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Study of Effects of Gravity on Crystallization

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Over the course of this research effort two primary tasks were accomplished. The result helped in confirming the theory that the absence of gravity aids in preventing for formation of crystallites in ZBLAN optical glass fiber. Secondly, an important step has been taken in being able to draw fiber from a ZBLAN preform on the Space Shuttle. Section 1 reports on the effort of confirming the effect gravity has on the formation of crystallites in ZBLAN. Section 2 deals with the development of a sting system to initiate a fiber draw by remote control during low gravity maneuvers aboard NASA's KC-135 aircraft.

Section I

Worked Performed on the Glass Anealing Furnace

Introduction

ZrF4-BaF2-LaF3-ALF3-NaF (ZBLAN) optical fiber was flown on board NASA's KC-135 aircraft to determine the effects of microgravity on crystal growth in this material. Fiber samples were placed in evacuated quartz ampoules and heated to the crystallization temperature in 0-g and on the ground in 1-g. The 1-g samples had many regions of crystallites, while the 0-g samples showed no evidence of crystallization.

Mid infrared fiber optics have many promising applications in both the industrial and military sectors. These include ultralong, repeaterless, transcontinental and transoceanic links, nuclear radiation resistant links, high capacity wavelength multiplexed fiber optic devices, remoting of infrared focal planes, infrared laser devices, infrared power delivery, long-length fiber optics sensor systems and nonlinear optical systems.[1] Heavy metal fluoride glasses have shown the most promise to date. The most stable heavy metal fluoride with respect to crystallization appears to be those in the ZrF4-BaF2-LaF3-AlF3-NaF family, commonly referred to as "ZBLAN" glasses.[2]

Intrinsic and extrinsic processes limit light propagation at low powers in ZBLAN.[3] Intrinsic properties include band gap absorption, Rayleigh scatter and multiphonon absorption. Extrinsic processes include impurities such as rare earth and transistion metal ions and crystallites formed during preform processing and fiber pulling. The theoretical loss coefficient for ZBLAN is 0.001 dB/km at 2 microns. Achieving this lower limit is hampered by both the intrinsic and extrinsic processes.

All of the intrinsic processes and extrinsic impurities can be controlled through processing of the initial raw materials and in preparation of the glass preform. The devitrification of ZBLAN is due to a narrow working range and low viscosity at the pulling temperature.[4] These two factors make this glass unstable and prone to crystallization.

Microgravity processing offers the potential to minimize these losses in ZBLAN glass.[5] Improved purity of raw materials and the possibility of containerless processing in microgravity in the future are expected to expand glass forming regions and minimize the formation of microcrystallites during synthesis of the glass.[5] Microgravity processing also offers the potential of minimizing phase separation and crystallization during subsequent glass forming steps.[5] Fluoride glass synthesis has not been attempted in microgravity to date due to the corrosive nature of the process.[5]

Canadian work has indicated the enhanced crystallization of certain ZBLAN formulations under 2-g and no evidence of crystallization in 0-g using a T-33 aircraft.[5,6]

Experimental

Two meter lengths of ZBLAN optical fiber were obtained from two different sources.* The protective polymer coating was removed chemically, and then the fibers were cut into 25mm lengths. Individual fibers were placed in an evacuated quartz ampoule and sealed. Fiber diameters were nominally 300 microns.

A fiber annealing furnace (FAF) was designed and constructed for use on the KC-135 aircraft (figure 1). The FAF consists of a preheat furnace, annealing furnace and a quench block. The sample is translated manually through each component using a stainless steel push rod. In operation, a single quartz ampoule is placed at the end of the push rod, and then translated into the preheat furnace for a period of two minutes. This allowed the fiber to reach a temperature of 250°C. Then, during the microgravity portion of the aircraft parabola the ampoule is translated into the annealing furnace for 15 seconds allowing the sample to reach a temperature of 415°C. This temperature is approximately 20°C above the crystallization temperature of ZBLAN. At the end of 15 seconds the ampoule is translated into the perforated brass quench chamber. Water was then used to quench the ampoule via a plastic 60 cc syringe. Cooling rates were generally around 40° C/sec.

Ground tests were performed to determine the time necessary to reach the nucleation temperature and to run 1-g studies. A thermo-couple was inserted into a glass ampoule and translated into the preheat furnace until the temperature reached 250°C and then into the annealing furnace until a temperature of 415°C was obtained. In this manner, the times necessary for preheat and annealing during the parabolic manuever were established. Five samples of each manufacturer's fiber were then heated at 1-g.

During the KC-135 flights ten samples of each manufacturer's fiber was heated during the 0-g portion of the parabola. Actual gravity levels during the 20 to 25 second period of 0-g range in the 0.01 to 0.001 g level.

* Infrared Fiber Systems(Silver Springs, Md.) and Galileo Electro-optics (Sturbridge, MA.)

The processed ZBLAN samples were examined using optical microscopy, scanning electron microscopy and EDX analysis.

Optical fiber communication offers an exciting alternative to traditional wire communications. Extensive research over the past two decades helped in bringing down the transmission loss in silica fiber close to its theoretical limit of about 0.2 dB/km. However, the high density optical communication systems in the future would require optical fibers with losses far below those of silica fibers. Many infrared transmitting materials, such as heavy-metal oxides, halides, and chalcogenides, have the potential of having losses below 0.01 dB/km[2,3,4]. Amongst these, heavy metal fluoride glasses based on zirconium fluoride are most promising.

Fluorozirconate glasses have a broad transparency range, low refractive index, small dispersion, low Rayleigh scattering, and ultra-low thermal dispersion. In addition to fiber optics, they can be used in infrared remote sensing, laser power transmission, control systems for nuclear power plants, and various other applications. They offer a great deal of compositional flexibility, which could allow their properties to be tailored to a broad range. Although the theoretically predicted loss factor in fluoride glasses is around 0.001 dB/km[5], the practical limitations in material purification brings this value up to around 0.02 dB/km.

