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HIGH TEMPERATURE **MATERIALS** FOR
VENUS BALLOON ENVELOPES

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Abstract

These are several attractive concepts for relatively low cost Venus balloon experiments which would allow in situ measurements near the surface of Venus, as well as in the atmosphere. Venus balloon exploration concepts require balloon materials which can withstand the rigors of the Venus atmosphere, including temperatures of about 450°C. This requires evaluating and development of materials under conditions that match those encountered in Venus balloon experiments.

High Temperature Polybenzoxazole (PBO) based liquid crystal polymer films and fibers have been evaluated to establish their suitability as candidate Venus balloon envelope material.

Preliminary results from thermogravimetric (TGA) and thermal mechanical (TMA) evaluation show that the material is thermally stable at 500°C with promising mechanical properties. The effects of thermal cycling and the coefficient of thermal expansion (CTE) on PBO film and fiber were measured. Preliminary Venus balloon material requirements has been developed.

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INTRODUCTION

In keeping with the desire for "smaller, faster, cheaper, better missions", JPL has been exploring the possibility of using balloons to make in situ measurements of the Venus atmosphere and surface. Studies are underway of various types of Venus aero vehicles including aerostats and balloons, blimps and aircraft. Such vehicles could carry out a wide range of Venus exploration tasks including atmospheric structure circulation, composition, surface morphology, and mineralogy.

A number of concepts^{1,2} for balloon altitude control have been developed such that temperature differentials in a planet's atmosphere can be used to provide the energy necessary to ascend and descend. In the most promising concepts, a fluid is evaporated in the lower, warmer atmosphere, thus filling a balloon and generating buoyancy for ascent. The fluid condenses in the upper, cooler atmosphere, thus decreasing buoyancy and allowing redescent.

Aero vehicle concepts include systems which can bob up and down between lower, hotter altitudes where surface measurements can be made, and higher (cooler) altitudes where phase change materials (used to protect electronics at lower altitudes) can cool, solar energy can be collected and stored, and where data can be transmitted to the Earth. Such concepts must be able to withstand the temperature and pressure near the surface, traverse the cloud layers, and survive exposure to corrosive sulfuric acid, cloud droplets.

The Venusian atmospheric environment is shown schematically in Fig. 1. The surface temperature is a scorching 460°C and has a pressure of over ninety times Earth's surface pressures. The Venusian upper atmosphere between about 55 km to 60 km altitude, however, has temperatures and pressures quite close to those of Earth's lower atmosphere.

One particularly attractive concept [Venus Cloud Bobber (VBOB)] under study can go all the way to the surface where composition measurements can be made. These concepts rely on phase-change materials (typically water) to protect conventional electronics and sensors from the high temperature (460°C) environment. In addition, systems must be designed to withstand the 90 atm surface pressure. Such a balloon must make periodic excursions to high altitudes to replenish the water ice used to cool electronics. Electrical energy may also be extracted from turbines during this bobbing motion through the atmosphere.

Another VBOB can traverse the Venus atmosphere from a height of 60 km to a depth of from 30 km to the surface. Water ice is used as the Phase Change Material (PCM) for temperature control of the hardware in the gondola. Water ice is generated while at high altitudes, and gradually melts at lower altitudes. When the balloon is at high altitudes (60 km, -10°C) a diode reflux heat pipe insures refreezing of the water Phase Change Material.

Hydrogen is used as the primary buoyant gas to supply most of the buoyancy. The hydrogen is generated by the heating of beryllium hydride ($\text{BeH}_2 \rightarrow \text{Be} + \text{H}_2$) when the VBOB first enters the Venus

