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K-RB LASER PUMP LAMP

LC TECHNOLOGY
64 COMMERCIAL ST.
SUNNYVALE, CA 94086

NOVEMBER 1975

TECHNICAL REPORT AFAL-TR-75-112
FINAL REPORT FOR PERIOD NOVEMBER 1973 – FEBRUARY 1975

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This technical report has been reviewed and approved for publication.

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Alkali Vapor Lamps
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20 ABSTRACT (Continue on reverse side if necessary and identify by block number)

A program was conducted to continue development of a potassium-rubidium arc lamp for laser pumping. Significant progress was made. Improved lamp bake-out, improved filling procedures and incorporation of an internal uranium getter prevented attack of the polished sapphire lamp envelope by the potassium vapor. A new, single envelope, air compatible lamp with nickel endcaps and novel brazed endseals was successfully tested. Dramatic improvement in laser pumping efficiency was achieved by → over

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substituting xenon starting gas for argon. Difficulties were encountered in fabricating an alternative lamp with double endcaps (protected end seals). This prompted a development effort to improve joining technology for this lamp. Encouraging results were obtained with a fused silicide oxidation protective coating for niobium endcap lamps. The best life test result on nickel endcap lamps was 85 operating cycles and 464 hours. Work is continuing on a subsequent contract.

FOREWORD

The work reported herein was performed under Contract F33615-74-C-1027, Project 20280116 for the Air Force Avionics Laboratory, 405B Group, Wright-Patterson AFB, Ohio. The Air Force technical monitor was Mr. James Heitman (AFAL/405B).

The program was carried out from November 1973 to February 1975 in the Engineering Division of ILC Technology, Sunnyvale, California 94086, which is directed by Dr. Len Reed.

Mr. Norman Anderson was principal investigator. The program manager was Mr. Hal Sowers. Mr. James Gaspar and Ms. Linda McLaughlin were responsible for lamp fabrication and testing, with assistance from Mr. William Warren. Dr. Paul Lovoi conducted acoustic emission experiments with sapphire. Valuable contributions to the program were made by Mr. Heitman of the Air Force, who conducted experiments on lamp efficiency and by Mr. Barry Fitzgerald of McDonnell-Douglas Astronautics Company, who was largely responsible for work on oxidation protective coatings. These contributions are gratefully acknowledged. Dr. John Waymouth of the GTE-Sylvania Lighting Division provided technical advice in the area of arc physics for which the author is also indebted.

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1.0 INTRODUCTION

This is the final report on a contract for work at ILC Technology concerned with the development of an alkali metal (K-Rb) vapor arc lamp for laser pumping. A reliable 250 watt CW lamp with a lifetime of several thousand hours is needed for optical pumping of a Nd:YAG laser in a satellite communications system under development by the Air Force. This program was directed at increasing the useful lifetime of single envelope lamps which are required for reasons of laser system efficiency. The original intent was to concentrate effort on improving a protected end seal (PES) lamp. During the program parallel development of another design concept, the nickel endcap lamp, was undertaken.

This program was conducted simultaneously with related work at GTE-Sylvania and McDonnell Douglas Astronautics Company to build and test an engineering feasibility model (EFM) laser transmitter for the same satellite communications system. Separate, concurrent lamp development programs were conducted by ILC under subcontracts with Sylvania and McDonnell Douglas. The Sylvania subcontract involved design and fabrication of K-Rb lamps specifically for use in the EFM laser. McDonnell Douglas partially supported development of the nickel endcap lamp. There was considerable interplay among the three programs at ILC; for the sake of completeness, this report draws to some extent from work sponsored by Sylvania and by McDonnell Douglas.

The report is organized as follows:

In Section 2.0, prior work on K-Rb lamps is briefly reviewed.

In Section 3.0, lamp design requirements are described. These requirements were established early in the project, partly as the result of a cooperative design effort by ILC and Sylvania, and were intended to ensure that lamp development effort would be consistent with EFM laser performance goals and interfacing constraints.

Significant progress was made to prevent lamp envelope degradation resulting from chemical attack by the alkali metal fill. This work is described in Section 4.0.

Primary attention was given to improving the structural reliability of lamps. As noted above, two different lamp design concepts were pursued, the protected end seal (PES) lamp and the new nickel endcap lamp. Work on these two lamp types is detailed in Sections 5.0 and 6.0 respectively.

In Section 7.0, progress in improving the reliability of sapphire for use in lamp envelopes is discussed.

Dramatic improvement in laser pumping efficiency of K-Rb lamps was obtained on the program. An account of this work is given in Section 8.0.

Evaluation of oxidation protective coatings for lamp endcaps is reported in Section 9.0.

A brief summary of accomplishments on the program is given in Section 10.0.

2.0 BACKGROUND

Early investigations of alkali metal arc discharges for optically pumping Nd:YAG lasers demonstrated their superiority over more conventional krypton arc discharges.[1,2] Noble and colleagues at ILC Technology found in subsequent work [3-5] that potassium-rubidium mixtures were particularly efficient sources for laser pumping because of the good spectral match between the broadened, self-reversed resonant line output from potassium and rubidium and the excitation bands for Nd:YAG, as shown in Figure 1.

Early ILC lamps had translucent polycrystalline alumina, as-drawn tubular sapphire, or cored, polished sapphire envelopes. Endcaps of niobium metal were brazed to the envelopes with a Zr-V-Nb alloy. The lamps were either operated in bell jars under vacuum or were enclosed in fused quartz vacuum jackets to prevent oxidation of the endcaps and brazements.

Pumping efficiency considerations eventually dictated the exclusive use of cored, polished sapphire for envelopes because of its superior optical quality.

Use of the fused quartz vacuum jacket was found to be undesirable because of its detrimental effect on arc image quality, a critical parameter in specularly reflective pump cavities. The operation of bare lamps in pump cavities backfilled or purged with inert cover gases was explored with discouraging results. Impurities in the cover gases causes embrittlement and early failure of the niobium endcaps of the lamps.

In response to the need for an air compatible, single envelope lamp, the protected end seal (PES) lamp was conceived. This lamp employs oxidation resistant ceramic-metal end enclosures, frit-sealed to the primary lamp envelope, and evacuated to protect the niobium endcaps and Zr-V-Nb brazements from oxidation. After minimal development work, ILC was able to supply PES lamps to several Air Force laser system contractors for use in the brassboard laser development work that preceeded the present EFM phase. Early bare, quartz-jacketed and PES lamps are shown in Figure 2.

At the beginning of this program PES lamps had two primary life-limiting problems:

1. The solder glass frit seals used to attach the protective end enclosures to the primary envelope were unreliable in service; frit seal leaks occurred early in life and led to failure of the niobium endcaps by oxygen-induced embrittlement.

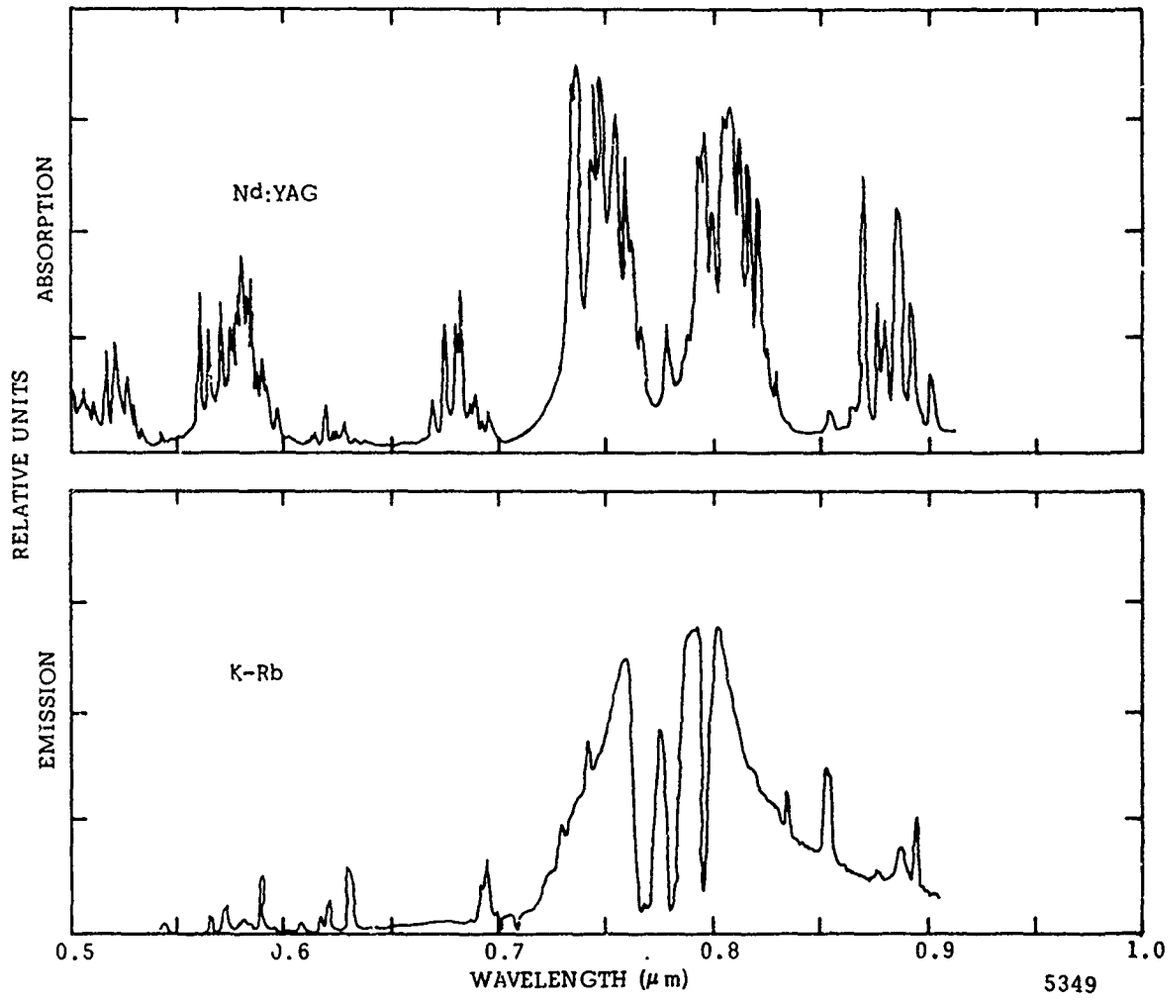
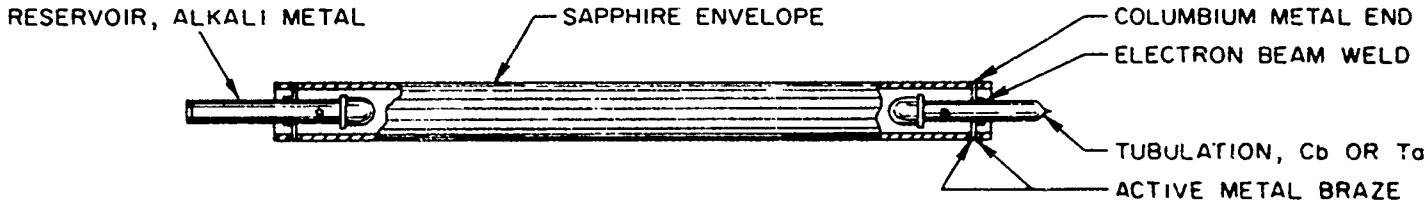
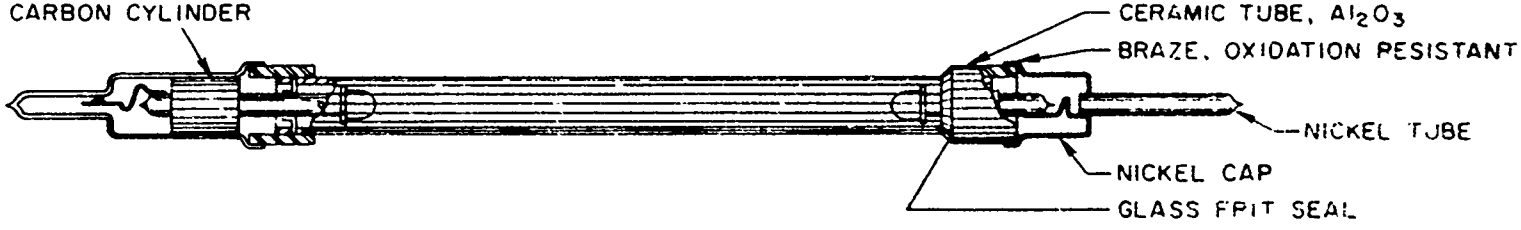


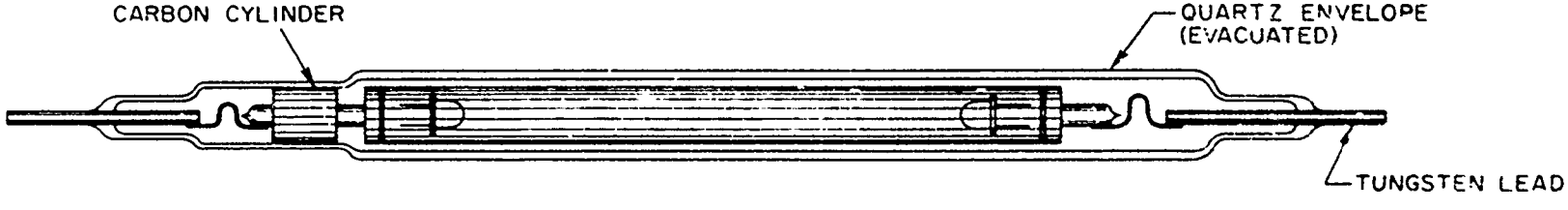
Figure 1. K-Rb Lamp Output and Nd:YAG Excitation Spectra



(a) BARE METAL LAMP - FOR USE IN VACUUM



(b) PES TUBE DESIGN - FOR USE IN AIR



(c) QUARTZ ENVELOPE - FOR USE IN AIR OR LIQUID

Figure 2. Early K-Rb Laser Pump Lamps

2. Bake and fill procedures were inadequate to prevent contamination of the lamp interiors with oxygen, water vapor, and other gases; nor was an internal getter used to scavenge the contaminant gases. Consequently, the sapphire envelope bore would rapidly degrade during lamp operation as the result of chemical reactions with the K-Rb fill (generally referred to as "frosting" reactions because of formation of a whitish, diffuse coating on the sapphire i.d. surface).

The major objective of the program reported herein was to develop and implement solutions to these problems.

3.0 LAMP DESIGN REQUIREMENTS

At the beginning of the program a number of lamp design requirements were established so that concurrent development of the K-Rb lamp and EFM laser would proceed in compatible fashion. These requirements were as follows:

1. Lamps would have a 2.5 inch arc gap and a 5 mm envelope bore diameter. Prior lamps had 1.9 inch arc gaps; the increase to 2.5 inches was made to reduce thermal loading of the envelope and, therefore, increase lifetime potential. Also, the 5 mm bore diameter represented an increase in diameter from the most efficient prior lamps, again for the purpose of reducing thermal loading.

2. Lamps would be capable of sustained operation in air to ensure survivability in laboratory and eventually satellite environments.

3. Cored, polished sapphire would be used for envelopes for best arc image quality.

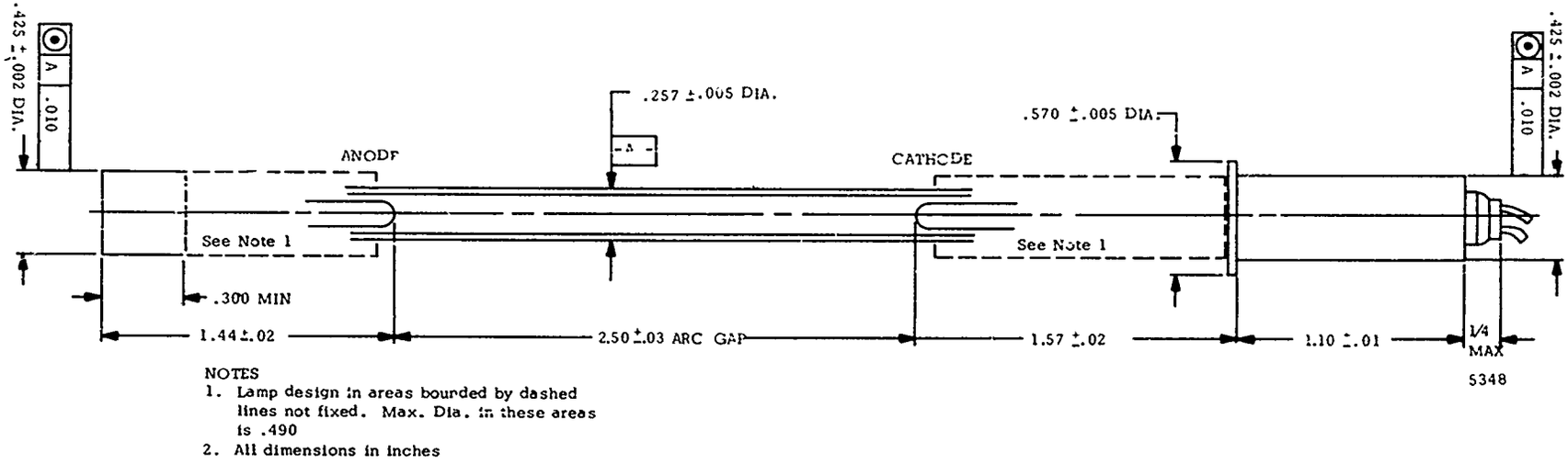
4. Lamps would be of a single envelope design, again for best arc image quality.

5. Lamps would operate with 250 watts of dc electrical input power.

6. Lamps would be equipped with an integral auxiliary heater for controlling the alkali metal reservoir temperature so that an optimum K-Rb pressure for laser pumping could be sustained. Ten watts of additional electrical power were allowed for powering the heater.

7. The lamps would be designed for operation in the EFM laser pump cavity. A cooperative design effort between ILC and GTE Sylvania would be undertaken to generate a practical lamp/laser interfacing design. The resulting dimensional interfacing requirements for the lamp are shown in Figure 3.

Satisfying many of these requirements involved relatively conventional exercises in mechanical and thermal design. However, development of a structurally reliable, single envelope, air compatible lamp, especially with an envelope of sapphire (an ostensibly strong but crack-prone material), called for advancements in the state of the art and would be by far the most challenging aspect of the program.



NOTES
1. Lamp design in areas bourded by dashed lines not fixed. Max. Dia. in these areas is .490
2. All dimensions in inches

Figure 3. Lamp Dimensional Requirements for EFM Laser Interfacing

4.0 LAMP PROCESSING AND INTERNAL GETTERS

A consistent characteristic of K-Rb lamps fabricated on previous programs had been the occurrence of chemical reactions between the alkali metal fill and the sapphire envelope very early in lamp life. The reactions produced a hazy covering of fine crystallites on the envelope bore. This "frosting" effect is detrimental to laser performance because the haze on the envelope scatters pump radiation from the K-Rb arc, reducing the optical coupling efficiency between the lamp and laser rod in specularly reflective pump cavities.

Frosting reactions were studied extensively on the previous program [5]. A tentative conclusion was that much could be gained by eliminating free oxygen from the lamp interior since only reactions involving free oxygen seemed particularly likely.