Fluoride glasses have a narrow glass forming region. The large density difference between different components could lead to rapid phase separation and crystallization in these glasses under gravity. The microcrystallites formed in these glasses during synthesis or subsequent processing give rise to undesired scattering and higher than expected losses. Presently the losses obtained in these glasses are in the range of 1 to 100 dB/km, with best reported values of 0.7 to 0.9 dB/km[6,7].

Previous studies of this nature[1] were incomplete in that the researchers observed no crystallization during flight. The conclusions drawn from that set of experiments noted that several variables, including the temperature profile were inadequately controlled for good scientific observations. The results did suggest that nucleation of microcrystallites does have a strong dependency on the temperature gradient at the solidification interface and any experimental work in this area needs to have that parameter under control.

Synopsis of the Glass Annealing Furnace

The purpose of this KC-135 experiment is to determine the effects of gravity on nucleation and growth of crystals in optical fiber. It is believed that in microgravity that there will be an absence of nucleation and growth and that these phenomena will be enhanced in the 2g portion of the parabola. The GAF will be used to anneal (not melt) the fibers in either low or high gravity. During the annealing process the growth of crystals maybe enhanced or retarded by the influence of gravity. The results of this experiment will be used in the development of Space Shuttle experiment. This research is intended to help develop a process to improve the transmission properties of commercial fiber optic cables.

The optical glass fibers will be sealed within a quartz ampoule to prevent deterioration from moisture while being heated. The ampoule is 60 mm long and 3 mm in diameter.

Test Objectives

To process approximately 30 samples total, 15 in low-g and 15 in high-g. Results of the effects of low and high gravity upon crystal nucleation will be determined in the laboratory at MSFC, Alabama.

Test Description

For each run one quartz ampoule containing one 24 mm long optical grade quartz fibers will be placed into the preheat furnace set to 300° C. At the appropriate time the ampoule will then be pushed into the annealing furnace set to 400° C and allowed to heat soak for approximately 10 seconds. Then it will be pushed into the quench chamber where water will be sprayed onto it and rapidly quench the sample. Quartz is very resistant to thermal shock at this low temperature, therefore the risk of the ampoule shattering is very minimal. A 0.125" diameter stainless steel rod is used to secure the ampoule and move or push the ampoule from one zone to the next in a straight line.

Equipment Description

The GAF is a relatively small package measuring 22"L x 10"W x 7.25"H and weighs less than 25 pounds. Its basic components are two furnaces, a water quench chamber and associated temperature control circuitry. The unit will be bolted down to a framework assembly fabricated from AMCO Engineering Co. stock materials which will act as a pedestal structure. This pedestal has flown twice in support of the KC-135 Fiber Pulling Apparatus in the past. It's safety information is contained within documents associated with the FPA experimental hardware and has already passed a JSC TRR.

The experiment processing area is contained within a Plexiglas housing and is vented to the aircraft's overboard dump system. The dump system is utilized for two reasons: (1) To provide a means of removing the 50 to 60 ml of quench water and (2) to maintain a slight negative pressure within the housing in the unlikely event that one of the sample ampoules should break. If an ampoule should break then no further action would be taken during that particular flight and the experiment would be shut down and recycled on the ground.

The electrical system is comprised of two temperature control systems - one for each furnace. An Omega model CN132 temperature controller will monitor the temperature of the particular furnace and apply or remove power to the heating element by controlling a International Rectifier solid stage relay model TD1225 which is rated to 25 amps. There is only one 7.5 amp circuit breaker switch to control power to the entire system.

To quench the ampoule a 60 cc plastic syringe located outside the Plexiglas housing is connected via a plastic hose to the brass quench chamber located within the housing. The syringe contains only pure water and does not utilize any type of needle.