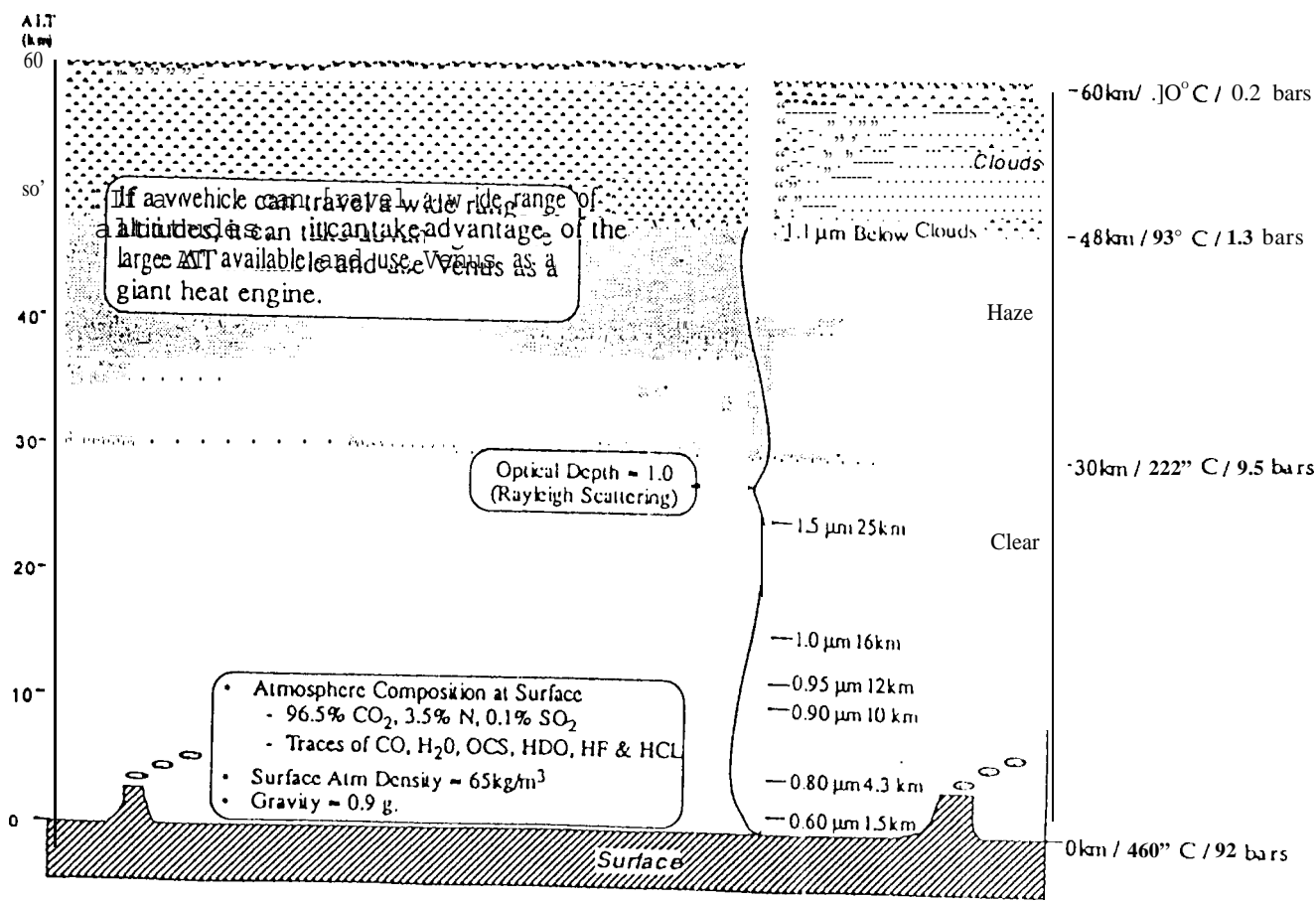


Fig. 1. Venus Environment.

atmosphere at the start of the mission. Methylene chloride (CH_2Cl_2) is used as an altitude control fluid by adjusting the buoyancy. As the balloon descends, the methylene chloride heats up. When the water ice used to cool the payload starts to become depleted, the balloon must ascend. To achieve this, a valve is released which allows the methylene chloride vapor to fill the secondary balloon, thus providing added buoyancy. When the balloon reaches higher altitudes, the methylene chloride again condenses and returns to its small pressure vessel giving the balloon negative buoyancy for its return descent to the surface. The high surface temperature and pressure and the thick clouds of the Venus environment provide a significant challenge to the design of an in situ science instrument. However, the large temperature difference between the upper atmosphere and the surface presents an opportunity to use the atmosphere as an energy source. Balloons can tap this energy source. There are several other candidates for altitude control fluids as well as for the primary buoyant gas. There are also other Venus aero vehicle concepts. A detailed description of these concepts is given in reference 2. Concepts for short duration, very low altitude blimp or aircraft are also being investigated. Venus aero vehicles are very exciting concepts which could open the entire planet up to in situ observational measurements to take the next step beyond Magellan in Venus exploration.

BACKGROUND

Zero pressure balloons which are currently fabricated from polyethylene materials for the most part meet the needs of the scientific community in earth observation experiments.

Nonetheless, the desire to increase float altitude, payload weight, and duration of flight, continue to fuel the search for new and better materials for scientific research balloons.

The materials recently tested³ as candidates for new balloon films (for use on earth) are summarized below:

Poly-plus - dacron thread reinforced polyethylene film - for heavy lift applications

Biaxially oriented nylon-6 (Emblem-6) - an unreinforced superpressure balloon material

Nylon-12 - for use in heavy lift, extreme altitude, and superpressure balloons

Unreinforced polyester materials - thin film laminates to overcome pinholing and crack

propagation problems seen in polyester single ply films

Aramid fiber reinforced polyester 6-m x 6--m laminated polyester with Aramid yarn

Testing of the reversible fluid altitude control concept is possible on earth and most of the materials listed above can be used buoyancy control principles. This testing is now underway and polyethylene film passed compatibility tests with Freon R-114. However, the use of these materials for Venus near-surface balloon mission is not possible.

previous studies of balloon materials for use on Venus included those by **Jacque Blamont (CNES)** of balloons made from a metal (aluminum) skin in the shape of a Chinese lantern or accordion for use around 20 km. According to **Blamont**, there was always a concern about crack formation and leakage with such a metal balloon. In June 1985 instrumented balloons were placed in the atmosphere of Venus as part of the VEGA mission at a float altitude of near 54km where the temperature is <100°C. The balloons were fabricated from specially woven Teflon plastic cloth coated with Teflon plastic (limit to 200°C) .

A past CNES/JPL Venus Warm Region (15-20km) mission study identified a fabric of aluminum coated Kapton as possible balloon material stable at temperature below 350°C. This is the most thermally stable balloon material for Venus application described in the literature to date. For this study the first material under consideration was Polybenzimidazole or PBI. Developed by the Air Force for supersonic parachutes for reentry system, Hoechst **Celanese** PBI Product Division currently manufactures the fiber. Some of the current applications include firefighters' suits, race car driver suits, aircraft. fire blocking layers, rocket-motor insulation and others. Contacts with Hoechst **Celanese**, however did not recommend PBI for Venus near-surface applications. It was indicated that the useful life of PBI at temperature of 450°C is less than 5 minutes.

After evaluating several materials on the market, Polybenzoxazole (PBO) appears to be the leading candidate that could meet the

materials requirements at this time. A similar material, Polybenzothiazole (PBT) might also be an excellent candidate; however it is available only on a laboratory scale.

