INVESTIGATION OF ELECTRONIC CERAMIC FIBERS FOR NON-DESTRUCTIVE EVALUATION OF ADVANCED COMPOSITES (U)

by

E. C. Henry

FINAL REPORT

COVERING THE PERIOD OF 16 FEBRUARY 1971 TO 15 FEBRUARY 1972

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15 FEBRUARY 1972

PREPARED FOR THE

NAVAL AIR SYSTEMS COMMAND, WASHINGTON, D.C.

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ABSTRACT

The objective of this program is to investigate the feasibility of developing and using crystal-bearing filaments as sensors in the non-destructive testing of fiber-reinforced plastics and related composites. The fibers or filaments contain crystalline oxidic materials commonly referred to as electronic ceramics - these compositions include several soft and hard magnetic ferrites, and ferroelectric potassium sodium niobate and lithium niobate.

Work for this period included the synthesis of these compositions by high temperature solid-phase reaction. These were then dissolved in different glasses and in various concentrations, by melting. The resulting glassy solutions were shaped into test disks for devitrification studies. Significant quantities of filament were drawn and spooled, and a technique was developed for devitrifying them in 300-foot batches of 6-inch long fibers. From 3000 to 5300 feet of continuous filament was drawn of three compositions, while from 200 to 500 feet was drawn of four other compositions.

A study was made of the devitrification behavior of the key compositions, to show the effects of time, temperature, kind of glass and concentration of electronic ceramic on the exsolution of the active phase. The primary phases were identified by x-ray diffraction techniques. The major index of ferroelectric material crystallized was the dielectric constant of the resulting sample.

The chief compositions investigated were potassium sodium niobate dissolved in potassium sodium silicate and in a recrystallizable insulator glass; and a magnesium, manganese, zinc spinel-type ferrite dissolved in the recrystallizable glass. Problems were encountered in the poling of the ferroelectric samples and in obtaining magnetostrictive response in the ferrite samples, but further work is planned, with different approaches, in both these areas.

The devitrified filaments were incorporated into composite test tile, with an epoxy-novalac matrix. Data are presented for the initial tests of these composites; further evaluation of these samples and the preparation of improved composites are under way.

I. INTRODUCTION AND SUMMARY

This report describes the activities during the period 16 February 1971 to 15 February 1972, under Contract N00019-71-C-0247, which is monitored by Mr. Maxwell Stander, Naval Air Systems Command.

The purposes of this program are to develop and investigate the feasibility of using crystal-bearing (crystal-containing) filaments as sensors in the non-destructive evaluation of fiber-reinforced plastic structural materials. Potential applications of the sensors include determination of induced stress, determination of vibration, detection of filament breakage, observation of debonding effects between the reinforcing fibers and the matrix, or the monitoring of changes in the characteristics of the plastic matrix.

The technical approach is based on the fact that one can prepare homogeneous melts consisting of various electronic ceramic (oxidic) compositions dissolved in appropriate and compatible glasses. Fibers and continuous filaments prepared from such molten solutions are clear and amorphous. When the fibers subsequently are given a specific heat treatment, a crystalline phase is developed within the fibers.

Electronic ceramic compositions prepared in filament form at the Space Sciences Laboratory include those capable of having magnetic, ferroelectric or high-dielectric-constant characteristics. Materials investigated include compositions equivalent to lithium ferrite in a lithium silicate glass; a manganese, magnesium, zinc ferrite in a commercial recrystallizable glass; potassium sodium niobate in potassium sodium silicate; lithium niobate in lithium silicate; and barium strontium titanate in barium strontium aluminum silicate glass. The freshly-drawn filaments of these compositions are essentially inert. They are non-magnetic, have no ferroelectric characteristics and/or have low dielectric constant. X-ray diffraction patterns indicate no crystalline phase present. After the partial devitrification resulting from the heat treatment, the compositions may become magnetic, give a piezoelectric response to an electrical signal or a mechanical stress and/or show an increase in dielectric constant. X-ray diffraction patterns now indicate the presence of identifiable crystalline material.

In essence, one produces a fiber or a continuous filament containing crystallites of a precipitated electronic ceramic phase suspended in a glassy (amorphous) matrix. If desired, one may generally use a crystallizable glass for this matrix and in a subsequent heat treatment, to a somewhat higher temperature, devitrify the matrix also. This combination produces still a different material, that is, one consisting of an electronic ceramic phase dispersed in a polycrystalline ceramic matrix.

For convenience in measuring electronic properties, glass test disks were prepared of a number of these solution compositions, representing each of the types of electronic performance: magnetic, ferroelectric, or dielectric.

Significant lengths of filament were drawn from several of these same compositions. More than 3000 feet of filament were drawn of each of three compositions (FF-30, FF-32 and 19R30) while from 220 to 500 feet was drawn of others (including FF-61, FF-62, FF-63 and 19R40). Disks and fibers were devitrified on various heat treatment schedules for the purpose of developing their electronic properties, and work is continuing on the optimization of the more promising compositions. X-ray diffraction patterns were used in following the progress of devitrification and in identifying the crystalline phases produced. Two new compositions of potassium sodium niobate and glass were investigated. Both have a new high content of PSN in solution (65 weight per cent), one in $KNaO.1 \cdot 5SiO_2$ glass and one is General Electric Company's Re-X glass, and both are yielding improved values of dielectric constant after being devitrified.

