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PREPARATION OF FOAM COMPOSITES

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for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
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SUMMARY

Methods were developed for the fabrication of fire resistant panels utilizing polybenzimidazole (PBI) and Kerimid 601 resins along with glass, quartz, and Kevlar reinforcements. Stitched truss structure, both unfilled and filled with PBI foam, were successfully fabricated and tested. Second generation structures were then selected, fabricated, and tested, with a PBI/glass skin/PBI foam sandwich structure emerging as the optimum panel concept. Mechanical properties, smoke generation, and fire resistance were determined for the candidate panels.

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1,0 INTRODUCTION

An area of great concern in present-day aircraft is the flammability and offgassing characteristics of the structural materials. Even when an aircraft fuselage survives a crash intact, lives may be lost in the subsequent fuel fire, as flames penetrate the cabin and/or gaseous by-products of combustion overcome the passengers within. A prime objective of NASA-Ames Research Center is to develop aircraft structure that maintains structural integrity and a viable cabin atmosphere for a minimum of 10 minutes under fuel fire conditions. Such structure would provide the time required for a spilled fuel fire to burn out or for fire crews to extinguish the flames.

Another area of concern involves the fires that originate in the rest rooms of commercial aircraft. Such fires, i.e. in trash receptacles, etc., often remain undetected until they develop into major conflagrations and pose a serious threat to the lives of those aboard. Here, the fire must be contained to the rest room and must not penetrate into the main cabin, while the evolution of poisonous gasses from the burning structure cannot be tolerated.

These stringent performance requirements are not the only considerations that must be taken into account. For a design/material concept to be viable, its economics must be attractive to the prime aircraft manufacturer. Light weight (low density) is another imperative characteristic. In addition, many applications require highly cosmetic structures with smooth, even surfaces or attractive color schemes needed for the concept to achieve wide acceptance. The following report details the efforts by Whittaker Corporation, Research and Development Division (WRD) to develop attractive, moderately priced aircraft panel concepts that exhibit outstanding fire resistance and performance in a fuel fire environment.

2.0 TECHNICAL DISCUSSION

2.1 Initial Concepts

Detailed design of aircraft structure was beyond the scope of this program. Work was limited to the basic simple concept of a flat panel unit. Early tests of conventional sandwich panels by NASA-Ames showed a tendency for panels to delaminate; i.e. for the skin to warp and debond, under impingement of the heat flux.

The general opinion was that a three-dimensional truss structure with the skin mechanically attached to the truss cross members would be required to maintain structural integrity throughout a 10-minute exposure to fuel fire conditions.

An obvious and direct approach is to utilize woven 3D reinforcements. A typical woven panel configuration is shown in Figure 2-1.