TECHNICAL APPROACH

The technical approach involved development of balloon material requirements acquisition of PBO film, fabric and *Polyimide benzoxazole* ~~Polyimide~~ Polyimidebenzoxole (PIBO). For evaluation. The initial testing was planned to be on film to see if the film itself is adequate for Venus applications. This would be simpler than developing composite films with a fabric core, but such a composite film will likely be considerably more rugged. Finally, an appropriate composite film will be manufactured from the fabric core, and tests will be conducted on the composite. The following tasks are planned:

- Characterization of PBO film, PIBO film and PBO adhesive. Primary emphasis will be placed on measurements in the range 400-500°C.
- PBO composite fabrication and baseline evaluation

The fabrication of the balloon material. from fabric will require a high temperature adhesive sealer for laminating PBO film on the surface of the PBO fabric. After discussion with Dow Chemical, PIBO was identified as a candidate coating/sealing material. PIBO is a film forming PBO copolymer, soluble in organic solvents. It has high temperature properties comparable to PBO and can be applied on the fabric using a commercial coater.

- Impact of mission temperature profile (cycling) on material properties.

As previously indicated, the mission may require periodic, short (less than two hours duration) excursions to the surface where the temperature is 460°C. At high altitudes, the temperatures could be as low as -10°C. This temperature cycling may have a

profound effect on the mechanical properties. It may induce morphological changes and improve mechanical properties. or, it may destroy beneficial molecular order built in the film **during** manufacture. Impact of high-temperature annealing **on** this material is also unknown. Cycling and annealing test will be conducted to evaluate it modification of mechanical properties can be made.

Chemical compatibility of candidate materials.

Balloon material must be compatible with trace amounts of sulfuric acid present in the Venus clouds as well as with the altitude control fluids planned to be used; e.g. , methylene chloride, ammonia, water or hydrazine.

This paper presents the preliminary results of the evaluation.

BALLOON MATERIAL REQUIREMENTS

The desired characteristics ~~(Category A requirements)~~ Of any balloon film are:

- High strength to weight ratio
- High tensile strength
- Toughness in tear resistance
- Toughness in impact resistance
- Maintenance of mechanical and physical. properties at low temperature
- Resistance to degradation from UV radiation
- Low radiation absorbtivity/emissivit.y ratio

All of these properties, of course, pertain to the environment in which the material is placed. This list of desired characteristics is both long and demanding and challenges conventional materials to the very limits of their performance.

At this time, the specific requirements ~~(Category B)~~ for Venus balloons are not fully defined. A first cut of balloon material requirements includes:

- 1) balloon deployment from a small. package while hanging from a parachute at 0.5-3 bar pressures and 20°-130°C temperatures,
- 2) storage in a small package at temperature as low as -5°C (could be heated if necessary),

- 3) must pass through Venus clouds which contain sulfuric acid droplets,
- 4) make periodic short (less than 2 hrs. duration excursions to the surface where the temperature is 460°C and pressure is 92 bars while maintaining mechanical and physical properties,
- 5) compatibility and low permeability to candidate altitude control fluids e.g., methylene chloride (CH₂Cl₂) , ammonia (NH₃) , water, hydrazine (NH₂NH₂) or others,
- 6) Maintenance of mechanical and physical. properties during temperature cycling from ~-5°C to 460°C.

In addition, the material must be available commercially, and fabrication processes must be feasible.

PBO CHEMISTRY AND TECHNOLOGY

The development of PBO is based on the pioneering work in polymer synthesis and fiber spinning funded by the U.S. Air Force.

PBO is a conjugated aromatic heterocyclic liquid crystalline polymer (LCP) with a chemical structure as shown in Figure 2.

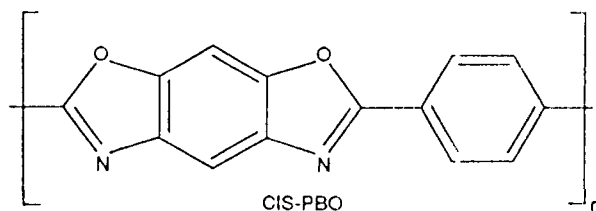


Figure 2. Chemical Structure of PBO

The chemical synthesis⁴ of PBO, results in a LCP solution which is processed^{5,6,7} to fiber or film by various techniques. The high strength and superior physical properties of PBO are due to the rod-like nature of the PBO molecule and the orientation that can be built into the polymer film. PBO film's self-reinforcing microstructure results in a "molecular fabric" with properties

comparable to those of advanced, fiber-reinforced materials, but without the drawbacks of distinct fiber and matrix components. The neat polymer has no melting point or glass transition temperature.

At the start of this study Dow Chemical Corporation produced PBO resin for internal evaluation with the intended commercialization at a future date. Pilot plant quantities of PBO resin (4000 lb) have been produced for this evaluation.

Recently Dow transferred the production capability to Toyobo Co., LTD which will start fiber production and will provide sample quantities of PBO for evaluation.

Dow had concentrated their effort on development of fiber manufacturing technology with a limited effort toward film production.