Technical problems include (1) precipitation of the maximum amount of electronic ceramic phase in the fibers, (2) optimization of procedures for determining the electronic properties of the processed fibers, (3) selection of specific means of exploiting these properties and (4) development of experience in interpreting signals obtained from the sensor fibers.

The preparation of composites of devitrified fibers in an epoxy-novalac matrix was started. Tests units are approximately 2 inches by 1/4 inch by 1/8 inch for measurements of piezoelectric and magnetostrictive response and 2 inches by 1 inch by 1/16 to 1/8 inch thick for determination of dielectric constant.

II. DEVITRIFICATION STUDIES

Most electronic ceramic compositions have a very strong tendency to crystallize suddenly while being cooled from the melt. It is nearly impossible, for example, to prepare various titanates and niobates in an amorphous form. In many instances the only practical expedient for preparing fibers and filaments is to introduce a limited amount of glass-forming ingredients with a view to producing a homogeneous, viscous melt. For ease in drawing filaments and because of the potentialities for control of crystallite size during devitrification, the ability to dissolve the electronic ceramic completely in the glass phase is very attractive. It is important, therefore to study the effects of the time-temperature schedules during the devitrification step, on the amount of electronic ceramic developed and the properties of the resulting materials.

A. Ferroelectric Compositions

Studies have been made of the melting behavior, fiberization characteristics and devitrification relationships of a series of compositions of potassium sodium niobate, PSN, $K_{0.5}Na_{0.5}Nb0_3$, in one of several potassium sodium silicate glasses. The range of compositions investigated include 60, 40 and 20 weight per cent (w/o) niobate and 40, 60 and 80 w/o glass. The glasses included $KNa0 \cdot 3Si0_2$, $KNa0 \cdot 2Si0_2$, $KNa0 \cdot 1.5Si0_2$ and $KNa0 \cdot Si0_2$. All combinations of these were not tested, but on balance it appeared that the most promising were compositions FF-30, 60 w/o PSN and 40 2/o $KNa0 \cdot 3Si0_2$ and FF-32, 40 w/o PSN and 60 w/o, $KNa0 \cdot 3Si0_2$. Finally, because of the higher PSN content, work was concentrated on the FF-30 composition.

(a) Devitrification of FF-30 Composition

A number of samples of composition FF-30 were melted, poured into disk molds, annealed, and devitrified. Roughly 3000 feet of filament of this composition were pulled (one continuous length of 1500 to 2000 feet and another of approximately 1200 feet) having a diameter of about 1 to 3 mils. Devitrification studies were made of disks first, in order to determine a temperature for the devitrification of the filament. At too low a temperature (such as 600°) the amount of PSN precipitated was too small. At too high a temperature (such as 750°) the disks softened and tended to deform extensively.

It was found that the test disks had improved appearance if poured from glass that previously had been fritted twice. (Fritting is the process whereby mixed heterogeneous raw materials are melted together for the purpose of creating a homogeneous, reacted material. The melts usually are cooled rapidly from the molten state, commonly by pouring them into water. Materials that have been fritted twice are considered to be more homogeneous than those fritted only once). These glass disks were exceptionally clear, having almost optical glass quality. Significant differences in electrical properties were not found, however, between devitrified samples of material that had been fritted twice and disks made from material that had been fritted once. Several examples of the similarities in dielectric constant between materials fritted once and those fritted twice are seen in the series shown in TABLE I. Disks C-2 and C-6 were made of glass fritted once; disks C-3 and C-7 and C-8 were poured from glass fritted twice.

The practice of fritting twice was continued because it appeared to result in a better quality of drawn filament, although it was felt that it could have been discontinued in the preparation of test disks.

A procedure was adopted whereby the glass disk samples were carefully annealed immediately after being poured in a graphite mold. Gold electrodes were painted on, dried, and fired to 400°C. Liquid bright gold was used for these electrodes, from Hanovia Liquid Gold Division of Englehard Minerals and Chemicals Corporation, East Newark, New Jersey. The material, apparently an organometallic solution of gold, is painted like a varnish. Between 300° and 400°C the material is converted to a film of metallic gold.

The capacitance of each electroded glass disk was measured in the "as annealed" condition, the thickness of the sample and the electrode areas were determined, and the dielectric constant was calculated. Disks were then devitrified at various temperatures, and the capacitance was measured again, to help establish relationships between devitrification

TABLE 1

DIELECTRIC CONSTANT VS. DEVITRIFICATION TREATMENT

	Dielectric Constant Values					Avg.
1. Glass $KNaO\cdot 3SiO_2$: Disk No.	C1					10
Annealed only	10					
2. Composition FF-30: Disk No.	C-2	C-3	C-6	C-7	C-8	
Annealed only	18.0	16.6	15.3	15.7	13.6	15.8
Devitrified 600°/16 hrs.	-	-	-	-	-	-
Devitrified 650°/16 hrs.	23.9					23.9
Devitrified 675°/16 hrs.		23.3				23.3
Devitrified 650°/64 hrs.			26.8	20.6		23.7
Devitrified 650°/64 hrs.					22.8	22.8

temperature and dielectric constant. The gold electrodes painted and fired on the annealed glass disks were not adversely affected by devitrification temperatures up to 725° at least, with the result that it was not necessary to replace electrodes before measuring the capacitance of the devitrified disks.

