to the He-Ne laser beam by having the reflected beam coincident with the incident beam. The mirror/Q-switch assembly is then aligned to achieve concentricity of resonator interference fringes on the viewing screen. A drop of purified water may be used as an index matching fluid to assist in this alignment. After alignment a final mirror cleaning is performed in place.

The entire laser assembly is contained within an insulated enclosure with a small aperture for coupling the optical beam. The entire enclosure is preheated to 55°C (the curing temperature of the adhesive) and if necessary, the mirror/Q-switch assembly readjusted to compensate for thermal distortion of the mounting structure and to reestablish the concentric interference pattern. A drop of the outgassed adhesive is then applied to the end of the Nd:YAG rod and the mirror/Q-switch mount translated toward the rod until contact is made with the adhesive. A force of 150 grams is applied to the assembly to provide the correct curing pressure. The pattern on the viewing screen is observed to make certain that alignment has not been disturbed and if necessary the mirror/Q-switch is immediately realigned. This realignment must be completed within 2 minutes of adhesive application. The adhesive is cured for 4 hours during which the alignment must not be disturbed. After curing, the mirror is released from its mount and the cartridge assembly allowed to cool to room temperature. This completes the cartridge fabrication.

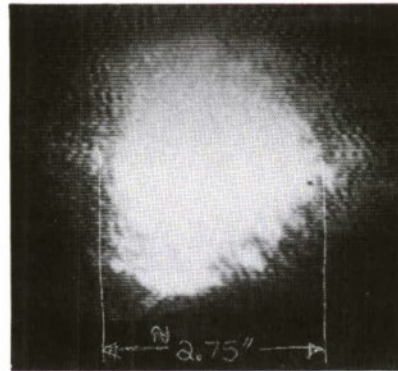
It was observed that the YAG rod to Q-switch bond resulted in a small meniscus at the rod end approximately 1 mm in length which interfered with insertion into the pump cavity. Therefore, the later cartridges were made with 16 mm rods to allow for the adhesive meniscus.

2.7 Performance Evaluation

Cartridges were evaluated for output energy, pulse duration, divergence, performance over temperature, and operating life.

The pulse duration of all cartridges was in the 10-12 nanosecond range at the half power point with a waveform as shown in Figure 5. This waveform is typical of all cartridges.

CARTRIDGE NUMBER 4 - 3 x 15 MILLIMETER ROD
HIGH REFLECTANCE MIRROR RADIUS - 1 METER
500 VOLT INPUT TO STORAGE UNIT
≈ 1 MILLIJOULE OUTPUT ENERGY
DISTANCE FROM LASER TO SCREEN = 260 INCHES

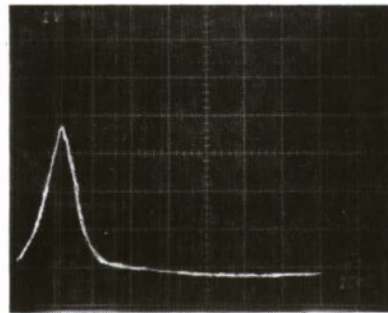


$$\theta = \frac{2.75}{260}$$

$$= 10.6 \text{ MILLI-RADIANS}$$

← 10.6 MR → DIVERGENCE

VIDICON IMAGE OF BEAM PATTERN



VERTICAL SCALE
1 VOLT/DIVISION
HORIZONTAL SCALE
10 NANOSECONDS/
DIVISION

PULSE DURATION

Figure 5. Typical Beam Pattern and Pulse Duration

The divergence was measured with a silicon target vidicon viewing the image of the laser energy on a screen at least 5 meters from the output mirror. No beam expanders or other optics were employed. All cartridges exhibited a pattern width in the range of 10 to 15 milliradians. Figure 5 shows a typical divergence pattern as viewed by the T.V. system.

The use of mirror radii from 1 to 5 meters made no discernible change in the pattern. It does not appear that the divergence of the cartridge radiation can be readily controlled by changes of Q-switch density (and corresponding output power) or mirror radius. Possible approaches for obtaining control of this factor are outlined in the recommendations of Section 4.

The output energy was measured with a Scientech Model 362 power energy meter operated on the 1 or 3 millijoule scale. All cartridges with threshold voltage below 500 volts (into the standard ECOM storage network of 13 microfarads and 40 microhenries) produced an output power in the range 0.5 to 1.0 millijoule. Units with threshold voltage in excess of 500 volts provided output power in the 1-2 millijoule range. The output power of each cartridge is tabulated in Table 4.

Cartridge number 6 was used for a combined temperature life test. The cartridge in its cavity was placed in a temperature controlled chamber with the output mirror facing the energy meter through an antireflection coated window. During these temperature-life tests, the energy meter was used as a qualitative monitor of the cartridge performance. Dry nitrogen was flushed through the chamber during low temperature runs to prevent the build up of condensation on either the laser or exit window. The laser was first operated at room temperature for approximately 1500 cycles at a rate of 17 pulses per minute. The chamber temperature was then raised to 125°F and after a 1/2 hour soak at 125°F the cartridge was subjected to another 1300 cycles of operation at the 17 pulse per minute rate. The chamber was then raised to 160°F and the operating cycle repeated for approximately 1000 pulses. The laser operated throughout the test. The chamber was then lowered to 0°F and after a 1/2 hour soak an 800 pulse test was performed. The chamber was then lowered in 10°F steps to -20 -30 -40 and -50°F with 50 pulses at each temperature. The chamber was returned to room ambient and after an additional 1200 cycles the power was measured and found to be 0.45 millijoules, down approximately -2.5 dB from the initial 0.8 millijoules. The total number of cycles on the cartridge at this time was approximately

Table 4. Laser Cartridge Fabrication and Performance Summary

CART. NUMBER	MONTH (1975)	2-WAY TRANS. (%)	MIRROR RADIUS (METERS)	ROD LENGTH MM	DEL TO ECOM	THRESHOLD VOLTS	INITIAL ENERGY (MJ)	RESULTS
1 ¹	JAN	70	1	15	NO	-	-	ALIGNMENT LOST DURING CURE
2 ¹	JAN	70	1	15	NO	~430	0.25	ROOM TEMP. ADHESIVE CURE, FIXTURE SPRING LOAD CAUSED MISALIGNMENT.
3 ¹	JAN	70	1	15	NO	390	0.5	BOND AND ALIGNMENT GOOD. DYE ABSORPTION LOW.
4	JAN	50	1	15	YES	500	1	DAMAGE IN BOTH BOND AND Q-SWITCH. OUTPUT DOWN AFTER 200 CYCLES.
5 ¹	MAR	50	1	15	NO	480	2	PMMA DAMAGE. 1 MJ AFTER 60 CYCLES
6	MAY	50	5	15	YES	530	0.8	EVALUATED FROM -50°F TO +160°F 0.45 MJ (-2.5 DB) AFTER 6000 CYCLES
7 ¹	JUNE	50	5	15	NO	500	0.2	ALIGNMENT POOR - ONLY 1 FRINGE @ 5 METERS
8	JUNE	50	2	16	YES	530	1.8	NO DAMAGE AFTER 150 CYCLES
9 ¹	JUNE	60	2	16	NO	460	0.6	NO PMMA DAMAGE WEAK BOND. YAG RECYCLED TO #12
10	JULY	60	2	16	YES	480	0.6	NO PMMA DAMAGE - 2 TEMP. CYCLES TO 0°F
11	JULY	60	2	15	YES	460	0.75	NO PMMA DAMAGE - 2 TEMP CYCLES TO 0°F
12	JULY	60	2	16	YES	520	0.7	NO PMMA DAMAGE - HIGH THRESHOLD FROM BAD FLASHLAMP

NOTES ¹ YAG Recycled to later cartridge

² Divergence of all cartridges 12-15 milliradians - halfpower

6000 cycles. At this point the flash-lamp used for the test was examined and found to be darkened as a result of the test. It is, therefore, difficult to assess how much of the 2.5 dB degradation was due to cartridge deterioration and how much was the result of flashlamp deterioration. This particular cartridge showed definite evidence of a growing damage site which was initially observed at 75 pulses. The growth of the damage site with the number of pulses approximated an inverse exponential i.e. it grew rapidly at first and then slowly approached its final size asymptotically.