On this program, solutions to the frosting problem were implemented. Two complementary approaches were utilized with very encouraging results. These were:

1. Glove box processing of lamps, including high temperature vacuum bake out and filling under purified argon; and

2. Use of a uranium metal getter in each lamp.

4.1 Chemical Reactions

In the presence of free oxygen (including oxygen from CO, H_2O, and the like), alkali metals will react readily with Al_2O_3 at elevated temperatures to form aluminates. Potassium, for example, will combine with oxygen and Al_2O_3 to form either $K_2O \cdot Al_2O_3$ or beta alumina, $K_2O \cdot 11Al_2O_3$, by the following reactions:

$$2K(g) + 1/2\ O_2(g) + Al_2O_3(s) \longrightarrow K_2O \cdot Al_2O_3\ (s) \qquad (1)$$

$$2K(g) + 1/2\ O_2(g) + 11Al_2O_3(s) \longrightarrow K_2O \cdot 11Al_2O_3(s) \qquad (2)$$

Both reactions are energetically favorable at envelope temperatures and potassium vapor pressures typical of K-Rb lamp operation. Beta alumina is apparently more stable than the equimolar compound, so reaction 2 is more likely. It is these reactions that can be prevented by elimination of oxygen from the system.

Unfortunately, direct reaction between Al_2O_3 and potassium in the absence of oxygen is also possible. The reaction:

$$6K(g) + 4Al_2O_3(s) \longrightarrow 3\left[K_2O \cdot Al_2O_3\right](s) + 2Al(s,l) \qquad (3)$$

was considered previously [5]. Free energy data for the equimolar sodium aluminate ($Na_2O \cdot Al_2O_3$) were found [6] and corrected to give approximate free energies of

formation for $K_2O \cdot Al_2O_3$ using the differences in ΔF_f^o for K_2O and Na_2O, i.e.,

$$\Delta F_f^o (K_2O \cdot Al_2O_3) = \Delta F_f^o (Na_2O \cdot Al_2O_3) - \Delta F_f^o (Na_2O) + \Delta F_f^o (K_2O)$$

For a potassium vapor pressure of 0.05 atm., reaction 3 has a negative free energy change below approximately 300°C, but is unfavorable (positive ΔF) above this temperature as shown in Figure 4. Kinetics for such a reaction below 300°C. are probably unfavorable.

A similar reaction with aluminum vapor as the product can be written:

$$6K(g) + 4Al_2O_3(s) \longrightarrow 3\left[K_2O \cdot Al_2O_3\right](s) + 2Al(g) \qquad (4)$$

Assuming again a potassium pressure of 0.05 atm., the equilibrium vapor pressure for aluminum was calculated and also is plotted in Figure 4. The pressures are low at temperatures of interest (800-1300°C) indicating that the reaction is not likely to be driven by mass action effects, i.e., transport of aluminum vapor away from reaction sites.

By contrast, formation of beta alumina by the reaction

$$6K(g) + 34Al_2O_3(s) \longrightarrow 3\left[K_2O \cdot 11Al_2O_3\right](s) + 2Al(l) \qquad (5)$$

is considerably more favorable. Again, free energy data for the analogous sodium aluminate, $Na_2O \cdot 11Al_2O_3$, were found [7] and corrected by the same procedure as before. ΔF_r for reaction 5 is plotted in Figure 5. Depending on the accuracies of the original free energy data and the correction procedure, this reaction may be favorable at temperatures of interest and is at least nearly favorable. Equilibrium vapor pressure of aluminum for the reaction

$$6K(g) + 34Al_2O_3(s) \longrightarrow 3\left[K_2O \cdot 11Al_2O_3\right](s) + 2Al(g) \qquad (6)$$

is also plotted in Figure 5. The aluminum vapor pressure is relatively high, suggesting the likelihood of vapor transport of aluminum formed by the reaction to cooler regions of the lamp. Such transport would encourage the reaction to proceed.

The potassium beta alumina product of reactions 2, 5 and 6 is one of a generic class of such aluminate compounds which includes several alkali and alkaline earth aluminates. An interesting characteristic of beta aluminas is that their crystal structures are very similar to and compatible with that of alpha alumina, i.e., sapphire. Beta alumina structures have anion (oxygen) stacking arrangements that are only slightly modified from the hexagonal structure of alpha alumina normal to the close packed (basal) plane. [8] This is significant in light of the fact that envelope frosting observed on this program (detailed in Section 4.4) involved formation of crystallites generally oriented along the sapphire envelope basal planes. Basal plane cleavage cracking observed in some sapphire envelopes may also be associated with beta alumina formation (see Section 7.2).

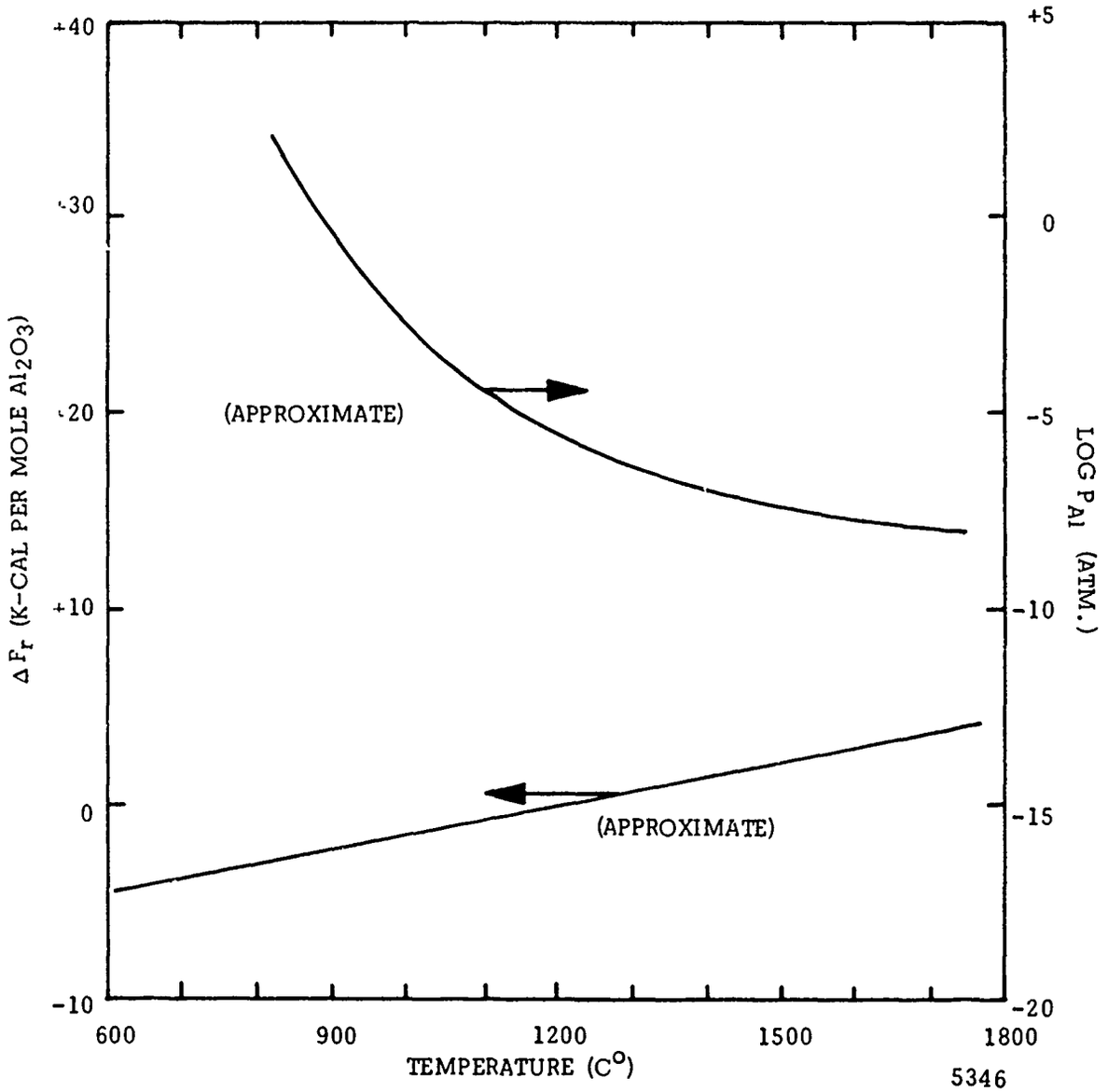


Figure 4. Reaction Free Energies and Equilibrium Aluminum Vapor Pressures for Formation of $K_2O \cdot Al_2O_3$

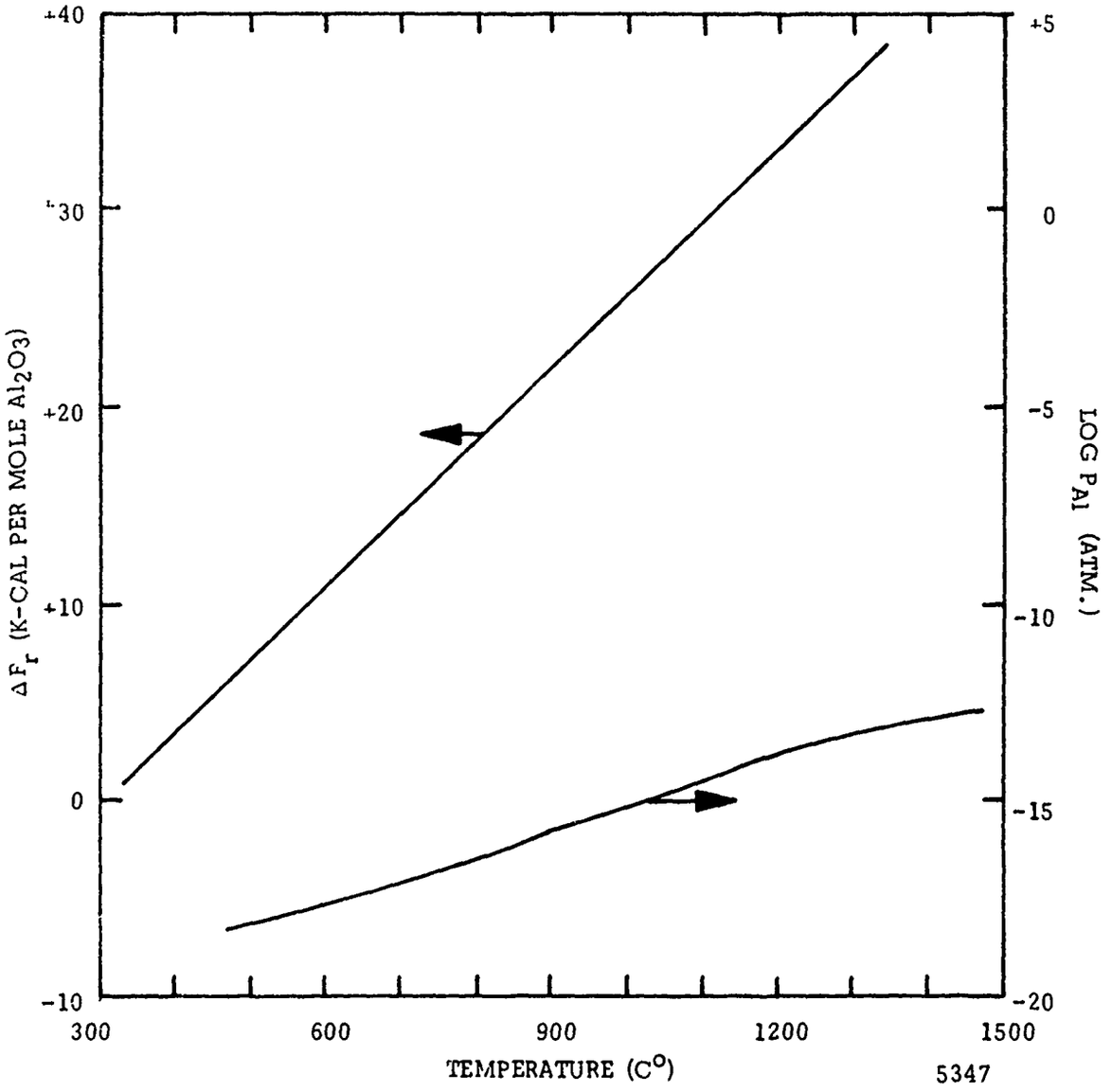


Figure 5. Reaction Free Energies and Equilibrium Aluminum Vapor Pressures for Formation of $K_2O \cdot 11Al_2O_3$

4.2 Lamp Filling Procedures

Various methods of introducing the alkali metals and inert starting gas into lamps had been used on prior programs. Most lamps were baked out on a vacuum manifold using heating tapes and then filled with K-Rb within a polyethylene glove bag purged with argon cover gas. The lamps were reattached to the vacuum fill manifold, also within the glove bag (after bake-out), for final evacuation, backfilling with starting gas, and pinch off.

This procedure was felt to be inadequate because:

1. The argon cover gas was of questionable purity; oxygen and water vapor at relatively high levels were probably present.

2. Prior vacuum bake-out of the lamps was limited to 300°C.

A more elaborate loading system was designed and constructed. The system involved transfer of K-Rb into the lamps by vapor transport in a specially designed, high temperature, valved manifold. The system was equipped with a guard vacuum and furnace to allow lamp bakeout in situ prior to loading. This system, although sound in concept, developed several problems. These included unreliable valves (to meter vapors at up to 600°C) and cold spotting (K-Rb would condense somewhere in the manifold instead of in the lamp).

A commercial, state-of-the-art glove box had been obtained by ILC for lamp processing on another program[9] and was available for use with K-Rb lamps. A decision was made at the beginning of the present program to abandon the vapor transport loading system and utilize the glove box. All lamps fabricated on the program were filled in this glove box under purified argon. The glove box, shown in Figure 6, continually recirculates the argon through hot titanium and copper purifiers to maintain low levels (a few ppm) of oxygen, nitrogen and water vapor.

The box also contains an integral vacuum furnace for lamp bakeout prior to filling, a vacuum/gas manifold for backfilling of starting gas, a manual pinch off tool, and a TIG welder for back-welding of lamp appendage pinchoffs.

The following step-by-step procedure for lamp filling was and is presently used:

1. Empty, open tubulated lamps are placed in the vacuum furnace and baked for two hours at 600° C under a vacuum of 5×10^{-6} torr or better.

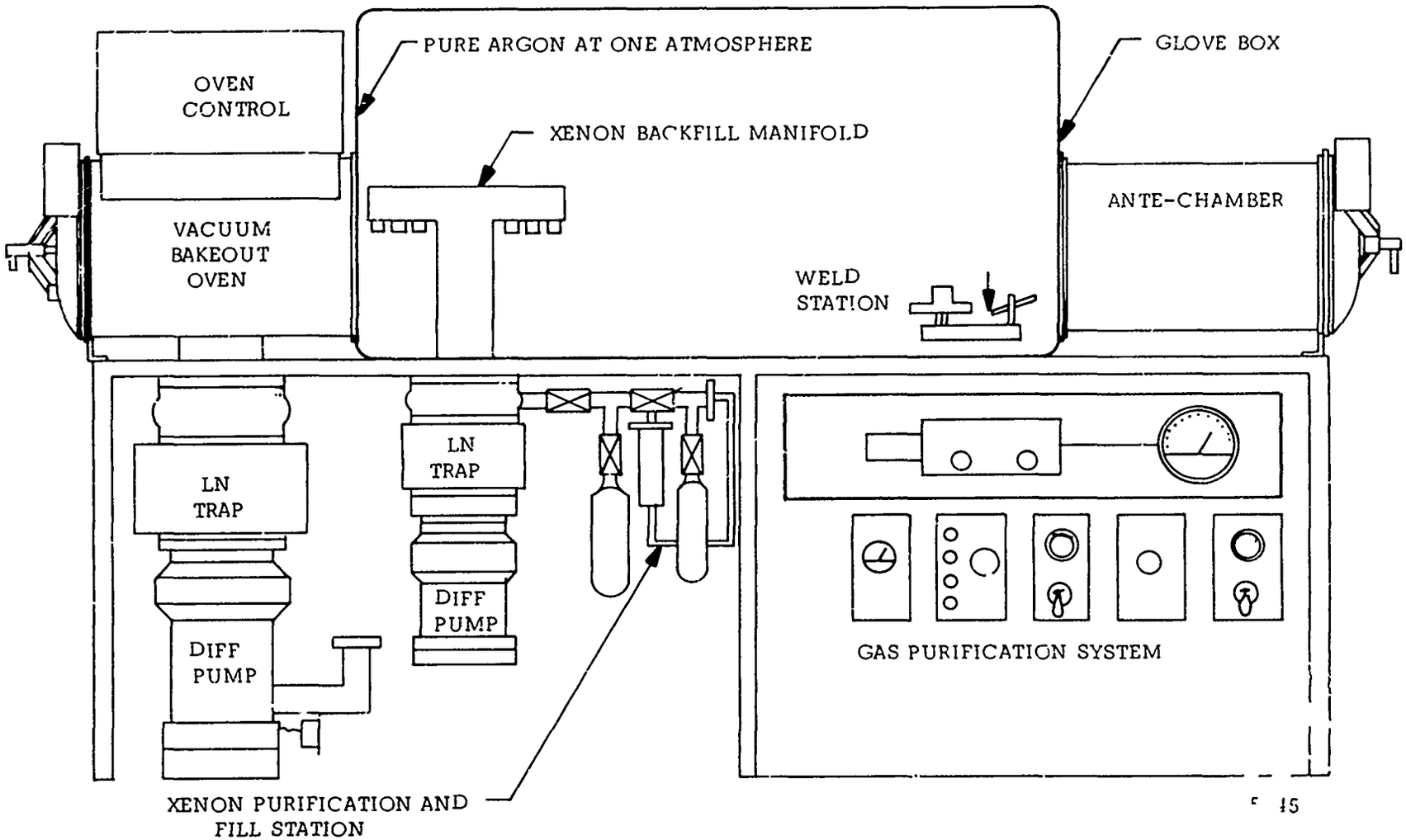


Figure 6. Argon Atmosphere Glove Box for Lamp Filling Operation

2. The lamps are transferred to the main argon chamber where a 10 mg charge of alkali metal is inserted using a syringe-type device followed by insertion of a strip of uranium metal (getter).

3. The lamps are then attached to the vacuum/gas manifold, evacuated, and back-filled with starting gas (either xenon or argon) to the desired pressure.

4. The lamp tubulations are pinched off using a manual pinch off tool that squeezes the tubing to a cold-welded knife edge.

5. The tubulation knife edges are carefully TIG welded within the box for added reliability.

6. The lamps are transferred back into the integral vacuum furnace for a 300°C, two-hour final bake designed to promote reaction of residual oxygen with the internal uranium getter.

4.3 Internal Getters

Although glove box processing ensures that the lamp interiors and K-Rb are not exposed to sources of oxygen contamination during filling operations, consideration had to be given to the presence of residual oxygen in lamp parts (especially tantalum and niobium), in the starting gas, and in the alkali metal charge. Use of a getter internal to the lamp was considered essential in order to deal with residual oxygen from these sources.

Getters of various sorts are commonly used in electron vacuum tubes [10], primarily to tie up residual gases that would otherwise poison electron emitters. Most getters are chemically reactive metals or alloys that will combine with undesirable gases (hydrogen, oxygen, nitrogen, water vapor, carbon monoxide, etc.) to form stable, condensed phase compounds.

Getters are employed in commercial high pressure sodium lamps to scavenge residual oxygen [11]. Use of such getters is undoubtedly a major factor in the very long useful lifetimes exhibited by these lamps.

Metallic uranium, depleted to 0.22 percent U_{235}, was selected as the first getter material for evaluation in K-Rb lamps. Uranium had been used successfully for purifying alkali metals by other investigators [12] in the laboratory and seemed well-suited for lamp use. It combines readily with oxygen at relatively low temperatures (200-300°C) to form very stable oxides. More importantly, the uranium oxide layer formed is permeable to oxygen [13]. Thus the gettering capacity and rate are not limited by the formation of impermeable oxide skins such as is the case with titanium, zirconium, thorium, and other common reactive metals.