In earlier measurements, disks of FF-30 fired to 700° and 725° deformed extensively, and it was felt that 675° was a top temperature for this composition. Toward the end of the report period, however, it was found that embedding the sample in powdered zirconia during devitrification gave promise of permitting subjecting the disks successfully to higher temperatures. Additional disks of FF-30 thereupon were poured, annealed, and electroded, and are being devitrified to at least 700 and 725°C. The embedding of these materials in zirconia powder is discussed further in Section IIb and the progressive increases in dielectric constant with increased devitrification temperatures, within limits, are discussed in connection with TABLE III, Section IIC.

Although the dielectric constant of about 24 for disks of FF-30 devitrified at 650° for 40 hours was considered quite low, a significant quantity (some 600 feet) of the 3000 feet of filament of this composition originally spooled was so devitrified (650° for 40 hours) and incorporated into composite test pieces.

(b) Devitrification of FF-32 Material

Approximately 3000 feet of filament of composition FF-32 was drawn and spooled. The material, having the composition 40 w/o potassium sodium niobate, $K_{0.5}Na_{0.5}Nb0_3$ and 60 w/o potassium sodium silicate, $KNa0 \cdot 3Si0_2$, fiberized readily, probably because of the high content of glass. Fibers of this composition were devitrified at 650, 720 and 750°, being held at these temperatures for 16 hours. To the unaided eye, the 720° samples do not seem to be extensively devitrified.

Two clear thin disks of FF-32 composition fritted twice and only annealed, and a clear disk of once-fritted material were equipped with gold electrodes. Devitrification at 600, 650 and 675° yielded no increase in dielectric constant. Other disks of this composition deformed when devitrified at 700 and 750°, but these heat treatments were not made with disks embedded in powdered zirconia. Nevertheless, inasmuch as FF-32 contains only 2/3 as much PSN as FF-30, further work is not planned with FF-32 at present. In the following brief table, disks C-4 and C-9 were of material fritted twice; C-5 was poured of once-melted frit. Dielectric constant is tabulated against devitrification temperatures that are too low to be effective.

Composition FF-32: Disk No.	C-4	C-5	C-9	Dielectric Constant
Annealed only	16.2		13.4	14.6
Devitrified 600°/16 hrs.		13.3		13.3
Devitrified 650°/16 hrs.		13.7		13.7
Devitrified 675°/16 hrs.	11.7			11.7
Devitrified 650°/40 hrs.		13.1		13.1

(c) Studies in the system $LiNbO_3$ - $Li_2O \cdot 2SiO_2$ and $LiNbO_3$ - $Li_2O \cdot 3SiO_2$

Four compositions in the system received attention:

FF-60	Lithium niobate ($LiNbO_3$) Lithium silicate glass ($Li_2O \cdot 3SiO_2$)	40 w/o 60 w/o
FF-61	Lithium niobate ($LiNbO_3$) Lithium silicate glass ($Li_2O \cdot 2SiO_2$)	40 w/o 60 w/o
FF-62	Lithium niobate ($LiNbO_3$) Lithium silicate glass ($Li_2O \cdot 2SiO_2$)	30 w/o 70 w/o
FF-63	Lithium niobate ($LiNbO_3$) Lithium silicate glass ($Li_2O.3SiO_2$)	30 w/o 70 w/o

Of these, composition FF-61 proved to be of greatest interest as a potential source of high-dielectric constant filaments. The dielectric constant of poured and annealed disks appeared to be raised regularly as the devitrification temperature was increased, but studies were not carried beyond 700°, where the disks tended to crack. When work is resumed with FF-61, the disks should be embedded in zirconia powder while being devitrified, as discussed in Sections II Aa, IIB, and IIC. This should permit the studies to be extended to higher devitrification temperatures.

X-ray patterns showed that lithium niobate was the primary phase precipitated during devitrification. Fibers of FF-61 devitrified at 650° for 16 hours (film 2574) and at 700° for 18 hours (film 2575) showed well - crystallized $LiNbO_3$. The 700° material had greater intensity of pattern, some evidence of preferred orientation of crystallites, and a small amount of weakly crystallized second phase. The latter might have represented the start of the crystallization of the $Li_2O \cdot 2SiO_2$ glass. However, because of the interest in developing a piezoelectric filament (one exhibiting electromechanical transducer characteristics) and because of the known high Curie temperature of lithium niobate, which would make polarization difficult, it was decided to place more emphasis on the preparation of fibers containing ferroelectric PSN (such as composition FF-30 and related compositions).

B. DEVITRIFICATION OF LARGER AMOUNTS OF FIBER

After several furnace runs to devitrify sample lengths of various filament compositions for study, an improved technique was adopted that permits the devitrification of larger quantities of filament at a time, as needed for incorporation into test composites. Initially, some 40 fibers, five to seven inches long, representing 16 to 20 feet of filament, were devitrified in each of several 7 1/2 x 2 x 1 inch alumina boats. It was found necessary to embed the fibers in powdered zirconia to prevent their sticking to each other during the heat treatment.

