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Non-Conducting Hermetic Coatings for Optical Fibers

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A number of military systems incorporating fiber optics require long lengths of high strength fibers. These fibers, like other silicate derived glasses, suffer from the phenomenon of stress corrosion or static fatigue. The observed fatigue occurs when fibers are subject to tensile stresses in a humid environment. None of the currently used organic buffer and overcoating materials is impermeable to water and, therefore, to provide hermetic protection, one must look to other materials. Among the more promising materials are metals, silicon nitride, and diamond-like carbon. The requirements and possible behavior of suitable coatings will be discussed in terms of performance, expected reliability, and economic considerations.

## INTRODUCTION

Many of the applications of fiber optics to military systems involve the use of long lengths (> 5 km) of optical fibers having high strength and long term durability.<sup>[1]</sup> As opposed to permanently installed, static data links, these applications require cables which may be frequently moved or, perhaps, used as a data link between moving platforms. Consequently, an extra measure of survivability is needed. One may roughly categorize fiber cables into two types: those having load-bearing members and those in which the optical fiber, itself, is the load-bearing element. Table I represents a selected number of such systems according to this division.

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Table I.  
OPTICAL FIBER CABLES

Cabled structures with load-bearing members:

- Towed array.
- Bottom laid communications (retrievable).
- Mine countermeasures.
- Work Systems.
- Intelligence Gathering
- Submarine Communications Buoy.

Load-bearing fiber:

- Torpedo
  - Missile
  - Sonobuoy
  - Bottom laid (nonretrievable)
  - Swimmer
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In the case of cables with load-bearing elements, an important consideration is the strain at which a load-bearing material yields or fails. In order to maintain optical continuity, it is requisite that the optical fiber strain-to-failure exceed that of the load-bearing members in tension. Table II lists a number of load-bearing materials and their approximate failure strains. Most optical fibers are composed of doped-silicate glasses protected by organic buffers. Since the modulus of silicate glasses is approximately  $10^7$  psi (or 70 G Pa), a 1% strain on a silica glass fiber represents a stress of 100,000 psi.

Table II  
LOAD-BEARING MATERIALS

<u>MATERIAL</u>	<u>STRAIN AT FAILURE %</u>
Ultra Strength Steel	1.0
Boron Composite	1.0
Graphite Composite	1.0
Kevlar-49 Composite	2.2
S-Glass Composite	4.5

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In the last few years, considerable progress has been achieved in improving the strength of optical fibers.<sup>[2]</sup> For many years it has been known that brittle ceramic materials fail under tension because of the presence of microscopic surface and volume defects. By carefully controlling the fiber drawing process, optical fiber manufacturers have been able to reduce the densities of larger flaws which lead to failure. As a result, the strengths of fibers have steadily increased. Many vendors now routinely proof-stress their fibers to a moderately high level, say 75,000 or 100,000 psi. While these levels are satisfactory for most non-military applications, the severe requirements of defense systems demand higher levels of assurance. It is reasonable to ask, however, what the limits of this improvement effort may be or, indeed, if it is practical and economically feasible to demand higher proof levels.

The theoretical bond strength of silica is approximately  $2.5 \times 10^6$  psi or approximately 25% tensile strain.<sup>[3]</sup> Thus, from the standpoint of ultimate fiber strength, the limits of attainability have not been approached. In short lengths, fibers have been drawn which have median strengths approaching  $1 \times 10^6$  psi (7 G Pa). Longer lengths are weaker because the probability of occurrence of flaws is proportional to the length. The longer the fiber, the more apt one is to have a large flaw which leads to tensile failure. In terms of the stress,  $\sigma$ , at which a fiber of length  $L$  fails, the probability of failure is given as:<sup>[4]</sup>

$$\psi(\sigma, L) = 1 - \exp \left\{ - \left( \frac{\sigma - \sigma_p}{\sigma_0} \right)^M \left( \frac{L}{L_0} \right) \right\} \quad (1)$$

In Equation (1),  $\sigma_0$  is a scale parameter,  $\sigma_p$  is the proof-stress, and  $L_0$  is the initial length. Thus, the chance of failure increases exponentially with length.