Later cartridges, with improved processing in the Q-switch preparation did not exhibit the damage sites but efforts to life test these units were thwarted by lack of a suitable flashlamp. All lamps purchased for the test phase were experimental units made by EG & G. One lamp functioned well and was used for most of the tests but was unservicable after the life test. The remaining lamps triggered irregularly and became inoperative in a few hundred cycles. Thus later cartridges could only be given basic functional tests. The flashlamp problem prevented obtaining accurate data on laser threshold voltage. The values in Table 4 for cartridges 9 through 12 are, therefore, best values available. These cartridges were subjected to 2 cycles of temperature down to 0°F to test bond integrity.

Table 4 summarizes the performance of all cartridges made on the program. Cartridges 9 through 12 were made with Q-switch having 60 percent round trip transmission to assure operation below 500 volts for use in ECOM tests. All cartridges made in June and July used the Q-switch fabrication technique described in the report and did not exhibit the Q-switch damage of earlier cartridges. While testing of these units was not as extensive as for cartridge 6, the number of cycles on each (at least 200) greatly exceeded the number of cycles where earlier cartridges exhibited initial signs of Q-switch damage.

SECTION 3 ALTERNATES INVESTIGATED

3.1 Direct Dye Sublimation

In this technique, the nickel complex dye is sublimed directly on the YAG rod, with the high reflectance dielectric mirror deposited over the dye. In the event that the dielectric mirror deposition temperatures exceed the dye decomposition (or sublimation) temperature, an alternate for this assembly procedure is to bond a mirror (on a glass substrate) to the dye-coated rod end. If a glass with a thermal expansion coefficient close to that of the YAG rod is used for the mirror substrate (such as Schott BK-7), a good bond can be readily achieved with commercial optical adhesives. The sublimation study was conducted at United Technologies Research Center (UTRC). A detailed report of the investigation is presented in Appendix B.

In summary, the dye was successfully sublimed, but when the thin film with transmission of the order required for laser action was subjected to laser energy, the high energy density in the film caused the dye to vaporize. It is, therefore, required that the absorption be distributed in a larger volume than that of the dye itself. UTRC outlined several polymerization techniques which would accomplish this end. However, the development schedule (in excess of six months) and cost were inconsistent with the present program objectives and further work was postponed. The technique remains extremely promising as a long term solution to laser cartridge fabrication, since the individual cartridge alignment required for all other techniques is eliminated.

3.2 Adhesive Host Q-switch

Representative samples of various types of optical adhesives were selected for evaluation as potential candidates to host the nickel complex dye. Table 3 lists the commercial names of the selected adhesives and their generic types. Physical data on these adhesives are given in Appendix C.

The dye was found to be insoluble (directly) in all of the above adhesives.

A solution was obtained by dissolving the dye in methylene chloride, then adding the adhesive. (In the case of the two-part adhesives, only the resin part was added.)

For all of the two-part adhesives, the dye bleached before the methylene chloride could be removed by evaporation. The Opticon UV - 57 and Summers UV - 71 adhesives accepted the dye/methylene chloride solution without bleaching the dye during solvent removal, but the dye bleached during the ultraviolet irradiation curing process.

Butylmethacrylate also was considered as a possible host for the Q-switch dye. However, since the softening point of bulk polymerized butylmethacrylate is 30°C, parts formed from this material would not be mechanically stable over the required temperature range.

The adhesive host technique for Q-switch fabrication was therefore abandoned.

SECTION 4 CONCLUSIONS AND RECOMMENDATIONS

4.1 Conclusions

The laser cartridge successfully developed on the program consists of an integral bonded assembly of a Nd:YAG laser rod (3 mm diameter x 15 long), a Q-switch, and a resonator mirror. The Q-switch is an organic nickel complex dye in a polymethylmethacrylate (PMMA) host. This unit has been tested and shown to satisfy the ECOM technical guidelines requirements. A beam divergence specification was not included in the technical guidelines because the beam divergence could not be determined a priori and it would have been beyond the scope of this program to attack such a problem. Complete static and limited operational tests were conducted over temperature, and a life test was run.

The key elements in the program were: the development of a total process for producing the PMMA/dye Q-switches with accurate control of optical density in a laser damage resistant high purity material; the test and selection of an optical quality adhesive with the necessary dimensional stability and strength which could permit the relative expansion and contraction of the joined materials without failure; the development of alignment and assembly techniques for consistently obtaining laser cartridges with predictable performance.

A total of 12 laser cartridges were fabricated during the program, 6 of which were delivered as required by the contract. Three of the delivered cartridges were specifically tailored to the ECOM mini-rangefinder program requirements, with the remaining three demonstrating significant stages in the development program.

Two alternate methods for cartridge manufacture were investigated. They were direct Q-switch dye sublimation on to the laser rod, and use of a Q-switch dyed adhesive. An adhesive to accept the dye was not found. The direct dye sublimation technique showed promise, but required substantially more research and development effort than was possible under the contract, and was therefore terminated.

The program has demonstrated that the ECOM laser cartridge concept is technically feasible. Working cartridges satisfying the technical guidelines have been manufactured which meet ECOM performance requirements in all areas except divergence.

4.2 Recommendations

Recommendations for continued laser cartridge work are in four areas:

- a. Beam divergence investigation
- b. Mini-Transmitter module development (excluding flash lamp development)
- c. Q-switch investigation
- d. Alignment fixture improvement

The beam divergence investigation should consider both unstable and stable resonator techniques. Unstable techniques include hybrid configurations and conventional negative branch resonators. Stable resonator investigations should include divergence versus Fresnel number, divergence versus Q-switch type, and cost-weight-size trade-offs using a beam expander.

The mini-transmitter task should develop a module which integrates the final laser cartridge and output optics, if required, with the flash lamp and pump cavity, pulse forming network, trigger circuits and energy storage elements being developed by ECOM on its minirangefinder program. Tradeoffs of performance, cost, weight and size should be paramount in this investigation.

The Q-switch fabrication task should provide further data on ultimate power limitations, and production type techniques for controlling Q-switch parameters and quality.

A cartridge alignment fixture should be developed to simplify the final assembly procedure and provide improved accuracy in both alignment error sensing and fixture adjustment. The cartridge should be mounted vertically to eliminate the asymmetric effects of gravity on the adhesive. The alignment controls should be brought out through the walls of the thermally controlled enclosure. An improved screen viewing system will provide higher contrast interference patterns by eliminating ambient illumination of the viewing screen.