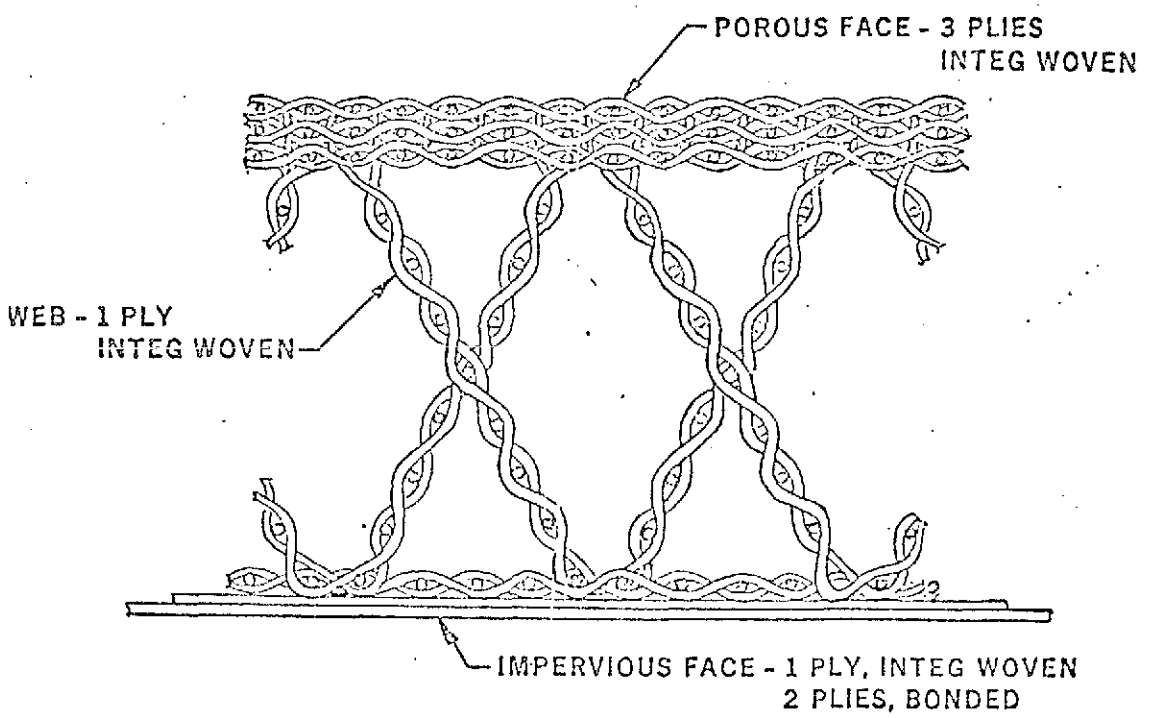

Figure 2-1. Panel Construction - Integrally Woven Fiberglass

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Note the integral construction with the cross members and skins woven as one unit. Unfortunately, such structures proved too costly to be included in the program. High set-up charges, long lead times, and high material costs led to the decision to seek an alternate approach to 3D structure.

An attractive approach was then developed. It involved a stitched structure with the same general configuration of the woven 3D truss. The use of a one-ply fiber reinforced skin stitched as a 3D truss is economical, light weight, and lends itself readily to automated processing. Figure 2-2 shows a rough schematic for the fabrication of stitched 3D structure.

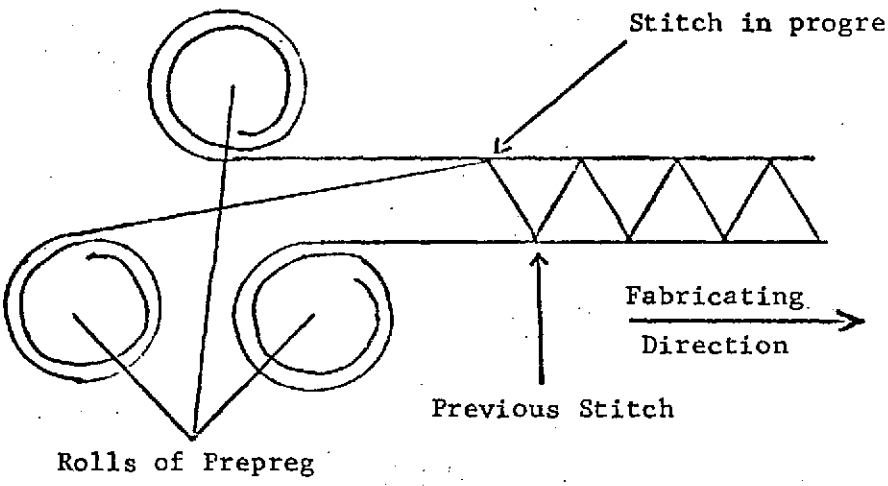

Figure 2-2. Schematic for Fabrication of Stitched 3D Structure

It was possible that such structure in itself would prove an effective fire barrier if suitable resins and reinforcements were chosen. It was also obvious that filling the truss sections with polybenzimidazole (PBI) foam would greatly increase the insulation characteristics of the final structure (retard heat transfer, etc.) and in general improve performance and enhance the fire resistance of the panels. Figure 2-3 shows such a structure.

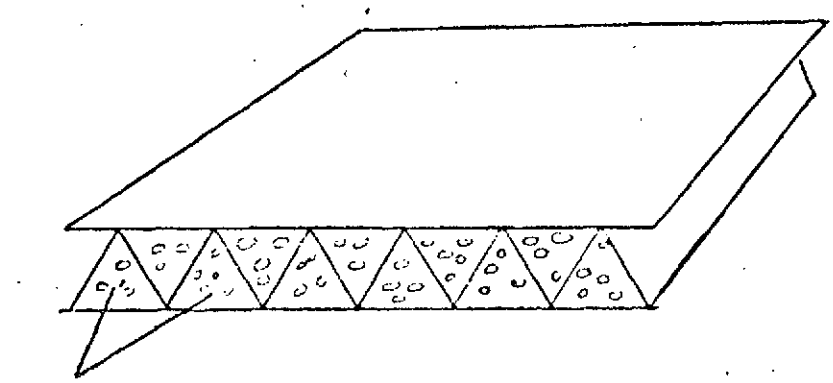

Figure 2-3. PBI Foam Filled Truss Structure

The trade-off would be additional material and fabrication cost versus improved fire resistance and performance. The empty and PBI foam filled stitched truss structures were therefore selected as the benchwork concepts for this study. The target densities for the final structures are 96.1 and 134.6 kg/m^3 (0.5 and 0.7 lb/ft^3 of 1 in. thick structure), respectively.

2.2 Material Selection

2.2.1 Resins

The candidate resins for the work were identified by NASA-Ames and again represent a trade-off of cost versus performance. Thermally stable, high char forming polymers are needed for such structures, but unfortunately high performance is usually matched or exceeded by high cost. Figures 2-4 and 2-5 summarize typical correlation of performance, cost, and structure.