Subsequently, refractory slabs 8 x 4 1/2 x 3/8 - 1/2 inches in size were being used. Alternate layers of zirconia powder and 6-inch lengths of fiber placed side by side were built up to the desired thickness of load. When three such slabs were placed in a small furnace, it was possible to devitrify about 600 fibers, equivalent to 300 feet of filament, in each run.

Roughly 620 feet of "ferroelectric" composition FF-30 and of the "magnetic" composition 19R30 were so devitrified (40 hours at 650°C). These fibers were then used in the preparation of composite test tile.

C. NEW FERROELECTRIC COMPOSITIONS PREPARED

Measurements made in late October indicated that the electrical resistivity of composition FF-30, devitrified at 650° (disk C-6, TABLE I) dropped very rapidly with increase in temperature. More complete data are given in TABLE II, but in essence the values change as follows:

TEMPERATURE	RESISTANCE
24°C	50,000 megohms
110°C	1,500 megohms
180°C	12 megohms
232°C	3 megohms

It was anticipated that replacing the $KNaO \cdot 3SiO_2$ glass of composition FF-30 with Re-X glass, a General Electric Company recrystallizable glass used in the production of some electrical insulator bushings, might improve the temperature-resistivity relations. Accordingly, composition FF-43 was made up, consisting of 65 w/o potassium sodium niobate and 35 w/o Re-X cullet. The Re-X seems to dissolve the additional PSN readily, and it is known, from previous experience, to have good fiberizing characteristics.

TABLE II

CAPACITANCE, DISSIPATION FACTOR AND RESISTANCE OF DISK C-6 VS. TEMP.

Temperature °C	Capacitance (pfd)	d.f.%	Resistance, Megohms
24	57.6	.48	50,000
43	58.1	.54	50,000
66	59.1	1.0	30,000
81	59.8	2.0	11,000
110	63.2	4.9	1,500
132	68.0	9.7	300
150	75.8	19.7	82
173	140	44.6	22
180	112	42.7	16
186	122	46.8	12
191	130	50.0	11
197	137	53.0	9.5
203	251	52.0	6.1
205	165	62.0	6.5
208	189	65.0	5.0
210	200	69.0	4.5
213	217	75.0	4.0
232	250	82.0	3.0

At the same time, as a back-up material in the effort to prepare solutions containing a larger quantity of PSN, composition FF-42 was made. This composition consists of 65 w/o PSN and 35 w/o $KNaO \cdot 1.5SiO_2$ glass. This composition also appeared to dissolve the full amount of PSN introduced.

In order to simulate the heat treatment received by filaments while being devitrified, and to prevent distortion at top temperatures, disks of both FF-42 and FF-43 were poured, annealed, equipped with gold electrodes, then devitrified at various temperatures while embedded in zirconia powder. Higher dielectric constant values were realized than with FF-30 at devitrification temperatures of 600° to 675°. Disks of FF-42 and FF-43 devitrified in this manner did not show evidence of overfiring even at 725°. The results are compared in TABLE III. Samples on hand currently are being devitrified at still higher temperatures, with a view to determining the point of maximum yield. Likewise, samples of FF-30 are being devitrified in zirconia at 700° and above to provide the data for these temperatures, missing but currently under study as shown in TABLE III.

Arrangements are presently being made to have measurements made on compositions FF-42 and FF-43 leading to tabulations of data similar to those furnished for FF-30 in TABLE II. When these studies are completed, they will help determine further the relative merits of FF-42 and FF-43.

D. DEVITRIFICATION OF MAGNETIC SAMPLES

Two disks were poured and annealed of composition 19R30 (30 weight per cent ferrite No. 19 and 70 weight per cent Re-X glass). The dielectric constant values of the annealed disks were 10.8 and 11.1. Devitrification at 600° for 40 hours produced a dielectric constant of 16.9. Forty hours at 650° likewise led to a dielectric constant of 14.6. Inasmuch as the dielectric constant of the ferrites does not run as high as that of the usual ferroelectric materials, the precipitation of a large amount of ferrite in these disks would not cause a large increase over the dielectric constant of the original glass-ferrite solution. Consequently, these values are not out of line. Both disks were non-magnetic as poured and annealed, and magnetic after being devitrified.

Some 5300 feet of continuous 1-mil filament was drawn of composition 19R30. Approximately 600 feet of this material (two 300-foot quantities) were devitrified by heating for 40 hours at 650°C. The fibers were used in the preparation of composite test pieces. This was enough fiber for four test tile, two with the fibers embedded transversely, two with the fibers arranged longitudinally.

A short length of filament of composition 19R40, about 200 feet, was pulled from the platinum bushing. At the top melting temperature a uniform, apparently homogeneous liquid was formed of this composition. However, on reducing the temperature so that the melt would have sufficient viscosity and surface tension for drawing filament, numerous crystallite of ferrite nucleated and grew, and, remaining in suspension, caused the filament to be rough and lumpy. The filament was of such poor quality that the drawing operation was discontinued for this composition.

III. POLING PROCEDURES

In order to determine the electromechanical, or transducer, properties of a ferroelectric material, it is necessary first to apply an electrical field and align the ferroelectric dipoles in some common direction. These dipoles establish themselves spontaneously as the material is cooled through the ferroelectric Curie temperature (they do not exist above this temperature), although they are not necessarily aligned in a preferred direction. The alignment of the dipoles is referred to as poling, and is commonly accomplished by applying a field in the desired direction while the material is being cooled through the Curie point. If this is not feasible, for example if the Curie temperature is too high, one poles at as high a temperature as is practical. The coercive force, the field required to switch the dipoles, tends to become lower as one approaches the Curie temperature.