Ideally, one would desire proof-tested fibers. Because of the aforementioned length dependence of the survival, however, the additional manufacturing cost of assuring defect-free fibers also rises exponentially. A practical compromise, therefore, would seem to be to splice together proof-tested segments of, say, 5 km if a fairly long, high strength fiber was needed. The solution to these problems appears to be within reach. Recent work at Bell Telephone Laboratories was reported to have exceeded 12 km at a 200,000 psi proof level in a continuous run.<sup>[5]</sup> This is an important milestone with promising implications for military applications.

#### STRESS CORROSION IN OPTICAL FIBERS

Assuming that strong optical fibers can be drawn is the first half of the problem. The second part is to preserve this initial strength and this leads to the issue of hard, insulating coatings. It is well known that ceramic materials, under stress in the presence of water, suffer a diminution of strength through the phenomenon of stress corrosion or static fatigue.<sup>[6]</sup> Fibers are coated with buffering materials as they are drawn. These coatings serve to prevent micro-bending optical losses and to protect against abrasion. There is a trade-off as to the thickness of such coatings, the greater the thickness, the better the fiber is protected against abrasion damage (which introduces flaws, thereby weakening the fiber). On the other hand, it should not be so thick that one ends up with dimensions on the order of conventional coaxial or twisted pair wires which fibers are intended to replace. However, from the standpoint of strength preservation, these coatings, which are organic polymers, are all permeable to water and, therefore, do not prevent stress corrosion.

The time to failure,  $t_f$ , of a fiber under a static stress,  $\sigma$ , can be estimated from the fracture mechanics relation,<sup>[7]</sup>

$$t_f = \frac{2}{YA(N-2)\sigma^N} \left( \frac{K_{IC}}{\sigma_{IC}} \right)^{2-N} \quad (2)$$

in which Y is a dimensionless geometric constant,  $K_{IC}$  is the characteristic critical stress intensity factor for the fiber material, and  $\sigma_{IC}$  is the strength of the fiber in an inert environment. The numbers A and N are constants, but N is environmentally dependent. In fact, N is a figure of merit as to the resistance of the material to static fatigue. For silica materials in an ambient environment N has a value of about 20. Hermetically-coated fibers have larger values of N and this translates into substantially longer times-to-failure.

Figures (1) and (2) are respectively plots of the cumulative failure probabilities of a non-hermetic fiber and a hermetically-coated fiber. These were taken in decades of stressing rate. The data points indicated by the symbol N were taken in dry nitrogen. It is clear from a comparison of the two plots that the hermetically-coated fiber is less dependent on stressing rate and more closely approaches the behavior observed in an inert environment.

If one now plots the median strengths versus the stress rate or, alternatively fitting the differentiated Equation (2) to stressing rates, the diagrams in Figures (3) and (4) are obtained. The inverse of the slope is the parameter N discussed above. The larger value of N, for hermetically-coated fibers, is indicative of stress corrosion resistance.

To achieve hermeticity in optical fibers, the Department of Defense has supported several research efforts aimed at coatings other than polymer-based materials. One approach involved the in-line application of metals. While these metallic coatings offered a fairly high level of protection from stress corrosion, they suffered from other problems which offset the advantage of improved resistance to static fatigue. Among these problems were excessive microbending losses, poor concentricity, cyclic failure, plastic flow under tension, and electrical conductivity. While some of these objections have since been overcome, there still remains the problem of electrical conductivity which is totally incompatible with all-dielectric waveguides.

Alternative approaches using dielectric coatings are currently under investigation. The first of these is the in-line chemical vapor deposition of silicon nitride.<sup>[8]</sup> This technique has the advantage of rapid coating at fiber drawing rates. At the same time, results of fatigue testing yield values of N on the order of 100 which significantly extend the estimated times-to-failure. One of the disadvantages with the SiN, however, is that the median strengths of fibers is halved. While this intrinsic strength reduction is acceptable for moderate strength applications, it is unsatisfactory for those applications where proof-stress levels in excess of 200,000 psi are required. Figure (5) depicts the drawing system and Si<sub>3</sub>N<sub>4</sub> in-line reactor for hermetically-coating fibers as developed at Hewlett-Packard Laboratories.<sup>[8]</sup>

Another approach involves the ion plasma deposition of dielectric materials.<sup>[9]</sup> This technique offers the advantage of drawing and coating fibers in a high vacuum which should dramatically improve the attainable pristine strength, in addition to hermeticity, since a major source of surface flaws has been traced to airborne particulates. Preliminary results are very interesting, but whether deposition

rates will be fast enough is still uncertain. Inadequate quantities of coated fibers have been available for extensive mechanical or optical characterization. Another advantage of this approach is the fairly large number of dielectric, as well as metallic materials that could be investigated. To date, only hard carbon coatings and indium were attempted.