APPENDIX A LASER ROD REQUIREMENTS

Norden DIVISION OF UNITED STATES GOVERNMENT U A.	CLASSIFICATION UNCLASSIFIED	NUMBER 1249-R-0005 SHEETS - SHEET						
TITLE LASER ROD REQUIREMENTS	DATE 20 Sept 74 REVISED 20 Sept 74	BY H. NEYMAN BY D. D. D. D. D. CHECKED 20 Sept 74 CHECKED						
Host Material: YAG (Yttrium Aluminum Garnet) Doping Material: Nd³⁺ (Neodymium) Doping Level: Optimized for Pulsed--Q--Switched output. Diameter: 3.000 mm $\pm$.125 mm Length: See Table I End Parallelism: Better than 6 seconds of Arc. End Flatness: Better than 0.2 wavelength at the Sodium D-Line. End Finish: Better than 20-20 as defined by MIL-O-13830A, para 3.5.1.1 Rod Quality: Less than one (1) fringe, double pass. Rod Finish: Outer diameter of rod to be an optical fine grind finish. Coating: A. One 3.000 mm diameter face to be coated for 75% $\pm$ 2% reflection at 1.06 microns. B. One 3.000 mm diameter face to be uncoated and to have no stain. C. Coating Durability: Abrasion requirement of MIL-C-675A para. 3.9.4 Adhesion requirement of MIL-M-13508B para. 3.9 Operational Temperature Range: -54°C to +55°C Storage Temperature Range: -62°C to + 85°C TABLE I <table border="1"> <thead> <tr> <th>DASH NO.</th> <th>LENGTH</th> </tr> </thead> <tbody> <tr> <td>1249-R-0005 -1</td> <td>15MM $\pm$ 0.5MM</td> </tr> <tr> <td>1249-R-0005 -2</td> <td>16MM $\pm$ 0.5MM</td> </tr> </tbody> </table>			DASH NO.	LENGTH	1249-R-0005 -1	15MM $\pm$ 0.5MM	1249-R-0005 -2	16MM $\pm$ 0.5MM
DASH NO.	LENGTH							
1249-R-0005 -1	15MM $\pm$ 0.5MM							
1249-R-0005 -2	16MM $\pm$ 0.5MM							

N242 (9/99)

APPENDIX B

VAPOR DEPOSITION OF Q-SWITCH LASER DYE

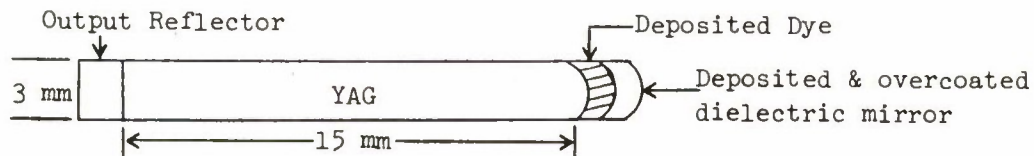
SUMMARY

This report presents the results of an investigation undertaken in support of Norden Division, the objective of which was to determine the feasibility and conditions for the sublimation of the organic chelate Q-switch dye, bis-(4-dimethylaminodithiobenzil) nickel. The work covers the period from July 1 to October 15, 1974 under UARL Project Number 121294. The major results of this investigation are listed below in summary form.

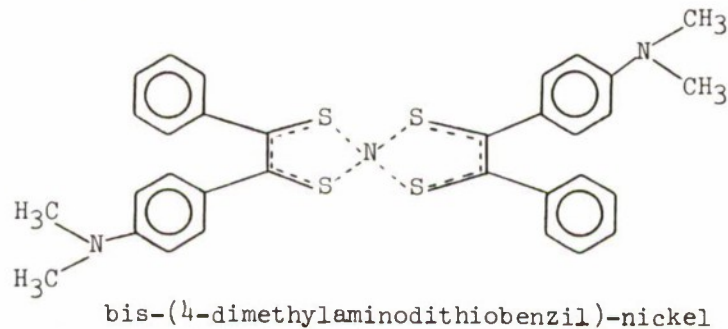
- The dye can be successfully sublimed at temperatures of 230-235°C and $1.0-6.0 \times 10^{-5}$ Torr pressure without decomposition.
- Optical densities on glass slides ranging from 0.1 to 1.2 can be readily obtained. The rate of sublimation was dependent on particle size; larger particles resulting in slower deposition rates. Optical densities in the desired range (0.12) were achieved in approximately fifteen (15) minutes using finely powdered material.
- Deposition on non-treated glass surfaces is characterized by dye crystallization resulting in microcracking of the dye film. Silane pretreatment of the glass reduced such cracking to a minimum so that a continuous dye film was formed.
- Preliminary lasing tests showed the dye rapidly sublimed (vaporized) from the glass slide. Open lasing was noted but no Q-switching occurred.
- Attempts to improve dye adhesion by glass sandwich construction or silver mirroring over the deposited dye did not produce satisfactory results during lasing tests.

I. INTRODUCTION

One of the four laser cartridge conceptual configurations in the Norden laser cartridge program being sponsored by the U.S. Army Electronics Command involves direct deposition of a passive Q-switch dye onto a Nd:YAG laser rod. A schematic of the proposed design is shown below:



The most promising Q-switch dye has been found to be bis-(4-dimethylaminodithiobenzil) nickel (Ref. 1). This dye, having the structural formula shown below, had been previously synthesized at UARL (Ref. 2). Preliminary evidence at that time (1972) indicated that the dye could be sublimed without decomposition at low pressures.



The objective of this investigation was to determine the feasibility of dye sublimation and to optimize conditions with respect to dye purity and optical density as they related to vacuum, temperature and deposition rate.

In order to determine optimum conditions for sublimation of the subject Q-switch dye it was necessary to first demonstrate conclusively that the dye could be sublimed congruently, i.e. without decomposition. To this end initial experiments were carried out on sodium chloride crystals so that infrared spectrum of the sublimed dye could be obtained. The majority of the work, however, involved deposition of the dye onto glass microscope slides (3/8 x 3/4 in.). Optical density measurements on the dye coated slides were made at 1.06 microns using the Cary 14 spectrometer.

The following sections of this report describe the procedure developed for dye sublimation and attempts to provide adequate adhesion of the dye to the glass substrate during lasing.

II. SUBLIMATION APPARATUS AND PROCEDURE

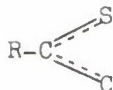
The assembled sublimation apparatus is shown in Fig. 1 and consisted of a standard vacuum pump, diffusion pump, an oil heating bath and sublimation tube. A schematic of the sublimation tube is shown in Fig. 2. The glass slide was attached by clips to the bottom of the water cooled copper disk suspended an inch above the powdered dye. The pressure was reduced to at least 5×10^{-5} Torr prior to insertion of the sublimation tube into the preheated oil bath at the desired temperature. Three to four minutes were required for the dye to reach the set temperature at which point sublimation time was measured.

Dye previously synthesized at UARL was used throughout the program. The solvent recrystallized dye was finely ground in an agate mortar and pestle prior to sublimation. A charge of 150 mg dye was normally employed and could be used for up to ten short sublimation runs before a decrease in sublimation rate was noted. Regrinding of the used material allowed further sublimation to occur without having to add additional dye to the tube. Infrared spectra of sublimation residues showed no decomposition of dye occurred during the use period.

III. DYE SUBLIMATION ON SALT PLATES

Since the infrared spectrum of an authentic sample of bis-(4-dimethylamino-dithiobenzil) nickel was available as a standard of purity, the solvent recrystallized UARL synthesized dye could be easily checked for decomposition after sublimation. Three dye sublimations onto sodium chloride plates were carried out and the infrared spectrum checked with that of the standard. In all cases the analysis showed the dye sublimed at the above-stated conditions without decomposition. The major peaks of the pure dye are listed in Table I.

Apart from bands due to the presence of phenyl groups, three characteristic absorptions, 1340, 915, and 880 cm^{-1} are observed in the 3000-600 cm^{-1} range. These are combination bands and probably coupled to each other. Rigorous assignment would require a normal coordinate treatment (Ref. 3). Qualitatively, the 1340 band may be assigned to the perturbed -C=C- stretch, the 1180 band to the perturbed C=S bond while the 880 band has been tentatively assigned to a stretching vibration of the



In a partially decomposed or flash lamp decayed sample of the Q-switch dye peaks at 2920 and 2840 cm^{-1} appear, which are not in the pure sample, as well as a change in the intensity ratio of the 1440-1540 cm^{-1} and 1140-1190 cm^{-1} bands.