In an independent effort using Dow resin, Foster Miller Corporation is developing technology for the manufacture of PBO film. Dow PBO fiber filament is 11 microns thick, and, currently, 250 denier yarn has been used to produce fabric material. The fiber has a tensile strength of 800 Ksi at room temperature and the tensile modulus is 20-25 Msi as spun and 40-45 Msi after a heat treatment. These are attractive properties. Foster Miller³ is currently capable of producing biaxially or triaxially oriented PBO film in 20 x 36 inch sheets and up to one mil thickness. Depending on the process conditions, PBO film can have balanced biaxial strength (for isotropic strength and stiffness applications) , or uniaxial strength (almost all

orientation in one direction for applications where directional strength and stiffness must be controlled) . The size of the film is equipment limited, but and can be scaled up by renting existing equipment. The tensile strength of this film is 70-80 Ksi at room temperature, tear resistance over 2000 lb/in (better than polyester by factor of 2 or more), very low permeabilities (500-1000 times less than polyester), and very good UV resistance (over 4 times better than polyethylene).

Weight loss studies of PBO films (Foster-Miller) found essentially no weight loss up to 500°C for 6 hours. In a separate study of PBO film for an application at 400°C application, no loss of mechanical properties has been observed. Table 1 and 2 show Dow Chemical Co. PBO comparative film and fiber data. Figure 3 illustrates the extremely low permeability of PBO film as compared to other polymer films (Foster-Miller data) .

Property	Unit	Kapton	Aramid	PET	PEN	PBO
Density	g/cm ³	1.420	1.500	1.395	1.355	1.54
Melting Temp	'c	none	none	263	272	none
Glass Transition Temp	°C	350	280	68	113	none
Young's Modulus.	kg/mm ²	300	1000- 2000	500- 850	650- 1400	4900
Tensile Strength	kg/mm ²	18	50	25	30	56-63
Tensile Elongation	%	70	60	150	95	1-2
Long-Term Heat Stability	°C	230	180	120	155	>300
Heat Shrinkage (200°C x % rein)	%	0.1	0.1	5-10	1.5	<0.1
Coefficient of Thermal Expansion	ppm/ 'C	20	15	15	13	- 2
Coefficient of Hydroscopic Expansion	ppm/ %RH	20	18	10	10	0.8
Moisture Absorption	%	2.9	1.5	0.4	0.4	0.8

Table 1. Comparative data for films

Property	PBO	PBO High Modulus	Aramid	Steel	Spectra® (HDPE)	Carbon (hi- tensile)	Glass (s-2)
Tensile Strength (KSi)	820	800	400-500	250	435	500-700	665
Tensil Modulus (Msi)	25-30	40-45	10-25	29	25	30-40	12.6
Compressive Strength (Ksi)	40	65	65	250	10	300-400	>150
Elongation, Break (%)	3.0	1.5	1.5-4.0	2.0	3.5	1.5-2.0	5.4
Density (g/cc)	1.56	1.56	1.44	7.86	0.97	1.8-1.9	2.4
Specific Tensile Strength (Ksi)	525	510	280-350	32	450	270-380	280
Specific Tensile Modulus	16	26	7-i8	4	26	16-22	5
Limiting Oxygen Index (LOI:%)	56	56	30		19	50-65	

Table 2. Properties of high performance fibers

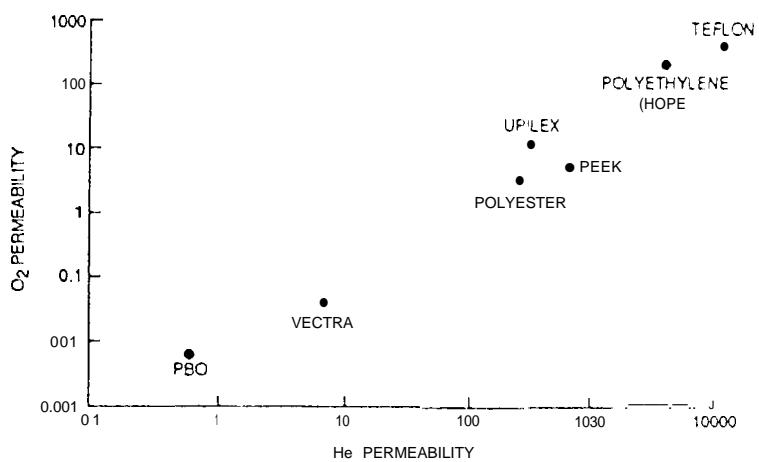


Figure ³~~4~~. Permeability comparison data

EXPERIMENTAL PROCEDURES

The tensile properties and CTE of PBO film and fiber were studied using a TA Instruments 2940 **Thermomechanical** Analyzer (TMA) equipped with a film and fiber tension assembly. The film used in these studies was provided by Foster-Miller and was not specifically prepared for a balloon application. The orientation of the film is $\pm 22^\circ$ and was determined to have a thickness of 0.4 mil. The fiber samples are from a 6"x6" piece of plain weave ballistic cloth with 45 x 45 yarns per inch which was provided us by the U.S. Air Force. Due to a lack of free fiber, samples for testing were removed from this cloth in the form of fiber bundles or yarns. Each yarn consists of 333 individual PBO fibers each with a diameter of 11 microns. Tensile specimens of the film in the form of strips in both the machine and transverse direction were cut from the original sample using sharp scissors. Paper was used as a backing for the film to aid in cutting. Specimens of varying width were cut (from 3mm to 0.2mm) and an optical microscope was used to inspect them and accurately measure the width. Samples were clamped and mounted onto the instrument using a special mounting fixture. The sample or gage length was ~8.8mm for all the tests. An initial weight of 100 to 250 grams was applied to the sample and the sample was then quickly heated to temperature (-3 min.) under a purge of He at 100 cc/min. Once the desired temperature was achieved the instrument applied an increasing force to the sample at a rate of (5g/min) until the sample broke. From this experiment both tensile modulus and

tensile strength at several temperatures were determined in both the machine and transverse directions.