A thin strip of uranium is inserted into the fill tubulation of the lamp following alkali metal filling under purified argon as noted earlier.

Results with the uranium getter were sufficiently encouraging (and its use so simple) that no other getters were investigated

4.4 Experimental Results

Except for four lamps fabricated early in the program, three of which were delivered to the Air Force for testing, all lamps tested had been baked and filled by the procedure outlined in Section 4.2 and employed internal uranium getters.

The first four (old process) lamps exhibited general envelope bore haziness early in life as had been typical of lamps from prior programs. In striking contrast, virtually none of the subsequent lamps exhibited early frosting and, except for lamp No. 451, did not develop frosting over lifetimes ranging to 464 hours. These results are summarized in Table I. Apparently, the combination of high temperature vacuum bakeout, filling under purified argon, and use of the uranium getter effectively tied up free oxygen and prevented reactions 1 and 2 from occurring.

Equally significant is the fact that reactions 5 and 6, involving direct reaction between potassium and sapphire, appear not to be occurring at rates sufficient to produce noticeable effects in the first few hundred hours of lamp operation, at least for single envelope (nickel endcap) lamps.

Envelope frosting on lamp No. 451, a quartz-jacketed K-Rb lamp, became noticeable after several dozen hours of operation. Its occurrence on this lamp, but on no others, is attributed to higher envelope temperatures associated with the vacuum-jacketed design (single envelope lamps operate at lower temperatures because of conductive and convective heat transfer to the surrounding atmosphere). The reaction products on the bore of the lamp were in the form of separate, distinct crystallites mostly oriented along the basal plane of the sapphire as shown in Figure 7. This is in contrast to a general cover of haziness typical of earlier, inadequately processed, ungettered lamps. The basal plane orientation is consistent with the assumption of beta alumina formation as discussed in Section 4.1.

After 300 hours of operation, lamp No. 451 was dissected and its internal envelope surface examined with a scanning electron microscope (SEM). Hexagonal shaped crystallites containing aluminum and potassium (as determined by concurrent x-ray analysis) were found at the reaction sites. A representative SEM photomicrograph is shown in Figure 8.

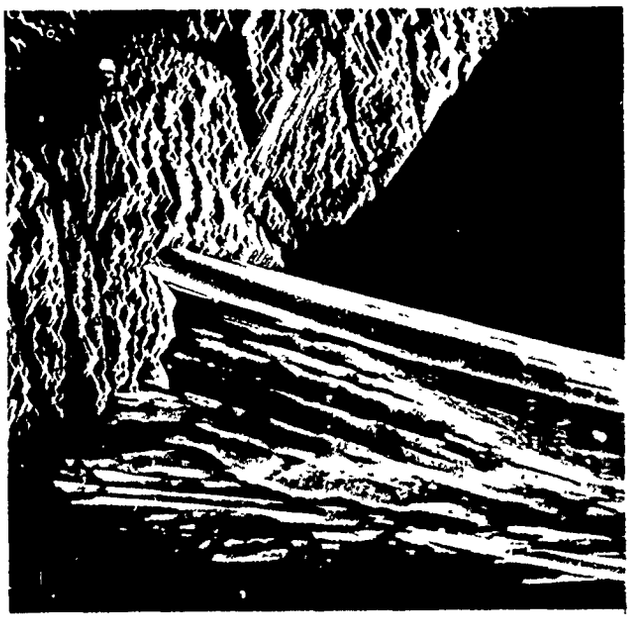
TABLE I

EVALUATION OF NEW PROCESSING PROCEDURES
AND GETTER, LAMP TEST RESULTS

Lamp No.	Lamp Type/Fill	600°C Vac. Bakeout?	Glove Box Fill?	Getter?	Results
419	Quartz-jacketed/K-Rb	No	Yes	No	Severely frosted after 120 hours
420	PES/K-Rb	No	Yes	No	Some haziness after 30 hours
421	PES/K-Rb	No	Yes	No	No data, not yet tested
422	Ni endcap/K-Rb	No	Yes	No	Early frosting, very heavy with increasing time to 1700 hours
426	Ni endcap/K-Rb	Yes	Yes	Yes	No frosting after 103 hours
439	Ni endcap/K-Rb	Yes	Yes	Yes	No frosting after 89 hours
440	Ni endcap/K-Rb	Yes	Yes	Yes	No frosting after 44 hours
442	Ni endcap/K-Rb	Yes	Yes	Yes	No frosting after 51 hours
446	Ni endcap/K-Rb	Yes	Yes	Yes	No frosting after 110 hours
447	Ni endcap/K-Rb	Yes	Yes	Yes	No frosting after 12 hours
451	Quartz-jacketed/K-Rb	Yes	Yes	Yes	Oriented frosting after few dozen hours, see text
459	Ni endcap/K-Rb	Yes	Yes	Yes	No frosting after 220 hours
460	Quartz-jacketed/Rb	Yes	Yes	Yes	No frosting in 300 hours
463	Ni endcap/K-Rb	Yes	Yes	Yes	No frosting after 3 hours
464	Ni endcap/K-Rb	Yes	Yes	Yes	No frosting after 16 hours
465	Ni endcap/K-Rb	Yes	Yes	Yes	No frosting after 464 hours
466	Ni endcap/K-Rb	Yes	Yes	Yes	No frosting after 3 hours
467	Ni endcap/K-Rb	Yes	Yes	Yes	No frosting after 7 hours
468	Ni endcap/K-Rb	Yes	Yes	Yes	No frosting after 131 hours
469	Ni endcap/Rb	Yes	Yes	Yes	No frosting after 3 hours
470	Ni endcap/K-Rb	Yes	Yes	Yes	No frosting after 217 hours
471	Ni endcap/K-Rb	Yes	Yes	Yes	No data, not yet tested
472	Ni endcap/K-Rb	Yes	Yes	Yes	No frosting after 209 hours

5357

Figure 7. Envelope Frosting on Lamp No. 451 (~9X)



5358

Figure 8. SEM Photomicrograph of Envelope Frosting Site (500X)

An identical lamp, No. 460, was constructed, but filled with only rubidium (no potassium). After similar operating times, no such frosting reaction had occurred.

4.5 Discussion

Lamp test results indicate that frosting reactions involving free oxygen have probably been eliminated from K-Rb lamps, presumably as the result of upgraded processing and use of an internal getter.

The energetic feasibility of direct attack of sapphire by potassium and results with lamp No. 451 suggest that long term degradation of lamp envelope bores remains a definite possibility. Prevention of such attack appears feasible by proper control of factors influencing reaction kinetics, especially envelope wall temperature. Continued attention to the frosting "problem" is recommended including determination of the effects of longer lamp operating times and of zero-gravity conditions (with attendant loss of free convection cooling). If a genuine problem exists, consideration can be given to lamp designs that keep envelope temperatures lower (e.g., a larger envelope o.d. to provide more area for heat dissipation), forced gas cooling in the pump cavity, use of helium (high thermal conductivity) cover gas, reduction of the amount of potassium in the fill, and perhaps even reduction in lamp input power (if efficiency considerations allow).

5.0 DEVELOPMENT OF PROTECTED END SEAL LAMPS

At the onset of the program, the protected end seal (PES) lamp represented the baseline lamp design concept. The intent was to redesign this lamp to satisfy requirements outlined in Section 3.0, fabricate lamps for life testing and support of EFM prototype testing, and concurrently upgrade fabrication technology.

In fact, the first EFM-compatible PES lamp design proved very difficult to fabricate. Constraints on lamp length and on mechanical design of the end bell structures had resulted in an end bell assembly sequence that required making awkward weldments and brazements. With perserverence and modifications to some joint designs, a series of prototype lamps survived the entire assembly sequence, but developed cracks and leaks in the solder glass frit seals during the final end bell pump-out and closure operations.

Pressing requirements for K-Rb lamps to support EFM laser development work prompted a change in emphasis to the backup nickel endcap lamp. Effort on PES lamp development centered on upgrading of fabrication technology for the balance of the program.

5.1 Design

The PES lamp design developed on this program, shown in Figure 9, reflected an attempt to adapt then-current fabrication technology, including Zr-V-Nb endcap brazements and solder glass frit seals, to a modified geometry that satisfied the design requirements outlined in Section 3.0.

The primary lamp structure was similar to previous designs (except for the increased arc gap and overall length). Most of the modifications were made in the end enclosure geometries to accomodate the auxiliary heater, provide proper mechanical interfacing with the EFM laser cavity, and (theoretically) improve the mechanical design of the frit seals.

Some specific details are worth noting:

1. Sapphire end bell sleeves replaced polycrystalline alumina (PCA) to minimize thermal expansion mismatch at the frit seal joints (now sapphire-to-sapphire rather than sapphire-to alumina).

2. Direct, rigid, mechanical connection was provided between external end mounting surfaces and the primary lamp rather than indirect support via the frit seal joints so that the

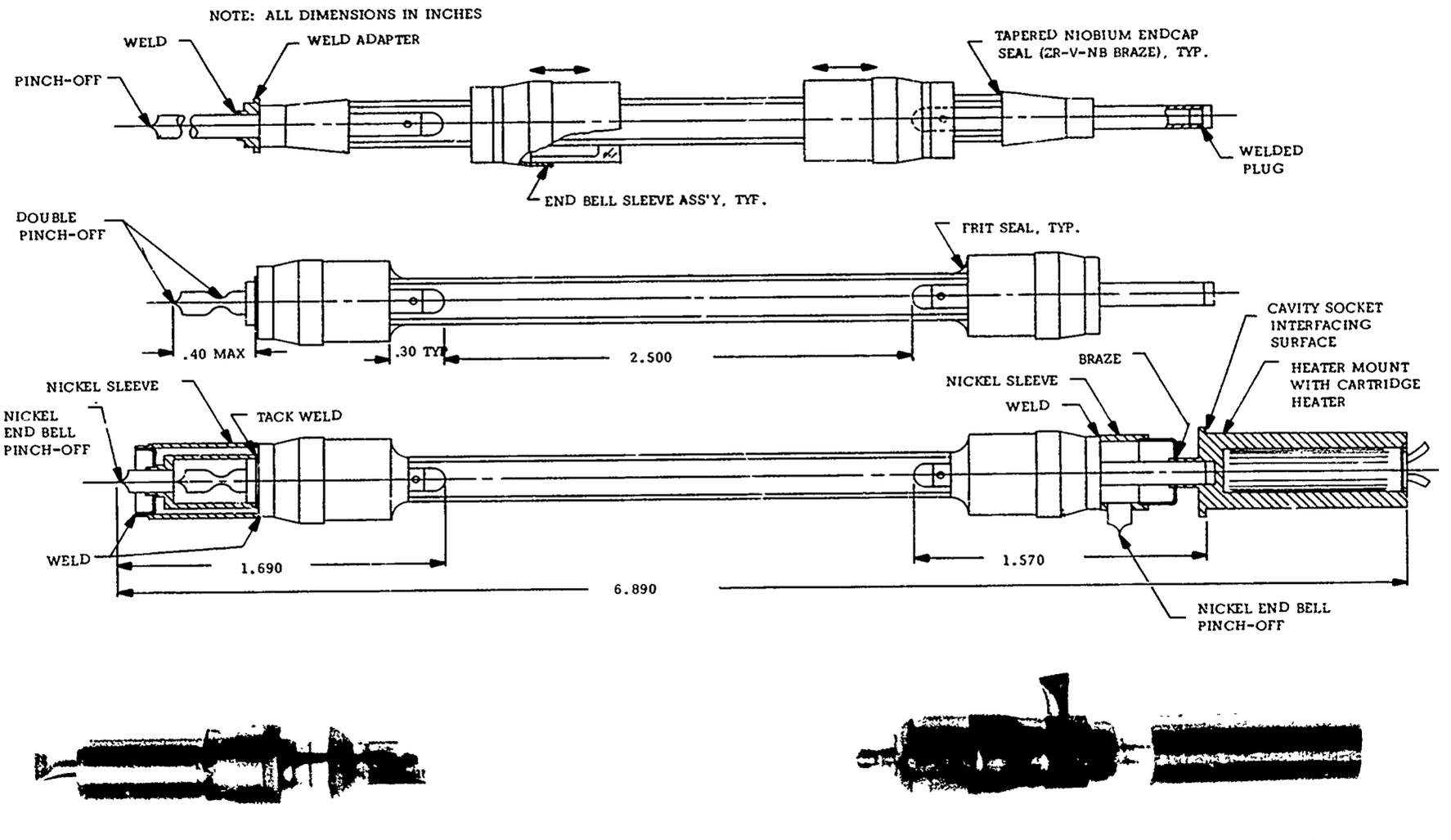


Figure 9. Design of Protected End Seal (PES) Lamp

solder glass would not be unduly stressed during shock and vibration.

3. Substantial clearance was provided between the sapphire end bell sleeves and the primary lamp endcaps to allow subsequent application of oxidation protective coatings to the niobium endcaps as an optional redundancy measure.

5.2 Fabrication

Ten PES lamp fabrication starts were made, all with an assembly sequence as follows (refer to Figure 9):

1. Tapered niobium endcaps are brazed to the sapphire envelope, one end at a time, in an rf-heated bell jar vacuum furnace with Zr-V-Nb alloy.

2. Electrode subassemblies, prewelded by electron beam (eb) welding, are inserted and joined to the envelope assembly, also by eb welding.

3. A weld adapter is eb welded onto the fill appendage.

4. The lamp is evaluated and the fill appendage temperature pinched off to protect the envelope interior during the subsequent frit sealing operation.

5. The sapphire sleeve subassemblies (with nickel adapter skirts pre-attached), which were slid onto the envelope prior to the second niobium endcap braze, are positioned and frit sealed to the envelope with a solder glass. The seals are made, one at a time, in an rf bell jar furnace under purified argon.

6. The fill appendage is reopened, the lamp is baked out in vacuum at 150°C, and is then filled and pinched off in the glove box

7. The pinch-off cover assembly is welded on.

8. The anode end bell nickel extension sleeve is attached by eb welding in two places.

9. The cathode end bell subassembly is slid into position and eb welded.

10. The heater mount is positioned and copper-brazed to the cathode end.

11. The end bells are evacuated and the lamp is operated at full power to promote degassing of the end bell interiors.

12. The end bell appendages are pinched off, completing the assembly sequence.

Initially, considerable difficulty was encountered in welding several of the end bell joints. By improving parts fits and utilizing eb welding rather than TIG welding as originally planned, these problems were eventually overcome.

The primary technical problem with the lamps developed during the final end bell pump-out step. Every lamp that had survived previous assembly operations developed cracks and leaks in the solder glass seal when cooled after the end bell degassing operation, although in at least one instance a frit seal failed during heat up. Frit seal cracking has always been more or less of a problem. In previous PES lamps the frit seal joints were located nearly adjacent to the electrode tips. Considerable glass was used to produce massive seal fillets. Cracks frequently developed in the glass du[illegible] lamp cool-down but often did not entirely penetrate through the fillet. During subsequent full power lamp operation the temperatures at the frit seals were high enough to cause the cracks to fuse together. In this way, previous PES lamps were often able to endure several on-off cycles.

The new lamp design had two features that apparently were contributing causes to the cracking problem. First, the frit seal fillets were situated further from the electrodes tips. This feature, combined with lower envelope temperatures resulting from the longer arc gap (lower thermal wall loading), probably precluded any "refusing" of the seals during service. Second, the sapphire end bell sleeve was considerably larger than the alumina sleeve in the previous PES lamp design (to provide space inside the end bell for application of a protective coating to the niobium as noted earlier). It is likely that temperature gradients in the seal region were more severe in the new design, thus imparting higher thermal stress to the frit joint.

The inability to successfully complete fabrication of the redesigned PES lamp led to a decision to postpone attempts to fabricate such lamps and concentrate efforts on upgrading joining technology, particularly the frit seal.

5.3 Frit Seal Development

A commercially available solder glass, Owens-Illinois Article 01338 (IN-3) glass, had been used for PES lamp frit seals. The glass has certain merits, principally its excellent thermal expansion match with Al_2O_3 and its relatively high softening point (710°C) compared with other common solder glasses. However, in the PES lamp application the 01338 glass is at best a marginal choice. Typical temperatures at the frit seals during lamp operation are close to or above the 710° softening point; under such conditions the seals can be expected to have little mechanical strength.

It was noticed that 01338 glass frit seals contained many fine bubbles; the presence of such bubbles undoubtedly further compromises the mechanical strength of the glass. This observation led to serious questioning of the procedures then used to make the seals.

The bubbles were suspected to be the result of water vapor desorption from the glass during melting. It was further suspected that excessive water content in the glass was associated with the use of a water-base slurry to apply the frit to the joint areas.

Also of concern was the potential for oxygen pickup by refractory metal components on the lamp and embrittlement during the frit sealing operations in an argon cover gas. Early work had already shown that the glass severely frothed if melted in vacuum, probably again because of excessive sorbed water from the original slurry.

In the first phase of frit seal development on the program, ways of improving frit sealing procedures with the 01338 glass were explored. It was quickly found that by using a slurry with methyl alcohol rather than water, bubble-free seals could be made, even in vacuum. Trial frit seal joints made in this way were subjected to thermal cycling (room temperature to 700°C). The seals survived several dozen thermal cycles without cracking. It should be noted, however, that in the cycle tests samples were heated and cooled in a relatively uniform manner, i.e., without significant temperature gradients.

After thermal cycling, one sample was held at 700°C in air overnight. The glass severely devitrified and developed leaks during this treatment. This result in particular indicated that 01338 glass would be a poor choice for prolonged high temperature service in PES lamps.

Meanwhile, an intact PES lamp structure was salvaged and resealed with 01338 glass using the new vacuum procedure. Again, the

frit seals cracked during the final end bell pump out and bake operation. Apparently the thermal stress conditions were sufficiently unfavorable to negate improvements in the sealing procedure (seals now free of bubbles) and in the thermal expansion match of seal members (now both sapphire).

It was clear that a much better glass was needed for PES lamp frit seals, a glass with a softening point safely above anticipated service temperatures and amenable to vacuum melting. Attention was, therefore, shifted to the evaluation of high temperature sealing glasses. Four candidates were selected.

1. Corning Code 1731 glass, a calcium aluminate composition containing some silica (SiO_2), which has been used successfully at ILC for joints between polycrystalline alumina members and has a 1590° C sealing temperature.

2. A CaO-Al_2O_3-SiO_2 composition developed for Al_2O_3 joints,[14, 15] which has a 1300-1500° C sealing temperature range.

3. Kingman Feldspar, which has been used successfully for sapphire joints[16] and seals at approximately 1425° C.

4. A proprietary calcium aluminate composition, generously provided to ILC by the GTE Sylvania Lighting Division, which is used with considerable success for Al_2O_3-Nb seals on commercial high pressure sodium lamps.

Several attempts to make sapphire-to-sapphire joints with Code 1731 glass were unsuccessful. The sapphire parts cracked, either during cool down from the sealing temperature or some hours later at room temperature.

At the end of the program, crack-free, vacuum tight, sapphire-to-sapphire seals had been made with Kingman Feldspar and the CaO-Al_2O_3-SiO_2 glass at 1450° C in a hydrogen-nitrogen cover gas mixture. The Sylvania glass had not yet been tried.

Continuation of work on these high temperature sealing glasses is being conducted on a subsequent program.

5.4 CVD Seals

There was strong interest in having an alternative to the brazed sapphire-to-niobium endcap seal in PES lamps. A very attractive substitute is the CVD seal, made by chemical vapor deposition of niobium directly onto the sapphire.

