DEVELOPMENT OF QUARTZ FIBER PARACHUTE MATERIALS

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ABSTRACT

In the realm of flexible space vehicle decelerators, few existing materials are capable of withstanding hyper-thermal environmental temperatures up to 2000°F. Conventional glass fiber structures normally have a temperature ceiling of 1200°F. Filamentous nickel alloys and fused silica are potentially serviceable to 1800°F or higher for short durations. Quartz fibers became semi-commercially available about two years ago. This research project was initiated to investigate the feasibility of utilizing these fibers for the fabrication of various parachute components.

Potentially, individual quartz fibers when handled with extreme care, have tenacities up to 15 grams per denier at room temperature. As much as 50% of this tenacity remains at 1800°F. Quartz fibers are even worse than fiberglass in that they are extremely brittle, sensitive to scratching, and have virtually no abrasion resistance when unprotected. Therefore, the initial portion of the research effort was directed to the development of a high temperature finish which would facilitate the processing of the fibers at room temperature as well as to afford protection at elevated temperatures. The first promising compound developed was magnesium acetate. Yarns treated with a size composed of starch and oil to which 3% magnesium acetate was added was found to be satisfactory at room temperature for winding, plying, and weaving.

However, this finish does not offer sufficient protection at temperatures above 600°F. Fabrics woven with quartz yarns thus treated lost up to 90% of their room temperature strength when tested at 1800°F, while under the same conditions the fibers lose only 50%. Thus, there is theoretical as well as practical opportunity to improve strength translation efficiency from 10% to at least 50%.

Preliminary data on a recently developed finish, an alumina 8-hydroxy quinoline complex, show considerable further improvement over the magnesium acetate. Analysis of mechanical properties of quartz indicates possible cause of poor fiber to yarn and fabric translation due to the crushing and bending behavior of fibers in aggregates.

I. INTRODUCTION

In the realm of flexible space vehicle decelerators, few existing materials are capable of withstanding hyper-thermal environmental temperatures up to 2000°F. Conventional glass fiber structures normally have a temperature ceiling of 1200°F. Filamentous nickel alloy and fused silica (quartz) are potentially serviceable to 1800°F. Continuous filament quartz fibers became semi-commercially available about two years ago. This paper describes the results to date of a research project, sponsored by the Aeronautical Systems Division, Wright-Patterson Air Force Base, Ohio, to investigate the feasibility of utilizing these fibers for the fabrication of various parachute components.

Potentially, individual quartz fibers when handled with extreme care have tenacities of up to 15 grams per denier* or approximately 420,000 p.s.i. at room temperature. Unfortunately, however, this high strength value for fibers is rarely attainable in the laboratory, nor is the equivalent strength achieved in yarn and fabric under use conditions. From the time the fiber is produced, it is immediately subjected to a multitude of unfavorable environmental as well as physical abuses. While the mechanism of strength loss is still unclear, it is known that glass and quartz yarns are brittle and easily scratched. These scratches, together with other invisible defects, become sources of crack propagation and fiber failure. (This failure mechanism is briefly discussed in Section VI.) It is further believed that certain gases such as carbon dioxide, sulfur dioxide and particularly atmospheric moisture are also detrimental to the strength retention of quartz fibers. The damage caused by normal mechanical processing and handling during yarn twisting and fabric weaving are usually unavoidable. As a consequence, quartz fibers generally yield a tenacity of approximately eight or nine grams per denier. This is by no means outstanding when compared with the tenacities of the various high strength synthetic fibers. Nylon and high tenacity rayon, for example, have tenacities in the seven to nine grams per denier range. On the other hand, none of the conventional fibers, including the new high temperature resisting HT-1, retains even a small fraction of its original strength at temperatures in excess of 1200°F. Since the softening point of quartz is even higher than that of glass, it is logical that the feasibility of utilizing this material for high temperature applications be fully explored.

II. OBJECTIVES

Quartz fibers are similar to glass fibers in that at room temperature, because of their extreme brittleness and scratch sensitivity, they have very low abrasion resistance when unprotected by finishing treatments. One of the objectives of this program is the development of finishes which will protect the quartz and maintain its tenacity throughout manufacturing and subsequent assembly processes at room temperature, as well as at elevated temperatures during and subsequent to deployment.

With the present conventional starch-oil size applied by the fiber producer, the strength translational efficiencies of fiber-to-yarn and yarn-to-fabric are very low. This translational efficiency is defined as the ratio of the actual strength of a woven textile structure to the total theoretical strength of all the load-bearing individual fibers contained therein. For example, in a yarn containing 100 filaments, each having an average breaking strength of 13 grams, to be 100% efficient, the yarn must have a breaking strength of 1300 grams. In reality, 100% translation is almost never attainable even in conventional textile materials, primarily due to a condition called "elongation balance" or, actually, the lack thereof. Consider the case of a yarn containing 100 separate filaments, each of which is extruded from a multi-hole spinnerette. While each filament comes from the same polymer melt, it is extremely unlikely that all filament breaking strengths and elongations are absolutely identical. The weaker or less extensible

* Denier is a direct measure of weight per unit length, where one denier is equal to one gram per 9000 meters.

filaments will fail first, and the load which they were carrying must then be borne by the still intact filaments. However, this load, when added to that which the intact filaments are already carrying, may be excessive, and so some more filaments fail. Thus, the failure is progressive and the yarn breaks at a strength value lower than that theoretically achievable. A second important elongation-balance inefficiency is due to the fact that not all fibers within a yarn are of the same total length, a variable often introduced during yarn plying, twisting and winding operations. Because of these unequal lengths, all filaments do not reach their limiting loads and break at the same time, and a low break results. In the case of quartz yarns, the effect of elongation unbalance is much more serious than with conventional textiles. Wherever there is differential elongation, relative motion must occur between individual fibers and their immediate neighbors when strained, and this causes friction. Quartz fibers, particularly if not finished, simply can not afford to be touched or rubbed at all without creating catastrophic damage.