Thin coatings of diamond-like carbon would seem to offer solutions to the optical fiber problem. In addition to hermeticity, the hardness should serve to prevent abrasion enabling one to reduce the thickness of the polymer overcoating. Chemical inertness and insulating behavior are further attractive features. Table III lists some of the properties that would be essential for such coatings.

Table III

PREFERRED CHARACTERISTICS OF HERMETIC COATINGS

Thin (300 - 600 Å )  
Uniform/smooth/concentric  
Pin-hole free  
Stress-free  
Chemically inert  
No cyclic fatigue  
Fast in-line deposition  
No diminution of fiber strength  
Easily spliced  
Low cost

FUTURE WORK

Numerous ideas have been proposed by several investigators for hermetic coatings. These proposals suffer primarily from the fact that the investigator does not have access to a fiber drawing facility. In order to fully and properly characterize coatings, one must have the capability of in-line coating and control of the many variables involved in drawing fibers. To date, many of the large optical waveguide vendors have been unresponsive to the DOD's need for fatigue-resistant, high strength fibers. However, as the military and commercial markets expand for these fibers, greater interest in solving these problems can be expected.

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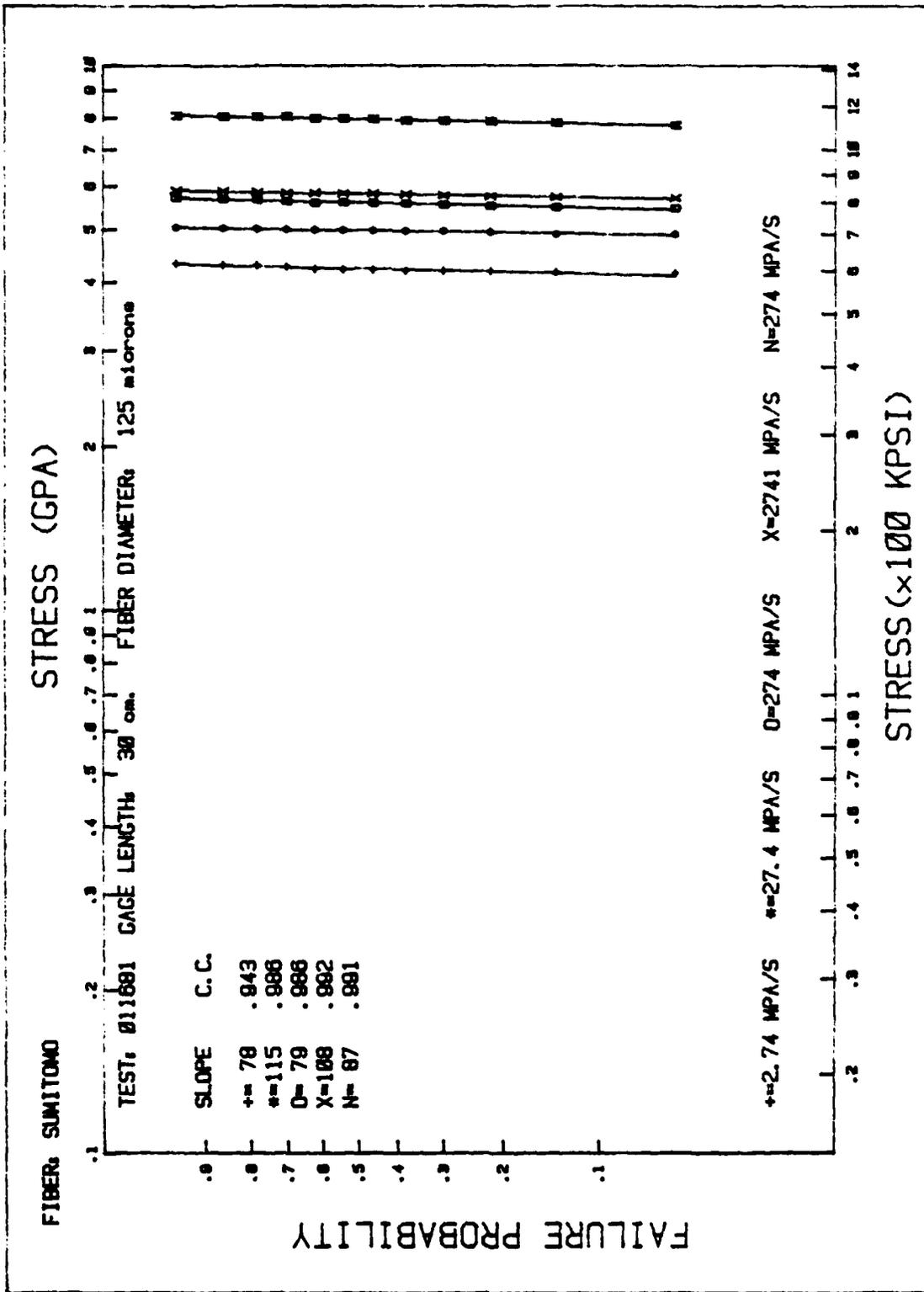