Barium titanate ceramic transducers frequently are poled by applying a field of approximately 30 volts per mil of thickness of the ceramic while the material is allowed to cool from 130°C (which is above the Curie point of 120°) to about

$80^{\circ}C$. Potassium sodium niobate, PSN, in comparison, has a phase change from one ferroelectric form (tetragonal) to another (orthorhombic) in the neighborhood of $200^{\circ}C$. It is not inconvenient to apply a field while cooling PSN through this temperature range. The field required for ceramic PSN is slightly higher than that for barium titanate and is of the order of 50 volts per mil.

The question arose as to whether the field needed to pole glass-bonded PSN, as in FF-30 and FF-32, may be considerably higher than had been used in this work to date. There had been a tendency to keep the field intensity or voltage gradient within the limits of the state-of-the-art for polycrystalline electronic ceramics. It was therefore considered significant that a recent study showed that the dielectric strength (resistance to dielectric breakdown) of a glass-bonded electronic ceramic can be several times higher than that of the simple polycrystalline composition. Burn (Reference 1) reported that 60 KV/cm., or about 152 volts per mil, is considered the limit of dielectric strength of sintered pressed disks of lead zirconate, $PbZr0_3$. However, he found it possible to impose 400 KV/cm (more than 1000 volts per mil) when 10 volume per cent of a lead, bismuth, borosilicate glass was used to bond the material. As a consequence he was able to impress a field high enough to convert the lead zirconate from an antiferroelectric to a ferroelectric structure. In the antiferroelectric condition the dipoles are arranged in 180° opposition to each other;

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1) "Field-Enforced Ferroelectricity in Glass-Bonded Lead Zirconate", by Ian Burn, American Ceramic Society Bulletin 50(5) 501-505 (1971).

in the ferroelectric state they are aligned in a common direction. Belding and McLaren (2) also have alluded to the behavior of piezoelectric ceramics under high fields.

In light of this information, it was planned to employ higher voltage gradients than heretofore, when attempting to pole the thin disks of devitrified FF-30 or FF-32 in preparation for measuring the radial electromechanical coupling coefficient.

However, when it was attempted to pole disks of devitrified FF-30 difficulties were encountered and no piezoelectric response was obtained. Measurements of dielectric constant vs. temperature had shown a peak at 203°C, indicating a Curie point just about where it would be expected. When the temperature was raised toward this point, it was found that the samples did not respond properly.

The major difficulty in the poling of FF-30 is believed to lie in the fact that the continuous glass matrix of this composition, essentially $KNaO \cdot 3SiO_2$, has excessive electrical conductivity at the elevated temperatures where poling usually is carried out. As a consequence, it was not possible to establish and maintain an electrical field, at such temperatures, high enough to cause the necesary realignment of the ferroelectric dipoles of the PSN. The capacitance in pfd, the per cent dissipation factor and the insulation resistance of disk C-6 are presented as functions of temperature in TABLE II.

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(2) "Behavior of Modified Lead Zirconate - Lead Titanate Piezoelectric Ceramics Under High Electric Fields", by J. H. Belding and M. G. McLaren, ibid., 49 (12) 1025-1029 (1970).

It was for these reasons, as pointed out in Section IIC above, that it was decided to investigate compositions FF-42 and FF-43. Poling experiments will be carried out with these latter compositions as soon as data are obtained for them similar to the information on FF-30 provided in TABLE II. Additional procedures will be investigated, such as longer-time poling at lower temperatures, where the resistance of the material is higher and larger fields can be maintained. Systematic reversal of field polarity has been found effective in many instances.

IV. STABILITY OF $KNaO \cdot 3SiO_2$ GLASS

In earlier work (and as reported on pages 12 and 13 of the final report on Contract No. N00019-70C-0416) drops of $Li_2O \cdot 2SiO_2$ and $Li_2O \cdot 3SiO_2$ were heated for 16 hours at 600° and at 700°C. The disilicate glass was significantly devitrified at 600° and more extensively at 700° (x-ray films 2262 and 2272.). The crystalline phase obtained from these heat treatments was identified as $Li_2O \cdot 2SiO_2$, lithium disilicate.

Lithium disilicate was the primary crystal phase precipitated also from the trisilicate glass, according to the relationship

$$Li_2O \cdot 3SiO_2 = Li_2O \cdot 2SiO_2 + SiO_2$$

The excess silica, in turn, crystallized in the form of the mineral cristobalite (films 2261 and 2273).

However, when similar studies later were made with $KNaO \cdot 3SiO_2$ glass, the glassy constituent of FF-30 and FF-32, different results were obtained. Samples of the glass were heated to 550, 600 and 700°C. The sample heated to 700^{o}C was quite clear. It had melted and pulled up into a rounded mass. This temperature apparently is well above any likely short-time devitrification temperature for this glass. The sample heated to 600^{o} also softened considerably and showed well rounded edges. Likewise, the sample heated to 550^{o} showed some rounding of sharp edges but was not otherwise deformed. No evidence of crystallization were found when the 550^{o} and 600^{o} materials were examined with the petrographic microscope.