Infrared spectrum of the dye residue after several runs also indicated that no decomposition had occurred during heating. Regrinding of the residues allowed additional dye to sublime. This indicates that all the dye charged to the system would be sublimable and dye losses due to mechanical manipulations only would be encountered. Thus, the sublimation process appears to be a highly efficient method of obtaining high purity material.

An additional check on the sublimation technique was carried out by subliming sufficient dye (60 mg) for incorporation into a polymethylmethacrylate (PMMA) wafer by Norden personnel. Comparison of laser results of this material with that obtained with a PMMA wafer made using the solvent recrystallized dye should verify the infrared analysis for dye purity.

IV. DYE SUBLIMATION ON GLASS SLIDES

The remaining sublimation studies were carried out using microscope glass slides cut to 3/8 x 3/4 in. size. Each slide was washed with green soap and water, followed by a distilled water rinse prior to use. Additional surface treatments are discussed below.

A measure of the sublimation rate of the dye was obtained by determination of the optical density obtained per time. Optical density was measured using a Cary 14 spectrometer. Some shift in the broad curve in the 1.06 μ region was noted in some of the runs which was probably due to non-uniform dye deposition or light scattering because of microcrystallites. Similar shifts have been noted in dye solutions. The change in spectra with time of deposition is shown in Fig. 3 and with optical density in Fig. 4. The sharpness of the peak is increased with the heavier deposition. It should be noted that the four hour run was carried out using a freshly ground dye sample rather than that used for the shorter runs since the initial dye batch had been used for ten short sublimations prior to these runs. Regrinding the initial dye residue and subliming for two hours gave an optical density slightly lower than would have been expected based on the above results (Fig. 4). This indicates the importance of particle size and uniformity in sublimation and if further use of this technique for either dye purification or Q-switch preparation is to be considered, emphasis on achieving uniform, high surface area dye powder should be considered.

Norden Division had specified an optical density of 0.12 for performance in the final laser rod design. Samples of glass coated dye having O.D. in this range and higher were submitted to Norden for testing. The slides having close to 0.12 O.D. were found to open lase once or twice prior to sublimation or evaporation of the dye. Slides of higher O.D. would not lase due to insufficient power input related to the test equipment.

One cause for the dye evaporation during testing is the noncontinuous film of material deposited during sublimation. Rather than a clear plastic film like that made using PMMA resin with dissolved dye, the sublimed dye crystallizes into small isolated crystallites apparently associated with nucleation sites. A microphotograph of sublimed dye (0.125 O.D.) on glass is shown in Fig. 5 at 200X magnification.

Two additional surface treatments other than soap cleaning were tested to see if a decrease in surface cracking and improved dye adhesion could be achieved. The first, a hydrofluoric acid etch proved ineffective in that the surface flatness of the glass slide was destroyed (increased roughness) and resulted in nonuniform dye distribution. No improvement in dye adhesion was noted.

The second treatment did improve dye uniformity and gave a noncracked film on the glass surface. The treatment consisted of applying a silane coating on the glass surface after initial cleaning. This was done by dipping the slide into an alcohol/water solution of gamma-aminopropyltriethoxy silane (A-1100) followed by drying at 100°F. This treatment is the same as that applied to nearly all fiber glass filament and cloth which is used as reinforcement in resin composites. The dye film deposited on the silane treated glass appears continuous with no cracking as shown in Fig. 5 at 500X magnification. Actually, the dye appears to provide a replica of the silane treated surface thus indicating the nature of the silicone polymer resulting from hydrolysis of the starting silane.

Although improved dye coatings were obtained by the silane treatment sufficient dye adhesion during lasing tests at Norden was not achieved.

V. DYE RETENTION ON GLASS SLIDES

Two approaches were tried to prevent dye evaporation during the lasing test. First a dye coated slide was covered by a second clear slide and while clamped together the edges were sealed with epoxy resin. The second approach was to deposit a silver mirror by evaporation over a dye coated slide. The silver mirror adhered well to the glass with no apparent effects on the dye layer. In each case the slide having the dye was silane treated.

Both specimens were submitted to Norden for testing. In neither case was Q-switching successful at the 0.11-0.12 optical density level used on these slides.

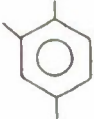
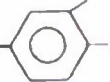
Norden's decision to employ the polymethylmethacrylate films containing the Q-switch dye for the fulfillment of the contract obligations led to termination of the sublimation approach at the present time.

VII. REFERENCES

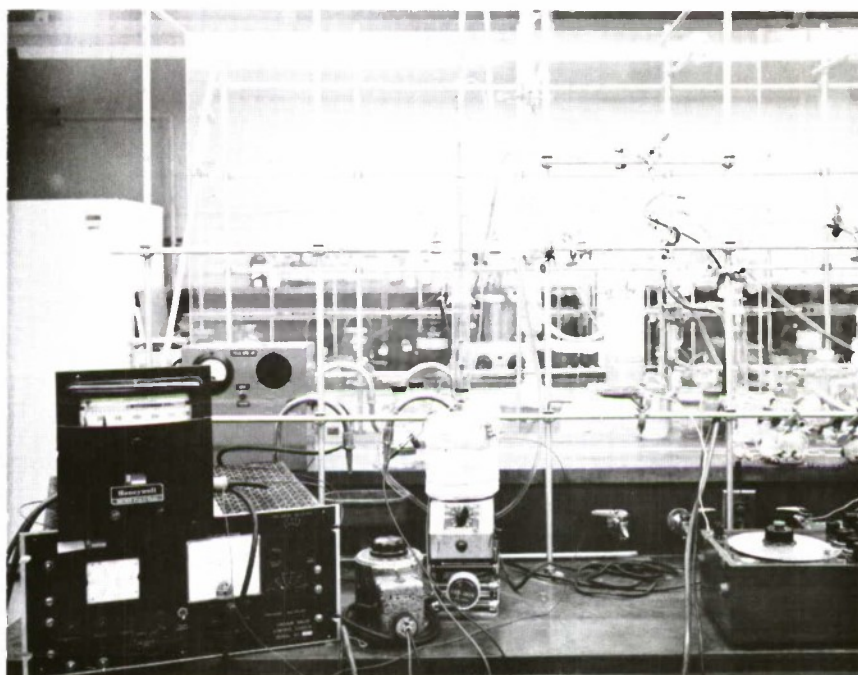
1. K. H. Drexhage and U. T. Muller-Westeroff, "New Q-Switch Compounds for Infrared Lasers", IEEE J. Quantum Elect., QE-8 (9) 759 (1972).
2. Internal Correspondence, R. Pike to G. Golden, "Synthesis of Bis-(4-dimethylaminodithiobenzil)-nickel, Dec. 14, 1972.
3. G. N. Schrauzer and V. P. Mazweg, "Preparation, Reactions, and Structure of Bisdithio- α diketone Complexes of Nickel, Palladium and Platinum", J. Am. Chem. Soc., 87 1483 (1965).