Figure 1. Cumulative Failure Probabilities of a Non-Hermetically-Coated Fiber at Different Stressing Rates

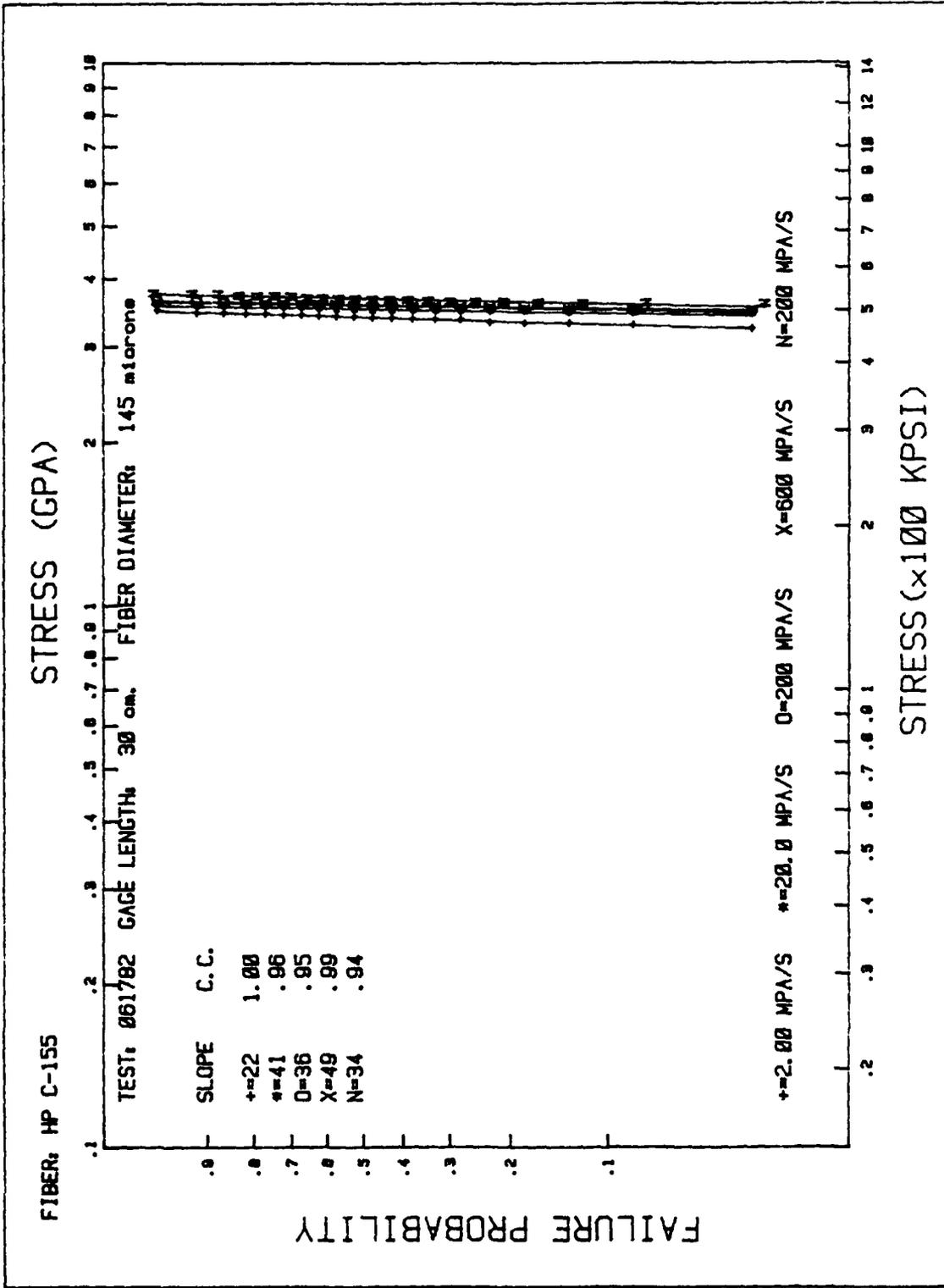


Figure 2. Cumulative Failure Probabilities of a Hermetically-Coated Fiber at Different Stressing Rates