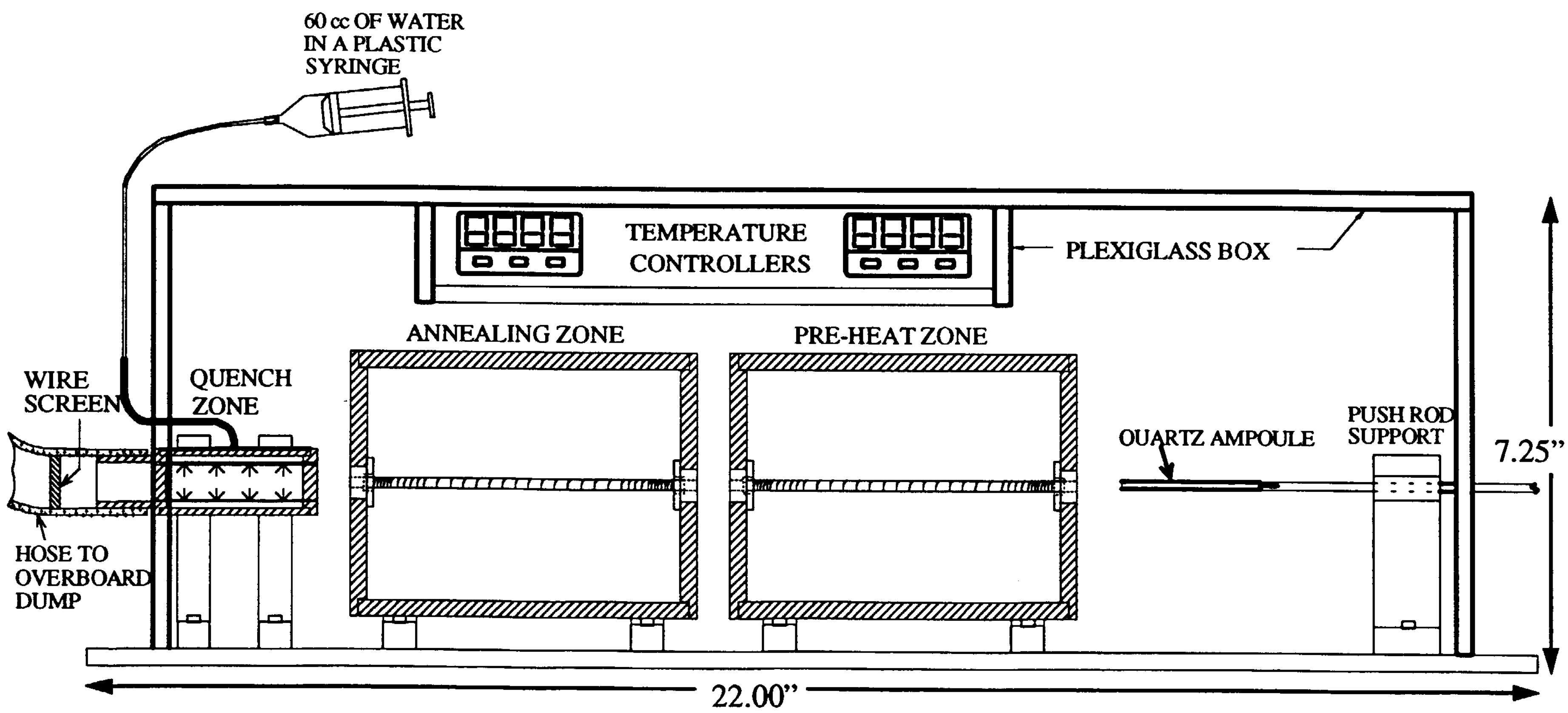


Figure 1. KC-135 Glass Annealing Furnace Design

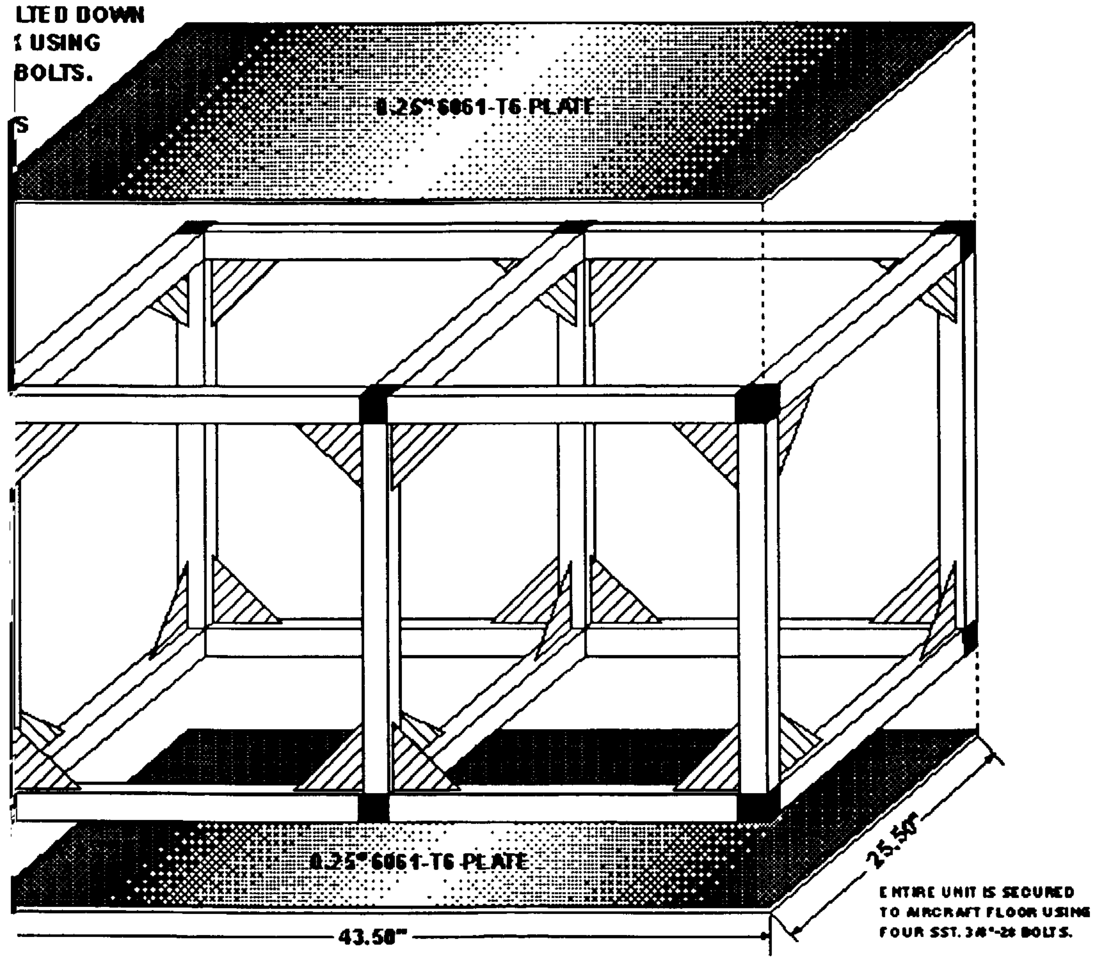
Structural Load Analysis

Two simplified case studies are presented dealing with the heaviest objects and the associated mounting points. The first case deals with the two bolts that mount the entire assembly (25 pounds) to the top of the support structure. This support structure has been flown aboard the KC-135 in the past for use with the Fiber Puller Apparatus and is referred to as the pedestal.

The second case shows an analysis of the heaviest object (the annealing zone furnace) and its associated hardware to the base plate. All total this item weighs 3.1 pounds and is the heaviest single item contained within the system.

It is our contention that these two studies represent the worst case conditions with this hardware. It should be pointed out that the reactive moment arm is included in the analysis with the center of gravity for the assembly at 3.5 inches up from the base plate, 9 inches from the left side and 5.5 inches from the front edge. It is assumed that the 9 g load is applied in the X and Z directions simultaneously along with a 2 g load in the Y direction.

Figures 2 and 3 provide sketches of the AMCO framework pedestal and where the GAF system is located with respect to the pedestal. The pedestal was originally designed to support the Fiber Puller Apparatus which weighed 146 pounds, therefore no structural analysis is considered necessary for this application since the GAF only weighs 25 pounds.



oded view of pedestal framework used on previous KC-135 Glass Fiber Pulling Apparatus.