The CVD seal eliminates the need for endcap brazing and thereby facilitates construction of PES lamps with new high temperature frit seals. A disadvantage of Zr-V-Nb brazed seals is that the new frit seals are made at or above the melting temperature of the brazing alloy. The time/temperature schedules for the two joining operations are not compatible, precluding concurrent brazing and frit sealing.

In addition, the Zr-V-Nb brazements, although reliable in service and characterized by high manufacturing yields, require careful pre-braze assembly techniques. There is considerable variability in the appearance of such brazed joints. Furthermore, the brazing operation apparently must be carried out in the rf vacuum bell jar furnace which subjects the sapphire envelopes to a high axial temperature gradient. Because of the temperature gradient, deposition of vapor from the molten brazing alloy on cooler portions of the envelopes occurs. Attempts to make Zr-V-Nb seals in a high vacuum, tungsten element (isothermal) furnace have been unsuccessful, probably because of the slower post-braze cooling rate inherent in this well insulated furnace compared with the apparently necessary quick cool-downs achievable in the bell jar furnace.

CVD seals are made by heating an assembly including the Al_2O_3 part and a sacrificial mandrel to 1000°C or higher in a stream of $NbCl_5$ vapor and hydrogen. Thermal decomposition of the pentachloride vapor on the assembly surface produces an arbitrarily thick coating of dense, pure, ductile niobium that is well bonded to the Al_2O_3. Subsequent dissolution of the mandrel in an appropriate acid leaves a CVD-sealed envelope assembly equivalent geometrically to the previous brazed assemblies.

Use of the CVD seal allows all of the above-mentioned disadvantages of Zr-V-Nb brazed seals to be avoided.

Trial CVD-sealed envelope assemblies were procured from Chemetal, Pacoima, California. The configuration is shown in Figure 10. Chemetal encountered some initial problems with mandrel design and process variables but was able to overcome them. Two complete assemblies were delivered to ILC.

Examination of the CVD assemblies revealed that the HCl vapor product of the CVD reaction had attacked adjacent high temperature sapphire surfaces causing severe etching of the originally polished finish. This phenomenon can probably be avoided by modification of the process gas stream and/or other process variables. It is also feasible to have the CVD seals made prior to final grinding and polishing of the sapphire so that etched regions can be removed during those operations.

The CVD seals were evaluated as follows:

One seal was used in a welding test. A standard PES lamp electrode assembly was TIG welded into the CVD assembly. This operation presented no problems.

Another CVD seal was thermal cycled twice to 1500°C in vacuum and remained vacuum tight. It was then sectioned for metallographic examination. A photomicrograph of this seal is shown in Figure 11.

The other double-ended assembly was used in the fabrication of a quartz-jacketed lamp. One of the CVD seals in this assembly developed a leak during lamp fabrication which prevented completion and testing of the lamp. This seems of no particular concern given the fact that the CVD assembly was among the first of this type ever fabricated and undoubtedly reflected a "learning curve" situation at Chemetal.

Results with CVD seals were sufficiently encouraging to justify additional evaluation for use on future PES lamps. This work is now underway as part of a follow-on program.

5.5 Discussion

Despite the difficulties encountered in the fabrication of PES lamps on this program, the design concept is still considered to be very attractive. A lamp with high temperature seals having near ideal thermal expansion matches seems, in principle, to be most capable of surviving hundreds of on-off cycles and thousands of hours of operation. It is believed that the present problems can be solved, very probably by incorporation of the new sealing technology presently under development (CVD seals, high temperature frit seals), and by improvements in mechanical design.

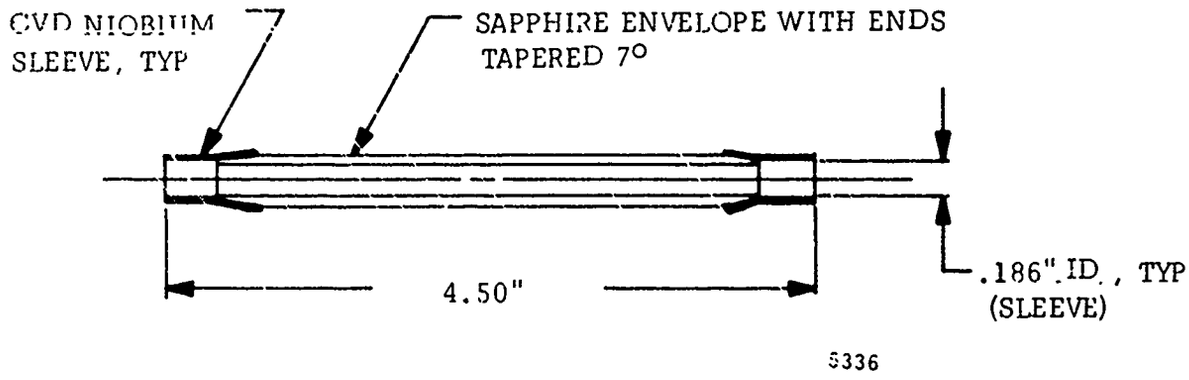


Figure 10. CVD Seal Specimen

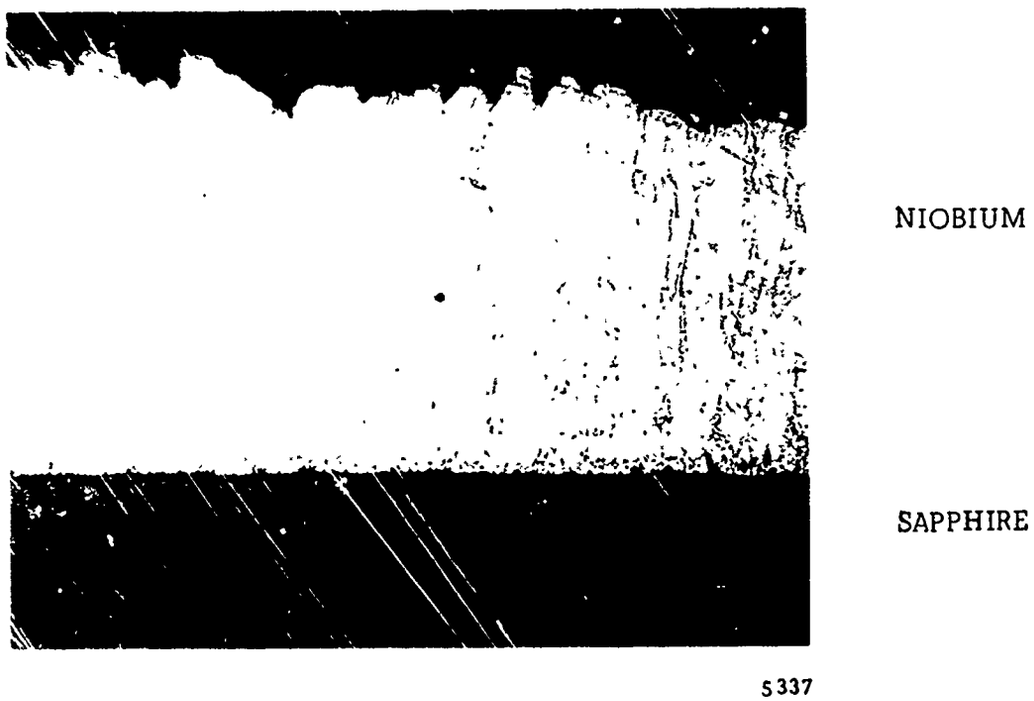


Figure 11. Photomicrograph of CVD Seal

6.0 DEVELOPMENT OF NICKEL ENDCAP LAMPS

Prior technology for fabricating of alkali metal vapor arc lamps has relied almost exclusively on the use of niobium metal end caps for two principal reasons:

1. Niobium, in pure form or as Nb-1% Zr alloy, has an excellent thermal expansion match with Al_2O_3 which, in combination with its good ductility, allows Nb-Al_2O_3 seals to be made with very low residual mechanical stress.

2. Substantial joining technology exists for making reliable Nb-to-Al_2O_3 seals that can function at elevated temperatures and resist chemical attack by alkali metals.

A major disadvantage of niobium endcaps, and often of the seals as well, is their vulnerability to oxygen-induced degradation. Niobium will oxidize catastrophically at elevated temperatures in air. Even when oxygen levels are relatively low, niobium will dissolve oxygen and become embrittled. Thus lamps with niobium endcaps must be operated in vacuum or with some other, often cumbersome, provision for protecting the niobium (e.g., the PES lamp design).

Prior to this program, ILC Technology had developed proprietary sealing technology for joining pure nickel members to high alumina ceramics. This technology was unique in that such brazed seals were found to be both compatible with alkali metals and oxidation resistant at high temperatures.

The availability of this technology prompted the introduction of a new K-Rb laser pump lamp design concept, the nickel endcap lamp. Such a lamp has a simple configuration, with exposed endcaps and seals. It is easy to fabricate and, because of its inherent simplicity, should be managable from a reliability standpoint than the complex PES lamp design.

Development of the nickel endcap lamp was originally undertaken as a backup approach to the PES lamp. When difficulties were encountered in fabricating PES lamps, the nickel endcap lamp became the primary lamp fabricated and tested on the program.

6.1 Basic Materials Considerations

6.1.1 Nickel Endcaps

Pure nickel has several attractive features as an endcap material. Its oxidation resistance is excellent up to temperatures well above the 600-700°C range typical of lamp service[17,18]. Pure nickel has excellent compatibility with alkali metals. Its solubilities in liquid sodium and potassium, for example, are in the few parts per million range[19,20]. As with niobium, dissolution and attack of nickel by alkali metals are strongly influenced by the presence of dissolved oxygen in both the metal member and the liquid[21]. In this regard nickel offers another significant advantage: oxygen and other dissolved gaseous impurities can be readily and nearly completely removed by high temperature vacuum bakeout operations[22] in contrast to the difficulty of removing dissolved gases from niobium. Furthermore, the solubility of oxygen in pure nickel is very low in comparison with its solubility in niobium[23].

A disadvantage of nickel for endcaps is its high thermal expansion coefficient, nearly twice that of Al_2O_3. However, this disadvantage may be largely offset by nickel's good ductility.

6.1.2 Brazed Seals

The nickel-to-sapphire seal is made by brazing in vacuum. The brazing alloy contains zirconium, which imparts to the braze melt high chemical reactivity, promoting good wetting of and bonding to the Al_2O_3 interface. It contains no constituents that have high solubility in alkali metals or are otherwise known to be subject to alkali metal attack. Because of this fact, no specific evaluation of the seal's resistance to alkali metal corrosion was undertaken on this program.

6.2 Oxidation Testing of Brazed Seals

Prior work[24] had indicated that the zirconium-containing brazement was inherently oxidation resistant. Further characterization of its oxidation behavior was undertaken on this program to determine whether such a seal could be expected to survive in marginal atmospheres for the duration of a space mission (thousands of hours) as well as for at least one hundred hours in ambient air during laser testing in the laboratory.

A group of representative nickel-to-Al_2O_3 seal samples were obtained from ILC's Ceramic-Metal Product Division for long term oxidation testing. These seals were placed in a laboratory air furnace

and heated to 800°C. Samples were pulled from the furnace after 72 hours, 272 hours, 504 hours, and 1005 hours, helium leak-checked and then sectioned for metallographic examination.

The results of these tests were extremely encouraging. Even after 1004 hours at 800°C the seals were intact and only superficially oxidized. Photomicrographs of tested seals, shown in Figure 12, show that oxide film h has developed on the braze fillet surface where exposed to air and has grown to approximately 1.5 mils thickness in 1004 hours. Beneath the oxide layer is a second zone apparently affected by a diffusion mechanism, including formation of scattered voids, that has penetrated into the brazement approximately 12 mils.

Since such seals in lamps would be operating at temperatures at least 100°C lower than the test temperature, would be approximately 70 mils wide, and would be exposed to an atmosphere relatively free of oxygen in ultimate space service, the results indicate that the seals would not fail from oxidation during lamp operation in the laboratory or in a satellite.

6.3 Prototype Nickel Endcap Lamps

An initial group of four nickel endcap lamps was fabricated for in-house evaluation at ILC. All employed sapphire envelopes procurred before the decision to increase the arc gap to 2.5 inches had been made; consequently, they had 1.9 inch arc gaps. The general configuration of these lamps is shown in Figure 13. The first two lamps had nickel eyelet endcaps. Subsequent lamps had endcaps of a cup configuration. The four initial lamps were filled with K-Rb and 100 torr of argon. They were operated until failure at a sustained input power level of 190-200 watts (to give approximately the same envelope thermal loading as 2.5 inch arc gap lamps operating at 250 watts) without on-off cycling.

The first lamp, No. 422, had been built with brazed metal-to-metal electrode and end assembly joints (while techniques for TIG-welding these joints were developing in parallel). The lamp was intended to be a vehicle for "weeding out" fabrication problems, and, although vacuum tight, was not vacuum baked prior to filling with K-Rb, nor was a uranium getter used. Very early in life the envelope bore became heavily frosted, reflecting the inadequate processing and absence of a getter. Nevertheless, the lamp operated in air for nearly 1800 hours at 190 watts before the anode contacted the envelope, causing it to crack.

In subsequent lamps of the series, Nos. 426, 439 and 440, cup-type endcaps, welded metal-to-metal joints, improved processing

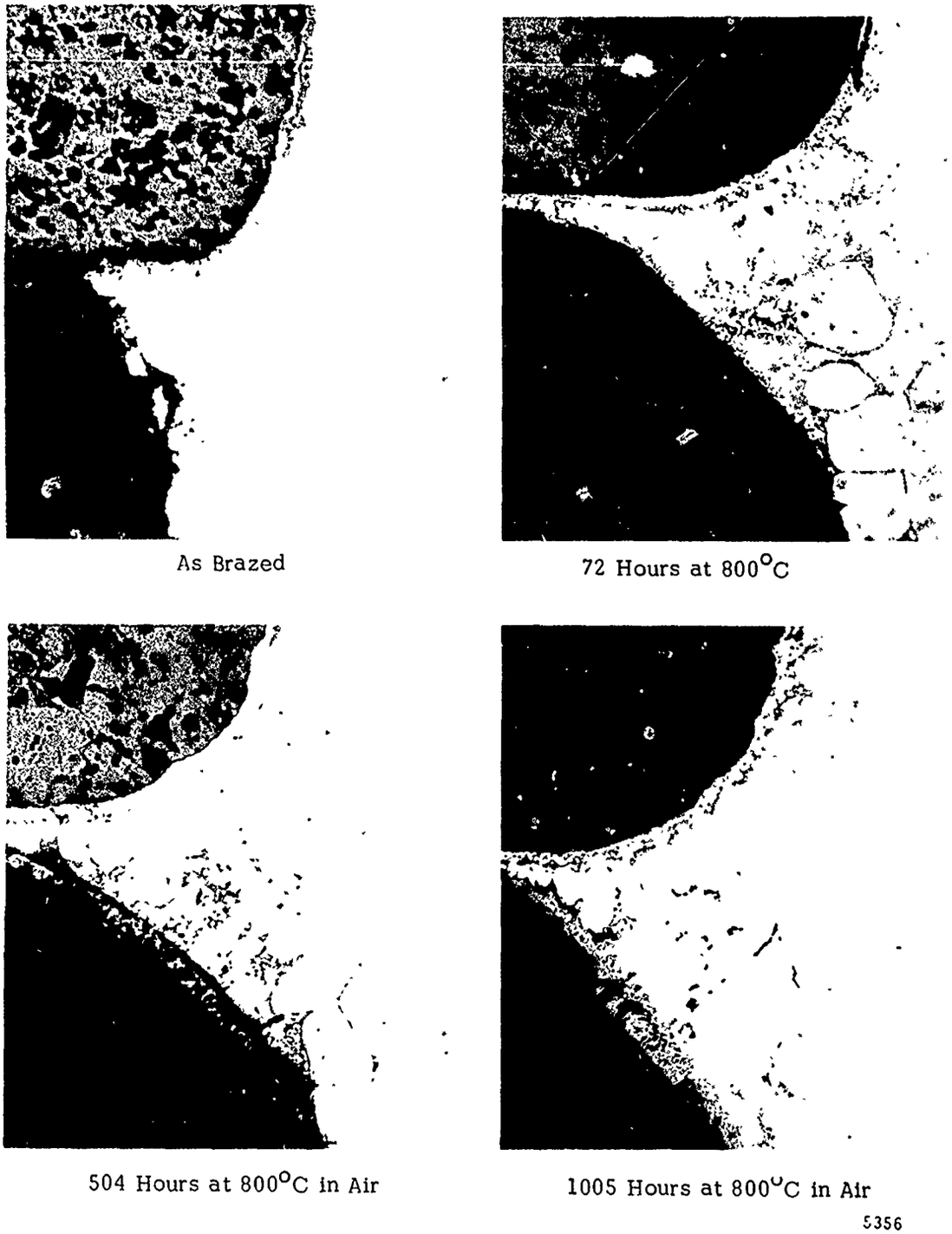


Figure 12. Photomicrographs of Ni/Al_2O_3 Seals Before and After Oxidation Tests (Magnification 200X)

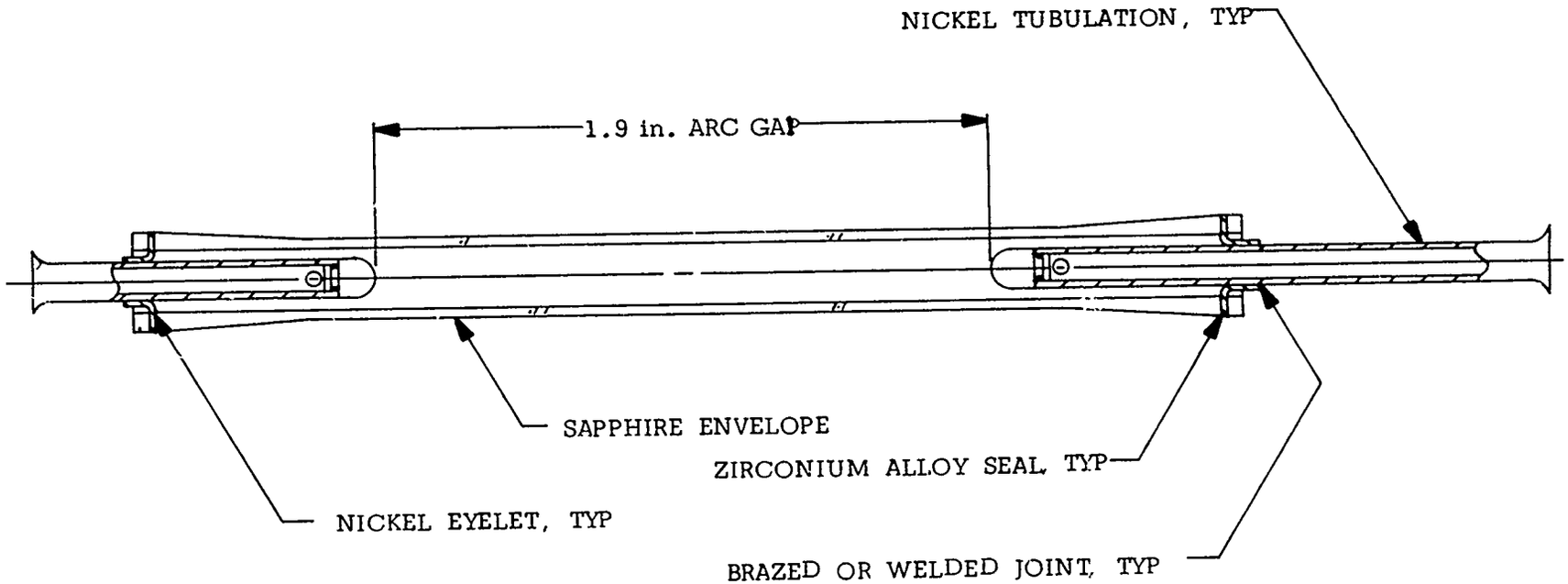


Figure 13. Initial Design of Nickel Endcap Lamps

(vacuum bakeout), and uranium getters were incorporated. These lamps operated for 103, 89 and 44 hours, respectively. Failures appeared to be the result of leaks in overbrazed or overwelded regions in the end structures.