Presuming that a satisfactory finish which will protect the fibers from abrasive damage at both room and elevated temperatures (1000 to 1800°F) were to be achieved, a second objective of the program is the fabrication and evaluation of the various parachute components manufactured from quartz yarns, utilizing optimum construction geometry. These include fabrics, webbings, tapes, braids and sewing threads which are to be evaluated at room temperature as well as at several elevated temperatures up to 1800°F. Attempts are to be made to make sewn joints of these components and then to determine seam strength efficiencies.

III. TEST METHODS

A. Characterization of Quartz Fiber and Yarn Properties

The mechanical performance of quartz yarns and fibers leave much to be desired when compared with ordinary textiles. There is no dispute that the material is brittle, difficult to handle, does not permit excessive flexing and is not readily weavable. To define all this in quantitative terms via measurement of fiber, yarn, and fabric strengths becomes complex. Differences among operators and specific testing techniques are as critically important as the prior history of the specimens. For example, FRL® was requested by ASD to provide some quartz materials to another Air Force contractor working on a related project. One would normally not expect inter-laboratory fiber and yarn results to differ significantly. Yet the fabric strength obtained by FRL was almost twice as high as the results obtained by the other contractor. Hence, at the risk of belaboring details, a brief discussion of the test procedures appears to be warranted.

Quartz is akin to glass, and so the yarn numbering system adopted to describe yarn size is the one commonly used in the glass fiber industry. For example, 300-2/2 designates a yarn composed of singles yarn weighing 300 **hundred**, i.e., 30,000 yards to the pound. Two such singles are plied together, and two of the resulting two ply yarns are again plied together. The nominal constructions of the semi-commercially available quartz yarns are as follows:

	Standard	Special*	Special*
Glass Yarn Number	150	300	600
Approximate yards per pound	15,000	30,000	60,000
Number of filaments	200	100	50
Filament diameter, inches	0.0004	0.0004	0.0004
Approximate yarn diameter, in.	0.008	0.006	0.004
Approximate yarn denier	300	150	75

All fiber data reported in this paper were obtained from fibers removed from these "producer's yarns. Except by special arrangements, quartz filaments in single fiber form are usually not available because of handling, transportation and storage difficulties. Additionally, unless otherwise specified, all yarns were finished with the producer's standard starch-oil size.

B. Fiber Tensile Tests

To minimize handling and flexing of the specimen, particularly during the process of clamping the fiber in jaws of the testing machine, FRL developed a technique whereby two nylon washers are placed in a jig with the distance between the edges of the washers accurately spaced one inch apart. The fiber specimen is then glued to the two washers with a dab of Duco cement. Thus the denier (tex) of the mounted specimens can be determined by the vibrascope** technique, and subsequently inserted in the tester for tensile testing.

In order to establish a valid basis for comparison, fiber tenacity at room temperature was determined by averaging 100 individual tests. Two series of fiber strength tests were made of: (1) nearly all individual fibers across a yarn cross-section; (2) sequential specimens selected from one continuous filament within a yarn. The results are as follows:

	Breaking Strength	Fiber Denier	Fiber Tenacity
Specimens across the yarn	13.7 grams	1.60	8.56 gm/den
Coefficient of variation	25.0%	12.0%	
Specimens along one filament	12.2 grams	1.60	7.63 gm/den
Coefficient of variation	32.4%	2.6%	

It is interesting to note that despite the fact that the filament denier was more uniform when all the specimens were taken from a single fiber, the breaking strength coefficient of variation was found to be higher along a filament than across the yarn.*** Since the breaking strength of 13.7 grams had a lower coefficient of variation and it represented the strength of many filaments, the tenacity of 8.56 grams per denier is probably more reliable, and this value is used as the

* Available on special order.

** Method is based on the principle which states that the denier of a fiber is a function of the frequency of vibration.

*** This may seem unusual, but is probably that the difference in coefficient of variation is not significant.

reference base for the calculation of yarn and fabric translation efficiencies presented herewith.

C. Yarn Tests

The tensile testing of quartz yarns is fairly straightforward. Using masking tape as a jaw liner, very little crushing was experienced. The starch-oil sized 100 filament producer's yarn used throughout the study has a strength of 652 grams and a tenacity of 4.07 grams per denier. Thus yarn efficiency, based upon available fiber strength, is 4.07/8.56, or 48%. The producer's standard yarn retains less than 50% of its theoretically available fiber strength.