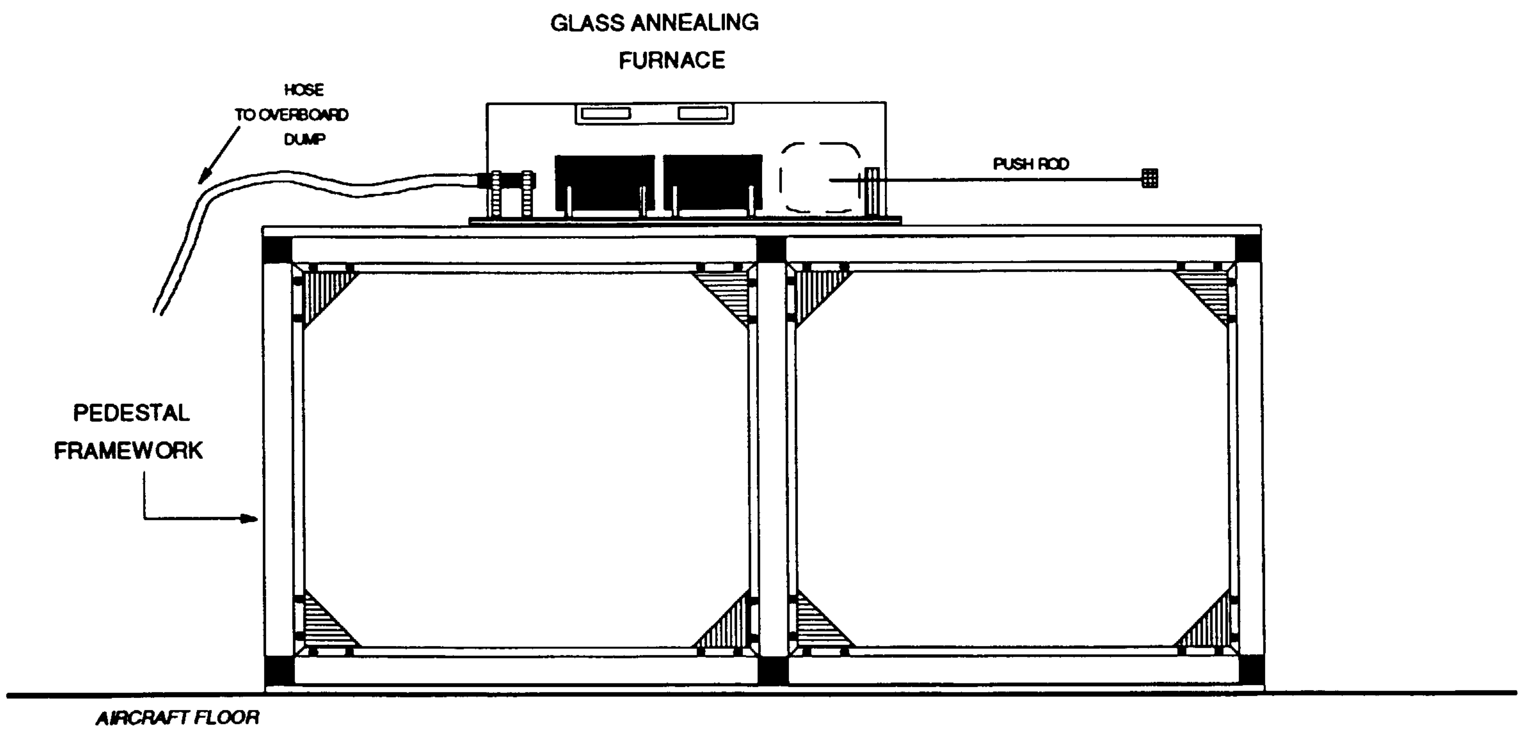


Figure 3: Assembly view of GAF attached to the pedestal framework.

Case 1: Glass Annealing Furnace Assembly to the Pedestal Framework.

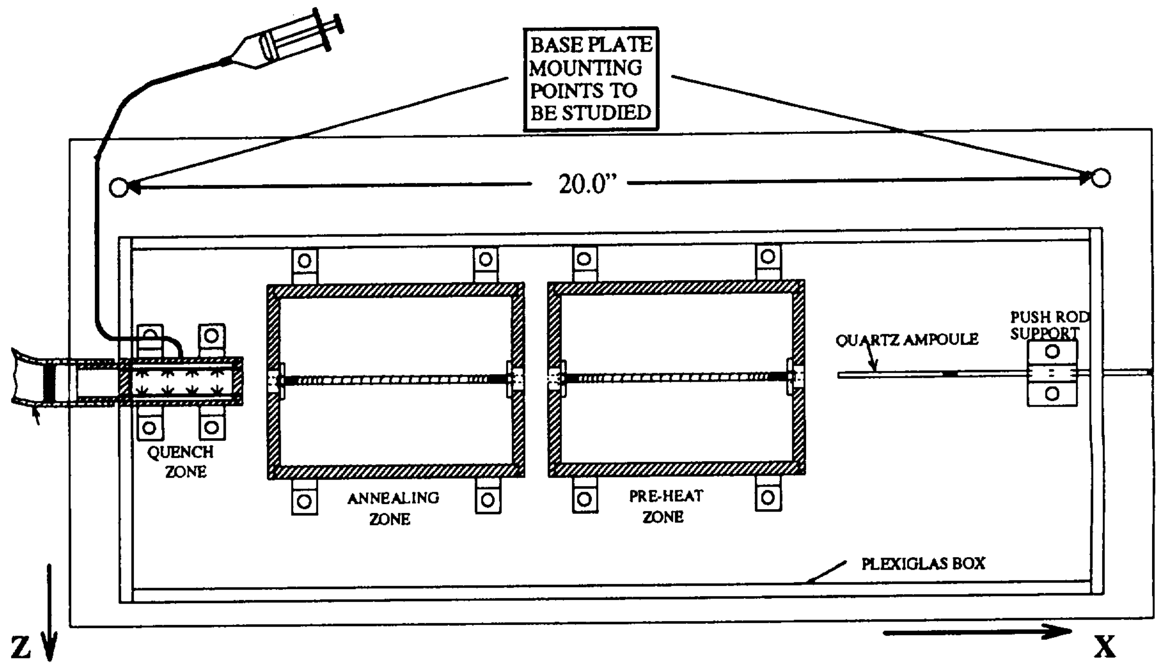


Figure 4: Top view of GAF assembly

TENSILE:

The reactive moment arm is calculated for the total tensile created which is distributed by the 2 bolts that mount the 1/4 inch thick base plate to the support structure. The center of gravity is located 9" on the X axis, 3.5" on the Y axis and 5.5" on the Z axis.