Table I

Major Infrared Adsorption Bands for
Bis-(4-Dimethylaminodithiobenzil) Nickel

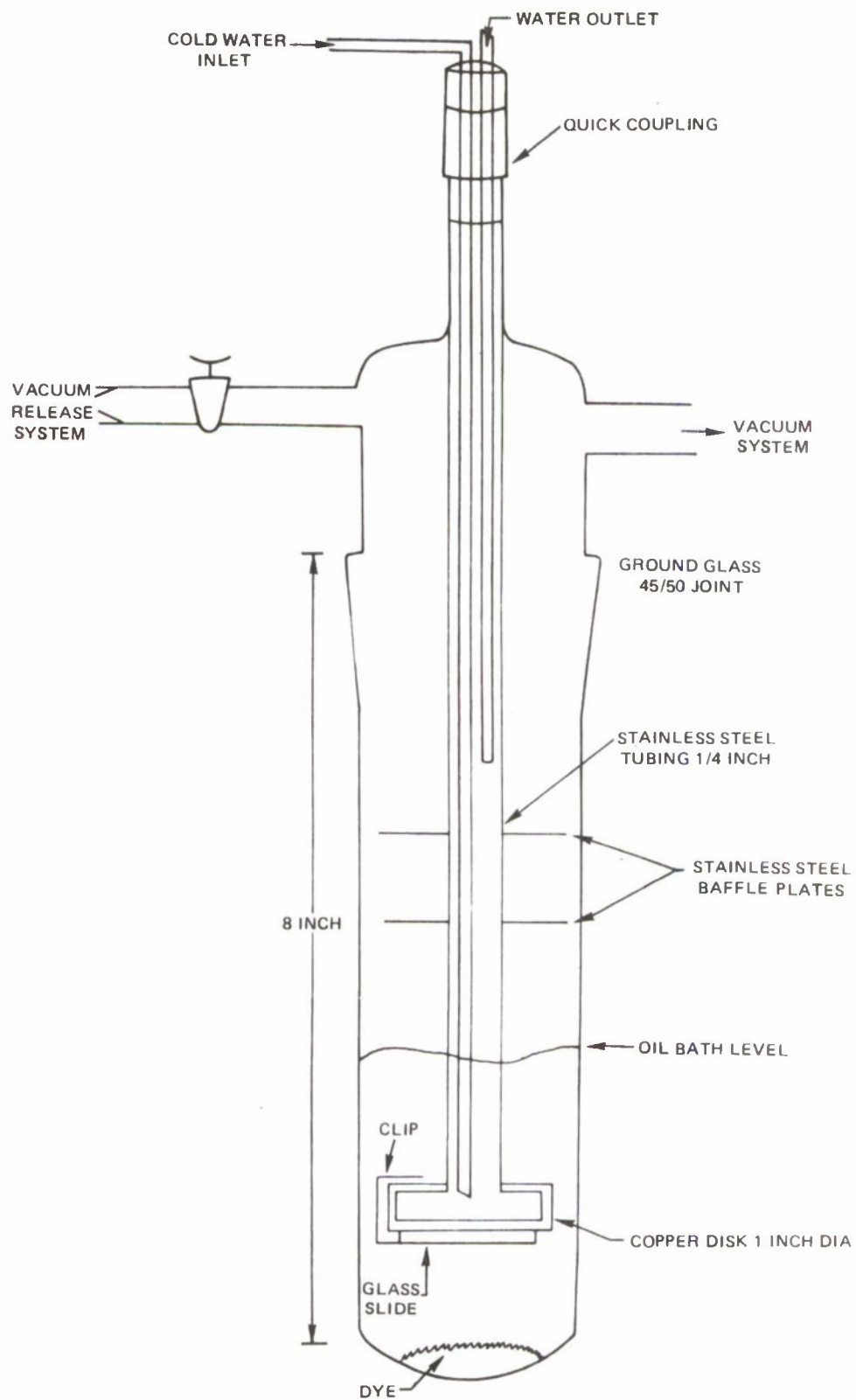
<u>Wave Number (cm⁻¹)</u>	<u>Intensity</u>	<u>Band Identification</u>
1590	strong	C-N bonds
1530	medium	Unsymmetrical
1460	medium	substitution on
1480	medium	aromatic ring 
1410	weak	
1395	weak	
1340	strong	Perturbed -C=C-
1320	} shoulder peaks on 1340	(CH ₃) ₂ N- 
1300		
1270		
1230		
1180	strong	Perturbed C=S
1165	strong	linkage & aromatic
1140	strong	substitution
1015	medium	
945	medium	
880	strong	
835	medium	substituted aromatic ring
815	medium	
795	medium	