Additional modifications to brazing and welding procedures were incorporated in the second series of prototype nickel endcap lamps, Nos. 442-447. These lamps had 2.5 inch arc gaps, auxiliary heaters, and all other features necessary for operation in EFM laser prototype test cavities. Their design is shown in Figure 14. The lamps were the first of the nickel endcap design to be released for use in laser experiments at GTE Sylvania and the Air Force 405B laboratory.

The cyclic on-off operation of these lamps during initial burn-in at ILC and laser testing at Sylvania and the Air Force 405B laboratory revealed that a serious structural deficiency existed, namely the inability of the nickel-to-sapphire end seals to withstand thermal cycling. The best of these lamps survived nine operating cycles, not nearly adequate for either laboratory use or for ultimate mission requirements. Consequently lamp fabrication work was postponed for several months while means of increasing seal cycle durability were explored.

Life test summaries of the prototype nickel end cap lamps are given in Table II.

6.4 Development of Low-Stress Seals

6.4.1 Analysis

Failure of the prototype lamps had often occurred by fracture of the sapphire envelope near either brazed end seal. It was apparent that mechanical stresses of high magnitude were present in the sapphire and were probably the result of the poor thermal expansion match between the sapphire and the seal components.

A theoretical analysis of thermomechanical seal stresses was performed by McDonnell-Douglas. Inadequate data were available on high temperature elastic and deformation properties of the seal components to permit a rigorous quantitative analysis to be performed. A semiquantitative treatment of the problem indicated that high bending forces were acting on the sapphire envelope near the seal with attendant high axial "fiber" stresses in its exterior and interior surfaces, as shown in Figure 15. The stresses develop during cooling from the original brazing temperature and from subsequent lamp operating temperatures.

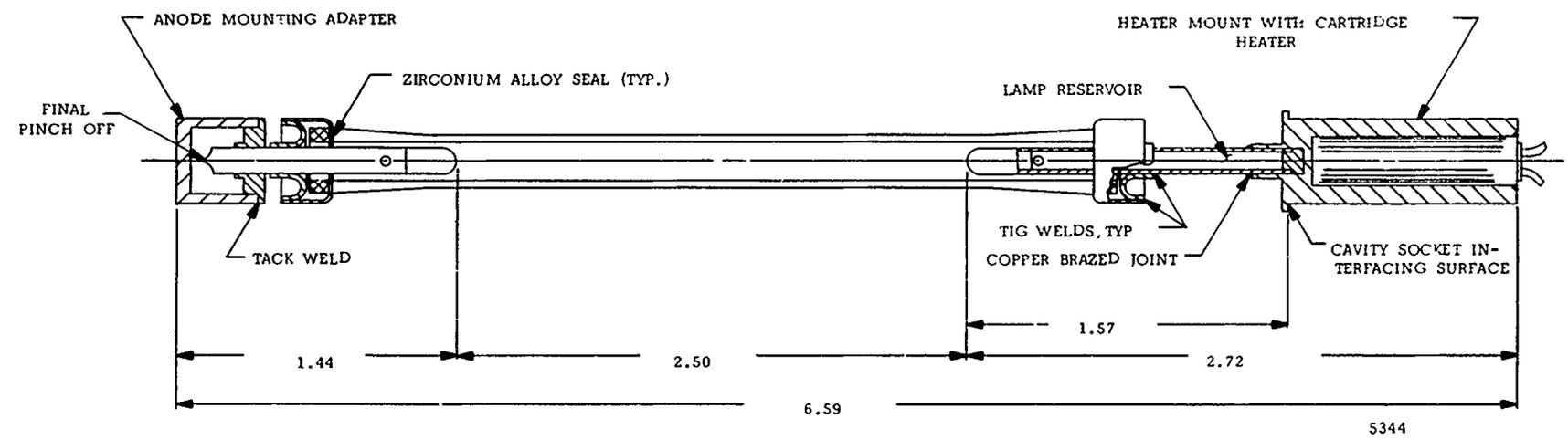


Figure 14. Design of First EFM-Compatible Nickel Endcap Lamp

TABLE II

LIFE TEST RESULTS, PROTOTYPE NICKEL ENDCAP LAMPS

Lamp No.	Description	Testing Mode	Lifetime Cycles	Lifetime Hours	Failure Mode
422	1.9 in. arc gap; all brazed construction; eyelet endcaps	continuous at 190 W	1	1792	cathode tip contacted and cracked envelope, due to melted electrode braze joint.
426	1.9 in. arc gap; welded construction	continuous at 200 W	1	103	cause of failure unknown; envelope cracked near seals during abrupt shutoff.
439	1.9 in. arc gap; cup replaces eyelet for endcap	continuous at 200 W	1	89	suspect leak in seal; anode support melted indicating excessive current before failure.
440	same as no. 439	continuous at 200 W	1	44	leak in endcap-tubulation weld
442	2.5 in. arc gap; modified brazing procedure for seals	during laser tests at Sylvania, 405B, at 250 W	9	51	envelope cracks in seal
443	same as no. 442	on bench at Sylvania at 250 W	1	6	envelope cracks near seals during shutdown.
444	same as no. 442	at 250 W, aging at ILC, laser tests at Sylvania	2	10	envelope cracks near seal during shutdown
445	same as no. 442	at 250 W, aging at ILC	1	3	envelope cracks near seal during shutdown.
446	same as no. 442	at 250 W, aging and laser tests at ILC, laser tests at Sylvania	7	110	leak due to envelope cracks in anode end
447	same as no. 442	at 250 W, laser tests at AF 405B	5	12	envelope cracks near seal

Various possible solutions to the seal stress problem were considered, including changes in the seal geometry to "knife-edge designs. The 'knife-edge" design, shown in Figure 16, seemed particularly attractive on the basis of calculated bending stresses in the sapphire envelope and was explored experimentally. Trial specimens with "knife-edge" seals failed by local shear fracture at the ends of the sapphire first. This approach was not pursued further.

Within the confines of the existing sandwich seal geometry, consideration was given to a variety of potentially beneficial design modifications including:

1. reduction of seal cup radial extension, i.e., smaller cup diameter

2. reduction in thickness of the braze-affected zone, i.e., use of less original brazing material, adjustments in brazing procedures, etc.

3. stress relief annealing during cool down from brazing

4. use of alloys with lower thermal expansion than nickel for the endcap material, e.g., Kovar

5. use of stress compensating members in the seal.

An experimental effort was undertaken to evaluate the various ideas proposed to reduce seal stress. This effort consisted of two parallel tasks, one to evaluate mechanical modifications of the seal design using existing brazing procedures, and the other to improve the brazing procedure to produce a seal with minimum mechanical effects on the adjacent sapphire (e.g., a thinner seal).

Considerable progress was made on the first of these tasks during the program. Work on both tasks is continuing on a follow-on contract.

6.4.2 Seal Design Improvements

Potential seal design improvements were evaluated experimentally using trial seal assemblies, shown in Figure 17, which were subjected to repetitive thermal cycling until failure by gross fracture or loss of vacuum tightness occurred. Thermal cycling was carried out in vertical tube furnaces. Samples were contained in wire baskets and mechanically raised and lowered into and out of the hot zone to produce an approximately sinusoidal time-temperature curve with 715° and 100° C maximum and minimum points in a twenty-minute full cycle.

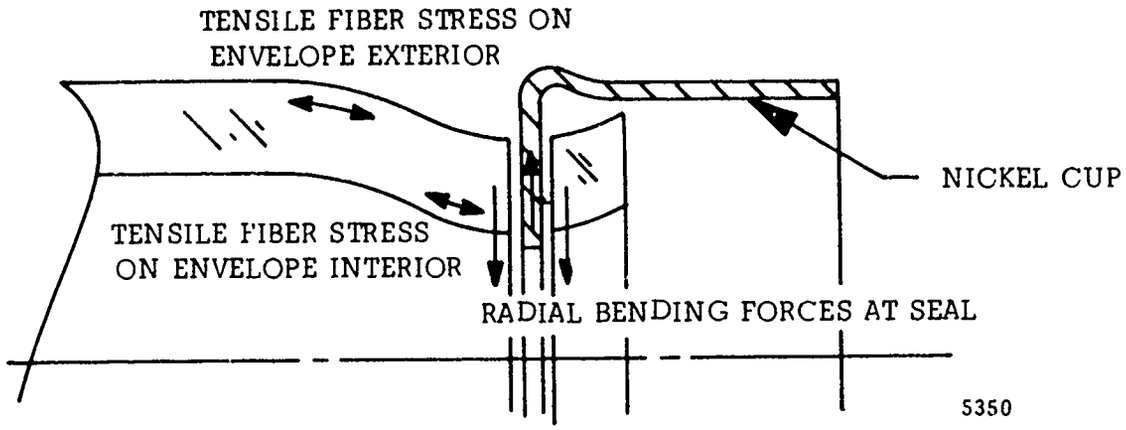


Figure 15. Forces and Stresses at Nickel Endcap Lamp Seals

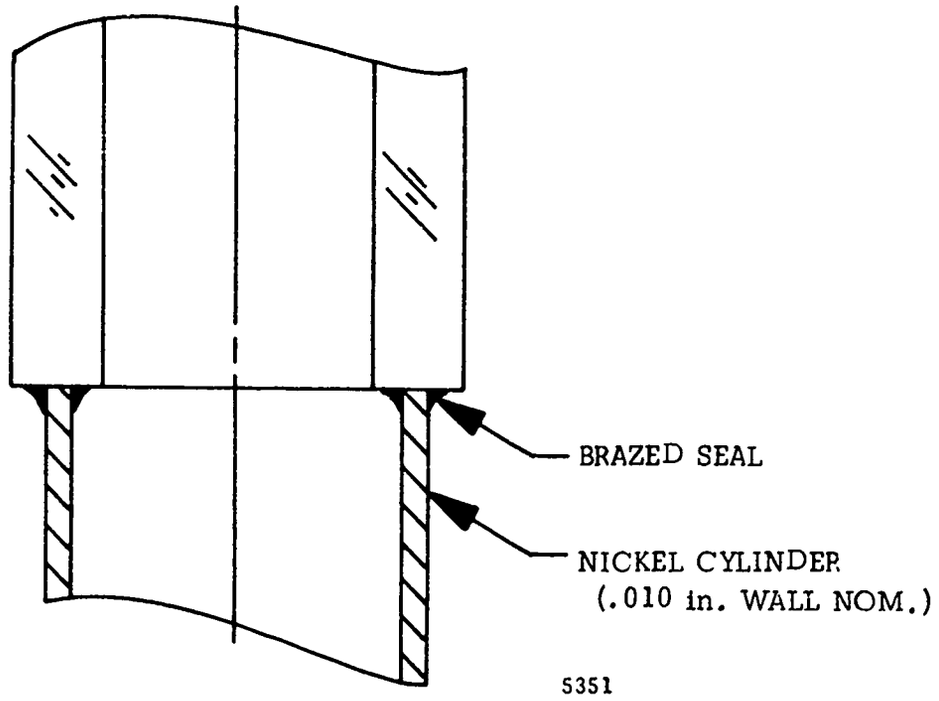


Figure 16. Knife-Edge Seal Configuration

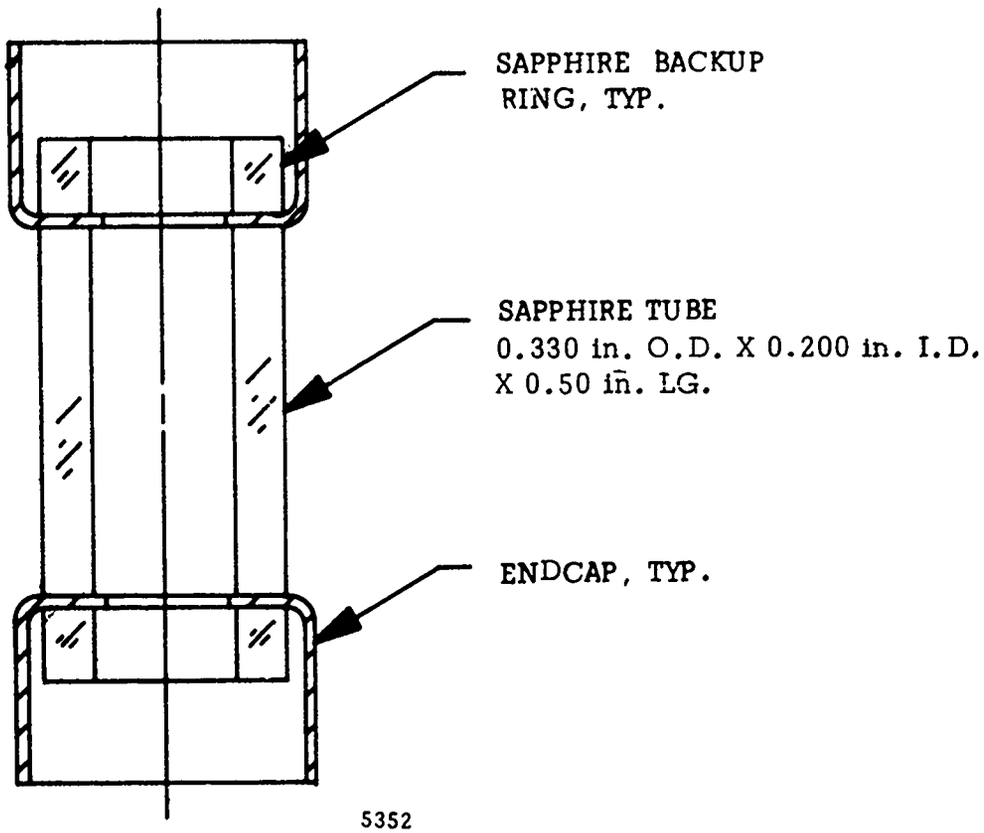


Figure 17. Brazed Seal Evaluation Specimen

To establish a baseline, the first specimens cycled were of the same seal design as in the prototype nickel endcap lamps. The best of these seals survived four thermal cycles and then cracked apart in the sapphire, as had the lamp seals.

Three modifications to the seal design had measureable beneficial effects:

1. Reduction in nickel cup diamter from 0.42 inch to 0.38 inch to reduce the radial force imparted to the sapphire.

2. Slight reduction in the diameter of the hole in the cup to provide room for a braze fillet on the i.d. of the sapphire. This eliminated localized cracking in the sapphire that occurred when the cup and sapphire were slightly misaligned as shown in Figure 18.

3. Use of a tungsten "stress compensating" washer. This was by far the most effective single modification. When a tungsten washer of approximately correct thickness was incorporated into the seal, cycle lifetimes extended to at least several dozen and in one case to over 300 cycles before failure.

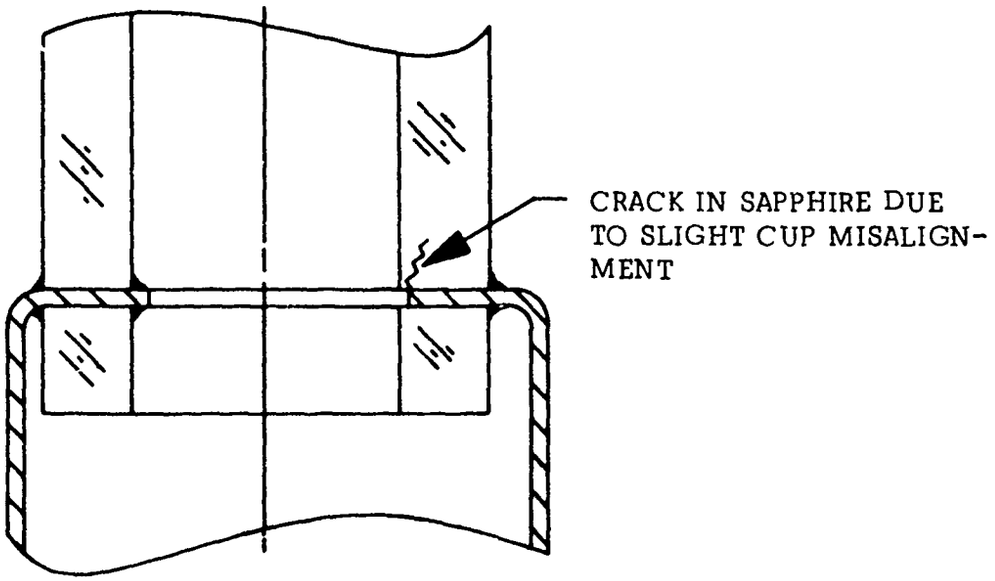
The design of the stress compensated seal is shown in Figure 19. Stress compensation results from the relatively low thermal expansion coefficient of tungsten that tends to balance the much higher expansion characteristics of the nickel member and the brazement. When the tungsten washer is joined to the back surface of the nickel cup during the brazing operation, a bimetal tungsten-nickel composite is formed having a thermal expansion coefficient intermediate between that of tungsten and nickel. With a proper tungsten washer thickness, a composite expansion coefficient is obtained that closely matches that of the sapphire, thus largely negating the forces that cause fracture of the sapphire.

Further work is required to evaluate other important variables associated with the seal, i.e.,

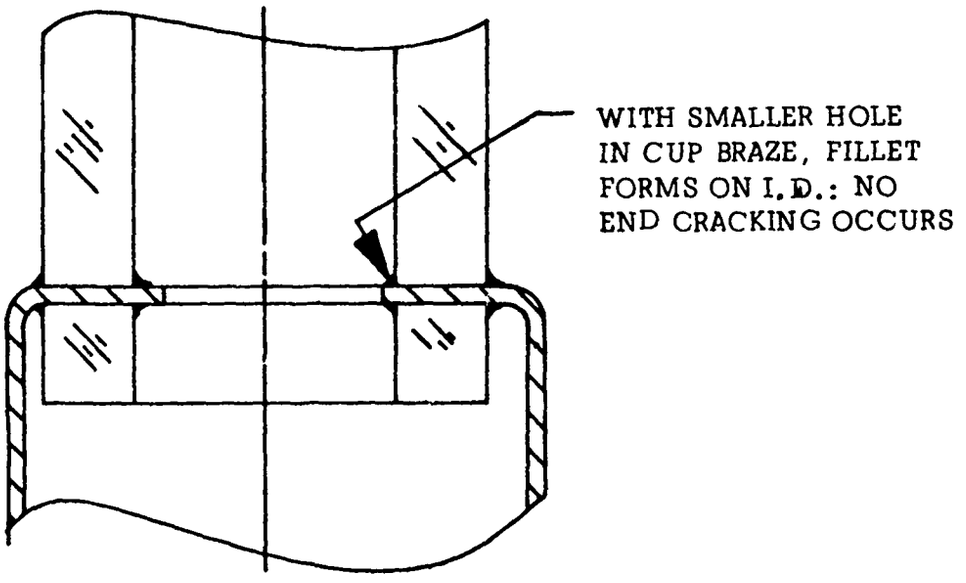
1. sapphire seal surface finish (ground or polished?)

2. sapphire thermal history (stress relief and/or surface smoothing anneals)

3. brazing fixture design, including its effect on joint loading force, concentricity and parallelism of mating seal parts, and time/temperature control during brazing.