Tensile force for the free standing structure seeing 9 g's eyeballs in, out, left, or right and simultaneously along with 2 g's eyeballs up pulling apart from the 1/4 inch thick base plate will be:

Direct Tension: (25 lb. x 2 g's) / 2 bolts = 25 lb./fastner

Moment from the X load:

M^x = 25 lb. x 9 g's x 6.519" = 1,466.78 in·lb

M^z = 25 lb. x 9 g's x 9.657" = 2,172.83 in·lb

Tension at "A" from M^x:

$T_A = M^x/(2 \times 20")=1{,}466.78/(2\times20") = 36.67$ lb.

Tension at "A" from M^z:

$T_A = M^z/(2 \times 20")=2{,}172.83 /(2\times20") = 54.32$ lb.

Total Tension = 36.67 + 54.32 + 25 = 115.99 lb.

The 3/8" stainless steel bolts used are rated to 13,798 lb. each. The force seen at 2 g's eyeballs up is 113 lb. pounds per bolt. Therefore, each bolt is 13,798/116 = 119 times stronger than required for a tensile force of 2 g's eyeballs up along with 9 g's eyeballs in, out, left, or right. This value is approximate since it does not take into account shear or pre-loading of the bolt.

SHEAR:

For this case, primary shear is considered for all bolts that attach the base plate to the top of the pedestal framework. Shear stress seen across the mounting bolts at 9 g's eyeballs in, out, left, or right will be:

Direct Shear: (25 lb. x (9^2 g's x 9^2 g's)$^{1/2}$) / 2 bolts = 159.10 lb./fastner

Total Shear = 159.10 lb.

Each bolt sees 159.10 pounds of direct shear force. The 3/8" stainless steel bolts used are rated to a shear strength of 8,279 lb. each. Therefore, each bolt is 8,279/159 = 52 times stronger than the shear forces expected at 9 g's eyeballs in, out, right or left. This value is approximate since it does not take into account shear or pre-loading of the bolt.

Case 2: Annealing Zone Furnace to the Base Plate

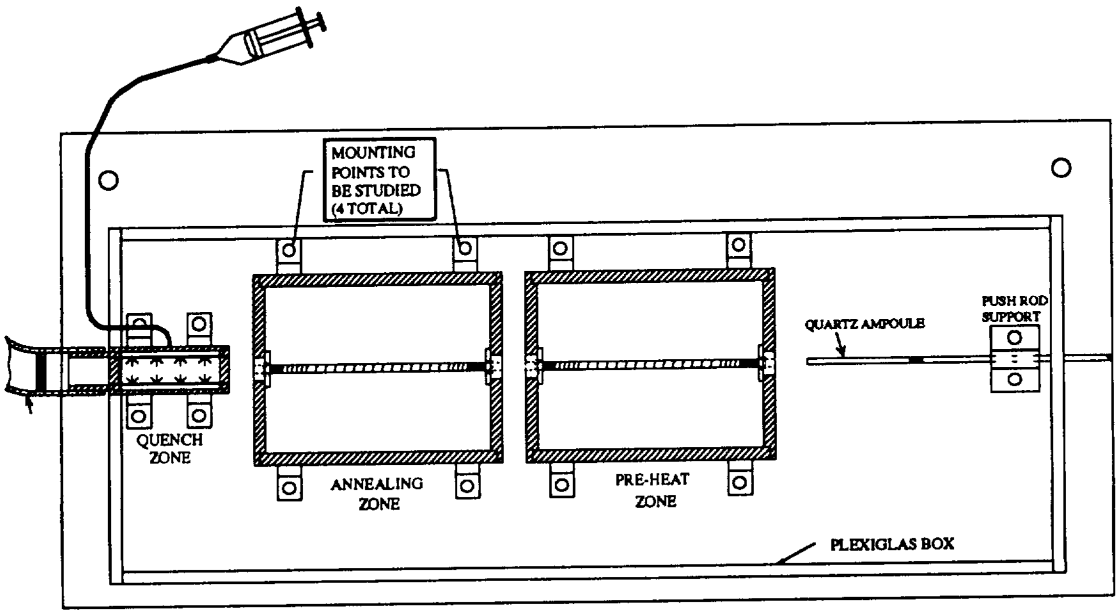


Figure 5: Top view of GAF assembly

The reactive moment arm is calculated for the total tensile created which is distributed by the four 10-32 stainless steel screws that attach the furnace mounts to the base plate. The center of gravity is located 2.5" on the X axis, 2" on the Y axis, and 2" on the Z axis.

Tensile force for the free standing structure seeing 9 g's eyeballs in, out, left, or right and simultaneously along with 2 g's eyeballs up pulling apart from the 1/4 inch thick base plate will be: (Assume moment arm is conservatively 4")

Direct Tension: (3.1 lb. x 2 g's) / 4 bolts = 1.55 lb./fastner

Moment from the X load:

M^x = 3.1 lb. x 9 g's x 2" = 55.8 in·lb

M^z = 3.1 lb. x 9 g's x 4" = 111.6 in·lb

Tension at "A" from M^x:

$T_A = M^x$/(2 x 3.75")= 55.8/(2x3.75") = 7.44 lb.

Tension at "A" from M^z:

$T_A = M^z$/(2 x 5")= 111.6 /(2 x 5") = 11.16 lb.

Total Tension = 7.44 + 11.16 + 1.55 = 20.15 lb.

The 10-32 stainless steel screws used are rated to 3600 lb. each. The force seen at 2 g's eyeballs up is 20.15 pounds per screw. Therefore, each screw is 3600/20.15 = 178 times stronger than required for a pure tensile force of 2 g's eyeballs up simultaneously along with 9 g's eyeballs in, out, left, or right. This value is approximate since it does not take into account shear or pre-loading of the screw.