Since the objective of the finishing treatment is to protect the fibers in the yarn from a variety of mechanical abuses including scratching, abrading, crushing and flexing, a need existed to develop a simple test whereby the efficacy of a new protective finish could be quickly ascertained. The following "flex-abrasion" test was adopted: one end of the test specimen is taped to a 50 gram weight, the other end is held by the operator's hand, the specimen is looped over a 1/4 inch glass rod clamped on a ring-stand, the specimen is bent around the rod in the form of an inverted "U". When the operator raises or lowers his hand, the yarn is subjected to simple tension as exerted by the 50 gram weight. Flexing and unflexing occurs as different portions of the yarn ride over the rod, with accompanying crushing of the interior fibers by the external fibers, as well as scratching caused by whatever relative motion develops between fibers. The action is continued until the yarn fails, at which point the number of strokes is counted. Admittedly, this is an empirical test, but it does give order of magnitude comparisons among the various finishes.

D. Fabric Tests

The breaking strength and elongation values for quartz fabrics were determined, employing a test procedure slightly modified from the standard strip tensile method in order to prevent premature specimen failure and spurious elongation measurements. The problem is paradoxical in that the jaws must be tightly clamped to minimize fabric slippage and "jaw penetration", but this will invariably crush the quartz yarns at the edge of the jaws, thus producing "jaw breaks". Lining the jaw faces with masking tape or leather, or looping the end of the specimen over a round rod, did prevent slippage without crushing the yarns. However, "jaw penetration" still persisted, giving falsely high rupture elongations. The best technique found was one where the ends of the specimens are firmly embedded in a mixture of ten parts Shell Epon 828 epoxy resin and one part tetraethylene pentamine catalyst. Fabric pieces were cut sixteen inches long by its full width and two parallel pencil lines were drawn, one inch from each edge, thus allowing fourteen inches for the gage length. The samples were laid over sheets of brown kraft paper. Sufficient resin was applied to the fabrics to provide complete penetration. Care was exercised to deposit the resin accurately along the penciled lines. The surface was smoothed with a spatula to obtain subsequent even clamping. The resin was allowed to cure at room temperature overnight, after which the fabrics, together with the kraft paper, were cut into strips 1-1/2 inches wide. The strips were then raveled to widths of exactly one inch for testing. The kraft paper backings were cut just prior to testing; their presence added dimensional stability to the fabric specimens, which permitted easier handling and clamping. This procedure, while time consuming, is well worth the effort. The elongation values thus obtained are quite reliable (as proven by an "Effective Gage Length"* test, utilizing specimens from 3 to 30 inches long). The actual gage length used for these tests was 14 inches long. This dimension was selected so that the same gage length could be employed in later high temperature studies where the specimens must extend beyond the high temperature test chamber. All tests were performed on an Instron Tester. Because of the inherent low extensibility of the material, the crosshead speed selected was 0.5 inches per minute.

The elevated temperature tests were conducted on the Instron with a clamshell type special oven. The oven consists of two deep stainless steel laboratory trays each measuring approximately eight inches long, six inches wide and four inches deep. The original rolled edges were flattened and welded to frames made of 1/2" x 2" flat stainless steel stock. The two mating frames were hinged together. Slots 1/8" deep by 2-1/2" wide were machined in the top and bottom of each frame, thus yielding an opening of 1/4" x 2-1/2" for the specimen. This opening is large enough for the future testing of webbings. The oven was clamped on the columns of the Instron, the hinged half was permitted to swing open for loading and unloading of the specimen. Closure was accomplished via toggle clamps. Thermal energy was supplied by specially made resistance elements, 1800 watts in each half, which were completely encased in refractory cement. The surfaces of the radiant sources were approximately 1/2" from the centerline of the oven and provided a heating zone of 6 inches. A thermocouple was used for sensing, while control was performed manually with a Variac. The oven was capable of reaching temperatures of up to 2200°F. Incidentally, the total cost for the complete unit was in the neighborhood of only $300 (See Figure 1).

The "at temperature" tests were conducted after the specimens were brought to the desired temperature for 15 minutes. It should be noted that since the clamps were outside of the oven and the heated zone was only six inches, while the gage length was 14 inches, the elongation values thus obtained were only relative.

IV. DEVELOPMENT OF IMPROVED FINISHES

Because of the nature of the quartz fiber manufacture which requires application of a size as the fibers are formed, all work had to be done on yarn as supplied by General Electric containing 2-3% of the starch-oil size. The question then arose as to whether this size should be removed prior to the application of experimental finishes. It seemed probably that several of the candidate high temperature finishes would be as effective when applied over the starch-oil as when applied to bare quartz. In such cases they would maintain their effectiveness at temperatures high enough to volatilize the starch-oil size. Other finishes, however, might require direct chemical reaction with the fiber and therefore must, of necessity, be applied to the bare fiber. Hence, various methods for removal of the starch-oil size were investigated. By analogy to normal practice with glass fabrics, the simplest method for removal of the size is heat cleaning. However, the literature indicates that quartz fibers or yarns lose considerable strength when heated to high temperatures in the presence of the starch-oil size. Photomicrographs(1) indicate that a surface reaction takes place between the size or its pyrolysis products, and the quartz, which results in a weakening of the fiber. Yarns thermally desized at 1000°F also showed virtually no abrasion

* A method determining true extension by extrapolating the results of tests with varying gage lengths.