A. With Original Cup



B. With Modified Cup (Smaller Hole)

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Figure 18. Cup Modification to Prevent Localized Envelope Cracking Due to Misalignment.

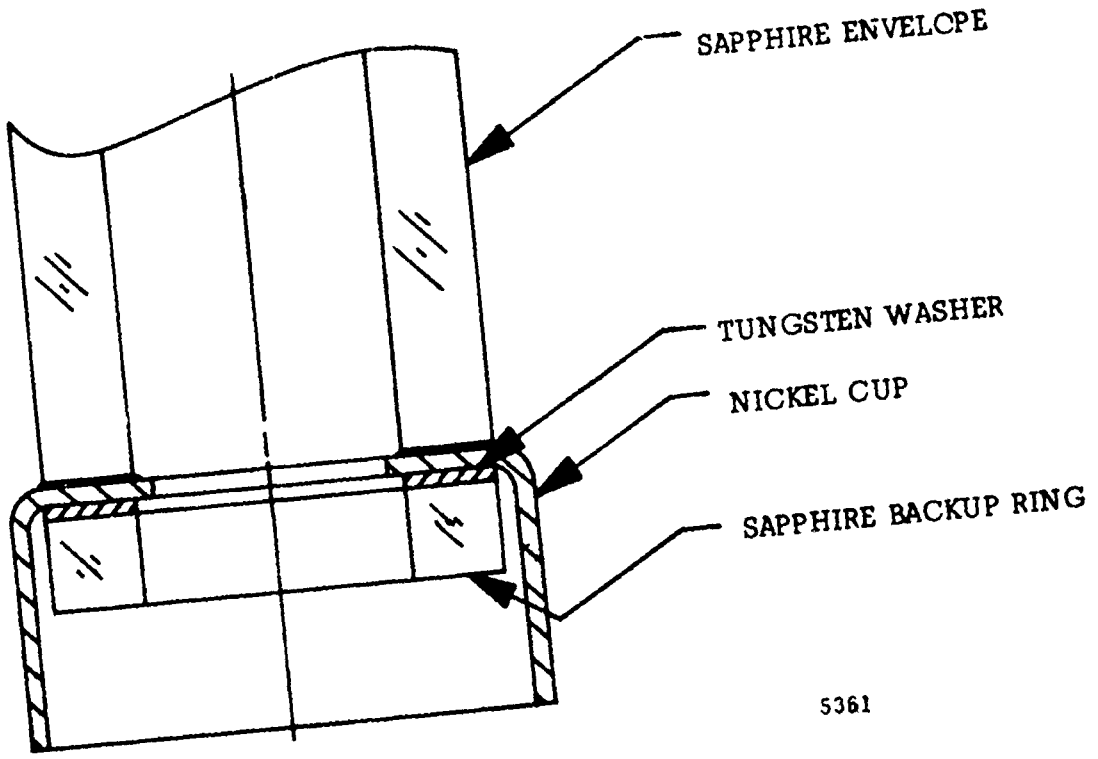


Figure 19. Design of Stress Compensated Seal

4. possible use of vacuum evaporated or painted metal coatings on the sapphire seal surfaces to promote wetting by the brazing alloy.

5. brazing parameters (time/temperature cycles, vacuum level, amount of alloy used, etc.)

6. purity of the nickel member

7. alternative endcap materials

The preliminary seal test results were encouraging enough to allow nickel endcap lamps to again be fabricated. These next lamps employed the improved seal design including the modified endcap cup and the tungsten stress compensation washer.

6.5 Second Generation Nickel Endcap Lamps

Ten nickel endcap lamps were built at the end of the program for life testing and to serve as test vehicles for evaluation of starting gas fills (see Section 8.0). In addition to the seal modifications described above, these lamps employed sapphire envelopes either gas polished in hydrogen (at General Electric Company, Vallecitos Lab) or subjected to a long high temperature anneal (at Union Carbide Corporation, Crystal Products Division). This enabled a comparison to be made between the two envelope pretreatment processes. The lamp design had also been modified to include a solid anode stem (with a provision for filling at the cathode end) to reduce the possibility of alkali metal transport to that end of the lamp (with attendent loss of reservoir control) and machined inner endcaps to provide more rigid support for the electrodes. The lamp design is shown in Figure 20.

The lamps were life tested with operating cycles of one hour or seven hours at the full 250 watts input power. The lamps were tested in prototype EFM-laser pump cavities to simulate realistic operating environments.

Results of the life tests are summarized in Table III. On the average these second generation lamps displayed a marked improvement in cycle durability over prototype nickel endcap lamps. Failures occurred either at the brazed envelope seals where the sapphire cracked or in thin portions of the nickel brazements or weldments where leaks developed.

Improvements in the seal design are undoubtedly responsible for improved lamp cycle durability. The substantial scatter in cycle-lifetime

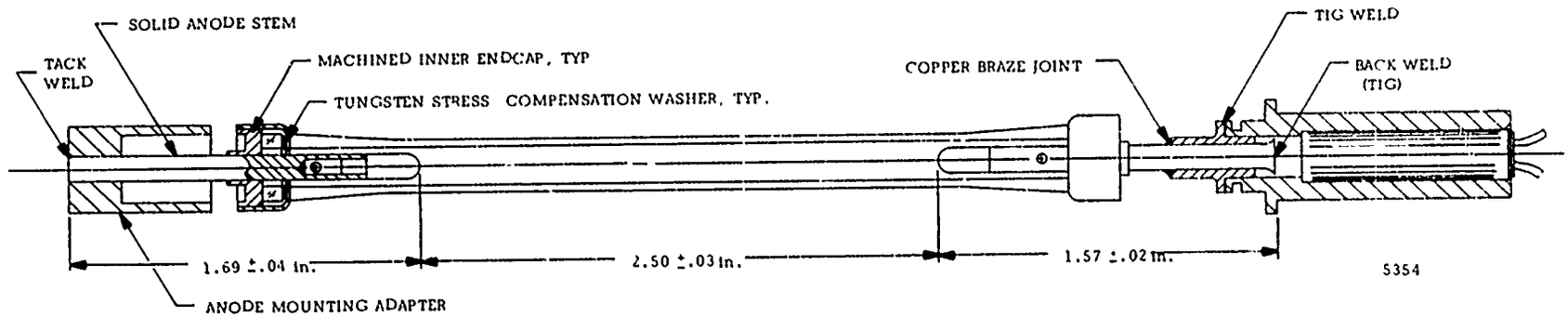


Figure 20. Design of Second Generation Nickel Endcap Lamps

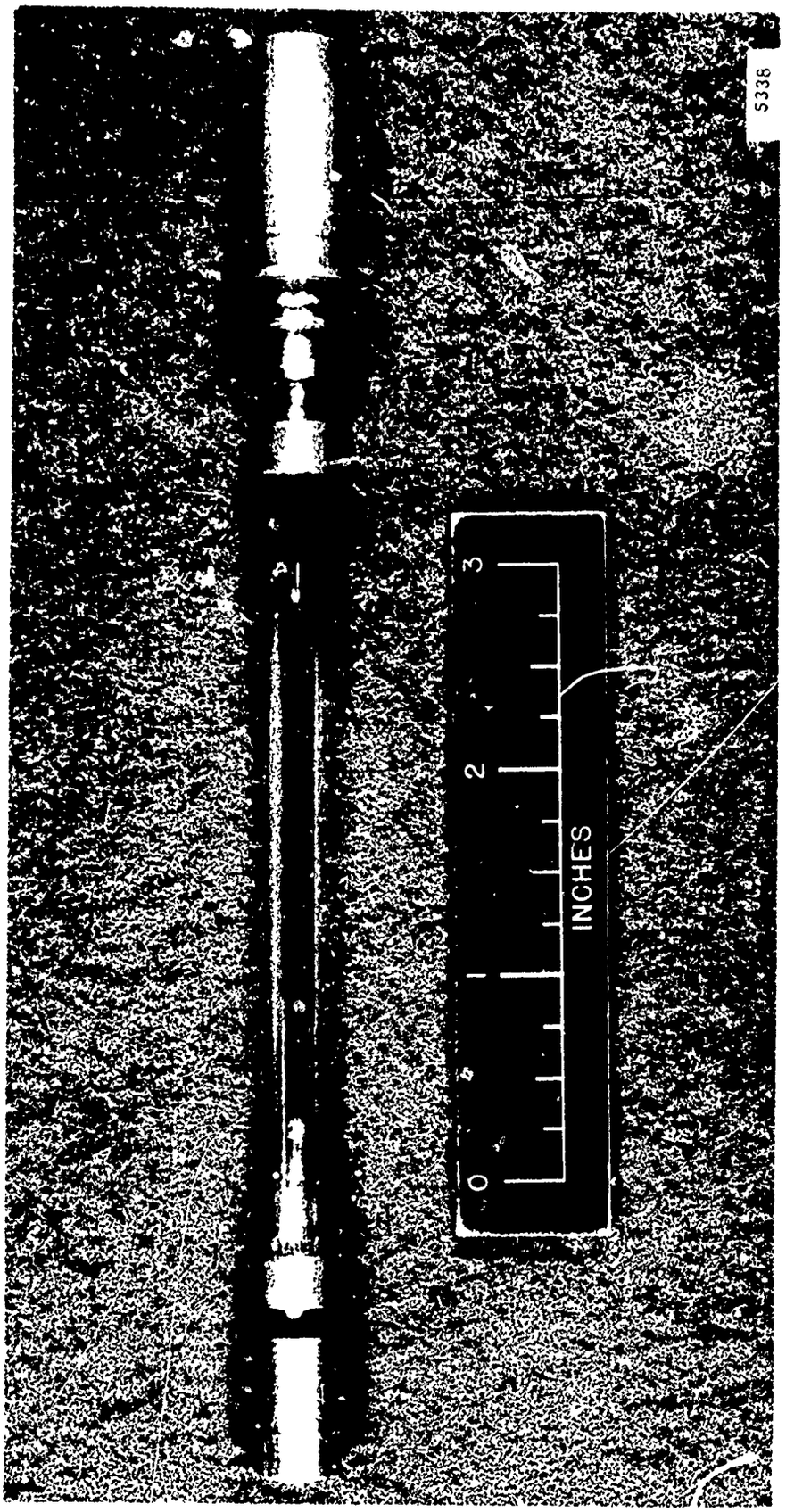


Figure 21. Nickel Endcap Lamp

TABLE III

LIFE TEST RESULTS, SECOND GENERATION NICKEL ENDCAP LAMPS

Lamp No.	Sapphire Treatment	Testing Mode	Lifetime Cycles	Lifetime Hours	Failure Mode
463	GE	1-hr cycles	3	3	envelope cracked at anode end; lamp subjected to two current runaways due to power supply problems.
464	UC	1-hr cycles	16	16	envelope cracked at anode end
465	GE	20 1-hr cycles plus continuous	21	464	leak in cathode endcap near seal; cracks also present in anode end seal.
466	UC	1-hr cycles	1	1	envelope cracked near seal.
467	GE	1-hr cycles	7	7	envelope cracked in cathode end seal
468	UC	7-hr cycles	19	131	leak in cathode endcap-tubulation weld
469	GE	1-hr cycles	3	3	leak suspected in cathode endcap-tubulation weld
470	UC	10 7-hr cycles, bal. 1-hr cycles	85	217	seal at anode end parted; leak suspected early in life at cathode end in endcap near seal
471	GE	-	-	-	not life tested yet
472	UC	Short cycles	157	209	envelope cracked near seal

reflects variability still present in the seal. Most seals had some visual defects as brazed, e.g., small glint cracks and unwetted areas at the sapphire interface, irregular fillets, eroded areas in the nickel endcaps, etc. In some cases these defects did not worsen during testing while in other cases relatively pronounced cracks developed.

The extraordinary cycle lifetime of lamp No. 470 in comparison with the other lamps is noteworthy. This lamp was the only one of the group with seals free of bare spots or localized glint cracks in the as-brazed condition.

Leaks in the nickel appendage weldment were probably due to a marginal joint design that caused thin sections of nickel to be present after welding.

There was no difference in performance between lamps with gas polished envelopes and lamps with "annealed only" envelopes that could be attributed to the sapphire treatments.

The average number of operating cycles before failure was thirty five. The average lifetime in hours (117) is not a meaningful measure of lamp performance in these tests because of the dominant influence of on-off cycling. Nevertheless, it is reasonable to assume that with 8-hour operating cycles, these lamps would average close to 200-hour lifetime.

Although the primary purpose of the tests was to determine cycle lifetime of the lamps, certain other results are also worth noting.

Auxiliary heater power required to sustain a lamp voltage of 70 volts was typically 15 watts, higher than the 10 watt design maximum. Minor thermal design modifications can be made in the lamps to solve this problem.

A discernible time-dependent slumping effect occurred on lamp No. 465 during its 464 hours of operation. Nickel support members at both the anode and cathode ends bent under the weight of the lamp, undoubtedly the result of a creep mechanism. Here again a simple design modification to strengthen these members can be incorporated.

Finally, in lamp Nos. 468 and 470, basal plane cleavage cracks appeared in the sapphire during testing. It is believed that these cracks were associated with slow leaks in the lamps. This subject is discussed in more detail in section 7.2.

7.0 SAPPHIRE IMPROVEMENT

In addition to the envelope frosting problem discussed in Section 4.0, other problems associated with sapphire were of concern at the start of this program. These were:

1. The presence of residual gouges and scratches on the internal surface of the tubes after mechanical polishing which are thought to be preferred nucleation sites for frosting reactions.

2. The frequent appearance of oriented cleavage cracks in the walls of the tubes that often became more pronounced during lamp operation and, because they represented mechanically weak areas, were believed to compromise long life potential of the lamps.

On the previous program at ILC, a high temperature gas polishing process was developed to improve the surface finish of sapphire tube bores. The process was developed in part by an outside laboratory under a service contract with ILC and utilized a high temperature atmosphere furnace at that laboratory. On this program the process was incorporated as a part of standard lamp fabrication procedure. A furnace was procurred and initial experiments were conducted. This effort is detailed in Section 7.1.

Cleavage cracking of sapphire continued to be observed, most often on tubes brazed with Zr-V-Nb alloy, one end at a time, in an rf induction heated bell jar vacuum furnace. Attempts to artificially induce such cracking by imposing severe temperature gradients and to detect crack growth by acoustic emission were unsuccessful. These results, coupled with other findings, led to the tentative conclusion that the cracking was chemically rather than mechanically induced. This work is discussed in Section 7.2.

The cracking problem was originally suspected to be associated with the original quality of grown sapphire stock from which tubes were machined. Accordingly, a procurement specification for sapphire was written and imposed on the vendor, Union Carbide. Details are given in Section 7.3.

7.1 Gas Polishing

A good mechanical polish on the internal surface of sapphire envelopes is apparently impossible to achieve because of inherent

difficulty in getting a polishing tool to work effectively in the long, small diameter bore.

By annealing the tubes in hydrogen at approximately 1900°C, sufficient surface and/or vapor phase transport is induced to eliminate lighter defects and to round the edges of heavier defects. Vapor transport of sapphire is believed to be induced by formation of volatile aluminum suboxide by the following reaction:

$$2H_2(g) + Al_2O_3(c) \longrightarrow Al_2O(g) + 2H_2O\ (g) \qquad (7)$$

This treatment also thermally relieves residual subsurface work stress and presumably smoothes surface microflaws that influence strength of the sapphire.

The process produces surfaces relatively free of thermal faceting that results from other chemical and gas polishing techniques for sapphire, perhaps because vapor transport occurs rapidly enough to overwhelm surface faceting mechanisms.

Similar hydrogen gas polishing of sapphire is described in the literature.[25,26] According to these references approximately 1 mm of material is removed at 1300°C. Annealing of subsurface work damage also takes place.[27]

The ILC gas polishing process was developed using a tungsten element furnace. All gas polished tubes used on this program were treated in this same furnace at an outside laboratory (General Electric Company, Vallecitos Nuclear Center, Pleasanton, California).

During this program, ILC procured a graphite element furnace. A series of experiments were conducted in this furnace, using as-received mechanically polished sapphire parts to evaluate gas polishing action.

The experiments showed that complex side reactions associated with the strong reducing potential of the graphite element prevented satisfactory sapphire smoothing from occurring. To solve this problem, an alumina muffle tube assembly was ordered for the furnace. The gas tight muffle tube will isolate the graphite element from the work and allow neutral conditions to be maintained, as in the original tungsten element furnace. The muffle tube had not been delivered at the end of this program. Further work on gas polishing is being conducted in a subsequent program.

7.2 Cleavage Cracks

During the previous program it was noticed that many lamp envelopes had feather-like cracks in the sapphire. The cracks appeared to be consistently oriented along a particular sapphire crystal plane and became discernible only after high temperature brazing. They always appeared to have nucleated on the internal surface of the tubes and then to have propagated into the wall.

At that time the cracking was tentatively attributed to either damage induced by grinding and polishing or to some deficiency in the original boule growing process, e.g., impurities, pulling rate, etc. Both the grinding house, Insaco Inc., and the original sapphire supplier, Union Carbide Crystal Products, were contact in regard to the problem. Neither vendor could explain the phenomenon, claiming that standard, proven techniques had been used to produce the envelopes supplied to ILC. Both indicated, however, that sapphire is prone to cleave on the rhombohedral plane under certain high stress conditions because of the lower fracture energy associated with this plane.

The problem was a major concern because the cleavage cracks tended to propagate during lamp service and, it was assumed, would eventually lead to failure of the envelopes. This concern was the principal reason for generation of the sapphire procurement specification described in Section 7.3.

7.2.1 Occurrence

During this program cleavage cracking was observed on virtually all of the brazed PES lamp envelope assemblies, on at least two of the similarly brazed quartz-jacketed lamps (Nos. 451 and 460) and on three of twenty-two completed nickel endcap lamps.

The presence of cleavage cracking on PES and quartz-jacketed lamp envelopes was always noted after endcap brazing. These lamps were all brazed with the Zr-V-Nb alloy in an rf vacuum bell jar furnace, one end at a time. Visual inspection prior to brazing had not revealed any defects or abnormalities on the sapphire envelopes that seemed likely to produce such cracking.

Cleavage cracking on three nickel endcap lamps, Nos. 426, 468 and 470, developed during lamp operation. Visual inspection of these lamps prior to testing again had revealed no indications of imminent cracking. Cracks in lamp No. 426 had become quite pronounced by the end of life although none extended completely through the wall.

7.2.2 Characterization

One brazed envelope assembly, intended for use in a PES lamp, had an agglomeration of cleavage cracks near one end, which to the naked eye appeared like a brown stain. This envelope was cut apart and examined by scanning electron microscopy. X-ray probe analysis was also performed on the cracked region.

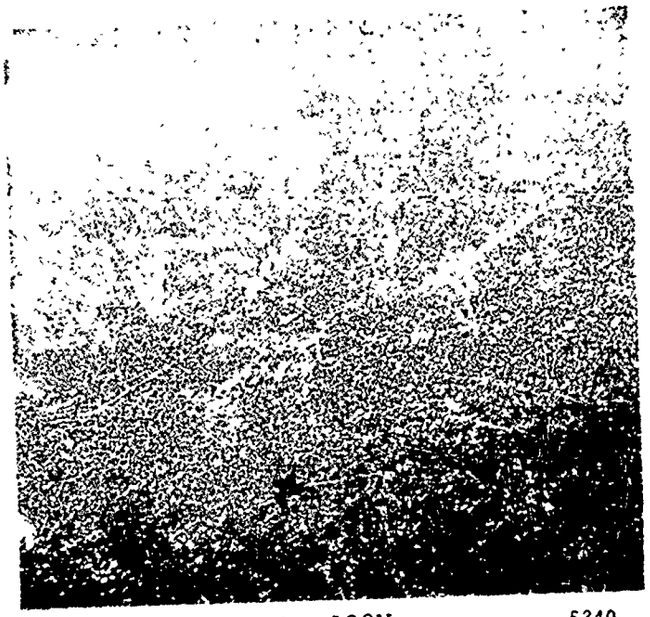
Examination with the SEM revealed the presence of what appeared to be secondary crystallites on the surface which were generally aligned with the cracks. No actual cracks could be seen with the SEM. A SEM photomicrograph of this region is shown in Figure 22. X-ray analysis indicated that calcium was present in the cracked region, but not elsewhere on the sapphire surface.