SHEAR:

For this case, primary shear is considered for all bolts that attach the furnace to the base plate. Shear stress seen across the mounting bolts at 9 g's eyeballs in, out, left, or right will be:

Direct Shear: (3.1 lb. x $(9^2$ g's x 9^2 g's$)^{1/2}$) / 4 bolts = 9.86 lb./fastner

Total Shear = 9.86 lb.

Each 10-32 stainless steel screw sees 9.86 pounds of direct shear force. The screws used are rated to a shear strength of 2,160 pounds each (60% of the tensile). Therefore, each screw is 2,160/9.86 = 219 times stronger than the shear forces expected at 9 g's eyeballs in, out, right or left. This value is approximate since it does not take into account tensile or pre-loading of the screw.

Electrical Load Analysis

This system uses 120 volts 60 Hz. AC only. A single 30 foot long 18 gauge power cord provides the AC power interface to the experiment. Total load for the experiment will be 5.0 amps maximum during the furnace heat up and will subsequently become intermittent once the furnace has reached temperature. The temperature controllers use timed proportioned control of the power to regulate temperature. Maximum current draw is limited by the resistance of approximately 50 ohms for each heating element.

The Omega model CN132 temperature controller maximum power draw is 2.5 watts and is internally protected with a 100 milliamp fuse.

On the following page is Figure 6 which identifies the AC power circuits, main power switch which is also a circuit breaker and wire gauges. Except for the power cord all wiring is TFE insulation and rated to 175° C. The 30 gauge thermocouple wire is insulated with glass braid insulation.

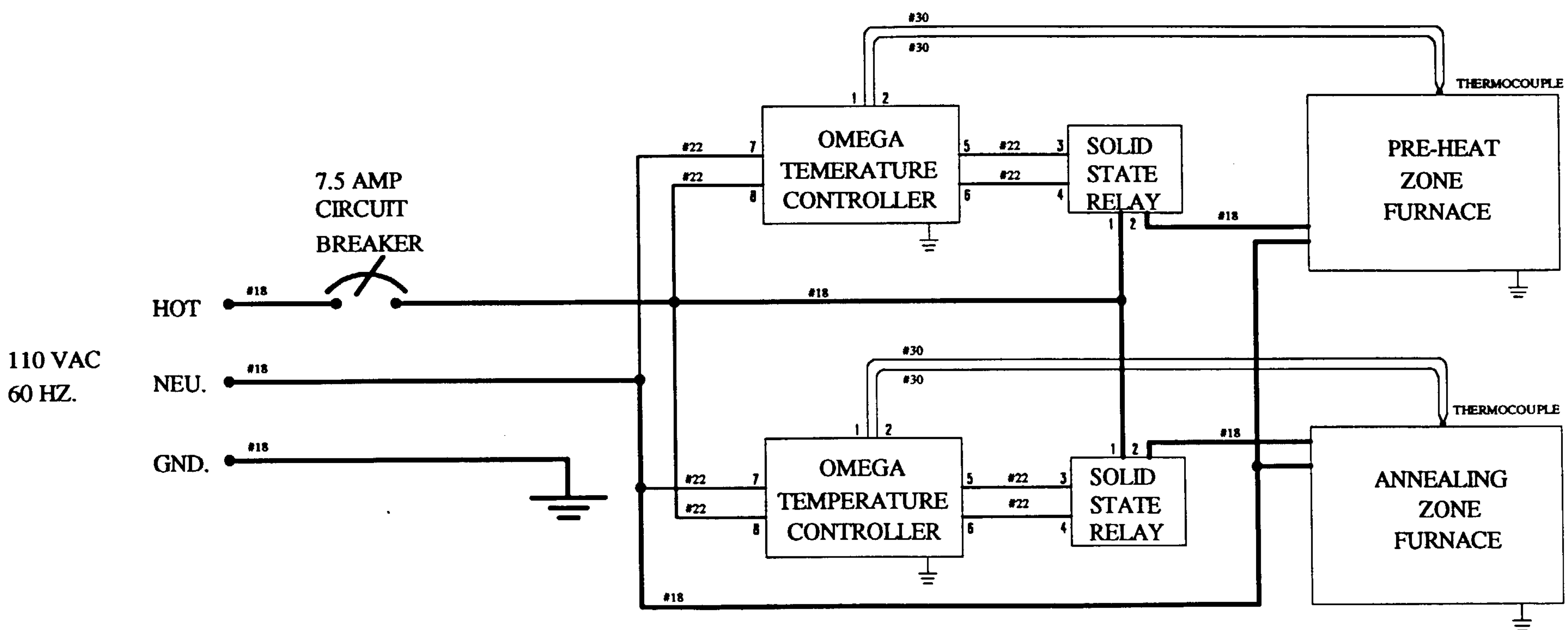


Figure 6: KC-135 Glass Annealing Furnace Electrical Diagram

In Flight Test Procedures

During level flight one quartz ampoule will be removed from the supply case and inserted into the end of the push rod. With the access door closed, the ampoule will be loaded into the preheat furnace set at 300° C and allowed to heat soak for at least 5 minutes. Upon entering a low or high-g period the ampoule will then be pushed or loaded

At the end of this time period the ampoule will then be pushed into the water quench chamber. Here the 50 to 60 milliliters of pure water will be sprayed onto the ampoule by the plastic syringe and thus rapidly cool down the sample.

To recycle the experiment a new ampoule and plastic syringe filled with water will be installed during level or high-g flight.

Parabola Requirements

During sample loading and unloading procedures a period of 1 to 2 minutes of high-g or level flight is all that is required. Only one parabola is needed to process anyone sample and can occur anytime after the required preheat period. It is hoped that 5 to 10 samples per flight can be processed. There are only two test support requirements: 1. The 110 Volt AC, 60 Hz, aircraft power be supplied at least 30 minutes prior to take off to buoyancy convection which is suppressed in microgravity. Therefore, one could expect little or no crystallization if the fiber could be produced in a microgravity environment.