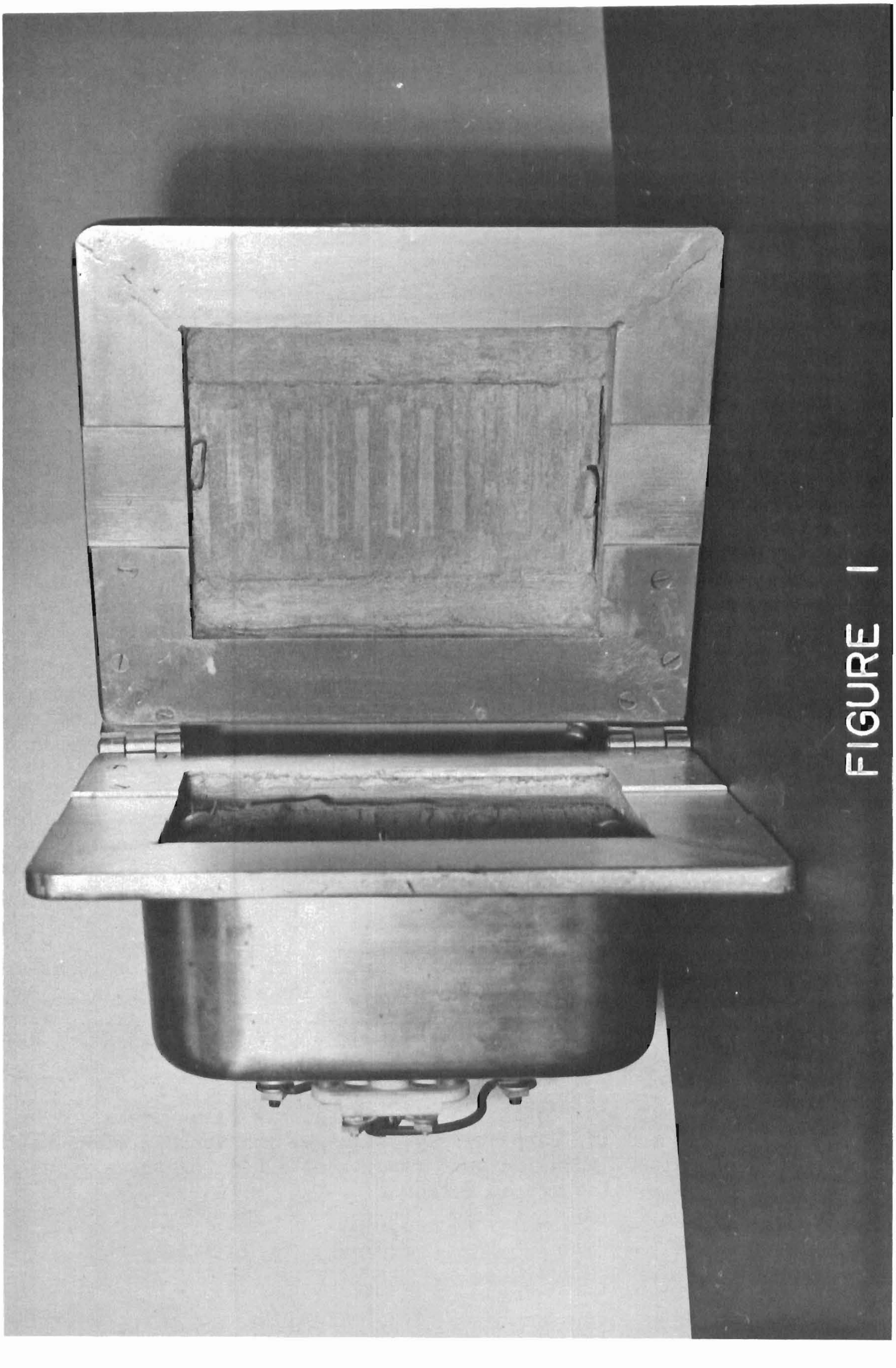

FIGURE I

resistance.

Therefore a simple non-thermal method of desizing was developed to remove the starch-oil size. It involved treating the yarn in a boiling 1/2% solution of a non-ionic detergent, Druterge W, at pH 4, followed by a water rinse. This treatment appeared to completely remove organic material from the yarn, with no loss in tensile strength, although its abrasion resistance is reduced markedly. Relubrication with a silicone oil (1.5% Dow Corning's DC-36) increases the abrasion resistance to a level over the untreated desized, but lower than the original starch-oil sized yarn.

In order to test the efficiency of finishes in conjunction with, and in place of the starch-oil size, experimental finishing trials were run using both yarn "as received" and "after desizing". The first materials investigated were silicone oils and resins, the former being used as glass fabric finishes for continuous use at 600°F. Two silicone oils, Dow Corning's DC-36 and XET-4327, and two resins, Dow Corning's No. QR-6-2000 silicone rubber, and No. 994 varnish were investigated. Only the DC-36 showed abrasion resistance comparable to the starch-oil sized yarn prior to heat treatment. The resin stiffened the yarn excessively. The DC-36 was apparently volatilized at 1000°F, since yarn treated with this oil had no abrasion resistance after heat treatment. Treatments which would lead to thermal formation of carbonaceous residues were equally unsuccessful. Starch, when treated with flame-proofing materials normally used with cotton, was pyrolyzed to an oxidation resistant char, which failed to protect the yarn from abrasion.