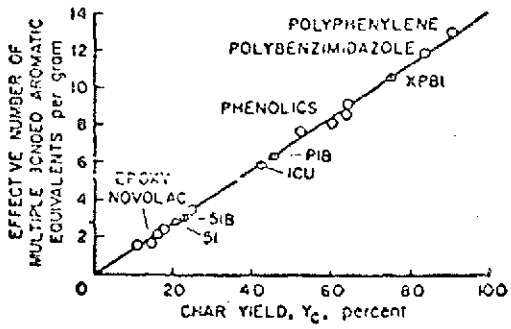

Figure 2-4. Correlation of Primary Thermomechanical Char Yield with Molecular Structure

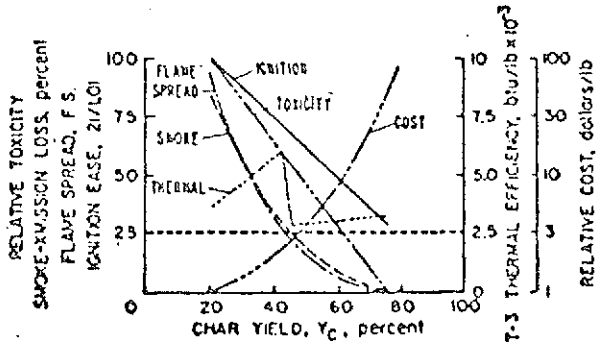

Figure 2-5. Summary of Properties of Char Forming Foamed Polymers

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The high performance resin selected for truss fabrication was the polybenzimidazole (PBI) system. The lower performance/lower cost resin was Kerimid 601, a bismaleimide type polymer. Figures 2-6 and 2-7 illustrate the chemistry involved with PBI and Kerimid 601 resins

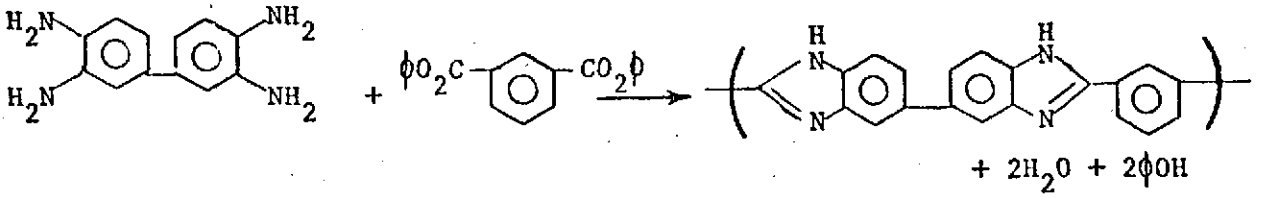


Figure 2-6. Typical PBI Synthesis

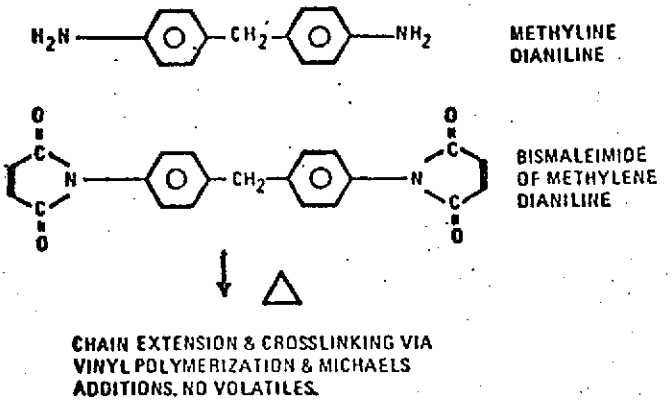


Figure 2-7. Kerimid 601 Chemistry

In addition, the PBI polymer was selected as the foam material for use in this study

2.2.2 Reinforcements

The selection of reinforcements was quite straightforward. Glass was the prime candidate as it is low cost, readily available, and has high temperature capability. Kevlar 49 fabric was included, as its low density offered an opportunity to achieve significant weight savings. Other materials such as quartz, graphite, fiberfax paper, alumina/boron/silica, and zirconium/silicon fibers were available if the high temperature performance of glass and Kevlar 49 proved inadequate.

2.3 Fabrication of First Generation Structure

2.3.1 Initial Flat Laminates

Small (15.3 cm x 15.3 cm), 4-ply, flat laminates were fabricated from various resin/reinforcement combinations to confirm curing cycles for the resin systems, check compatibility with the various reinforcements, and to provide display pieces for the NASA-Ames exhibit at the 19th Annual SAMPE Symposium held April 23 - 25 at Buena Park, California. Panels were fabricated with the following compositions:

(1) Kevlar 49/PBI

(2) Kevlar 49/Kerimid 601

(3) E Glass/Kerimid 601

(4) Quartz/PBI

In all cases 181-style weaves were used.