Based on this, it was conjectured that the cleavage cracking was the result of localized chemical reactions on the envelope during brazing. It is conceivable that a calcium aluminate compound is formed that "wedges" its way into the sapphire crystal lattice along preferred planes and causes high tensile stresses to develop which cleave apart the sapphire. This model is consistent with the fact that cracks do not propagate completely through the sapphire wall. In addition, other investigators have observed intragranular cracking in translucent alumina associated with attack by calcium impurities in sodium vapor lamps.[28]

The pronounced cleavage cracking in lamp No. 426 allowed crystallographic orientation of the cracks to be determined with the aid of a polariscope. The cracks appeared to be along the basal planes of the sapphire. This finding was confirmed by x-ray diffraction analysis on the same sapphire envelope by Union Carbide.

The formation of calcium aluminate along sapphire basal planes is consistent with characteristics of calcium beta-alumina, $CaO \cdot 6Al_2O_3$, alluded to in Section 4.1. In the case of envelope assemblies brazed in the rf vacuum bell jar furnace, a likely source of calcium is a high alumina (99.8%) axial alignment rod, a part of the brazing fixture. CaO is present in this ceramic to the extent of several hundred parts per million. Reactive zirconium vapor, generated at the braze melt, or spatter of braze alloy onto the alumina rod may have generated free calcium which could subsequently condense on the adjacent sapphire surface and there form calcium aluminate.

Cleavage cracking in the three nickel endcap lamp envelopes cannot be attributed to this mechanism. It is perhaps significant that two of these lamps were suspected to have slow leaks prior to dis-



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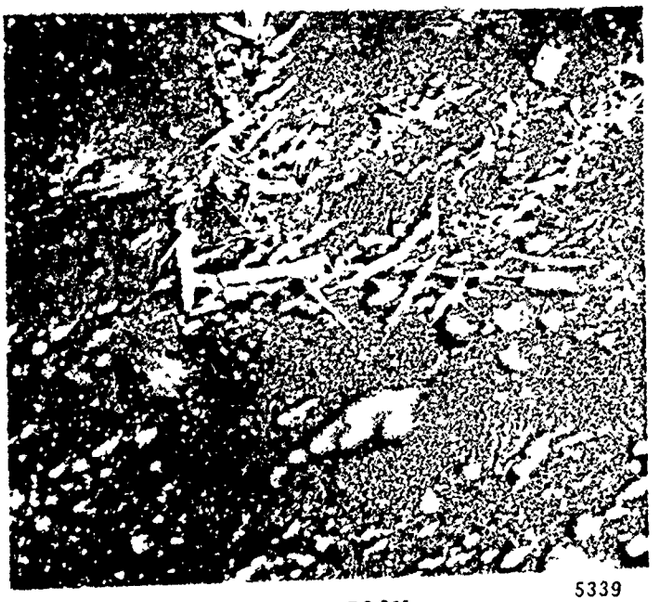

B. 500X 5339

Figure 22. SEM Photomicrographs of Cleavage Crack Nuclei

covery of the cleavage cracking. Possibly oxygen, admitted to the lamp through such leaks, is promoting a chemically-induced cracking phenomenon similar to that involving calcium, e.g., formation of very fine local crystallites of potassium beta alumina.

7.2.3 Acoustic Emission Testing

Prior to development of the chemical reaction model for explaining sapphire cleavage cracking, it was believed that intrinsic or machining induced defects in the sapphire were responsible. Acoustic emission measurements were proposed as a possible detection tool for deficient material. If a sapphire tube is connected to a sensitive acoustic amplifier, propagation of cracks can be sensed and recorded. By thermally cycling tubes while monitoring acoustic emission, "bad" tubes, i.e., those that undergo incipient cracking, can be weeded out.

Acoustic emission apparatus was rented for use on this program from Nortek Company, Richland, Washington. Included in the package were a transducer, preamplifier, signal conditioner, emission count analyzer and totalizer, and power supply.

In a typical experimental arrangement, shown in Figure 23, the sapphire tube was cemented or attached via a viscous, acoustically conductive medium to the transducer. The tube was then localiy heated in its middle or near its other end to generate thermal stresses and induce incipient cracking in "bad" tubes.

Sapphire tubes for acoustic emission experiments were drawn from the same lots as used for PES lamp fabrication, i.e., "bad" lots. Initial experiments were run using a clam shell furnace to heat the sapphire. At first, extraneous electrical noise was encountered that made output from the amplifier/counter apparatus meaningless. The test gear was moved into a rf screen room designed to isolate sensitive experiments from ambient electromagnetic interference and electrical line noise. The apparatus worked much better under these conditions. Nevertheless no cracking-induced acoustic emission could be detected in a number of tubes heated and cooled in the clam shell furnace.

Finally, tubes were heated by wrapping a few turns of Nichrome wire directly around the outside of the tubes. Even this intense, very localized heating did not produce acoustic signals or any visual indication of cleavage cracking. The results, although inconclusive, tended to reinforce the chemical reaction model of cleavage cracking.

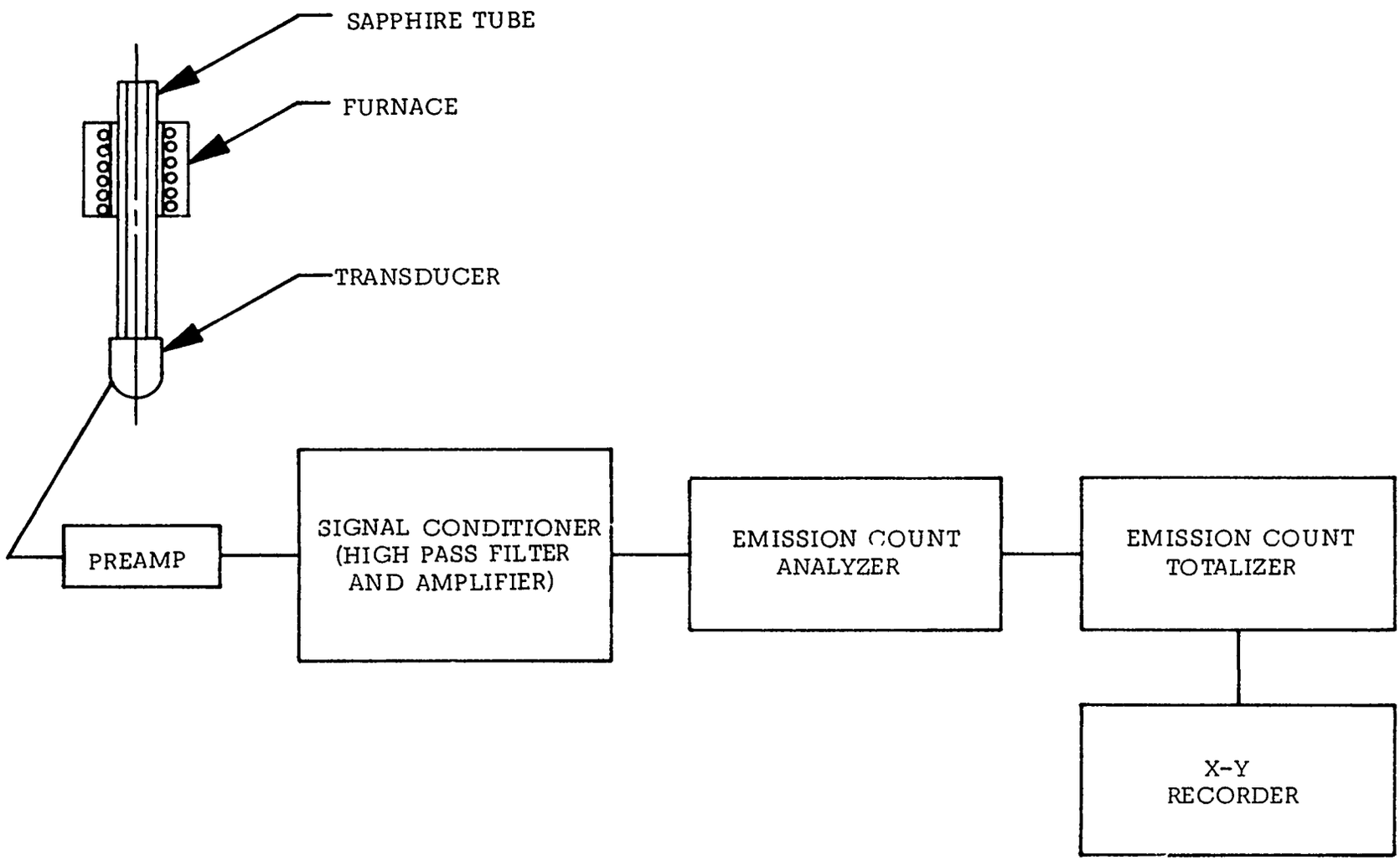

Figure 23. Arrangement for Acoustic Emission Tests

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7.2.4 Discussion

Inability to induce cleavage cracks by intense thermomechanical stressing of sapphire coupled with the plausibility of the chemical reaction cleavage cracking mechanism makes the latter model more attractive at this time.

Discussions with sapphire authorities cast further doubt on the thermal stress model(29, 30, 31). Opinions solicited from these sources were consistent in regard to the unlikelihood of stress-induced basal plane cleavage. This close-packed plane has high fracture energy compared with other crystallographic planes and will, under the proper circumstances, form twins but never cleavage cracks. If thermal stresses of high magnitude are present in sapphire, random fracturing or rhombohedral plane twinning and cleaving is much more likely.

The cleavage cracking phenomenon is by no means well understood at this time. More work is necessary to determine causes and develop solutions for the problem.

7.3 Sapphire Procurement Specification

A specification was written to cover procurement of sapphire rod stock and envelopes for use on this and subsequent K-Rb lamp programs. Quality requirements dictated by the specification in effect necessitate the use of UV grade sapphire. UV grade material, according to Union Carbide, is grown in much the same fashion as standard grade Czochralski sapphire but is made from much purer starting material (lower in Na_2O content) and is routinely subjected to careful visual inspection to detect and reject internal defects such as fine bubbles, striae, and the like.

The specification also provides traceability of sapphire lots to original grown boules. This potentially facilitates determination of the causes of sapphire problems (such as cleavage cracking) that occur sporadically.

The specification is included as Appendix I of this report.

8.0 LASER PUMPING EFFICIENCY

Concurrent development of K-Rb lamps and the EFM laser provided an opportunity for direct evaluation of lamp pumping efficiency in the laser system. Measurements of multimode laser output power at 1.06 μm were made with various K-Rb lamps in prototype EFM pump cavities, both at GTE-Sylvania and at the Air Force 405B laboratory.

The measured laser output power in such tests can be strongly influenced by several factors not directly related to lamp performance, e.g., laser rod and lamp positioning in the pump cavity, cavity reflector design, end mirror reflectivities and alignment, and laser rod temperature, among others. Nevertheless, multimode laser output power measured at Sylvania with the first EFM-compatible K-Rb lamps was lower than expected, particularly in comparison with results obtained in previous brassboard laser experiments. Although non-optimized laser parameters were subsequently found to be responsible in part for the initial low laser outputs measured, there was still concern about lamp pumping efficiency. Accordingly, lamp efficiency considerations were reassessed and led to significant improvements in this area as described below:

8.1 Effect of Increased Arc Length

As noted in section 3.0, the K-Rb lamp arc length was increased from 1.9 to 2.5 inches during this program in order to reduce thermal loading of the envelope and to enhance lifetime potential. The Nd:YAG laser rod was lengthened as well (to 60 mm). Lamp input power was held constant at 250 watts.

A direct consequence of increased arc length at constant input power is reduced arc brightness which, in turn, results in a reduced level of irradiance at the laser rod. Because laser output power is so strongly sensitive to the intensity of incident pump band radiance (as reflected by the steep slope efficiency curves for K-Rb pumped Nd:YAG[4]), it seemed likely that more laser power would be lost by reduced irradiance at the rod (due to an increase in arc length at constant arc power) than would be won back by the equivalent increase in active rod length.

By such reasoning it was concluded that the increase in arc length of K-Rb lamps probably compromised pumping efficiency.

8.2 Effect of Rare Starting Gas

A rare gas is routinely added to K-Rb and most other metallic vapor arc lamps to facilitate starting. Virtually all K-Rb lamps fabricated on prior programs at ILC and the first lamps on the present program were filled with argon at 100 torr absolute pressure.

A review of the literature on high pressure mercury and sodium lamps[32, 33] revealed that better radiant efficiency is obtained with these lamps when xenon rather than argon starting gas is used. This effect is attributed to reduced thermal conduction losses from the arcs in the presence of xenon. A significant fraction of arc discharge power is dissipated by radial thermal conduction from the arc column to the envelope wall. This conductive loss is proportional to the thermal conductivity of the gas/vapor mixture present in the lamp. Since the thermal conductivities of gases are in inverse proportion to their atomic weights, it follows that mixtures of xenon (atomic weight 131) with sodium or mercury will have lower conductivities than mixtures of argon (atomic weight 40) with sodium and mercury. Thus lamps with xenon will have less conductive loss, and (as a result) more radiative loss from the arc, i.e., increased arc radiance.

There seemed to be no reason why the same effect should not apply to K-Rb lamps. Accordingly a decision was made to use xenon starting gas instead of argon in some lamps on a trial basis. An additional consideration was the effect of xenon gas fill pressure. Although the thermal conductivity of a pure vapor or gas is independent of pressure, in the case of K-Rb-Xe or K-Rb-Ar combinations a mixture rule should prevail that is dependent on xenon or argon pressure. For example, mixtures of potassium (atomic weight 39), rubidium (atomic weight 85), and argon should have relatively high thermal conductivities, especially with argon pressures high in comparison to the few torr of K and Rb vapor present during lamp operation. By contrast, K-Rb-Xe mixtures should have relatively low thermal conductivities, especially at high xenon pressures. Presumably a point of diminishing return exists with respect to xenon pressure, i.e., where xenon atoms so dominate the lamp fill mixture that increases in xenon pressure have little or no effect on arc radiance. In addition, a practical limit on xenon pressure exists where lamp starting voltage requirements exceed power supply or pump cavity high voltage stand-off capabilities.

8.3 Experimental Results, Xenon vs. Argon

A preliminary experimental indication of the superiority of xenon-filled lamps over those with argon starting gas was obtained

with lamp Nos. 446 and 447 containing 100 torr of xenon. Measured multimode laser output with these lamps exceeded 1 watt under the same conditions where with prior argon-filled lamps laser output had never exceeded 600 mW.

More definitive tests were conducted at the Air Force 405B laboratory near the end of the program with a series of lamps (Nos. 466-472) filled with various pressures of either xenon or argon.

In these tests multimode output power was measured for a laser pumped, in turn, by each of the lamps from the experimental group. Laser conditions were, as nearly as possible, kept constant during the tests. Mirrors were prealigned and fixed in position. The laser rod was maintained at a constant +10°C. Lamps were positioned identically in the pump cavity using a locating jig. The fixed lamp position was not optimized for maximum laser output power but assured that comparative results would be meaningful. Measurements were made with each lamp at its optimal voltage at 250 watts input power.

As internal checks on the experiment, one lamp, No. 471, was tested twice and two lamps with identical fills were tested (No. 466 and 470).

In addition to measurements of laser output power, the lamps were characterized by other techniques. Broad band relative irradiance was measured for each lamp using a silicon photodetector. Spectral output curves and arc brightness profiles were also obtained.

Results of the tests are summarized in Table IV. The highest laser output was obtained with the lamp containing xenon at the highest pressure (No. 472, 760 torr xenon). Laser output dropped with decreasing xenon pressure and was substantially lower for argon-filled lamps. Differences between the two laser power readings with lamp No. 471 and between readings with Nos. 466 and 470 (with identical fills) were small in comparison with differences between lamps of various fills in the experimental group.

Irradiance values for the lamps generally reflect the same trend as is established with the laser power results, i.e., higher values for the xenon-filled lamps.

The arc diameters (at the 50 percent intensity point) measured for the xenon-filled lamps were larger than those for argon-filled lamps. However, the xenon filled lamps had significantly higher peak (axial) brightness than did the argon-filled lamps.

TABLE IV

RESULTS OF LAMP EFFICIENCY TESTS

Lamp No.	Fill	Optimal Voltage	Laser Power	Relative Irradiance	Arc Dia.	Relative Arc Peak Radiance
472	K-Rb; 760 torr Xenon	69	790 mW	129	2.95 mm	4.95
467	K-Rb; 400 torr Xenon	68	510 mW	120	2.90 mm	4.60
466	K-Rb; 100 torr Xenon	66	370 mW	116	2.80 mm	4.60
470	K-Rb; 100 torr Xenon	67	400 mW	--	--	--
468	K-Rb; 100 torr Argon	72	270 mW	101	2.75 mm	4.20
471	K-Rb; 760 torr Argon	70	160 mW	113	2.75 mm	4.50
469	Rb; 100 torr Argon	66	50 mW	103	2.70 mm	4.10

The basis for higher laser power with the xenon-filled lamps is perhaps most dramatically shown in the spectral output curves plotted in Figure 24 for lamp Nos. 472 (760 torr xenon) and 468 (100 torr argon, previous case). The spectral intensity of the xenon-filled lamp is significantly higher.

8.4 Discussion

The experimental results are in good agreement with qualitative predictions based on the gas/vapor thermal conductivity model. Somewhat surprising were the large quantitative differences in measured laser power between the best (xenon) and worst (argon) lamps and between lamps with various xenon fill pressures as well. The results suggest that significant additional improvement in efficiency can be obtained with further increases in xenon fill pressure. More work in this area is strongly recommended.

The use of xenon rather than argon gas in the lamps has two side advantages:

1. Reduced conductive heat loss from the arc column to the envelope wall will reduce the required heat dissipation from the envelope, resulting in a lower average envelope temperature.

2. Xenon-filled K-Rb lamps "behave" better than argon lamps during excursions to and from full power operation; the transition from the initial rare gas discharge mode (with associated anode overheating and deposition of electrode material onto the envelope walls) to the "safe" K-Rb arc mode occurred more quickly and at lower power levels in xenon-filled lamps.