Measurements of the CTE of the film in both the machine and transverse directions and of the fiber yarn were made using the TMA . The film samples used were 3mm wide and the sample or gage length for both the film and fiber was ~13mm. A static force of 15g (700 psi for the film and 675 psi for the fiber) was employed. For the analysis an initial heating to 125°C was used to drive off moisture, then multiple temperature cycles from room temperature to 500°C were applied with the heating rate of 10°C/min under a helium purge and the resulting dimensional changes were plotted.

RESULTS AND DISCUSSION

Tensil Properties

Figure 4 shows the dependence on temperature of PBO film tensil strength in the machine direction. Tensile strength decreased continuously as the temperature increased, resulting in approximately an 86% reduction at 600°C (as compared with room temperature) . Remarkably however, PBO film still retains enough strength at 500°C (44 ksi) so that it is stronger than PET at room temperature (-35 ksi) .

fig 4

Figure 5 shows the dependence on temperature of PBO film tensile modulus in the machine direction. Unlike tensile strength, the tensile modulus remains fairly constant to 500°C followed by a decrease to 600°C.

Figure 5.

Figures 6 and 7 show the temperature dependence of PBO film tensile strength and tensile modulus in the transverse direction. Although these films were not intended for a balloon application, measurement of tensile properties were made in the transverse direction in order to make some comparison to the machine direction properties.

Figure 6.

Figure 7.

While the tensile strength in the transverse direction shows a decrease with increasing temperature similar to the machine direction, the tensile modulus in the transverse direction has a very different temperature dependence than in the machine direction.

Lower temperature testing of the PBO yarn was beyond the capability of the TMA (200g maximum suggested force) because the fiber yarn has a 6 times higher tensile strength than the film at room temperature as well as a relatively large cross sectional area. If PBO fiber yarns longer than 6" were available the tensile strength could be determined using an Instron. By going above the recommended load of the TMA the yarn was broken at 600° giving a tensile strength at 600° of at least 150 ksi. This value represents an 83% reduction as compared with the value as shown in table 2 at room temperature of 800 ksi. This would indicate that the tensile properties of the PBO fiber have a similar temperature dependence as that of the film in the machine direction. , Therefore at the temperature of interest 450°C the PBO fiber can be expected to have a tensile strength of -300 ksi

making it stronger than room temperature **steel** and nearly as strong as room temperature Aramid.

The **thermomechanical** characterization of **PBO** was focused primarily on the Venusian atmospheric environment.

There are however, a number of potential future missions using a phase change balloon of argon or possibly some mixture for an altitude changing balloon on the Saturn moon, Titan, whose atmosphere is primarily nitrogen. Any number of fluids would work on Uranus and probably Neptune.

Therefore there was a lot of interest to test PBO at liquid nitrogen temperature. A simple test was performed by cutting a 1/4" strip of PBO film in the machine direction and attaching one end of it to a 50g weight. The film was immersed into a full LN₂ dewar while supporting the 50g weight and did not break or become brittle. The 50g weight applied 1.1 ksi to the PBO film which is equivalent to a 100kg gondola on a 4m diameter cylindrical balloon.

Unpublished Foster-Miller **tensile** strength data at liquid nitrogen temperature indicates that the material **retaining** mechanical properties at these temperature.

CTE of PBO Film and **Fiber**

It is well known that Kevlar⁸ and other highly oriented polymers⁹ possess a negative axial CTE. PBO fibers and film also have a negative axial CTE. Because of the special environmental conditions encountered on Venus and in order to reduce gas

permeability a protective coating on the PBO film on fabric of the balloon is necessary. For this reason, although the CTE characteristics of PBO have been studied⁷, it was important for us to determine CTE values of PBO film and fiber to 500°C in order to better understand the effects that the expected temperature excursions might have on a coating applied to the PBO balloon material. ⁷ ~~The~~ diagrams below show the dimension changes of PBO film on fiber as a function of temperature. For the film, after the first heating to 500°C which drove off moisture and may have induced a small increase in crystallinity, subsequent cycles of cooling and heating gave very reproducible values of CTE -7.6 ppm/°C in the machine (axial) direction and a CTE of 7.6 ppm/°C in the transverse direction. For the fiber some initial elongation was seen during the first heating to 500°C this was due to the straightening of the PBO yarn which had been kinked from the weaving process. Again, subsequent cycles of cooling and heating gave a very reproducible CTE of -5.9 ppm/°C.

Thermal Cycling

The PBO film and fiber were subjected to heat cycling in order to test the material under Venusian conditions. Samples of film and fiber were suspended in glass tubes with weights on the bottom of 120g (930 psi) and 25 g (1120 psi) respectively. These loads correspond to gondolas of 80kg for the film and 280kg for the fiber for a 4 meter diameter cylindrical balloon. In one tube the film and fiber were sealed with a vacuum of -10^{-5} Torr to prevent oxidation at higher temperature. The other tube was

filled with CO₂ at ambient pressure and sealed. The CO₂ filled tube was used because the atmospheric composition of Venus at the surface is 96.5% CO₂. The tubes containing the PBO fiber and film were placed in an oven and heated to 500°C at 1.2°C/min. After a 2 hour hold at 500°C the samples were cooled to room temperature at 1.2°C/min. and then held at room temperature for 8-10 hours (see figure 1.) This cycle was repeated 3 more times. At the end of four cycles the PBO film and fiber were still intact, but visibly darker in color. Upon opening the tubes an acrid odor was noticed. Further testing of the cycled material will be performed to determine changes in tensile properties i.e. modulus and strength.