The PBI prepreg was made to WRD specifications, while Kerimid was processed to low (3 - 5%) volatile contents as were used in the truss structures. Curing cycles were as follows:

Kerimid 601 - Bag for autoclave cure. Heat at 1 - 2°K (2 - 4°F) per minute to 455°K (360°F) under vacuum and 689 KN/m^2 (100 psi). Hold 1 hour at 455°K (360°F), cool. Postcure unrestrained for 16 hours at 511°K (450°F) in air.

PBI - Place in press at ambient temperature; heat under contact pressure at 1 - 2°K (2 - 4°F) per minute to 450°K (350°F), apply 1379 KN/m^2 (200 psi) pressure; heat in 28°K (50°F) increments to 642°K (700°F), holding for 30 minutes at each increment. Hold 1 hour at 642°K (700°F), cool.

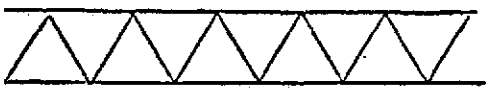
All laminates appeared well consolidated and of good quality. The Kevlar 49 darkened under the PBI cure cycle, but no quantitative evaluation of this change was made.

2.3.2 Truss Structure

The next goal of the program was to fabricate a series of twelve 3D first generation truss structures for test and evaluation. Table 2-1 lists the six initial types (2 structures per type) to be fabricated. Panel dimensions of 30.5 cm x 30.5 cm (12 in. x 12 in.) were required for this initial evaluation.

TABLE 2-1

SIX STRUCTURES FOR INITIAL FIRE PROTECTION EVALUATION

 Empty Truss Structure Target Density 96.1 kg/m^3 (.5 lb/ft^2 of 1 in. thick)	 PBI Foam Filled Truss Structure Target Density 134.6 kg/m^3 (.7 lb/ft^2 of 1 in. thick)
Compositions: 1. Resin: Kerimide 601 Reinforcement: Kevlar 49 2. Resin: Kerimide 601 Reinforcement: E Glass 3. Resin: PBI Reinforcement: E Glass	Compositions: 4. Resin: Kerimide 601 Reinforcement: Kevlar 49 Filler: PBI Foam 5. Resin: Kerimide 601 Reinforcement: E Glass Filler: PBI Foam 6. Resin: PBI Reinforcement: E Glass Filler: PBI Foam

An initial full-size truss structure was fabricated using one ply of 181 E glass/PBI prepreg. The apex of each triangular section was hand-stitched to the top and bottom skin plies using glass thread. Triangular shaped mandrels were cast of high temperature silicone rubber for the cure. It was planned to use the pressure generated by the thermal expansion of the silicone rubber during cure to consolidate the truss structure.

The initial cure with mandrels went to only 560°K (550°F). A later full postcure to 728°K (850°F) was accomplished with maranite mandrels.

A second, well consolidated truss was fabricated with this procedure, but two problems arose. First, the hand-stitching process was far too slow and laborious even for the purposes of the test program. Secondly, significant deterioration of the silicone rubber mandrels was observed, even in a 560°K (550°F) cure. The latter problem was readily solved by the use of aluminum mandrels for subsequent cures. An alternative to the hand-stitching was also found without major difficulty. An industrial

sewing machine, shown in Figure 2-8, was rented for the remainder of the program.