Graphite, molybdenum disulfide and nickel chloride, all good lubricants, the last two good at high temperatures, were applied to quartz yarns. Graphite and molybdenum disulfide gave fair, and nickel chloride good abrasion resistance prior to heat treatment, but all had poor abrasion resistance after heat treatment. Silver and nickel coatings were electroless plated on the yarn, but again both had poor abrasion resistance after heat treatment.

Investigation of metallic oxide coatings with extreme thermal stability showed magnesium oxide to be unique in improving abrasion resistance. The oxides of silicon, titanium, aluminum, chromium, zirconium, borium, calcium, and magnesium were applied, but only magnesium oxide showed improved abrasion resistance both before and after exposure at 1000°F.

At this point in the investigation the optimum finish obtained consisted of treating desized yarn with a 2% solution of magnesium acetate, drying, treating with dilute ammonia to form magnesium hydroxide and then heat treating at 1000°F. Yarn so treated had good abrasion resistance prior to heat exposure and fair abrasion resistance after heat treatment.

Small quantities of 300-1/0-1Z-100 filament quartz yarn were sized by the fiber producer with 2 and 4%, respectively, of magnesium acetate added to the standard starch-oil size. Abrasion test results obtained for these yarns and a control made at the same time are as follows:

Abrasion Resistance (strokes to failure)

Control (starch-oil size)	Starch-oil plus 2% magnesium acetate	Starch-oil plus 4% magnesium acetate
64	120	154

After treatment with 1% ammonium hydroxide to convert the acetate to the hydroxide and exposure to 1000°F for 15 minutes, the following abrasion results were obtained:

Control (starch-oil size)	Starch-oil plus 2% magnesium acetate	Starch-oil plus 4% magnesium acetate
0	11	22

These experimental magnesium acetate treated yarns were twisted, plied, and woven into narrow fabrics. In the plying and twisting operations, it was discovered that the yarns were considerably more abrasive than fiber glass. Nylon eyelets, for example, had to be replaced by stainless steel ones to withstand the abrasion.

Great difficulties were encountered in weaving a 6" wide narrow fabric from the control yarn. As far as is known, this was the first time an attempt was made to weave a quartz fabric from the 300's yarn; heretofore the fabrics were made from heavier 150's. Yarns treated with the magnesium acetate wove extremely well. According to the weaver, "there was as much difference as between night and day between the weavability of the treated and untreated yarns".

The results of abrasion tests on yarns and fabrics treated with the magnesium acetate consistently showed the 4% treatment to be slightly better than the 2%. However, the 2% appeared to perform better in yarn processing although it made no difference in weaving. Therefore it was decided that the yarn required for the production of experimental fabrics would be sized with the starch-oil size to which 3% magnesium acetate was added.

V. FABRIC EVALUATION

Six experimental fabrics were woven, consisting of three different weave patterns for each of two fabric weights. Three looms were set up; each had a different warp, namely 300-2/2 yarn, 300-1/2 yarn and 300-1/0 yarn. The 2/2 warp was intended for the three heavier fabrics, while the two-ply and singles warps were for the three lighter fabrics. It was soon evident that the singles warp could not be made to run on the loom because the yarns could not withstand the beating in the heddles.

The three styles of the heavy fabric were woven from the 300-2/2 warp; the filling was also a 300-2/2 construction. The three weaves were a plain weave; a four shaft satin and an eight shaft satin. Fabric weaving ran reasonably well, although according to the weaver it did not weave as well as glass fabrics. It was opined, however, that these fabrics could be woven on a satisfactory commercial basis.

Two of the lighter fabrics were woven with 300-1/2 yarns in both warp and filling. Many selvage breaks occurred when the loom was operating at 50 picks per inch. When the pickage was reduced to 48, satisfactory results were obtained. The third light weight plain weave fabric was woven with the 300-1/2 warp and 300-1/0 filling. This was done to see if the singles yarns could be successfully used in the filling direction.

These six experimental fabrics were tested for physical properties, including fabric analyses (Table I) and tensile strengths at 70°F, 600°F, 1200°F and 1800°F (Table II). The performances of these fabrics at the elevated temperatures were rather disappointing. Average strength retentions based on each fabric's respective room temperature strength, were only 58% at 600°F, 17% at 1200°F and 11% at 1800°F, while the fiber strength retentions were found to be 120% at 600°F, 59% at 1200°F and 19% at 1800°F. Thus it has been concluded that the magnesium acetate finish is not sufficiently effective at temperatures above 600°F due to the very poor abrasion resistance of the fibers at elevated temperatures. It was also concluded that the fabric strength losses at elevated temperatures which resulted from failure of the unprotected fibers were so large that no statistical nor practical strength differences could be established among any of the fabric constructions. It was believed futile to study the effects of yarn and fabric construction variables on physical properties at room or elevated temperatures, and to attempt to develop optimum quartz fabric constructions until the problem of the development of a protective finish was solved.

Subsequent effort has therefore been confined solely to finishes.