It is seen, therefore, that $KNaO \cdot 3SiO_2$ as a glass is decidedly more stable, being significantly more resistant to devitrification than either the $Li_2O \cdot 2SiO_2$ or the $Li_2O \cdot 3SiO_2$ glass. The resistance to deformation of the $KNaO \cdot 3SiO_2$ glass at a given temperature in the range 550 to 750^{o} was found to be increased by the presence of 30 w/o PSN (in FF-32) and even more so by the addition of 40 w/o (as in FF-30). These facts are considered favorable if the preparation of fibers of PSN in a glassy, rather than a crystalline, matrix is important.

The dielectric constant of the $KNaO \cdot 3SiO_2$ glass alone is 10. If composition FF-30 is devitrified in the temperature ranges shown in Table I the dielectric constant rises. X-ray diffraction patterns here show only the presence of well-crystallized PSN. It is apparent, however, that the amount of PSN recovered is not as high as would be desired. Hence the plan to devitrify FF-30 at higher temperatures or for longer times, as indicated in Table III.

TABLE III

DIELECTRIC CONSTANT VS. DEVITRIFICATION TREATMENT

Devitrification			Composition		
Temp.	Time, Hrs.		FF-30	FF-42	FF-43
Annealed only (average)			15.8	20.0	18.5
500	16			13.0	19.6
600	16			99	26
650	16		23.9	148	85
			23.7	126	
650	40	C-6	26.8	109	
650	64		22.8		
675	16		23.3	109	93
700	16		C	112	108
725	16		C	109	114
750	16		C	C	C
775	16		C	C	C
800	8		96.5		

C = Currently under Study

In contrast to $KNaO \cdot 3SiO_2$ glass is the General Electric Re-X glass. It is designed as a recrystallizable glass, but the crystallization of the glass does not commence until a temperature higher than that at which the precipitation of PSN from composition FF-43, for example, is already under way. For example, (film 2730) an x-ray diffraction pattern of FF-43 annealed at 400° and devitrified at 650° for 40 hours shows well-crystallized PSN and only a broad line characteristic of the glasses (as such) used in this investigation.

V. INVESTIGATION OF MAGNETOSTRICTIVE FERRITES

One of the proposed applications for implanted electronic ceramic fibers makes use of the phenomenon of magnetostriction. In the studies last year six ferrites were investigated, numbers 12, 15, 16, 17, 18 and 19. Their compositional characteristics were listed in the 12 February 1971 report of Contract N00019-70-C-0416.

Two of these were selected for further investigation: No. 18, a simple cobalt ferrite, $Co_{0.8}Fe_{2.2}O_3$ reported by Bozorth, Tilden and Williams (3) to be magnetostrictive, and No. 19, a typical magnesium, manganese, zinc spinel type experimental "square loop" ferrite composition. The latter had been made up as a solution in three glasses: (a) $Li_2O \cdot 2SiO_2$ glass, (b) lead bisilicate, "Ceraflux" and (c) Re-X. The maximum ferrite content for successful fiberization and devitrification was 30 w/o. As reported in Section IID, 5300 feet of continuous filament of composition 19R30 (30 w/o of ferrite 19 in 70 w/o Re-X glass) was drawn, and some 600 feet of the filament was cut into 6-inch lengths and devitrified for 40 hours at 650°C. The devitrified fibers were used in the preparation of composite test tile (2-inch by 1-inch panels).

A. New Ferrite Compositions

Three new ferrite compositions also were synthesized: Ferrite No. 20 is a composition described as strongly magnetostrictive in a patent by Kikuchi (4). The composition selected was

	50.00 mole % Fe_20_3	(80 gm)
	41.87 mole % Ni0	(31.2 gm)
	7.38 mole % Cu0	(5.9 gm)
and	0.75 mole % Co0	(0.6 gm)

Ferrite No. 21 is typical of magnetostrictive ferrites covered in a patent by Davis (5). It contains quantities of Fe_20_3, Ni0 and Co0 very similar to those in Kikucki's patent (82, 35 and 0.6 grams, respectively), but no Cu0. Ferrite No. 22 is a hard magnetic "magnetoplumbite" with the composition Ba0·6Fe_20_3.

The ferrite raw materials were weighed out, blended, and calcined at 950°C for 1 1/2 hours. They were then crushed and mixed with powdered Re-X glass cullet in the ratio 85 w/o ferrite and 15 w/o glass. The resulting compositions were designated 20R85, 21R85 and 22R85. These compositions

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3. "Anisotropy and Magnetostriction of Some Ferrites"; by R. M. Bozorth, Elizabeth F. Tilden and A. J. Williams, Phys. Rev. <u>99</u> (6) 1788-1798 (1955).
4. "Ferrite", by Y. Kikuchi, U.S. Patent 2,951,810 (Sept. 6, 1960).
5. "Magnetostrictive Ferrite", by C. M. Davis, Jr., U.S. Pat. 3,078,234 (Feb. 19, 1963).

were pressed in a steel die at about 2500 psi into disks 1 1/4 inches in diameter and 1/8 to 3/16 inch thick. The disks were fired to 980-1000°C for two hours and then allowed to cool. In this sintering operation the glass softened and served to bond the ferrite particles together to form a hard but somewhat porous ceramic structure.