# DYNAMIC FATIGUE FOR N VALUES

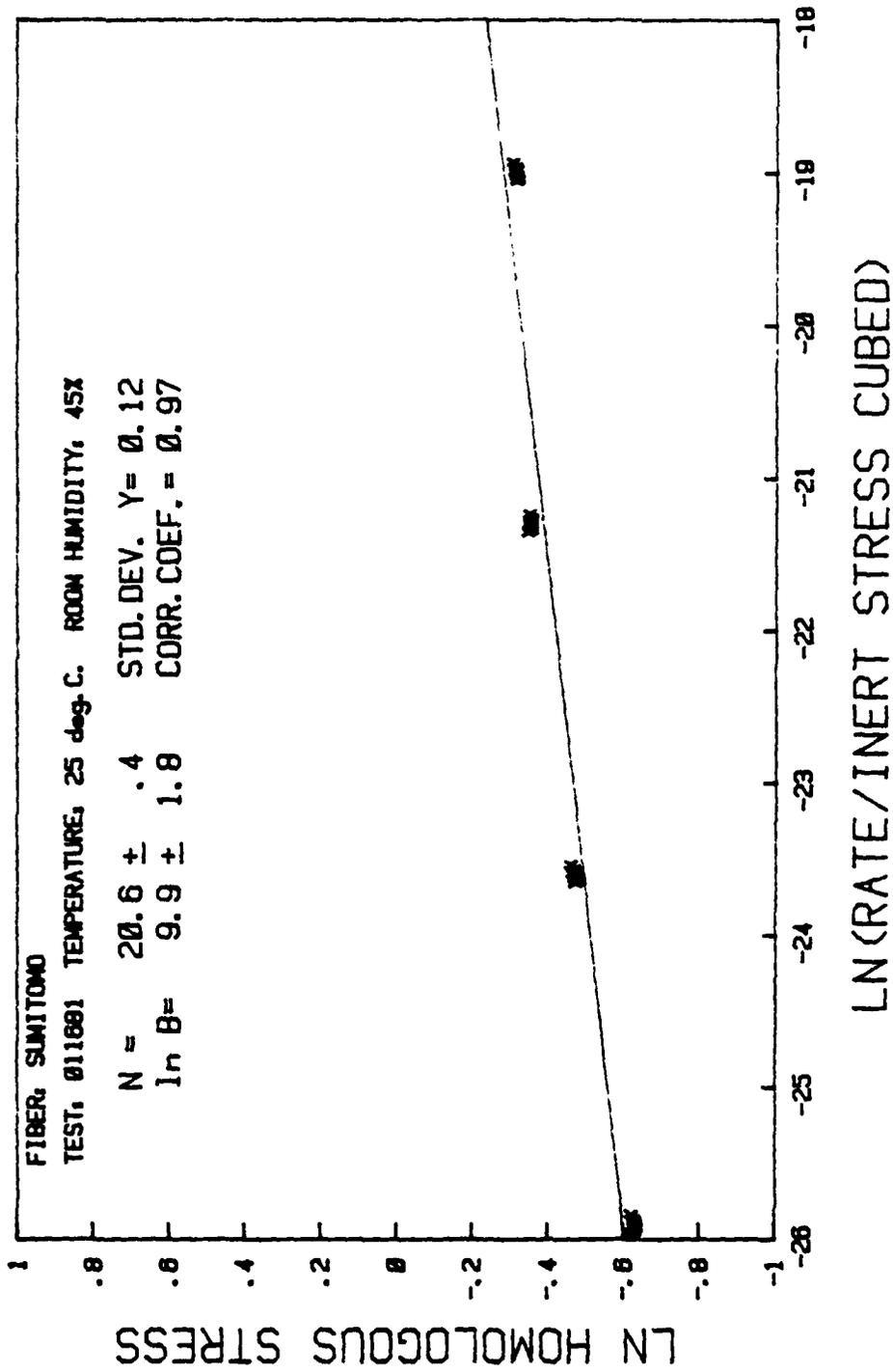


Figure 3. Plot of the Homologous Strengths (Determined at Different Stress Rates) of a Non-Hermetically-Coated Fiber versus Stress Rate