SUBLIMATION APPARATUS FOR Q-SWITCH DYE



SUBLIMATION TUBE

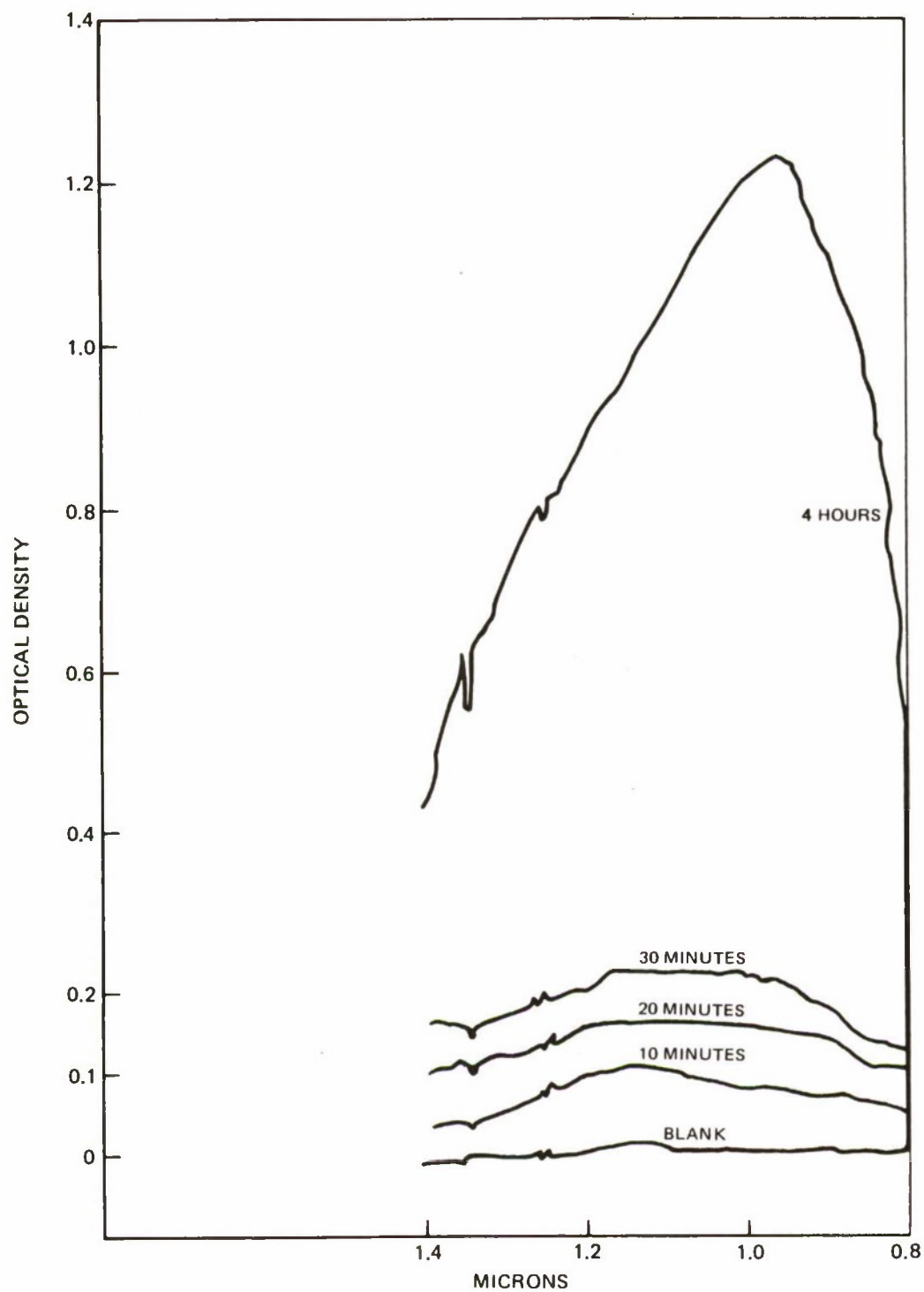
FIG. 2



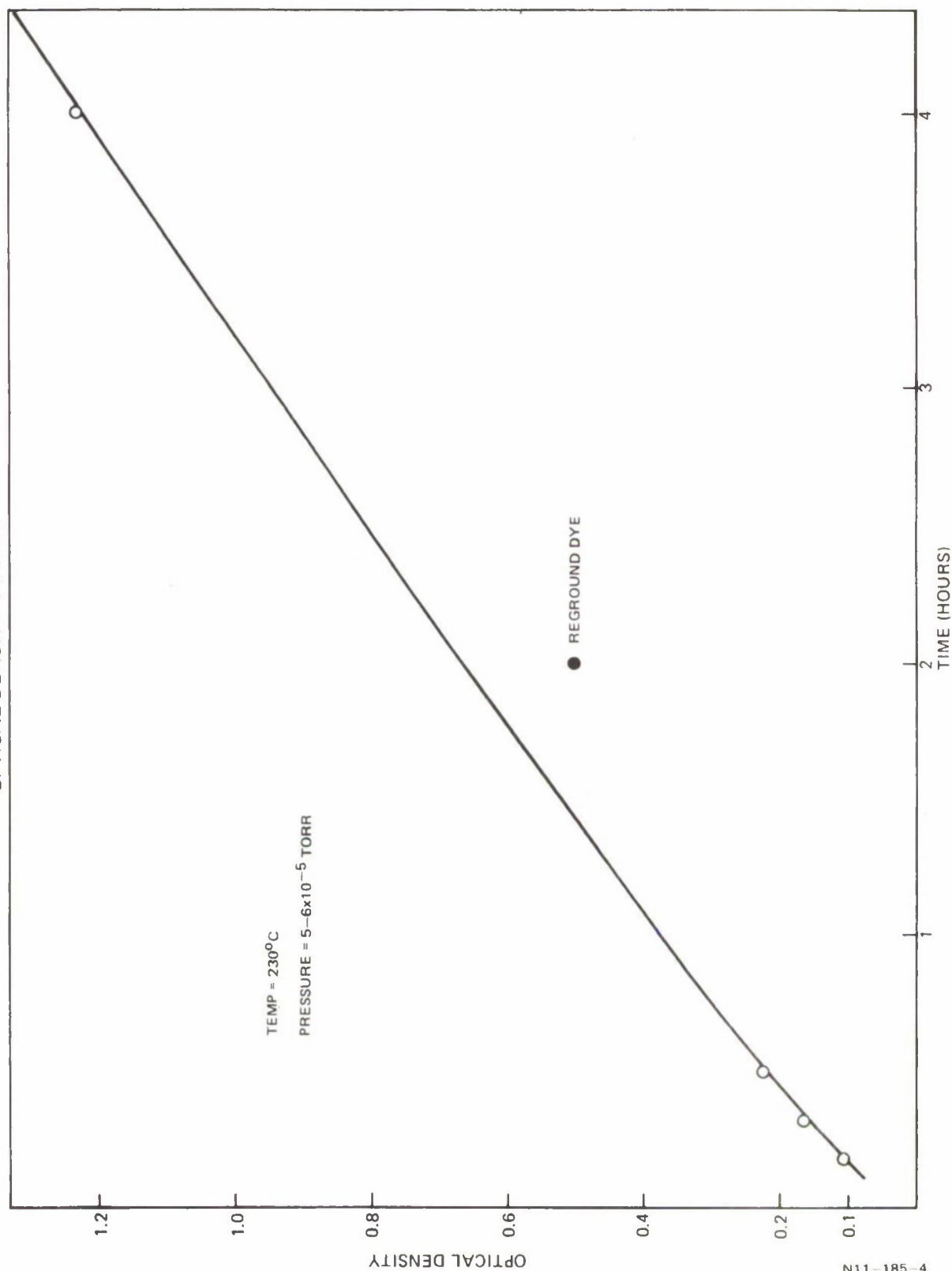
N11-185-2

SUBLIMATION OF Q-SWITCH DYE ON GLASS
NEAR INFRA-RED SPECTRUM

FIG. 3



N11-185-3

SUBLIMATION OF Q-SWITCH DYE
OPTICAL DENSITY VS TIME

N11-185-4

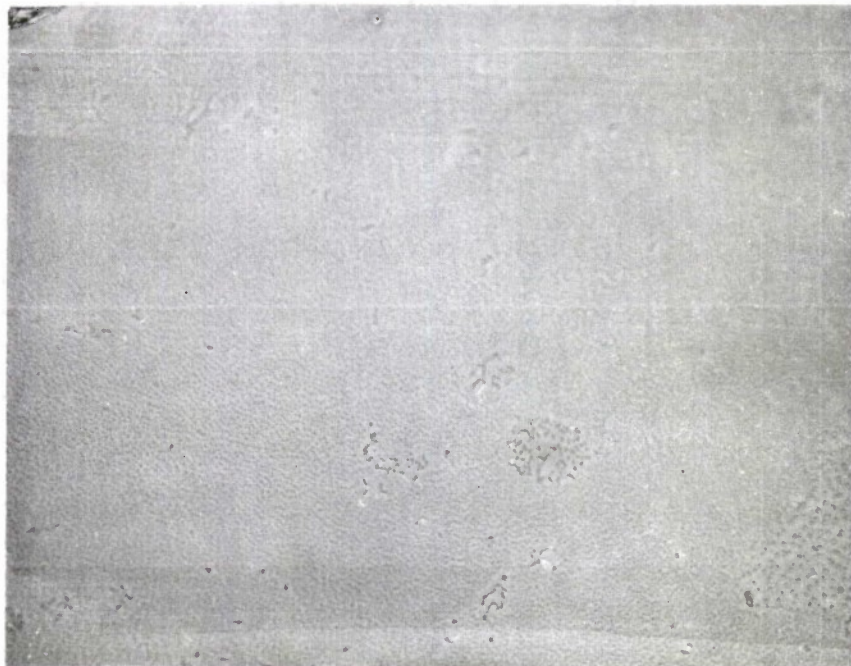
MICROPHOTOGRAPHS OF SUBLIMED DYE ON GLASS SLIDES



NO TREATMENT

OPTICAL DENSITY = 0.12

200X



SILICONE TREATED

OPTICAL DENSITY = 0.11

500X

N11-185-5

APPENDIX C
PHYSICAL DATA ON ADHESIVES STUDIED

1. Dow Corning R-63-489: a two-part silicone elastomer
Suggested operating temperature range = -65 to +200°C
Light transmission at 1.06 μ = 88%
Tensile strength = 900 psi
Refractive index = 1.430
Brittle point = -135°C
Coefficient of thermal expansion = 3.0×10^{-4} cm/cm-°C
(-55 to 150°C)

(DC Sylgard primer required for PMMA/YAG bonds)
2. GE RTV-602: a two-part silicone rubber compound
Suggested operating temperature range = -75 to +400°F
Tensile strength = 100 psi
Coefficient of thermal expansion = 16.2×10^{-5} in/in-°F
(0 to 350°F)
3. Hysol EA-9419: a two-part polycarbonate adhesive consisting of an epoxy/urethane paste and a liquid amine