Recently a finish has been developed which, according to preliminary test results, exhibits a vast improvement over the magnesium acetate. In the magnesium acetate study, there were indications that the physical form of the finish was an important factor which influenced its efficiency. The crystalline form and state of subdivision appeared to be of particular significance. Thus, a new molecular orientation of oxides was sought, using coordination complexes of metal alkoxides with 8-hydroxy quinoline. The most promising compound applied to date is the reaction product of aluminum isopropoxide and 8-hydroxy quinoline. It is applied continuously in a two-bath system. The yarn is fed from a bath of aluminum isopropoxide in toluene directly into a bath of 8-hydroxy quinoline in toluene. The complex formed is bright yellow and is apparently stable up to 500 or 600°F. Above this, the complex is destroyed leaving a deposit of alumina. It may be that it is stable enough to heat and hydrolysis so that a linear polymer is formed by selective pyrolysis or hydrolysis of the isopropyl groups. The importance of the method of deposition of the alumina coating is shown by the fact that finishing with collodial alumina or aluminum salts followed by treatment with alkali to form the oxide gave no significant improvement in abrasion resistance.

The 8-hydroxy quinoline-aluminum isopropoxide finish give best results when applied to starch-oil sized yarn. Desized yarn apparently does not pick up enough of the finish and magnesium acetate interferes with the finish.

Preliminary data, using a 2 ply yarn as a control - the 2 ply yarn being somewhat easier to manipulate in the laboratory - are as follow:

TABLE I

FABRIC ANALYSES OF EXPERIMENTAL QUARTZ FABRICS

		Fabric Style Number					
		2501-1	2501-2	2501-3	2502-1	2502-2	2502-3
Fabric Weight, oz/sq yd		4.08	4.14	3.08	9.56	8.78	7.34
Warp Yarn	Construction	300-1/2	300-1/2	300-1/2	300-2/2	300-2/2	300-2/2
	Equivalent Denier	330	305	355	649	685	694
	Ply Twist*, t.p.i.	3.5	3.5	1.0*	3.5	3.5	3.5
Texture	Ends per inch	51	50	51	57	56	56
	Picks per inch	47	49	49	53	45	33
Weave Pattern		Plain	Twill	Plain	Twill	Twill	Plain

* Singles twist: 1.0 t.p.i.Z

TABLE II

BREAKING STRENGTHS OF QUARTZ FABRICS
TESTED AT VARIOUS ELEVATED TEMPERATURES

(with 15 minute Dwell Period)

	Fabric Numbers					
	2501-1	2501-2	2501-3	2502-1	2502-2	2502-3
Breaking Strength, lb/in						
70°F	79.1	125.0	104.9	242.9	135.8	108.7
600°F	45.7	67.6	60.9	124.6	96.5	61.3
1200°F	11.9	18.4	15.6	36.0	25.6	25.1
1800°F	2.1	12.6	10.0	25.4	19.8	20.9
Elongation, %						
70°F	2.41	1.62	1.64	1.92	2.60	3.92
600°F	2.20	1.15	1.42	1.48	2.18	3.27
1200°F	0.91	0.66	0.71	0.80	0.85	1.31
1800°F	0.78	0.58	0.51	0.70	0.71	1.08
Fabric Tenacity, g.p.d.*						
70°F	2.13	3.72	2.63	2.90	1.63	1.28
600°F	1.23	2.01	1.53	1.49	1.14	0.72
1200°F	0.32	0.55	0.39	0.43	0.30	0.30
1800°F	0.06	0.38	0.25	0.30	0.24	0.25

*Based on the total warp yarn denier.

	Starch Oil Control	Starch-Oil plus 3% magnesium acetate	Starch-Oil plus 8-hydroxy quinoline
Breaking Strength @ 70°F, gm	1302	1478	1852
Tenacity @ 70°F, gm/den	4.07	4.57	5.78
Breaking Strength @ 1200°F, gm	281	464	927
Tenacity @ 1200°F, gm/den	0.88	1.45	2.90

Because of the apparent very poor abrasion resistance of quartz at elevated temperatures, the abrasion test was modified by replacing the 1/4" glass rod with a 3/8" nickel alloy cartridge heater so that yarns could be tested in abrasion over a surface up to temperatures up to 1200°F. The results for the three yarns above are as follow:

	Starch Oil Control	Starch-Oil plus 3% magnesium acetate	Starch-Oil plus 8-hydroxy quinoline
Surface temperature	800°F	800°F	1000°F
Abrasion strokes to failure	2.5	13.1	87

The tenacity of 2.90 grams per denier of the treated yarn tested at 1200°F is an impressive one. Additional work is in progress further to refine the treatment in preparation for possible mill trials.

Although the six experimental fabrics woven from yarns treated with the magnesium acetate did not perform up to expectations at the elevated temperatures, the experiment can not be considered a total failure because two important conclusions were obtained. First, it has been proven that fabrics as light as 3 ounces per square yard can be successfully woven, a fact of extreme significance in terms of parachute needs. Second, opportunity was afforded to evaluate the effects of fabric geometry at room temperature. Expressing the fabric strength at room temperature in terms of yarn to fabric translational efficiencies, the following comparisons are made:

Fabric No.	Weave	Filling Yarn	Picks/In.	Efficiency*
2501-1	Plain	300-1/2	47	45.8%
2501-2	Twill	300-1/2	49	74.0%
2501-3	Plain	300-1/0	49	60.3%
2502-1	Twill	300-2/2	53	63.1%
2502-2	Twill	300-2/2	45	35.9%
2502-3	Plain	300-2/2	33	28.8%

* Based on original warp yarn strength.