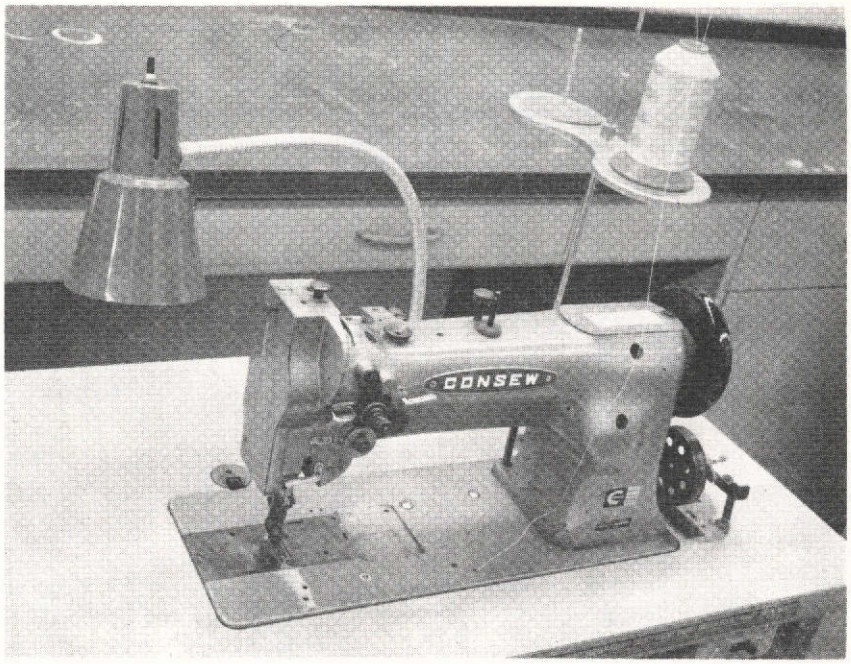


Figure 2-8. Industrial Sewing Machine

Glass thread was also readily available for this work. A detailed procedure for the rapid stitching of one ply of prepreg to form the truss structure was then developed and is attached as Appendix A.

The 181 E glass/PBI, 181 E glass/Kerimid 601, and Kevlar 49/Kerimid 601 structures (4 each) were then stitched, using the procedure and equipment described above. Aluminum mandrels were then used for initial cures below 560°K (550°F). Maranite mandrels were used for postcures of unfilled truss structures, while PBI foam triangular blocks were loaded into the remainder during the postcure. The truss matrix resin (PBI or Kerimid 601) was used to bond the foam blocks into place.

Figures 2-9 and 2-10 show the maranite mandrels and foam blocks, respectively. Figure 2-11 shows a cured Kevlar 49/Kerimid 601 truss, while Figure 2-12 shows an unfilled 181 E glass/PBI truss with both maranite mandrel and PBI foam block for comparative purposes. Figure 2-13 shows a cured 181 E glass/Kerimid 601 foam filled truss after cure but before trimming.