Detailed data not supplied by vendor.
4. Hysol 0151: a two-part epoxy resin
Suggested operating temperature range = -50 to +180°F
Tensile shear strength = 1850 psi.
5. Kodak HE-10: a two-part modified epoxy
Suggested operating temperature range = -65 to +185°F
Refractive index = 1.577
6. Loctite 04-E: a one-part thickened ethyl cyanoacrylate
Suggested operating temperature range = -110 to +185°F
Tensile shear strength = 2200 psi
Refractive index = 1.45 ± 0.03

7. Opticon UV-57: a single component photosensitive resin
Suggested operating temperature range = -65 to +200°F
Refractive index = 1.5316
Light transmission at 1.06 μ = 93%
8. Summers M-62: a two-part synthetic polyester adhesive
Suggested operating temperature range = -54 to 100°C
Refractive index = 1.55
9. Summers UV-71: a one-part photosensitive synthetic polyester adhesive
Suggested operating temperature range = -54 to 100°C
Refractive index = 1.55
10. Techkits E-7: a two-part flexible epoxy resin
Tensile shear strength = 3400 psi
Coefficient of thermal expansion = 4.8×10^{-5} cm/cm°C
11. Tra-Con BB-2114: a two-part epoxy resin
Suggested operating temperature range = -60 to +110°C
Refractive index = 1.565
Tensile strength = 7200 psi
Coefficient of thermal expansion = 61×10^{-6} cm/cm°C
12. Tra-Con BB-2129: a two-part epoxy resin
Suggested operating temperature range = -60 to +140°C
Tensile strength = 9400 psi
Coefficient of thermal expansion = 58×10^{-6} cm/cm°C
13. Tra-Con BB-2130: a two-part epoxy resin
Suggested operating temperature range = -60 to +140°C
Tensile strength = 8600 psi
Coefficient of thermal expansion = 52×10^{-6} cm/cm°C

DISTRIBUTION LIST

101	Defense Documentation Center ATTN: DDC-TCA Cameron Station (Bldg 5)	212	Cmd, Control & Communications Div Development Center Marine Corps Dev & Educ Cmd
012	Alexandria, VA 22314	001	Quantico, VA 22134
107	Director National Security Agency ATTN: TDL	215	Naval Telecommunications Command Tech Library, Code 422 4401 Massachusetts Avenue, NW
001	Fort George G. Meade, MD 20755	001	Washington, DC 20390
108	Director, Defense Nuclear Agency ATTN: Technical Library	220	Naval Research Laboratory Code 6503, LTP0(Dr. W. R. Sody)
001	Washington, DC 20305	001	Washington, DC 20390
200	Office of Naval Research Code 427	301	Rome Air Development Center ATTN: Documents Library (TILD)
001	Arlington, VA 22217	001	Griffiss AFB, NY 13441
201	Cdr, Naval Ship Systems Command Technical Library, Rm 3 S-08 National Center No. 3	307	HQ ESD(XRRI) L. G. Hanscom Field
001	Washington, DC 20360	001	Bedford, MA 01730
205	Director Naval Research Laboratory ATTN: Code 2627	312	AFSPCOMMCEN/SUR
001	Washington, DC 20375	001	San Antonio, TX 78243
206	Commander Naval Electronics Lab Center ATTN: Library	313	Armament Development & Test Center ATTN: DLOSL, Tech Library
001	San Diego, CA 92152	001	Eglin Air Force Base, FL 32542
207	Commander US Naval Ordnance Laboratory ATTN: Technical Library White Oak, Silver Spring	314	HQ, Air Force Systems Command ATTN: DLCA Andrews Air Force Base
001	MD 20910	001	Washington, DC 20331
210	Commandant, Marine Corps HQ, US Marine Corps ATTN: Code LMC	320	Air Force Systems Command Advanced Systems Div (ASD/ENAD)
001	Washington, DC 20380	001	Wright-Patterson AFB, OH 45433
211	HQ, US Marine Corps ATTN: Code INTS	401	Commander US Army Training & Doctrine Cmd ATTN: ATTNG-40
001	Washington, DC 20380	001	Fort Monroe, VA 23651
		403	HQDA(DACE-CMS)
		001	Wash, DC 20310
		405	OSASS-RD
		001	Washington, DC 20310

DISTRIBUTION LIST

406 Commander US Army Training & Doctrine Cmd ATTN: ATCD-SI 001 Fort Monroe, VA 23651 407 Commander US Army Training & Doctrine Cmd ATTN: ATCD-CI 001 Fort Monroe, VA 23651 408 HQDA(DARD-ARS-P/Dr. R. B. Watson) 001 Wash, DC 20310 409 Cdr, US Army Material Command ATTN: AMCMA-EE 5001 Eisenhower Ave 001 Alexandria, VA 22333 410 Cdr. US Army Material Command ATTN: AMCRD-TP (Dr. R. Zarwyn) 5001 Eisenhower Ave 001 Alexandria, VA 22333 411 Cdr, US Army Material Command ATTN: AMCRD-W 5001 Eisenhower Ave 001 Alexandriz, VA 22333 413 Cdr, US Army Material Command ATTN: AMCRD-FW 5001 Eisenhower Ave 001 Alexandria, VA 22333 416 Commander US Army R&D Group (Far East) 004 APO, San Francisco 96343 419 Commander US Army Missile Command ATTN: AMSMI-RR, Bldg 7770 001 Redstone Arsenal, AL 35809 420 Commander US Army Missile Command ATTN: AMSMI-RR (Mr. W. B. Jennings) 001 Redstone Arsenal, AL 35809	421 Cdr, US Army Missile Command Redstone Scientific Info Center ATTN: Chief, Document Section 002 Redstone Arsenal, AL 35809 422 Commander US Army Aeromedical Research Lab ATTN: Library Fort Rucker, AL 36362 423 Commander US Army Armament Command ATTN: AMSAR RDP (Library) 001 Rock Island, IL 61201 424 Commander Rock Island Arsenal ATTN: SARRI-LP-L-Tech Library 001 Rock Island, IL 61201 425 Commander, Rock Island Arsenal US Army Armament Command ATTN: SWERI-RET-E (Dr. L. Gardner) 001 Rock Island Arsenal, IL 61201 427 Cdr, US Army Combined Arms Combat Developments Activity ATTN: ATCAIC-IE 003 Fort Leavenworth, KS 66027 429 Commander US Army Logistics Center ATTN: ATCL-MA 001 Fort Lee, VA 23801 431 Commander US Army Intelligence School ATTN: ATSIT-CTD 001 Fort Huachuca, AZ 85613 433 Cdr, US Army Aviation Systems Cmd ATTN: AMSAV-G PO Box 209 001 St. Louis, MO 63166
---	---