Figure 4. SEM micrograph of ZBLAN fiber processed in 1-g, 80X magnification.

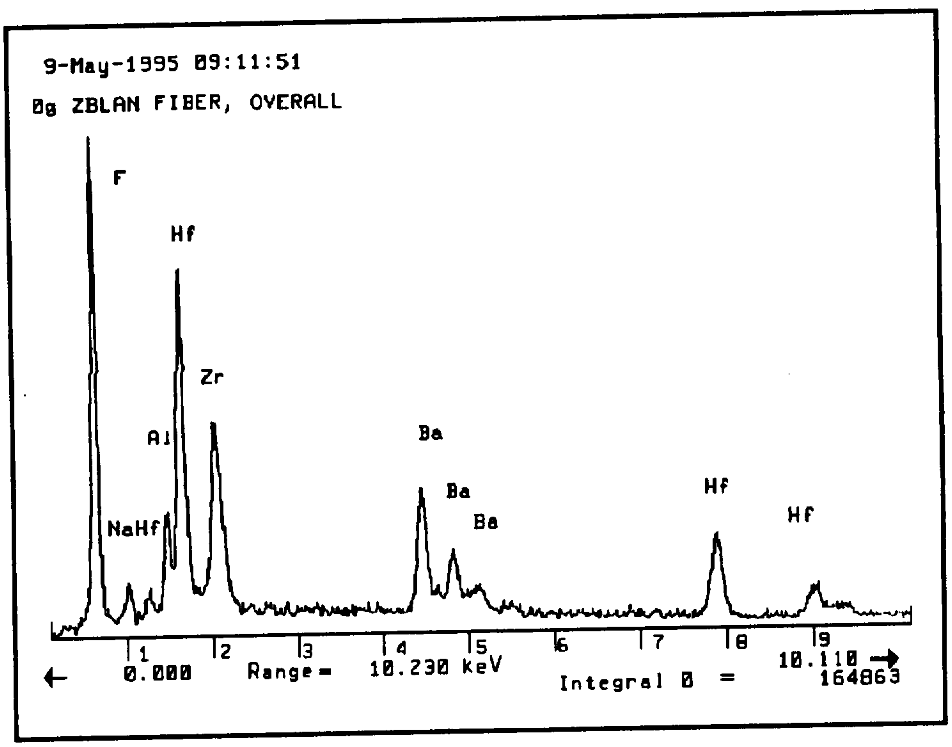


Figure 5. EDX Spectrum of low-g processed ZBLAN Fiber.

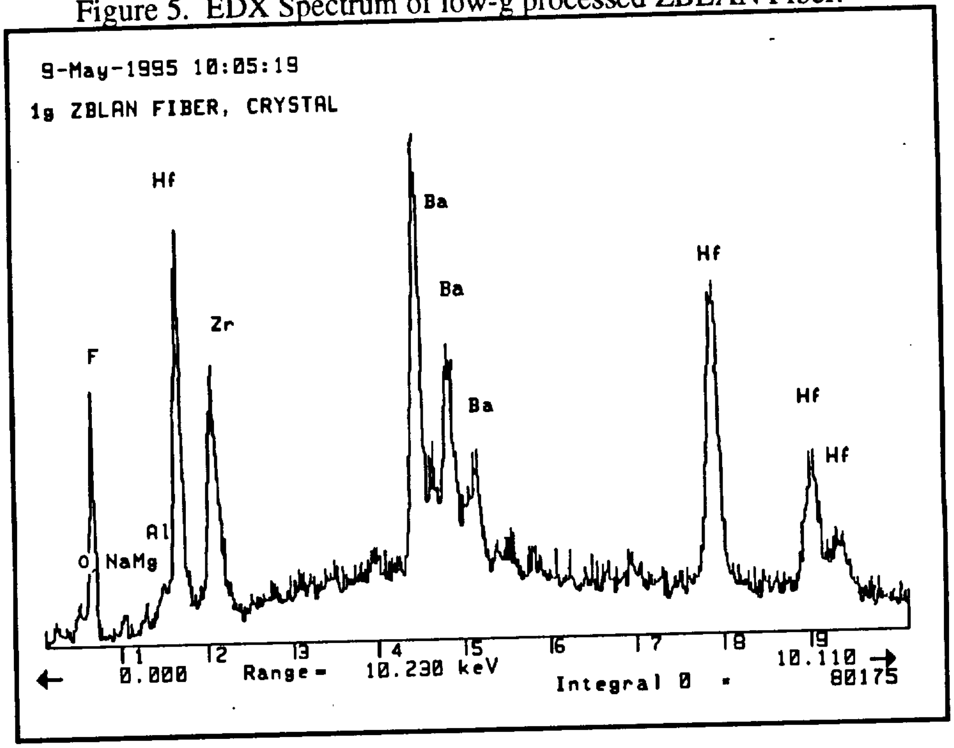


Figure 6. EDX spectrum of 1-g processed ZBLAN fiber.