Prior to the cycling test, Thermogravimetric Analysis (TGA) was performed on the PBO film using a TA Instruments 2950 TGA. A 14.5 mg sample was first heated to 100°C under a purge of dry nitrogen to drive off moisture. The sample was then heated at 10°C/min. to 500°C and then held at 500°C for 1 hour. Figure (-) shows a plot of weight loss vs. temperature. The sample lost less than 2% of its starting weight during this test.

Chemical Compatibility Tests

Preliminary chemical compatibility test of PBO film with concentrated and 85% sulfuric acid were performed. Strips of PBO were immersed in these liquids at 21°C. Within minutes the film becomes yellow and dull. Overnight exposure under these conditions it does not deteriorate or carbonize the film. This is expected since ordered polymers like PBO are processed from

their solutions in highly protonated strong acids including polyphosphoric acid, methanesulfonic and chlorosulfonic acid. The sulfuric acid however appears to plasticize the film with total loss of mechanical strengths.

CONCLUSION

The results from these preliminary high temperature thermal mechanical tests of PBO film and fiber on hand indicate that the material has significant retention of its tensile properties at elevated temperature making it a viable candidate for Venus balloon application. Based on results from preliminary low temperature test the material also appears promising for low temperature (LN₂) balloon applications. For the film these conclusions are based on the machine direction results. It is expected that an anisotropic film can be manufactured.

The CTE characteristics of PBO do not present any problems for the Venus balloon application

The Venus mission temperature cycle profile had no catastrophic effect on PBO film and film. The material survived 4 temperature cycles under a significant height. Further testing is required to determine exactly how well the material retained its tensile strength and modulus.

Initial chemical compatibility results of the test involving immersion in sulfuric acid showed that the material was attacked by the acid and indicates that some protective coating is warranted.

ACKNOWLEDGEMENTS

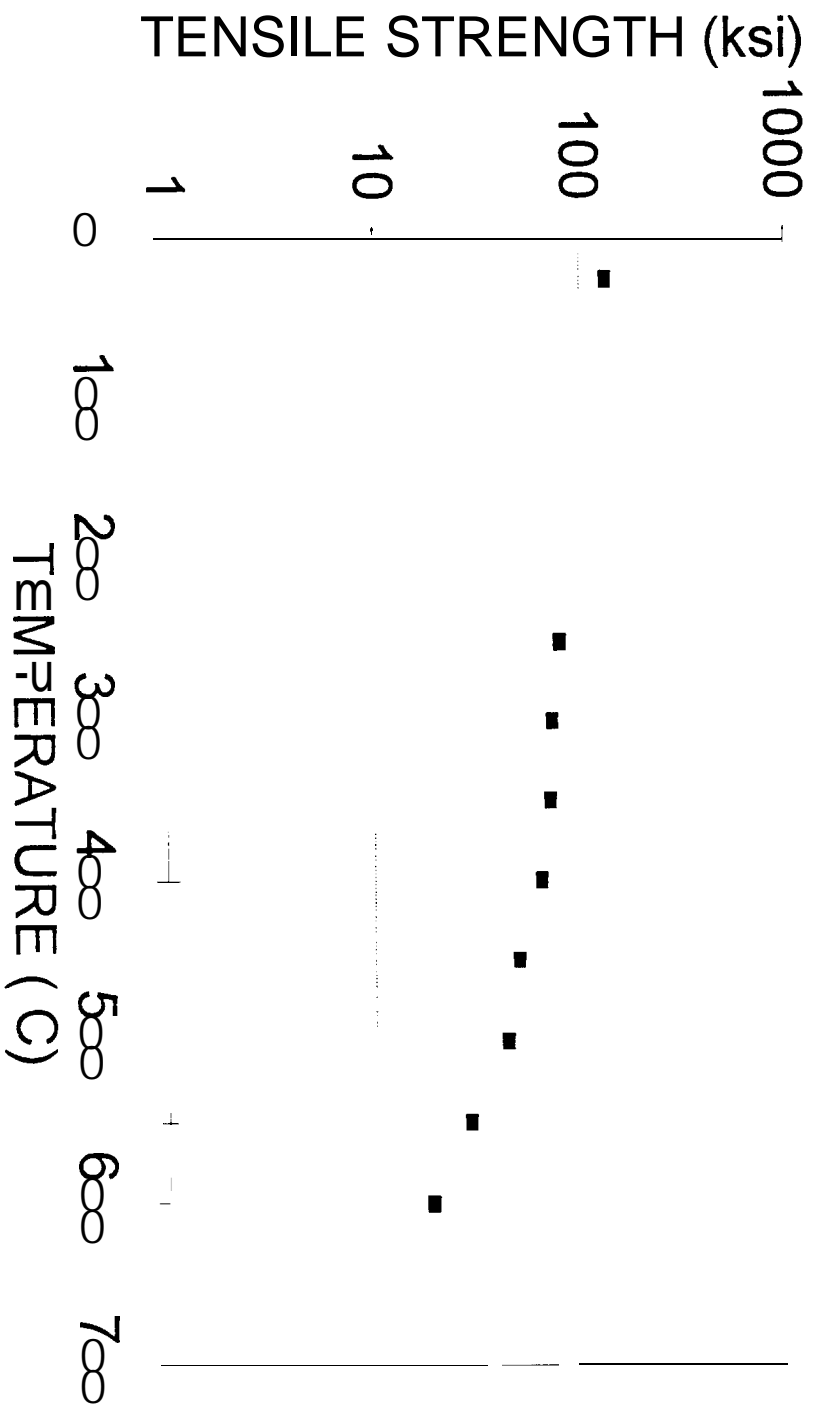
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F.M. PBO FILM MACHINE DIRECTION