Differences were noted in firing shrinkage and per cent water absorption from one fired composition to another:

Composition No.	% Firing Shrinkage	% Water Absorption
20R85	0.0	14.0
21R85	2.4	14.6
22R85	7.2	7.5

Small test bars, about 3/8 x 3/8 x 1 inch, were cut from the sintered disks and sent to the Electronics Laboratory of the General Electric Company for observation of magnetostrictive resonance. No resonance coupling effects were detected within the normal frequency range for this size test bar. It is very likely that perhaps these materials were too porous; the voids and the glass bonding phase may have served to dampen potential resonances. Two steps will now be taken: (1) additional disks will be sintered at a higher temperature (1050 to 1100°) with a view to developing a denser bonded structure, and (2) sample disks will be pressed of 100% ferrite and fired to appropriate temperatures within the range 1250°-1350° to form denser self-bonded ceramic structures. "Magnetostrictive" ferrite No. 18 should be included in this study, both as the 100% sintered material and as composition 18R85.

VI. STRENGTH AND MODULUS MEASUREMENTS OF FILAMENT SAMPLES

Sample lengths of filaments of several compositions were tested, to determine the general level of tensile strength and modulus of elasticity. The results are presented in Tables IV and V. In three instances (compositions 18R20, FF-61 and 19R30) comparisons are shown between as-drawn filaments and those that have been devitrified.

The strength of glass filaments depends, in part, on the speed with which they are pulled. Thus, since most of the fibers reported here were drawn by hand, some variations in diameter and strength are to be anticipated. Higher strength values should be expected from materials drawn at higher speeds. Of the batches reported here, only samples 5 and 6 were taken from the several thousand feet of each that had been spooled at approximately 80 feet per minute, and this is normally a low speed for the drawing of filaments.

VII. PREPARATION OF COMPOSITE TEST SAMPLES

The 600 feet of devitrified fibers of "ferroelectric" FF-30 (Section IIA and IIB) and like quantity of devitrified fibers of "magnetic" 19R30 (Section

TABLE IV

Tensile Strength of Selected Filament Compositions

Batch No.	Dia. in.	Strength psi	Batch No.	Dia. in.	Strength psi
1. Composition 18R20, As Pulled			2. Composition 18R20, Devitrified		
	.003	65,300		.004	9,500
	.003	71,000		.0025	7,400
	.0038	36,200		.0037	13,900
	.0035	73,000		.003	23,700
	.003	76,500		.003	13,700
	.0035	88,500		.0025	21,200
	.003	45,400	Avg.	0.0031	14,900
	.002	29,000			
Avg.	0.0031	60,600			
3. Composition FF-61 As Pulled			4. Composition FF-61 Devitrified		
	.009	33,800		.003	13,800
	.008	24,100		.005	10,200
	.009	46,200		.005	9,200
	.009	32,100		.002	124,000
	.010	93,100	Avg.	0.0038	39,300
	.010	44,600			
	.0085	124,300			
	0.0091	56,900			
5. Composition FF-32 As Pulled			6. Re-X Glass Filament As Pulled		
	.005	49,000		.003	47,000
	.004	60,300		.003	89,400
	.004	61,000		.003	79,400
	.006	65,900		.003	64,000
	.005	88,800		.003	105,000
	.004	66,700	Avg.	0.003	77,000
	.004	96,000			
Avg.	0.0046	69,700			

TABLE IV continued

Tensile Strength of Selected Filament Compositions

Batch No.	Dia. in.	Strength psi.	Batch No.	Dia. in.	Strength psi
7. Composition 19R30 As Pulled			8. Composition 19R30 Devitrified		
	.005	67,500		.008	15,300
	.004	52.500		.004	27,900
	.004	54,600		.006	1,770
	.005	32,600		.009	20,000
	.004	41,300		.003	2,130
	.005	50,600			
Avg.	0.0045	50,700	Avg.	0.006	13,400

TABLE V

Calculated Values of Elastic Moduli

Batch No.	$E_{avg.} \times 10^6$	Batch No.	$E_{avg.} \times 10^6$
1	8.4*	5	12.7**
2	5.2**	6	7.1**
3	9.1**	7	10.4*
4	9.9**	8	9.4**

* Corrected value based on 2.0 inch gage length

** Corrected value based on 1.0 inch gage length

(Section IID) were incorporated into test tile, approximately 2 inches by 1 inch by 1/16 inch, each mounted on an aluminum block of the same area, which served as an electrode. The fibers were encapsulated in a matrix of epoxy novalac modified with polypropylene glycol and with MNA curing agent. Ten test pieces were made in all: two consisting of matrix only, for reference; two of FF-30 fibers laid longitudinally, and two with FF-30 fibers embedded transversely to the long dimension of the tile; and two each of 19R30 fibers placed longitudinally and transversely. The capacitance values of the test pieces were measured, as was the dissipation factor. These data and the calculated dielectric constants are listed in Table VI.

It is planned next to subject these samples to electromagnetic signals of various frequencies for evidence of response. The FF-30 samples will be examined to see if they exhibit a ferroelectric hysteresis loop. The next ferroelectric or dielectric composite samples to be made will incorporate fibers having considerably higher dielectric constant than that of FF-30 devitrified at 650^{o} (Table III).