The effect of the plain weave with its frequent yarn crossovers is devastating. Fabrics Nos. 2501-1 and 2501-2 are practically identical except for the weave; the resulting difference in efficiencies is 28%. Fabric Nos. 2501-1 and 2501-3 have similar weaves but with different filling yarns. The smaller singles filling yarn in 2501-3 apparently permit the warp yarns to take a less tortuous path, and so give an increase in efficiency of 14%. This is further proven by the fact that the efficiencies of the heavier fabrics (woven from the 300-2/2 yarns) are much poorer. The difference of 34% in efficiencies between Fabrics 2502-1 and 2502-3 is more than the 28% in the case of 2501-1 and 2501-2. This is because, in addition to the weave change, there is also a large disparity in pickage. These phenomena are well known in conventional textiles, but they are seldom magnified to the extent seen here, which indicates that the effects of fabric geometry are even more important with quartz textiles.

VI. MECHANISM OF FAILURE

The poor translations shown in the previous table indicate that in addition to the fact that the magnesium acetate finish is not sufficiently effective at elevated temperatures, its room temperature performancealso leaves much to be desired. On the other hand, one might speculate that, the optimum translational efficiency of quartz materials is limited by the inherent properties of the fiber. It therefore appeared desirable to investigate the mechanism of failure.

Investigators generally agree that quartz and glass fibers are extremely brittle and sensitive to scratches caused by abrasion. The abrasion occurs not only when foreign objects are rubbed against the fiber, but when the fibers are touching each other as well. The development of a protective finish could and should certainly minimize the damage. This is substantiated by the improved abrasion resistance with the application of the magnesium acetate.

Another cause for the premature failure of the quartz fibers is due to crushing. The brittleness of the material dictates that the area of contact between fibers is extremely small. There is no opportunity for the fibers to deform or flow, thereby relieving the stress. Any normal (transverse) force applied to the fibers produces very high localized stress concentrations which can very easily exceed the crushing strength of the fibers. Transverse forces can develop in a yarn even in straight tension due to the helical configuration resulting from yarn twist. The presence of these forces in woven fabrics is obvious wherever the yarns cross over each other.

According to classical mechanics, the maximum crushing stresses of quartz fibers having a diameter of .0004 and a modulus of 11 x 10^6 p.s.i. under various load applications can be derived as follows:

1. Circular fiber against a flat plate:

$$Sc = 95{,}000 \sqrt{P}$$

where Sc is the crushing stress in p.s.i. and P is the external pressure in pounds per linear inch along the fiber axis.

2. Two parallel circular fibers pressed against each other:

$$Sc = 133{,}000 \sqrt{P}$$

where Sc is the crushing stress in p.s.i. and P is the external pressure in pounds per linear inch along the fiber axis.

3. Two fibers perpendicular to each other:

$$Sc = 4{,}430{,}000 \sqrt[3]{P}$$

where P = pounds per intersection.

Using a reported crushing stress of 283,000 p.s.i. the external forces which will cause failure are:

for case 1 P = 8.85 pounds/inch
for case 2 P = 4.54 pounds/inch
for case 3 P = 0.000256 pounds/intersection.

The crushing force for case 3 is low indeed. In the case of a woven fabric (say #2501-2) there are approximately 50 x 50 yarn crossovers. Assuming that the yarn twist is low enough to allow as many as ten filaments to be in direct contact with the perpendicular set of crossing yarns, the crushing force is then:

$$F = 50 \times 50 \times 10 \times 0.000256 = 6.4 \text{ p.s.i.}$$

The actual situation is probably not quite so severe, in that fibers in yarns seldom in fact cross each other precisely at 90° and any reduction in angle will significantly lower this stress concentration. Also there is evidence that the crushing strength of the small areas involved at the intersection may be greater than the strength of samples normally used for the determination of crushing strength.

In addition to crushing per se (i.e., pure normal force), the fibers within a fabric are subjected to a combination of bending and tensile forces. This is because the yarns of a tensile test specimen are actually being bent over the cross yarns as well as being extended. Since the rupture elongation of quartz fibers is approximately 3%, failure will occur if the combined strains (due to bending and tensioning) reach this level regardless of whether or not the breaking strength in pure tension has been exceeded. It can be shown that this premature failure will take place when the applied tension, T, is exerted on the specimen:

$$T = \text{Original Tensile Strength} \left(1 - \frac{\text{Bending Strain}}{\text{Total Strain}}\right)$$

Assuming the bending strain to be a ratio of the fiber to the idealized circular yarn diameter (Dn). T may be expressed as

$$T = 8.56 - \frac{0.112}{Dn} \text{ grams for the fibers in question.}$$

Carrying this to the extreme, if the quartz fibers are bent over a yarn with a diameter of less than 0.013 inches, the fibers should fail without any external tensile force. Experimental results, however, have shown that some residual strength still remains in the fiber when it is bent around a rod of 0.003 inches in diameter. The explanation probably lies in the fact that the strain is not confined solely to the bend, and the actual radius of curvature is much larger.

Figure 2 shows the calculated force against a rod or cross yarn over which the fiber bends necessary to cause failure of the fiber vs. the diameter of the rod or cross yarn.

Curve A is calculated for failure by crushing.