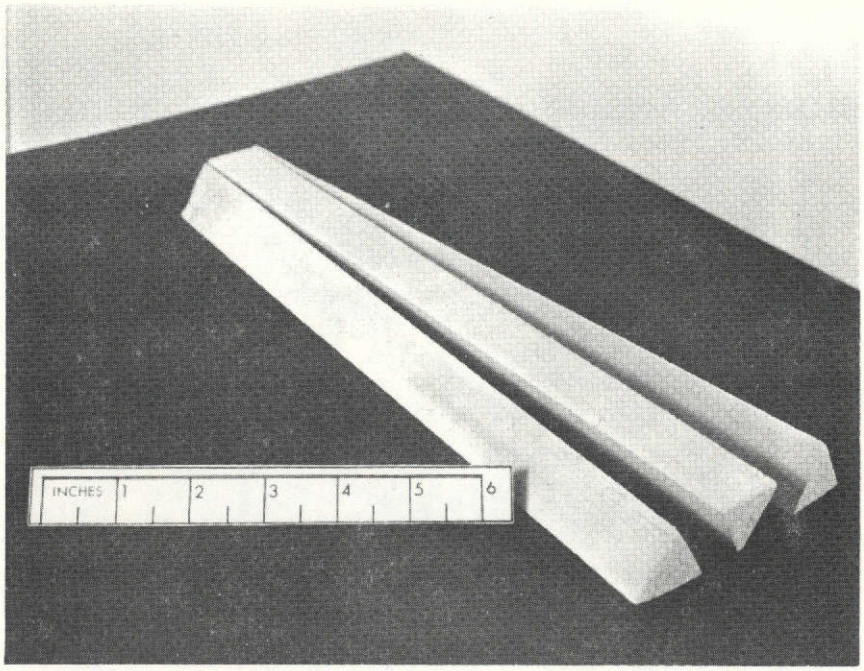


Figure 2-9. Maranite Mandrels

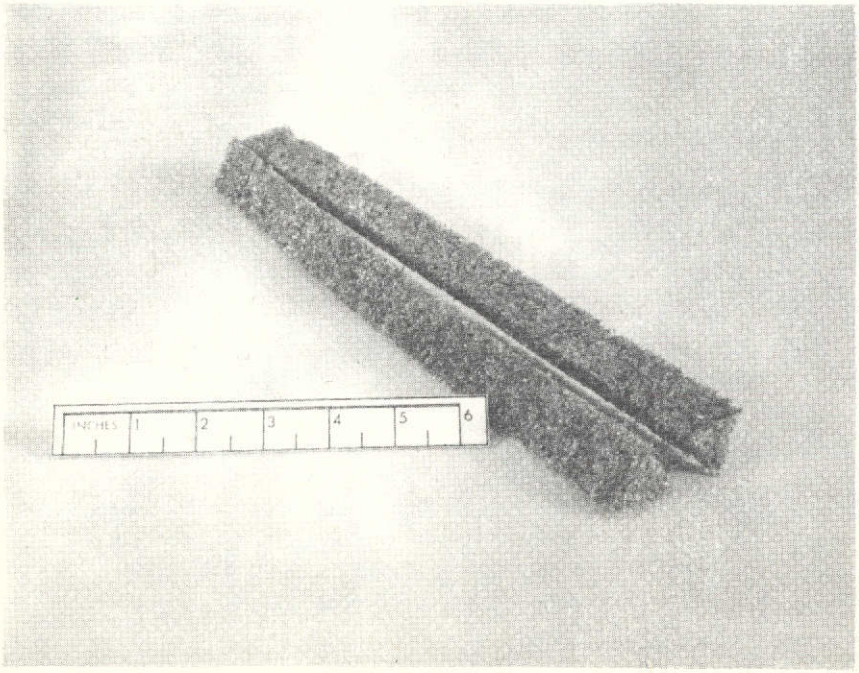


Figure 2-10. PBI Foam Blocks

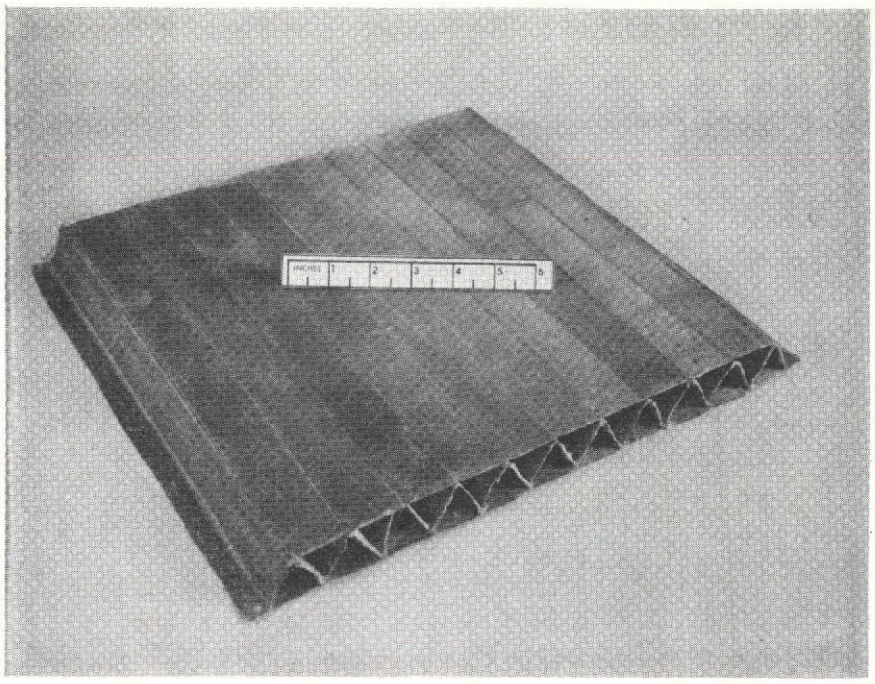


Figure 2-11. Unfilled Kevlar 49/Kerimid 601 Truss

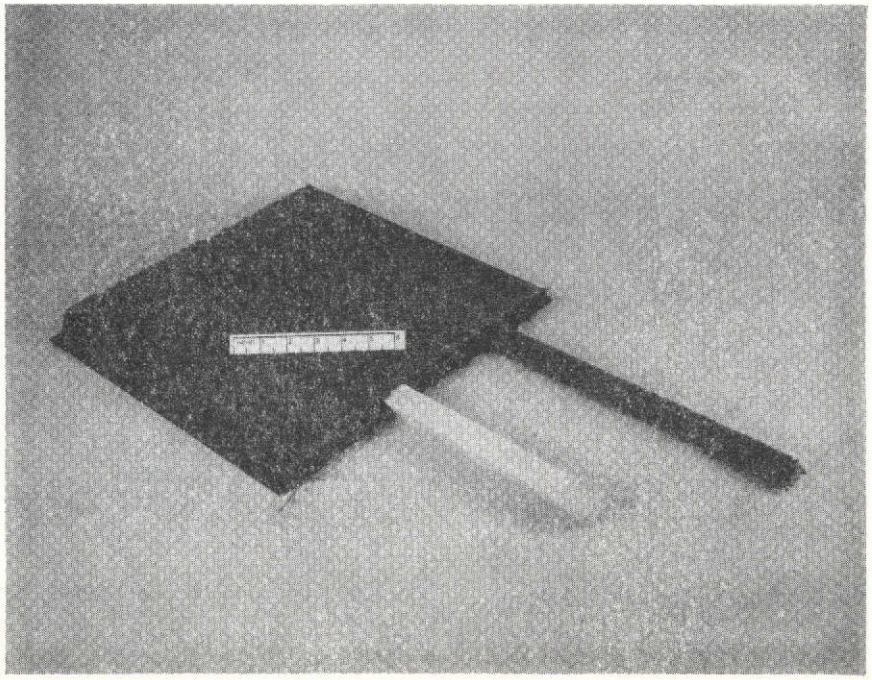


Figure 2-12. 181 E Glass/PBI Truss with Maranite Mandrel and PBI Foam Block

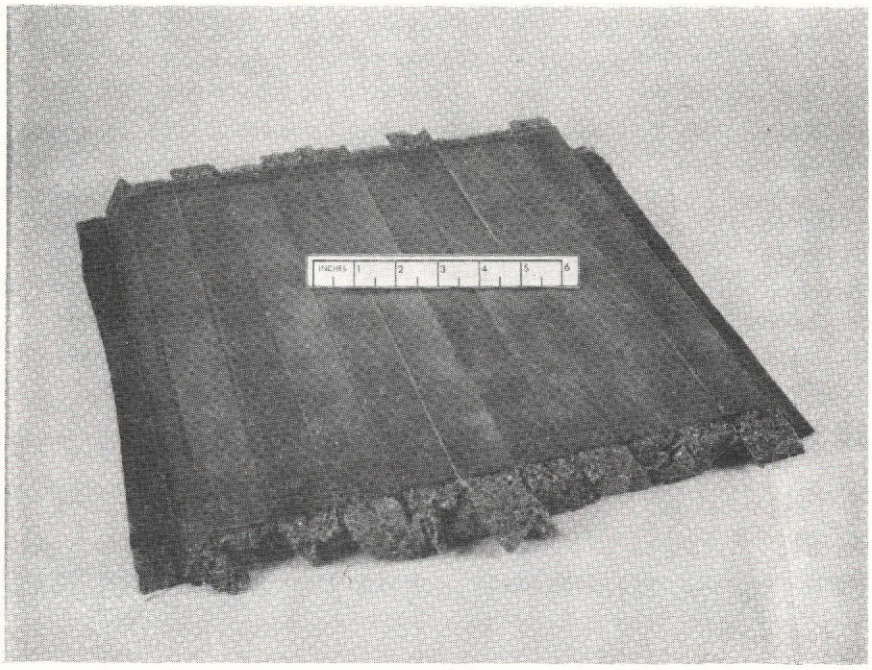


Figure 2-13. Cured 181 E Glass/Kerimid 601 Foam Filled Panel

The following cure schedules were used.

PBI Matrix Truss Cure - The initial step cure was accomplished using triangular aluminum mandrels and the following cure cycle:

> Place in press at ambient temperature; heat under contact pressure at 1 - 2°K (2 - 4°F) per minute to 450°K (350°F), apply 1379 KN/m^2 (200 psi) pressure; heat in 28°K (50°F) increments to 560°K (550°F), holding for 30 minutes at each increment. Hold 1 hour at 560°K (550°F), cool.

At this point the aluminum mandrels were removed. For the two foam filled structures, the PBI foam was coated with powdered PBI adhesive and placed in the truss. For the two unfilled structures, maranite mandrels were now inserted for the postcure in nitrogen. All structures were postcured as follows:

> Heat to 560°K (550°F), hold 1 hour. Heat in 28°K (50°F) increments to 728°K (850°F), holding 30 minutes at each increment. Hold 1 hour at 728°K (850°F), cool.

Kerimid 601 Matrix Cure - Aluminum mandrels were used for the initial cure of all structures. The following cure cycle was used:

> Heat at 1 - 2°K (2 - 4°F) per minute to 464°K (375°F) under vacuum and 689 KN/m^2 (100 psi). Hold 1 hour at 464°K (375°F), cool.

At this point, the aluminum mandrels were removed. For the PBI foam filled structures, the foam was coated with Kerimid 601 lacquer and placed in the structures. Maranite mandrels were placed in the unfilled structures for postcure.

All structures were then postcured under contact pressure for 16 hours at 511°K (450°F) in nitrogen.

Table 2-2 gives the densities and resin contents of the final structure.

2.4 Testing of First Generation Structure

2.4.1 Fire Resistance Testing

2.4.1.1 Panel Preparation. The twelve truss structures as described previously (two structures each of six types) were prepared for testing in the T3 tester, shown in Figure 2-14. Chromel alumel thermocouples were used. Figures 2-15, 2-16, and 2-17 show the placement of thermocouples on the filled and unfilled truss structures.