# DYNAMIC FATIGUE FOR N VALUES

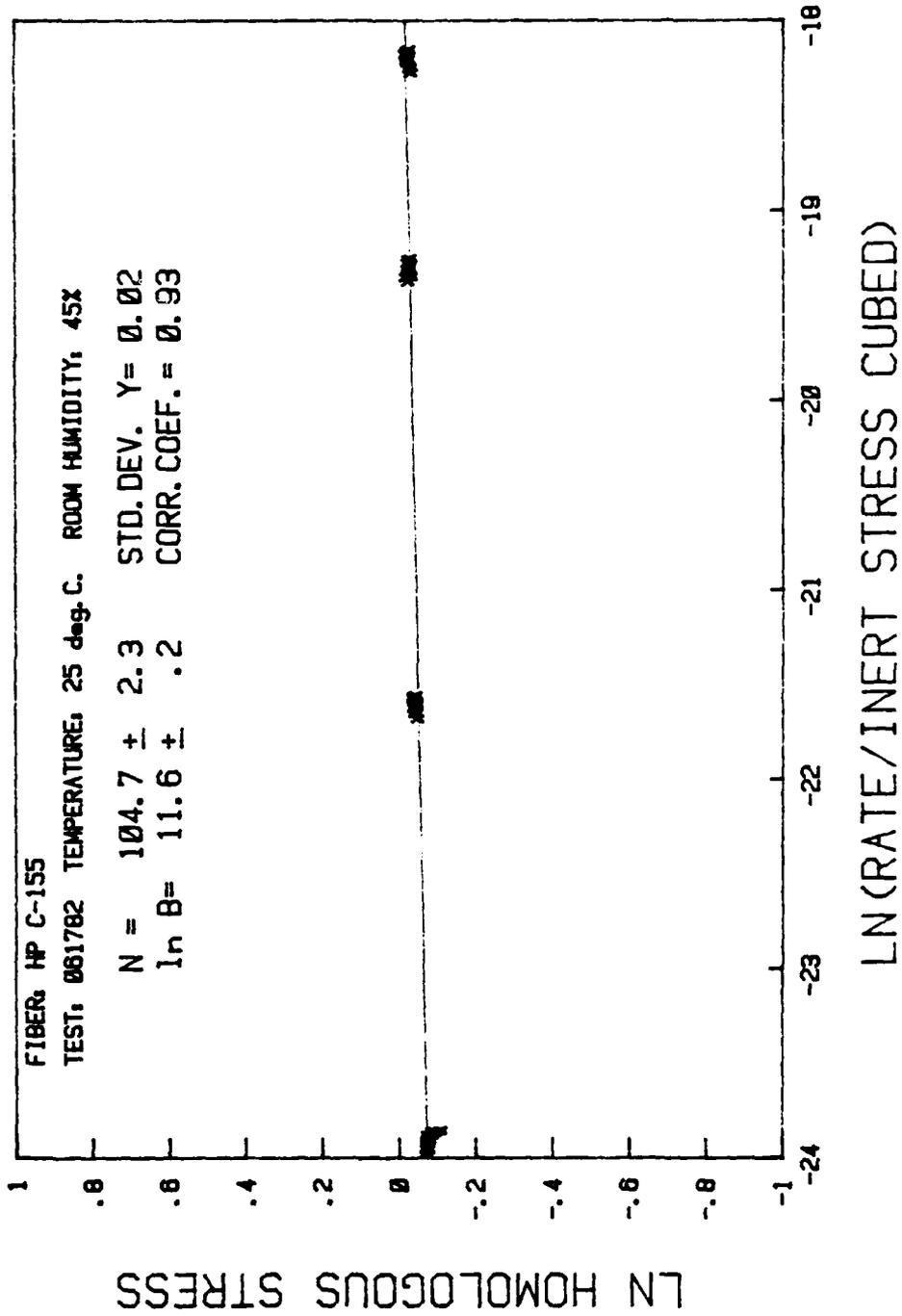


Figure 4. Plot of the Homologous Strengths of a Hermetically-Coated Fiber Versus Stressing Rate