TABLE VI

DATA ON COMPOSITE TEST TILE

Sample	Capacitance pf.	Dielectric Constant	Dissipation Factor
Matrix,	14.5	3.4	0.011
Unfilled	14.5	3.4	0.011
19R30	22	3.5	0.058
Longitudinal	19	4.1	0.046
19R30	17	3.9	0.048
Transverse	18	3.9	0.066
FF-30	25	4.1	0.011
Longitudinal	24	4.2	0.011
FF-30	25	4.5	0.012
Transverse	25.5	4.5	0.013

VIII. MEETINGS AND CONTACTS

1. L. R. McCreight, R. N. Griffin and E. C. Henry met with Maxwell Stander and P. R. Stone at Naval Air Systems Command offices in Washington, D.C. on April 15, 1971.
2. A paper "Ceramic Fibers as Sensors in NDE of Composites" was presented by E. C. Henry at the 74th Annual Meeting of the American Society for Testing and Materials (ASTM), at Atlantic City, N.J., June 29, 1971.
3. E. C. Henry presented a progress report at the Program Review of Navy-sponsored work on composite materials conducted by the Technical Standing Committee on Composites (TSCC) of the Naval Advisory Council on Materials (NACM). The program review was held at the Naval Research Laboratory, Washington, D.C., November 2-5, 1971.
4. L. R. McCreight, D. R. Ulrich and E. C. Henry met with Maxwell Stander and P. R. Stone on December 22, 1971, at Naval Air Systems Command offices in Washington, D.C. for a general progress report.

5. Dr. S. W. Tehon and Stephen Wanuga, of the General Electric Company's Electronics Laboratory in Syracuse, N.Y., visited D. R. Ulrich and E. C. Henry at the Space Sciences Laboratory as consultants on January 28, 1972. Tehon and Wanuga are experts in the measurement and evaluation of electrostrictive and magnetostrictive materials, and discussed the selection and operation of various test instruments and combinations thereof for the determination of significant parameters of electronic ceramic materials.

IX PLANS FOR FURTHER ACTIVITIES

In review, the key experimental procedures currently being carried out or immediately pending include the preparation of still further test disks of the "ferroelectric" compositions of FF-42 and FF-43 and measurements of their dielectric constant before and after devitrification in extension of the promising higher dielectric constant values reported in Table III. Studies will be made of the polarization behavior of these materials, including investigation of the effects of temperature on their electrical properties. Based on the results of these tests, continuous filament will be drawn of FF-42 or FF-43, or both. The filament will be devitrified and incorporated into composite test shapes, and the composite material will be tested for physical and electrical properties. Work will be continued also on the preparation and evaluation of composite materials containing "magnetic" fibers. The new technique of embedding disks in powdered zirconia during devitrification will be extended to composition FF-30 with a view to enhancing its dielectric constant beyond the values shown in Table I.

The longer-range plans include continuation of efforts to precipitate the maximum amount of electronic ceramic phase in the fibers, as discussed immediately above. With ferroelectric or dielectric materials, the primary criterion is the development of high dielectric constant. Closely related to this, however, and recently receiving little attention, is the study of the control of crystallite size and crystal orientation with a view to further enhancing the properties and performance of the electronic phase that has been precipitated (devitrified). It is known that if the crystallite size is too small (below about one micron in barium titanate, for example) adequate ferroelectric domain areas cannot be formed and the piezoelectric effect is suppressed or absent.

Examinations should be made, therefore, with the petrographic microscope to obtain more data on the range of crystallite sizes of PSN, in particular, developed during the various devitrification heat treatments. In general and within limits, the higher the temperature or the longer the time at temperature, the greater is the crystal growth and the coarser is the resulting microstructure.

The development of effective poling procedures is essential to the preparation of efficient piezoelectric electromechanical transducers. Further work is planned in this area, and several techniques are to be investigated including the use of higher voltages at more moderate temperatures, where the resistance will be higher, and systematic reversal of the direction of applied field. In the same category is the problem of developing, detecting, and exploiting magnetostrictive characteristics in ferrites, for use in magnetic transducers.

On balance, the compositions presently under study (PSN and the several ferrites) appear to be the most promising of the materials that have been investigated in this program. Problems of the extraction and interpretation of signals from implanted sensor fibers remain, but they are believed to be less pressing at the moment than the development of more efficient sensors. Hence the larger effort will be placed first on the further optimization of materials on which a considerable background of information has now been obtained, and the further refinement of processing techniques and procedures which are proving to be increasingly effective.

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13. ABSTRACT

The objective was to investigate the feasibility of developing and using crystal-bearing filaments as sensors in the non-destructive evaluation of fiber-reinforced plastic structural materials. The fibers or filaments contain crystalline oxidic materials commonly referred to as electronic ceramics. Compositions investigated include several soft and hard magnetic ferrites and ferroelectric potassium sodium niobate and lithium niobate.

These electronic ceramic compositions were dissolved in molten glass, shaped, annealed, and cooled to room temperature. The test shapes (filaments and disks) were then heat-treated to precipitate (exsolve) the material that had been put in solution. Progress is reported in the preparation and processing of these materials. Significant quantities (from 3000 to 5000 feet) of approximately 1-mil filament were drawn and spooled of several of the key compositions. Devitrified filaments are being incorporated into test composites, with an epoxy-novalac matrix, and will be subjected to electrical and mechanical measurements.

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