DISTRIBUTION LIST

434 Commander US Army Test & Evaluation Command ATTN: AMSTE-D5-E (Mr. J. Bialo) 001 Aberdeen Proving Ground, MD 21005	458 Dir/Dev & Engr Defense Systems Div ATTN: SAREA-DE-DDR, H. Tannenbaum 002 Edgewood Arsenal, APG, MD 21010
436 Commandant US Army Engineer School ATTN: ATSE-CTD-DT-TL 001 Fort Belvoir, VA 22060	465 Commander Aberdeen Proving Ground ATTN: STEAP-TL (Bldg 305) 002 Aberdeen Proving Ground, MD 21005
441 Commander Harry Diamond Laboratories ATTN: AMXDO-RCB, Mr. F. Harris 001 Washington, DC 20438	480 Commander USASA Test & Evaluation Center 001 Fort Huachuca, AZ 85613
442 Commander Harry Diamond Laboratories ATTN: Library 001 2800 Powder Mill Road Adelphi, Maryland 20783	483 Cdr, US Army Research Ofc ATTN: DRXRO-IP PO Box 12211 001 Research Triangle Park, NC 27709
447 Cdr, US Army Picatinny Arsenal ATTN: SMUPA-VC5 Bldg 350 001 Dover, NJ 07801	484 Cdr, US Army Research Ofc ATTN: DRXRO-PH(Dr. R.J. Lontz) PO Box 12211 001 Research Triangle Park, NC 27709
449 Commander Picatinny Arsenal ATTN: SARPA-TS-S #59 002 Dover, NJ 07801	485 Cdr, HQ MASSTER Technical Information Center ATTN: Mrs. Ruth Reynolds 001 Fort Hood, TX 76544
451 Commander Frankford Arsenal ATTN: Dr. Wm McNeill, PDS 64-4 001 Philadelphia, PA 19137	486 Commander US Army Mobility Eqpt R&D Cen ATTN: SMEFB-R 001 Fort Belvoir, VA 20060
454 Cdr. US Army Training Device Agcy ATTN: AMXPG Naval Training Equipment Center 001 Orlando, FL 32813	488 US Army Security Agency ATTN: IARD Arlington Hall Station 001 Arlington, VA 22212
456 Commander White Sands Missile Range ATTN: STEWS-RE-IO 001 White Sands Missile Range NM 88002	489 Commander US Army Tank-Automotive Command ATTN: AMSTA-RW-L 001 Warren, MI 48090

DISTRIBUTION LIST

492 Commandant US Army Air Defense School ATTN: C&S Dept, Msl Sci Div 001 Fort Bliss, TX 79916 496 Cdr, US Army Combined Arms Combat Developments Activity ATTN: ATCACC 001 Fort Leavenworth, KS 66027 500 Commander US Army Yuma Proving Ground ATTN: STEYP-MTD (Tech Library) 002 Yuma, AZ 85364 501 Commander US Army Arctic Test Center ATTN: STEAC-PL 002 APO, Seattle 98733 502 CO, US Army Tropic Test Center ATTN: STETC-MO-A (Tech Library) Drawer 942 001 Fort Clayton, Canal Zone 09827 511 Commander US Army Nuclear Agency 001 Fort Bliss, TX 79916 512 Directorate of Combat Dev US Army Armor School ATTN: ATSB-CD-AA 001 Fort Knox, KY 40121 514 Commandant US Army Inst for Military Assistance ATTN: ATSU-CTD-OMS 001 Fort Bragg, NC 28307 517 Commander US Army Missile Command ATTN: AMSMI-RE (Mr. Pittman) 001 Redstone Arsenal, AL 35809	518 Commander Harry Diamond Laboratories ATTN: AMXDO-RCB (Dr. J. Nemarich) 001 Washington, DC 20438 519 Commander US Army Systems Analysis Agency ATTN: AMXSY-T (Mr. A. Reid) 001 Aberdeen Proving Ground, MD 21005 520 Cdr, Frankford Arsenal ATTN: SMUFA-N-4100 Bldg 201 001 Philadelphia, PA 19137 596 Commandant US Army Signal School ATTN: ATSN-CTD-MS 002 Fort Gordon, GA 30905 607 Commander US Army Tank-Automotive Command ATTN: AMSTA-RHP (Dr. J. Parks) 001 Warren, MI 48090 608 Director Night Vision Laboratory (ECOM) ATTN: AMSEL-NV-VIS (Mr. L. Gillespie) 001 Fort Belvoir, VA 22060 609 Director Night Vision Laboratory (ECOM) ATTN: AMSEL-NV-SD (Mr. Gibson) 001 Fort Belvoir, VA 22060 610 Director, Night Vision Laboratory US Army Electronics Command ATTN: AMSEL-NV-D 001 Fort Belvoir, VA 22060
---	---

DISTRIBUTION LIST

614 Chief Ofc of Missile Electronic Warfare, Electronic Warfare Lab, ECOM 002 White Sands Missile Range, NM 88002 617 Chief Intel Material Dev & Support Ofc Electronic Warfare Lab, ECOM 001 Fort Meade, MD 20755 680 Commander US Army Electronics Command 000 Fort Monmouth, NJ 07703 1 AMSEL-PP-I-PI 1 AMSEL-PL-ST 1 AMSEL-NL-D 1 AMSEL-WL-D 1 AMSEL-VL-D 3 AMSEL-CT-LD (R. Wright) 1 AMSEL-BL-D 1 AMSEL-CT-L (Dr. R. Buser) 1 AMSEL-CT-LC (Dr. E. Tebo) 1 AMSEL-CT-L (Mr. B. Louis) 1 AMSEL-CT-LD (Dr. E. Schiel) 1 AMSEL-CT-LE (Dr. H. Hieslmair) 1 AMSEL-CT-LD (Mr. J. Strozyk) 1 AMSEL-TL-D 1 AMSEL-TL-I (Dr. H. Jacobs) 1 AMSEL-TL-B (Mr. M. Zinn) 1 AMSEL-TL-TG (Mr. S. Schneider) 1 AMSEL-NL-R-5 (Dr. E. Dworkin) 1 AMSEL-VL-E 1 AMSEL-WL-N 1 AMSEL- (Ofc of Record) 1 AMSEL-MA-MP 2 AMSEL-MS-TI 1 AMSEL-GG-TD 2 AMSEL-PA 1 AMCPM-TDS-SE 1 USMC-LNO 1 AMSEL-RD 1 TRADOC-LNO 1 AMSEL-CG (Mr. Doxey) 72 Originating Office	701 MIT - Lincoln Laboratory ATTN: Library - Rm A-082 PO Box 73 Lexington, MA 02173 703 NASA Scientific & Technical Information Facility ATTN: Acquisition Br (S-AK/DL) PO Box 33 001 College Park, MD 20740 705 Advisory Group on Electron Devices 201 Varick Street, 9th Floor 002 New York, NY 10014 706 Advisory Group on Electron Devices ATTN: Secy, Working Group D (Lasers) 201 Varick Street 002 New York, NY 10014 707 Target Signature Analysis Center Env. Research Inst of Michigan PO Box 618 001 Ann Arbor, MI 48107 708 Ballistic Missile Radiation Anal Cen, Env. Research Inst of Michigan Box 618 001 Ann Arbor, MI 48107 711 Metals and Ceramics Inf Center Battelle 505 King Avenue 001 Columbus, OH 43201 718 TACTEC Battelle Memorial Institute 505 King Avenue 001 Columbus, OH 43201
--	--

DISTRIBUTION LIST

720 Thermophysical Properties Res
Center
Purdue University, Research Park
2595 Yeager Road
001 Lafayette, IN 47906

Westinghouse Research Labs 1
ATTN: Dr. K. Steinbrugge
Pittsburgh, PA 15235

Sanders Associates, Inc. 1
ATTN: Dr. C. S. Naiman
95 Canal Street
Nashua, NH 03060

International Laser Systems, Inc. 1
ATTN: T. Crow
3404 N. Orange Blossom Trail
Orlando, FL 32804

Martin Marietta Aerospace 1
Orlando Division
P.O. Box 5837
Orlando, FL 32805

Bendix Research Labs 1
ATTN: G. E. Busch
Southfield, MI 48075

University of Dayton Research Inst. 1
ATTN: Dr. L. Huff
300 College Park Avenue
Dayton, OH 45469

Texas Instruments Inc. 1
ATTN: M. H. Luke
1500 Wilson Blvd
Arlington, VA 22209

EG&G 1
ATTN: Dr. D. Machennan
35 Congress Street
Salem, MA 01970