PBO FILM
MACHINE DIRECTION

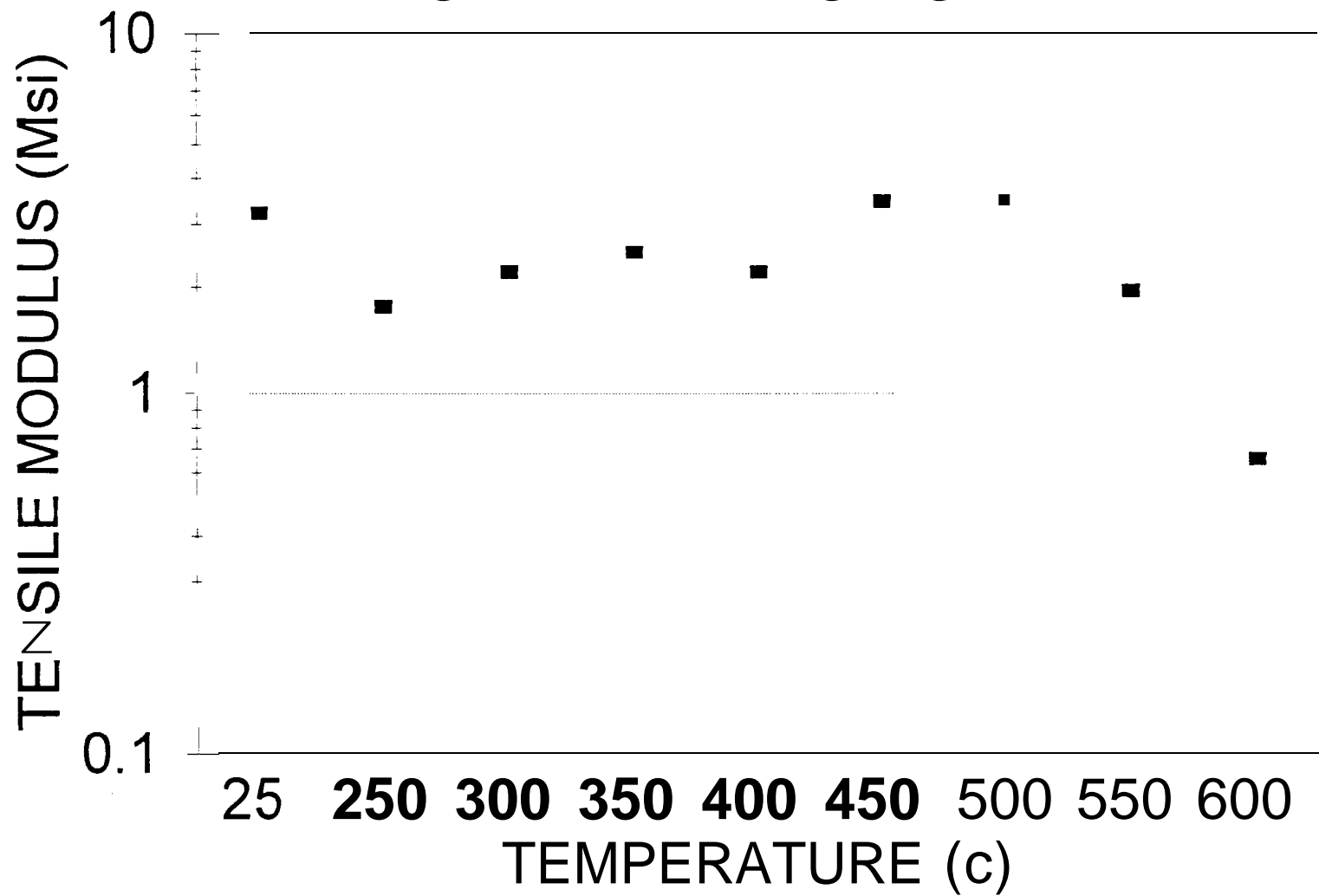


Fig 5

FM PBO FILM

TRANSVERSE DIRECTION

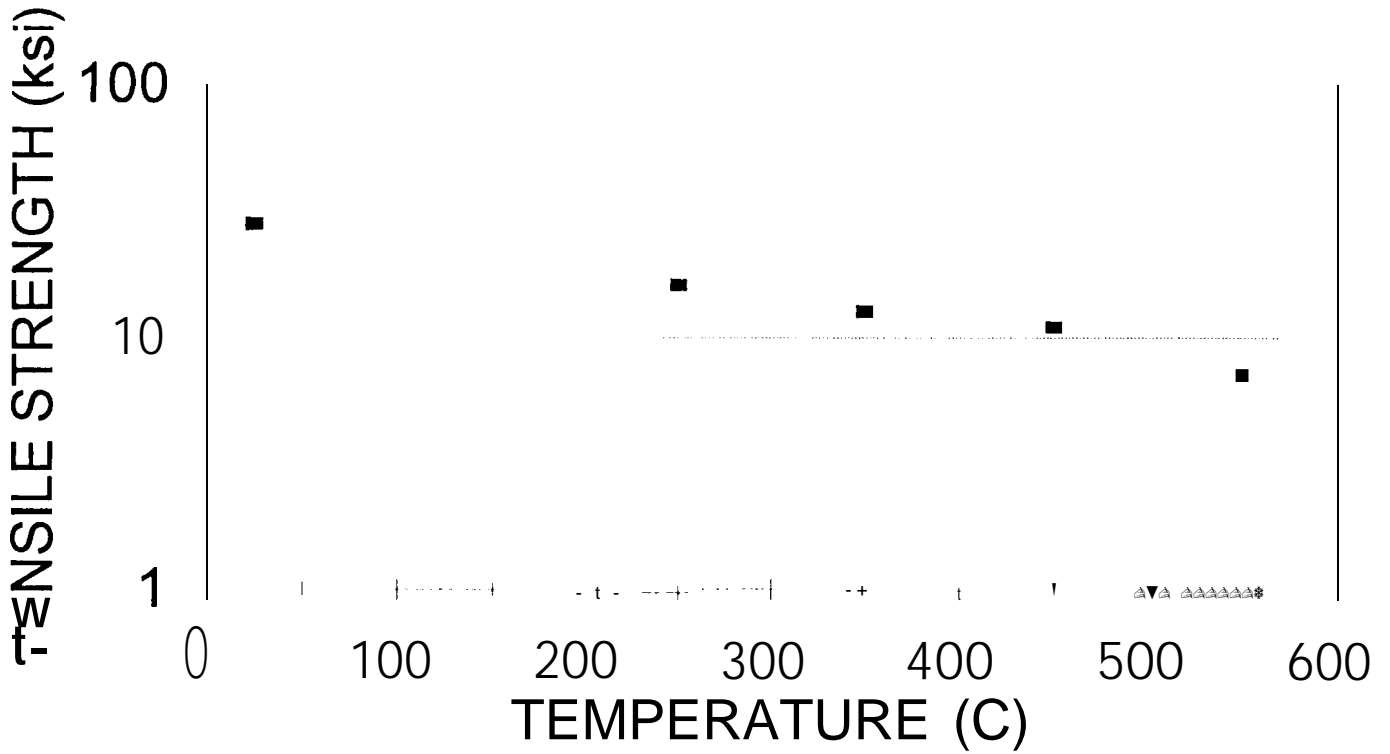


Fig 6

PBO FILM