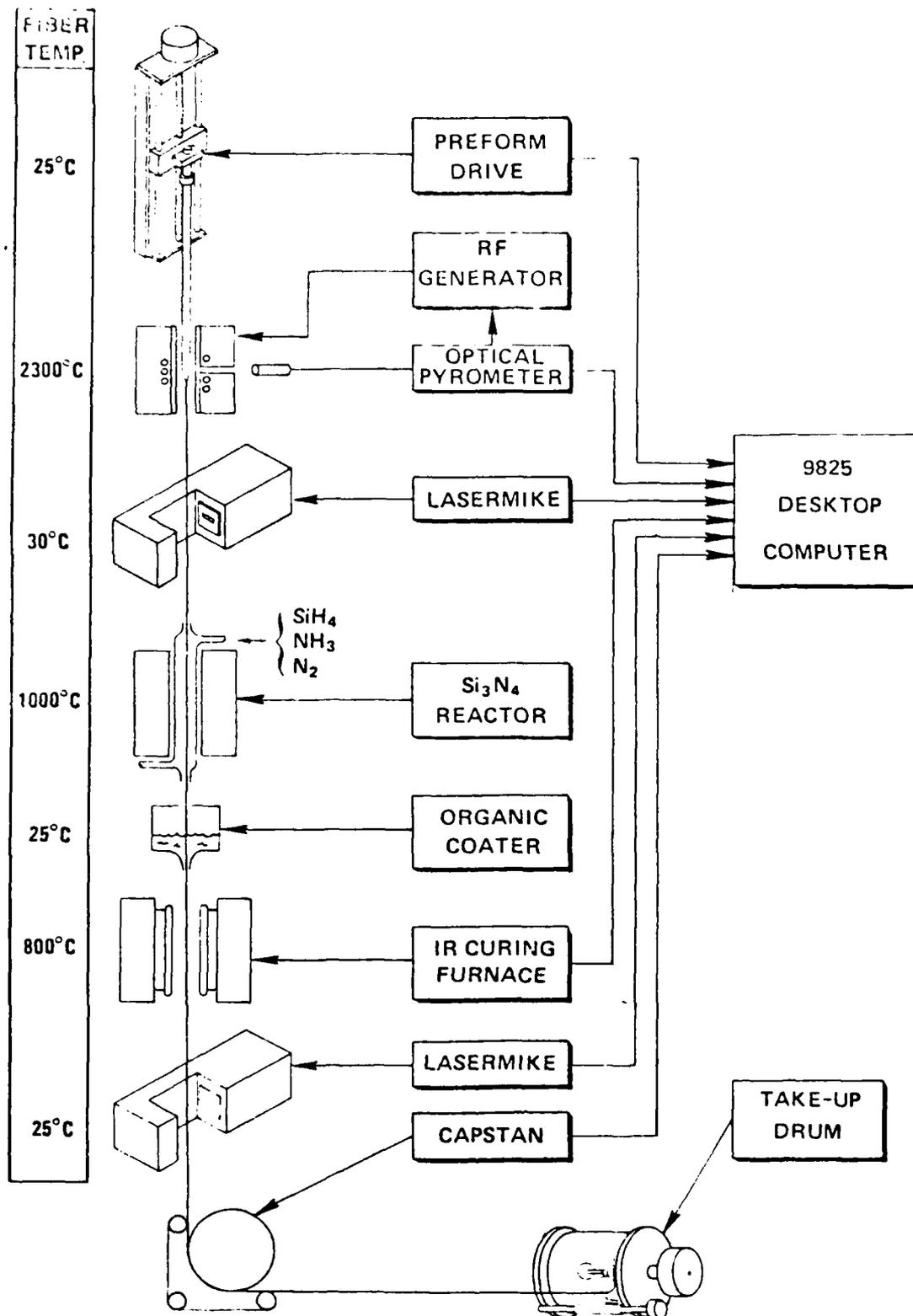


Figure 5. Diagram of the In-Line Coating of an Optical Fiber with  $\text{Si}_3\text{N}_4$  (from Hewlett-Packard, ref. 8)



# Preparation and Properties

PREPARATION AND PROPERTIES

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