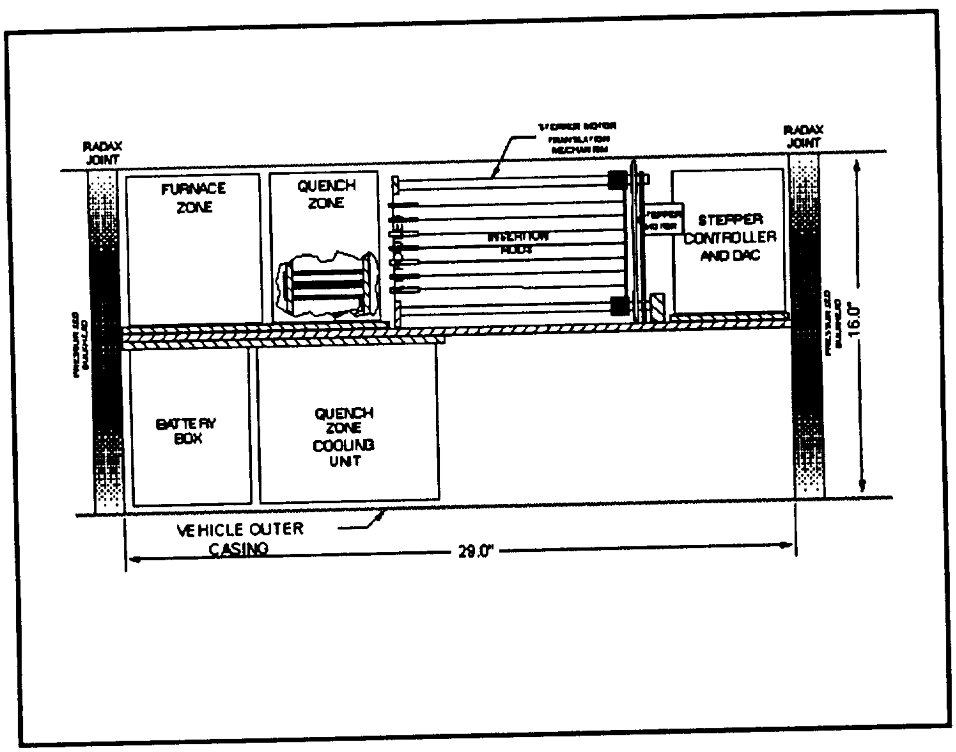


Figure 7. Schematic of sub-orbital rocket payload to be flown on Conquest 1 in February 1996.

Section II

Work Performed on the Fiber Pulling Apparatus

Introduction

A reduced gravity fiber pulling apparatus, henceforth FPA, has been constructed in order to study the effects of gravity on glass fiber formation. The apparatus was specifically designed and built for use on NASA's KC-135 aircraft. During this period of performance the FPA was modified to include a remotely controlled sting mechanism by which the operator could initiate a draw process from a glass preform. One flight has been completed to date during which ZBLAN glass fiber was successfully produced in a simulated zero gravity environment.

Initially the FPA was developed under contract NAS8-36955, Delivery Order 113 over the period of March through September of 1991. At the end of that six month period it was determined that several improvements could be implemented into the apparatus to aid in formation and study of micron diameter glass fibers. Hence, a follow on contact was obtained to continue the research. One of the major problems in drawing a glass fiber on the Space Shuttle is safety. Normally on the ground the draw tower operator will allow a bead of softened glass to fall and thus initiate the draw process. However in the absence of gravity this process will not work for obvious reasons. Hense a difference method had to be developed. The result was the integration of a remotely controlled sting apparatus into the existing FPA system. By developing this remotely controlled draw initiation process the payload specialist will not be in contact with a hot surface or material. The following provides the results of what has been accomplished.

Description of the FPA

Referring to figure 1, the FPA consists of a furnace and associated temperature controller, fiber take up reel and servo motor, video camera, fiber quenching and cooling system. These basic components are enclosed in a Plexiglas housing. Since this system flies aboard the KC-135 safety requirements dictated a containment system. There is a port for dry nitrogen gas so that the relative humidity of the apparatus can be reduced during fiber drawing. A hygrometer is located on the inside rear wall which reads relative humidity. The FPA is controlled by a data acquisition system. This system consists of an IBM AT industrial grade computer with Metrabyte model DAS8 eight channel 12 bit A/D board for recording data and controlling the servo winding motor and a video graphics overlay board. Also mounted within the rack is a super VHS video recorder, video monitor, keyboard, and associated control electronics. The video monitor displays the image of the glass fiber as it is being drawn and overlays on it the graphical data. This data includes three axes accelerometer data from a set of Sundstrand model 700 accelerometers, furnace and bushing orifice temperatures, fiber winding speed in cm/sec, date and time.

Quenching the fiber rapidly helps prevent nucleation events which can adversely affect fiber properties. The other option involves using dry nitrogen gas to quench the fiber. In this method, a brass annulus with a 13 mm diameter hole in the center and twenty 0.7 mm holes arranged around the inside perimeter is used to direct nitrogen gas onto the fiber as it is pulled. This not only quenches the fiber but aids in the drawing process.

During a reduced gravity maneuver in the KC-135 aircraft, the acceleration due to gravity can be as low as 0.001g and as high as 2g. Thus, the FPA was constructed to withstand the loads seen during the parabolic maneuvers, as well as 9g emergency crash landing loads. This reason, plus on-board safety considerations, is what dictated overall designs of the FPA system.

Results

Two types of ZBLAN glass material were used in this investigation: one provided by Infrared Fiber Systems and the other by Galileo Electo-Optics. Since the purpose of this test flight was to determine the effectiveness of initiating a draw by remote control the preform samples provided were the remnants of previous runs by the two companies. The left over preforms provided ample material to test the new design.

During the KC-135 flights several successful sting initiated draws were completed thus proving the concept that a draw could be initiated by remote control. This provided an important step in being able to accomplish the proposed research on the Space Shuttle and International Space Station Alpha.

Future Work

The next step in this research will be to fly samples of ZBLAN on a sub-orbital rocket flight. This experiment will have the advantage of providing 5 to 7 minutes of microgravity. Thus, a better understanding of the effects of microgravity on crystallization will be possible. Figure 7 shows the payload which will be flown in February 1996 on the University of Alabama in Huntsville managed Conquest 1 rocket. ZBLAN fibers will be placed in evacuated quartz ampoules and heated above the crystallization temperature during the microgravity portion of the rocket flight. Samples from each company will have DTA performed to determine their exact crystallization temperature. Past experience has shown this temperature to range from 385° to 394°C. The samples will be quenched below the glass transformation temperature before the rocket sees any gravity forces upon its decent to the ground.

Two shuttle mid-deck locker experiments are also planned. In the first, ZBLAN preforms will be heated to 800°C for 2 hours to dissolve all crystals and then rapidly quenched to below the glass transition temperature. The preforms will then be annealed at 300°C to remove any residual stresses. From some of the preforms optical fibers will be pulled on the ground using a conventional drawing tower to see if the attenuation coefficient has been lowered. The remaining preforms will be pulled on the second shuttle flight using a fiber pulling apparatus as shown in figure 8. The results of this research will determine the future course for the possible commercial production of ZBLAN fiber on the International Space Station.

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