TRANSVERSE DIRECTION

TENSILE MODULUS (Msi)

1

0.1

0

100

200

300

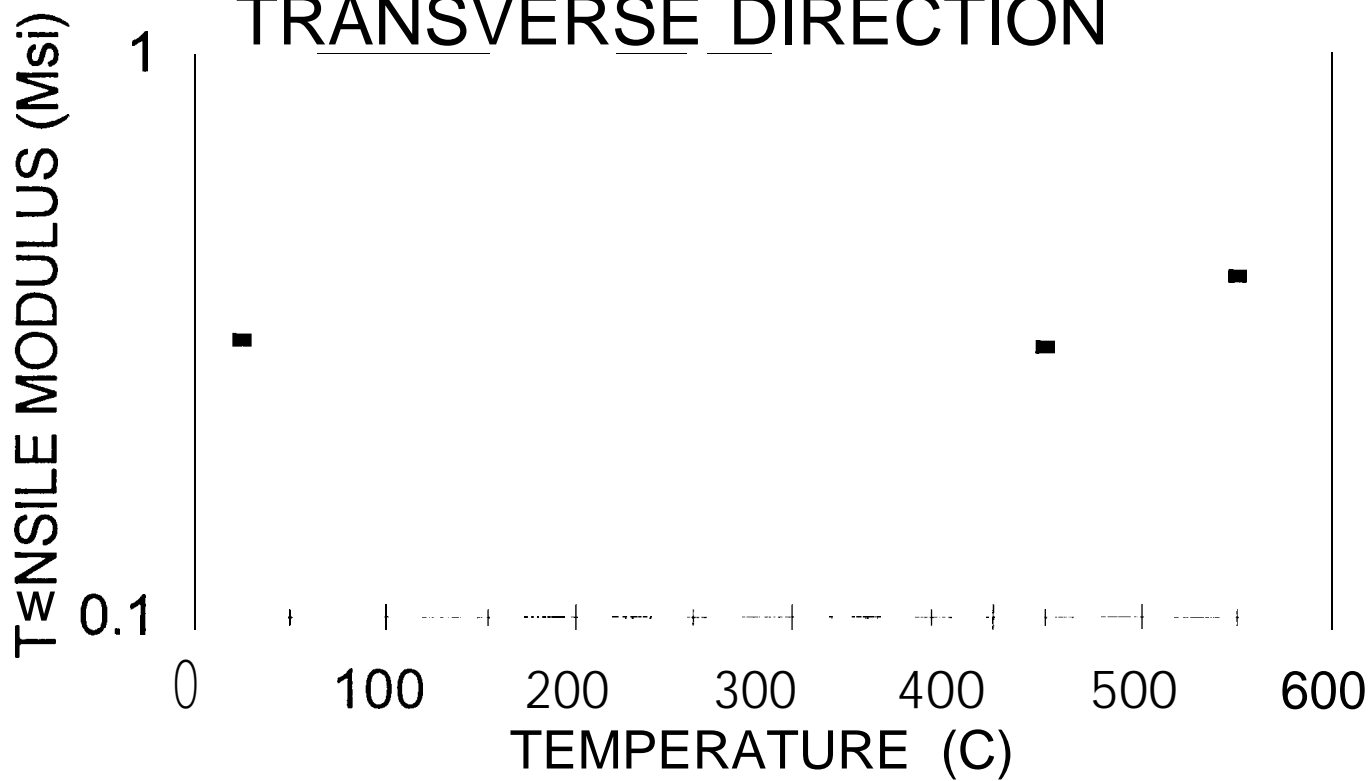
400

500

600

TEMPERATURE (C)

Fig 7



Venus Mission Thermal Cycling