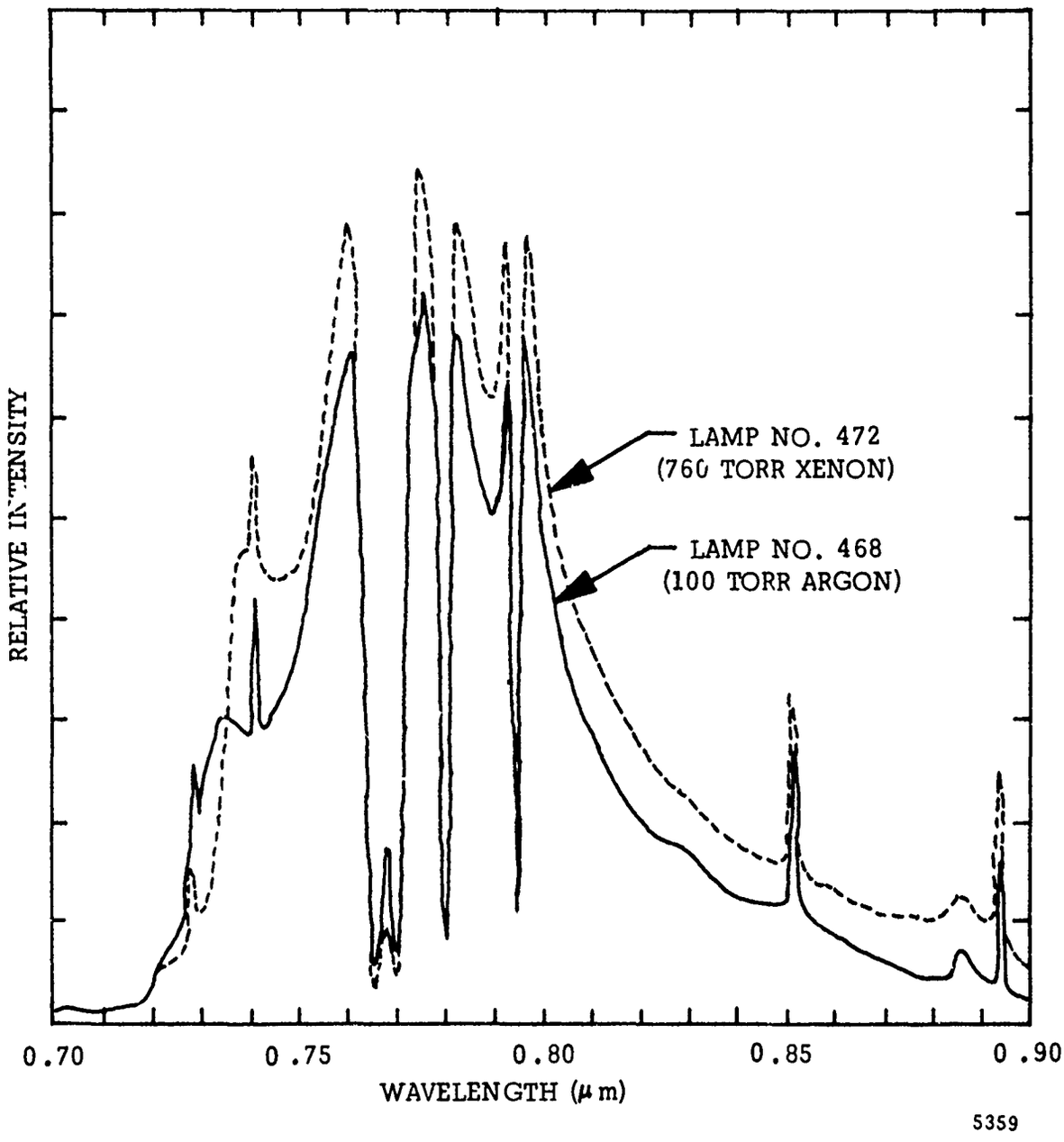

Figure 24. Output Spectra for K-Rb Lamps with Xenon and Argon Starting Gases

9.0 OXIDATION PROTECTIVE COATINGS

In addition to the protected end seal and nickel endcap lamps already discussed, a third approach to making an air compatible lamp was investigated: the use of an oxidation protective coating on the exposed niobium parts of a bare niobium endcap lamp.

A variety of effective protective coatings for niobium have been developed in recent years by the aerospace industry. Most of these are designed for service in severe environments, e.g., turbine blades, re-entry vehicles. The comparatively moderate service temperature of K-Rb lamp endcap would seem to indicate that any of several protective coatings could be utilized to easily produce an air compatible lamp. However, certain features of the lamp application present potential problems in regard to use of such coatings. These include:

1. Curing temperatures for coatings that may exceed lamp brazed seal melting points, cause the uranium internal getter to melt, cause excessive alkali metal pressure to develop in the lamp interior, or otherwise promote lamp degradation.

2. The possible development of cracks or other coating faults during lamp operation due to use at much lower than intended service temperatures with attendant absence of fluid, self-healing, surface oxides.

3. Unpredictable metallurgical interactions between the protective coatings and the Zr-V-Nb brazements that could cause, among other problems, lack of oxidation protection in these regions.

4. Likelihood of initial coating faults (cracks, thin spots, etc.) due to the complex surface topography of lamp exteriors.

5. Possible difficulty in provided reliable electrical contact to the lamp, i.e., via a transition region between coated niobium and uncoated mechanical cavity interfacing components.

The intent on this task was to evaluate promising coatings that had curing temperatures below lamp brazed seal melting points with particular interest in determining whether "low" temperature service and protection of Zr-V-Nb brazed seals would be problems.

9.1 Candidate Coatings

Two protective coatings were evaluated, a fused slurry silicide coating and a fused slurry tin-aluminide coating. Selection of these coatings for evaluation was based on the proven effectiveness of fused coatings (formed by fusion of applied slurries with the niobium substrate during elevated temperature curing) and the likelihood that these particular systems would retain self-healing characteristics at the comparatively low lamp service temperatures.

Active compositions for the two coating slurries are as follows:

Fused silicide: 60Si-20Cr-20Fe (weight percent)

Fused tin-aluminide: 67.5Sn-22.5Al-10Mo

Both coatings are applied by spraying acrylic lacquer-based slurries onto exposed niobium surfaces, dried and then fired.

Tin-aluminide coatings require two such application cycles, including firing under argon at 1038°C for 30 minutes and 10 minutes, respectively.

The silicide coating is ordinarily fired under vacuum at 1415°C. To avoid remelting the Zr-V-Nb brazed seals on lamps and specimens, the procedure was modified during this program to consist of a lower temperature, 1325°C, 10-minute firing followed by application of a glass overcoat containing 95 w/% pyrex and 5 w/% amorphous boron and refiring at 1000-1100°C for 2 minutes. The glass overcoat serves in place of the silica-base protective oxide layer that forms on the coating only above 1000°C during service in air.

9.2 Experimental Specimens

Representative end section assemblies of niobium endcap lamps were fabricated using standard techniques. A drawing of the test assembly is shown in Figure 25.

Protective coatings were applied to eight assemblies, four each with silicide and tin-aluminide. Coating thickness for the silicide coating was approximately 3 mils with an additional 1 mil glass overcoat; the tin-aluminide coating thickness was approximately 6 mils. All coatings were applied by HiTemCo, Hicksville, N.Y., who was responsible for their original development.[34]

9.3 Oxidation Life Tests

Coated specimens were tested in an air furnace at 800°C. Two tin-aluminide samples were tested in 24-hour cycles (removed from the furnace and inspected for coating failure every 24 hours); all other samples were tested in 8-hour cycles.

Results of these tests are summarized in Table V.

The tin-aluminide coating did not perform well. The coating seemed to be detrimentally affected by the presence of the Zr-V-Nb brazement, generally failing in this region (see Figure 26).

By contrast, the fused silicide coatings with the glass overcoats did not fail within the 400 hour test period. Aside from some cosmetic defects which developed during the tests on the coatings (easily seen in Figure 27) no indications of degradation were discernible.

9.4 Coated Lamps

A pressing need for deliverable K-Rb lamps to support EFM laser testing midway in the program prompted a decision to fabricate two silicide-coated niobium endcap lamps prior to completion of the first coating evaluation experiments described above.

To avoid the problem of excessive alkali metal vapor pressurization, the coating was applied and fired on the lamps prior to fill. The fill appendate was left uncoated to facilitate final pump, back fill and pinch off operations. One of the two lamps developed a seal leak during the coating process. The remaining lamp was filled and pinched off. Additional silicide coating was applied to the exposed pinch off surfaces and fired locally by immersing that end of the lamp quickly into an air furnace. This procedure was a radical departure from standard practice (i.e., vacuum firing) but was considered to be an acceptable compromise in the interests of obtaining a useful lamp quickly. It was believed that despite some superficial oxidation of the coating during the air-firing process, sufficient applied slurry would react with the substrate metal to form a sound, protective coating.

As an additional safeguard, a standard glass overcoat was applied to the air-fired coating region and fired.

Some difficulty was encountered in joining the nickel heater mount to the coated lamp. In several previous brazing tests various

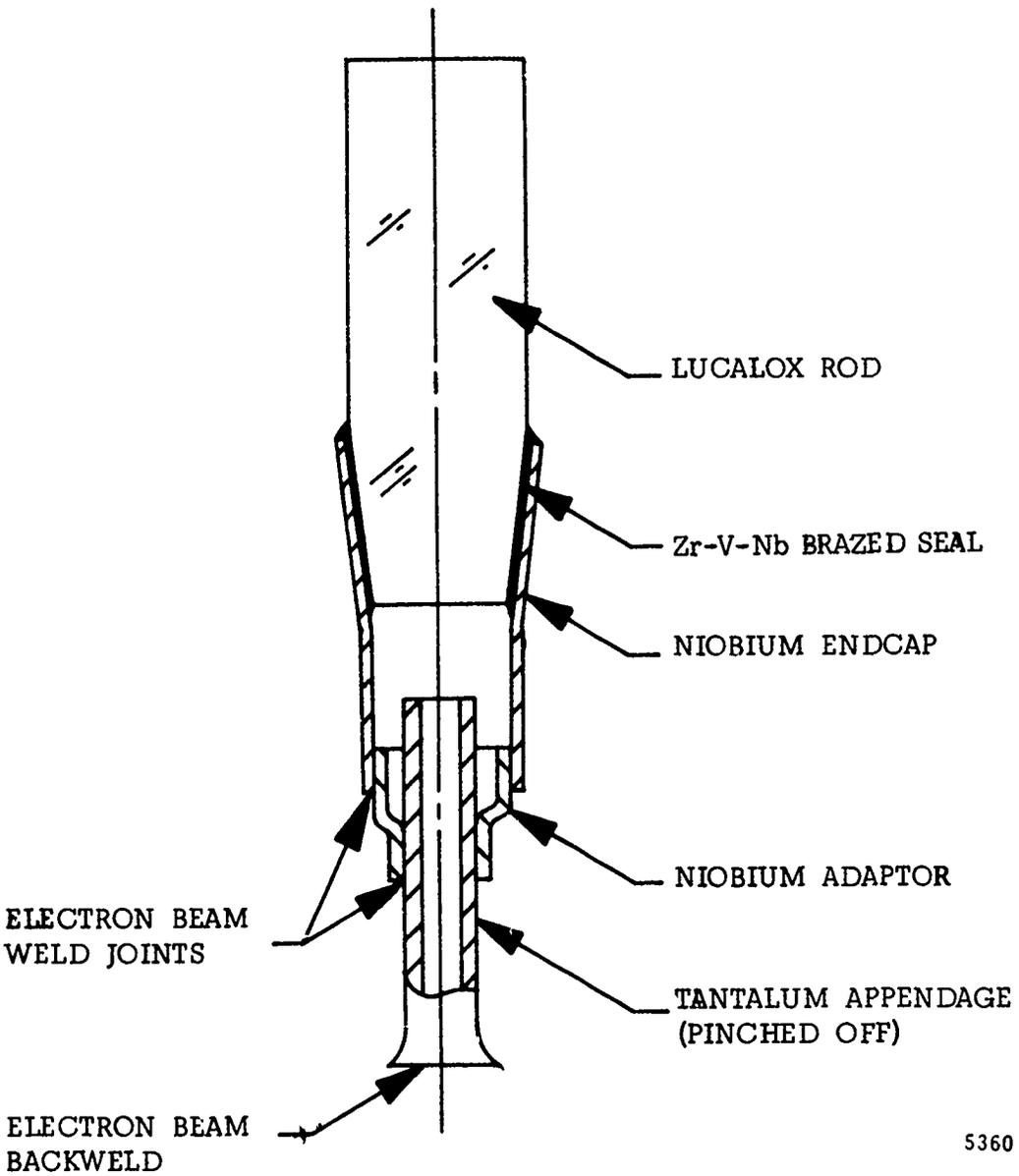

Figure 25. Specimen for Protective Coating Evaluation

Figure 26. Specimen Coated With Fused Tin-Aluminide After Oxidation Tests

Figure 27. Specimen Coated With Fused Silicide After Oxidation Tests

TABLE V

PROTECTIVE COATING OXIDATION RESULTS

Spec. No.	Coating Type	Cycle Mode	Lifetime (Hrs.)
1	Tin/Aluminide	24-hr	125-150
2	Tin/Aluminide	24-hr	24-48
3	Tin/Aluminide	8-hr	40
4	Tin/Aluminde	8-hr	88
5	Silicide with glass overcoat	8-hr	400+
6	Silicide with glass overcoat	8-hr	400+
7	Silicide with glass overcoat	8-hr	400+
8	Silicide with glass overcoat	8-hr	400+

tandard brazing alloys, including pure copper and nickel-gold alloys, 'ere found to give poor bonds to the cured silicide coating. The eater mount was finally brazed to the lamp with copper-silver alloy to rovide an electrical connection. Silicate cement was used to mechani- ally buttress the brazed joint connection.

The completed lamp was delivered to the Air Force but, because ıe need for a K-Rb lamp to support EFM laser tests had subsided, was ever operated.

.5 Discussion

Despite encouraging preliminary results, at least with the fused ilicide coating, two important design problems would have to be effec- vely addressed before a coated lamp can become an attractive alternative) present PES and nickel endcap designs. These are:

- The high temperature required to properly cure the silicide coating, which can cause melting of the brazed seal, excessive alkali metal vapor pressurization, melting of the uranium getter, etc.

- Difficulty in making positive, reliable mechanical and electrical connections to the lamp.

With the successful development of the nickel endcap lamp, an al- ernative lamp for laboratory use is no longer required. Concern has shifted :o the long term, multicycle lamp operational requirements of the satellite application. PES and nickel endcap designs presently appear to have greater potential for long term service than do coated lamps.

10.0 SUMMARY

Significant progress was made on the program in a number of technical areas. The effectiveness of the uranium getter and improved lamp processing in curbing envelope frosting reactions and the dramatic increase in laser pumping efficiency with the substitution of xenon for argon in the lamps are particularly important improvements in K-Rb lamp technology. The introduction of the nickel endcap lamp with its oxidation resistant, stress-compensated seals and simple design provides a second, viable design concept for potential use in space missions. Although problems were encountered in fabrication of PES lamps, encouraging preliminary results were obtained with new, improved endcap and frit seals for this lamp. If the performance of these seals meets expectations, the PES lamp can satisfy ultimate mission requirements. A better understanding of problems with the sapphire envelope material was developed. Finally, encouraging preliminary results with a fused silicide protective coating suggest that a coated niobium endcap lamp is a feasible backup design concept to PES and nickel endcap lamps.

Work on nickel endcap lamps (with emphasis on improving seal durability), PES lamps (including further development and evaluation of CVD and frit seals), laser pumping efficiency and sapphire problems is continuing on subsequent programs.

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APPENDIX I

REVISIONS				
SYM	SHEET	DESCRIPTION	DATE	APPROVED

1.0 PURPOSE

This specification outlines the requirements for clear synthetic sapphire tubes procured by ILC for use as alkali metal vapor lamp envelopes.

2.0 DEFINITIONS

2.1 The customer is ILC Technology, Inc.

2.2 The material supplier is responsible for original growth of the sapphire material and its subsequent delivery in the form of rod stock. Sections 3 through 7 of this document apply to the material supplier.

2.3 The fabricator is responsible for machining of rod stock to obtain the final configuration and surface finish as specified in applicable drawings and purchase requisitions.

3.0 CRYSTAL GROWING

3.1 Starting Material: Starting powders for use in crystal growing shall be of a quality consistent with the optical grade of final material specified in the purchase order.

3.2 Method: Boules shall be grown by the Czochralski method.

3.3 Crystal Orientation: The growth axis of the boules shall be approximately 60° from the C crystal axis ([0001] direction) of the sapphire.

NEXT ASSY / USED ON	
APPLICATION	
RELEASE DATE	
DRAWN / CHECK / ENGR. / PROJ. / APPR.	ILC INC. 164 COMMERCIAL STREET SUNNYVALE, CALIFORNIA 94086
APPROVED / APPROVED	SYNTHETIC SAPPHIRE ENVELOPES FOR LAMPS
	SIZE A · CODE IDENT. NO. 31573 · PS1006
	SCALE · SHEET 1 Of 4 OF

3.4 Boule Diameter: To be approximately 2 inches.

3.5 Growth Rate: To be consistent with the optical grade of final material specified in the purchase order.

3.6 Annealing: As-grown boules shall be annealed at high temperature in order to remove growth strain from the material. Annealing time and temperature shall be consistent with this goal. Heating and cooling rates shall be controlled so that attendant thermal transient stresses do not damage the material.

4.0 INSPECTION OF BOULES

4.1 Boules shall be inspected visually using the unaided eye a microscope, and crossed polarizers, as appropriate.

4.2 Boules shall be free of "smoke" as determined by visual inspection.

4.3 Boules shall be free of twins as determined by visual inspection using crossed polarizers.

4.4 Boules shall be free of other obvious defects including bubbles, inclusions, cracks, and the like which would compromise the mechanical integrity of the material.

4.5 Boules for use on the order shall be numbered for purposes of identification and traceability.

5.0 PREPARATION OF ROD STOCK

5.1 Rod stock shall be obtained by core drilling from annealed boule(s).

5.2 Rod stock required to satisfy the order shall be obtained from a minimum number of starting boules to facilitate traceability.

5.3 Rod stock pieces shall be labelled in an appropriate manner to indicate the starting boule from which they were cored.

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6.0 INSPECTION OF ROD STOCK

6.1 A representative rod from each boule shall be polished to facilitate visual inspection.

6.2 The polished rod sample shall be visually inspected for defects as described in Section 4.0.

6.3 The presence of defects in the sample rod that suggest marginal quality of the original boule shall be cause for rejection of all rods cored from that boule.

7.0 QUALITY ASSURANCE REQUIREMENTS - MATERIALS SUPPLIER

7.1 Rod stock shall be traceable to original boules from which it was cored. Record keeping and labelling shall be employed as necessary to satisfy this requirement.

7.2 Inspection reports shall be prepared and kept on file by the material supplier as evidence that the visual inspections called for in this specification were performed and that the material so inspected was found acceptable. These reports shall be available for examiniation by the customer at his request.

7.3 The material supplier will provide a certificates of conformance with Sections 3 through6 of this specification to the customer and to the fabricator with the completion of each order.

8.0 FABRICATION OF FINAL ENVELOPES

8.1 Workmanship: The best practical procedures and highest quality workmanship shall be employed to fabricate envelopes.

8.2 Polished surfaces shall be free of gouges and scratches inconsistent with the employment of best procedures and workmanship as specified in 8.1.

8.3 Care shall be taken to avoid overheating of the sapphire stock during grinding or polishing that will induce residual damage in the material.

9.0 INSPECTION OF ENVELOPES

9.1 The fabricator shall visually inspect completed envelopes

to ensure that they are free of gross scratches, gouges, cracks, or chips that would compromise the mechanical integrity of the envelope.

10.0 ANNEALING OF ENVELOPES

10.1 The customer will be responsible for annealing of final envelopes to relieve work strain induced during prior fabrication processes.

11.0 ACCEPTANCE TESTING

11.1 ILC Technology intends, at a later date, to develop a meaningful test(s) to evaluate the mechanical integrity of sapphire tubes as received. Strength tests, thermal cycling, and acoustic emission tests are under consideration. The tests are intended to measure quality of workmanship on finished tubes so that deficient envelopes can be "weeded out" before incorporation into lamps.

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