Curve B is calculated for failure by combined bending and external tensile forces.

The actual experimental points for testing fibers over the indicated rod diameters are also shown.

It is to be noticed that the experimental points fall between the two calculated curves.

The foregoing rudimentary analyses on two possible mechanisms of quartz fiber failure are obviously not all inclusive. It does show, however, that two alternatives are available to improve the performance of quartz textiles. First a decrease in modulus, i.e., a reduction in brittleness, and a reduction of diameter to give increased fiber flexibility would produce a tougher, less mechanically fragile fiber. Second, work should continue to develop protective coatings so that not only is the fiber surface completely protected from scratching, but the coating material will increase the radius of curvature through which the fiber bends and absorb the localized bending and crushing strains.

FUTURE WORK

At the current writing, work is still under way on the study of finishes. While the alumina - 8-hydroxy quinoline finishes appear promising, it is believed that further effort should be expended before carrying out another mill trial. Quartz fiber at $32. per pound is expensive. Because of the fiber's mechanical sensitivity, experimental yarn and fabric manufacturing costs are high. It is proper that the potentially best finish is developed before proceeding with fabric geometry studies. Whether this finish will be the alumina - 8-hydroxy quinoline or another finish remains to be seen. That finish ultimately selected will be applied, a rational series of fabric prepared, and again evaluated at room and elevated temperatures.

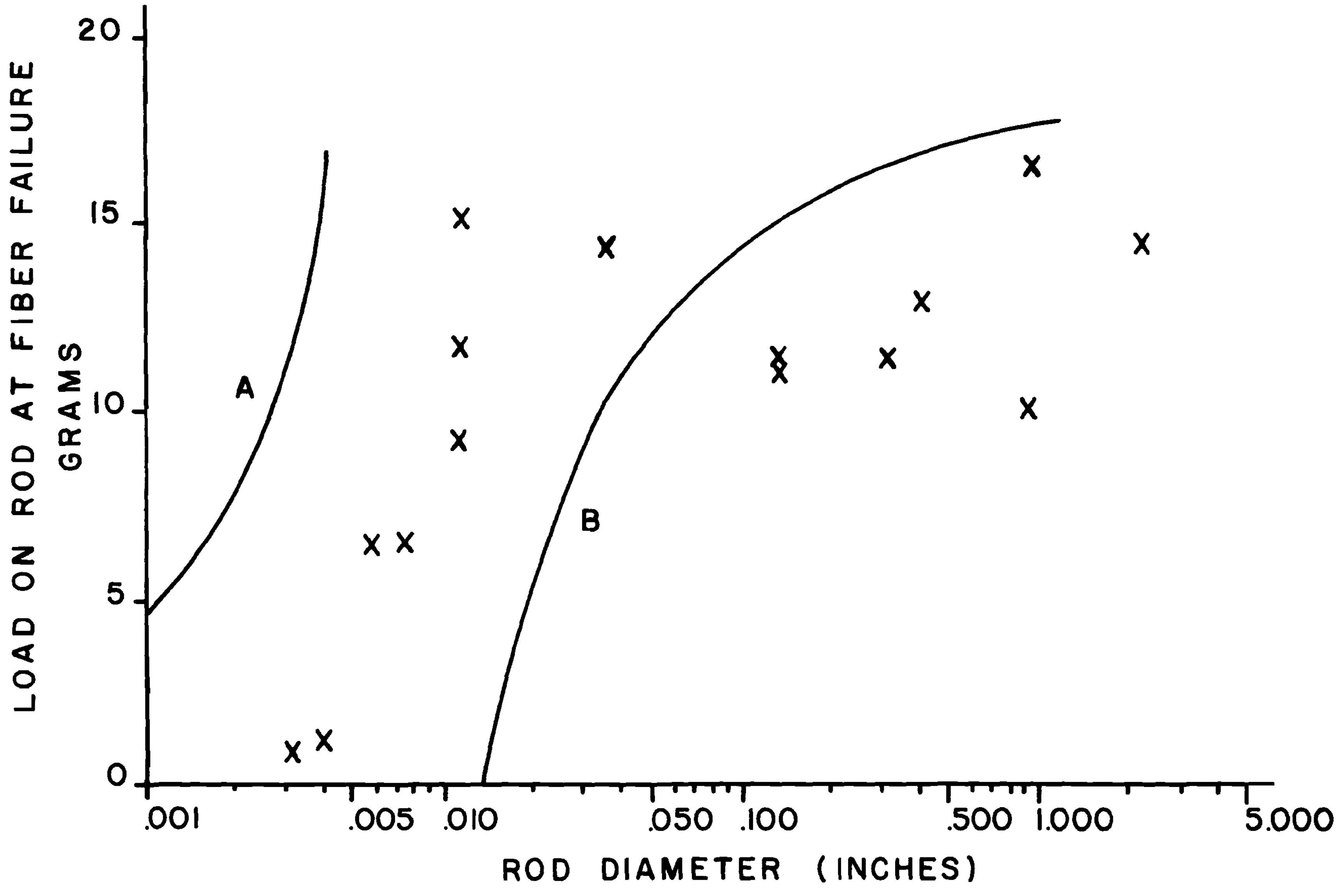

FIGURE 2 LOAD ON ROD AT FIBER FAILURE VS. ROD DIAMETER