TABLE 2-2

PHYSICAL PROPERTIES

Sample Description/ Configuration	Panel No.	Density, kg/m^3 (lb/ft^2 of 1 in. Structure)	Truss Resin Content (%)
PBI/Glass, unfilled	1	75 (.39)	35.1
	3	77 (.40)	31.3
PBI/Glass, PBI foam	5	131 (.68)	30.2
	6	129 (.67)	37.0
Kerimid/Glass, unfilled	9	87 (.45)	37.4
	10	90 (.47)	37.6
Kerimid/Glass, PBI foam	11	121 (.63)	33.2
	12	121 (.63)	37.4
Kerimid/Kevlar, unfilled	2	58 (.30)	54.4*
	4	52 (.27)	35.0*
Kerimid/Kevlar, PBI foam	7	98 (.51)	38.1*
	8	100 (.52)	44.4*
PBI Skin, 96 kg/m^3 (6 lb/ft^3) PBI core, no truss	13		32.6

* Calculated from weight data, as any known method that digests the Kerimid 601 matrix also attacks Kevlar 49.

Note that the target goals were met or exceeded in every case.

The additional panel, no. 13, will be described in the following section.

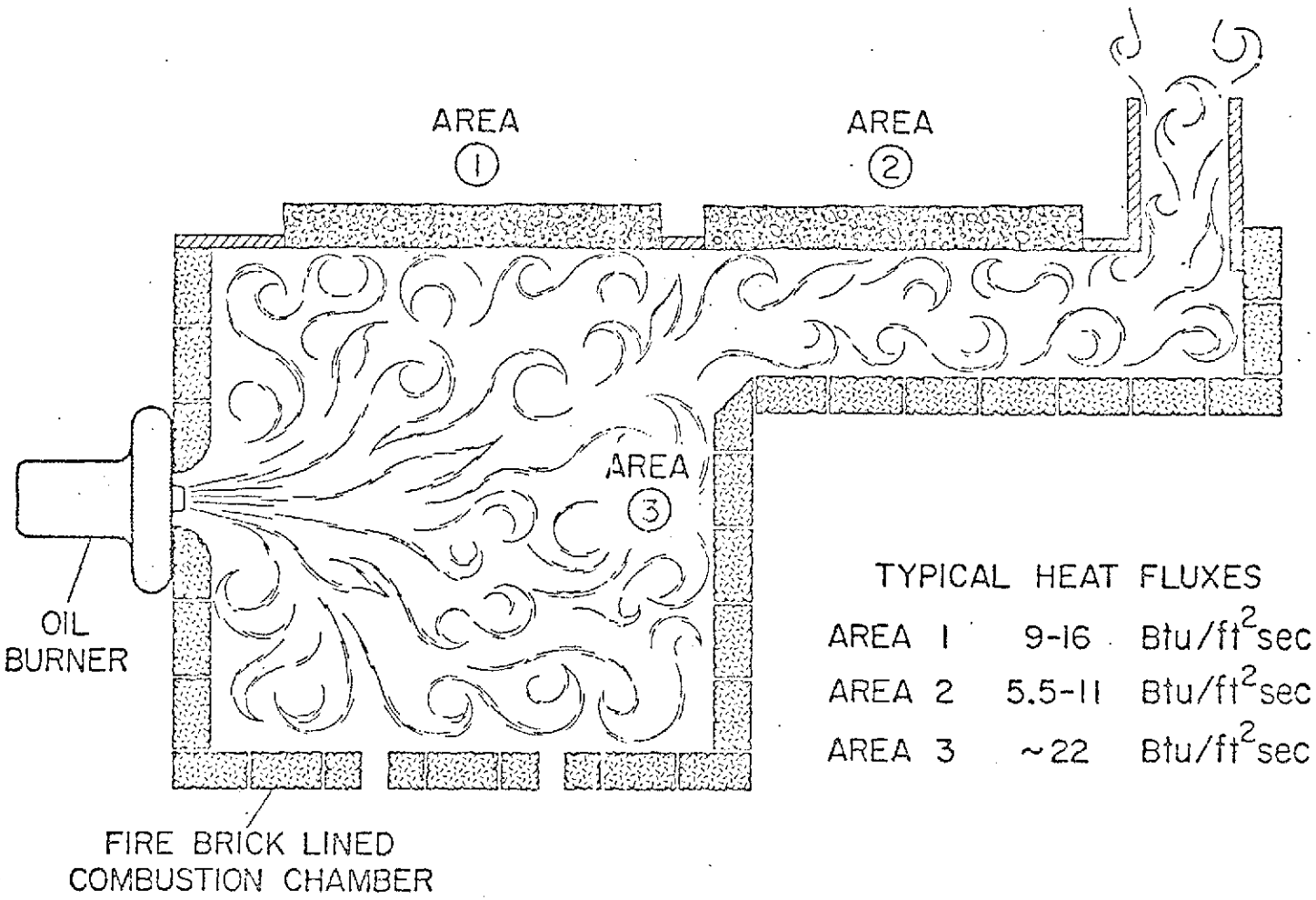


Figure 2-14. Ames T3 Thermal Test Facility
JP-4 Fuel

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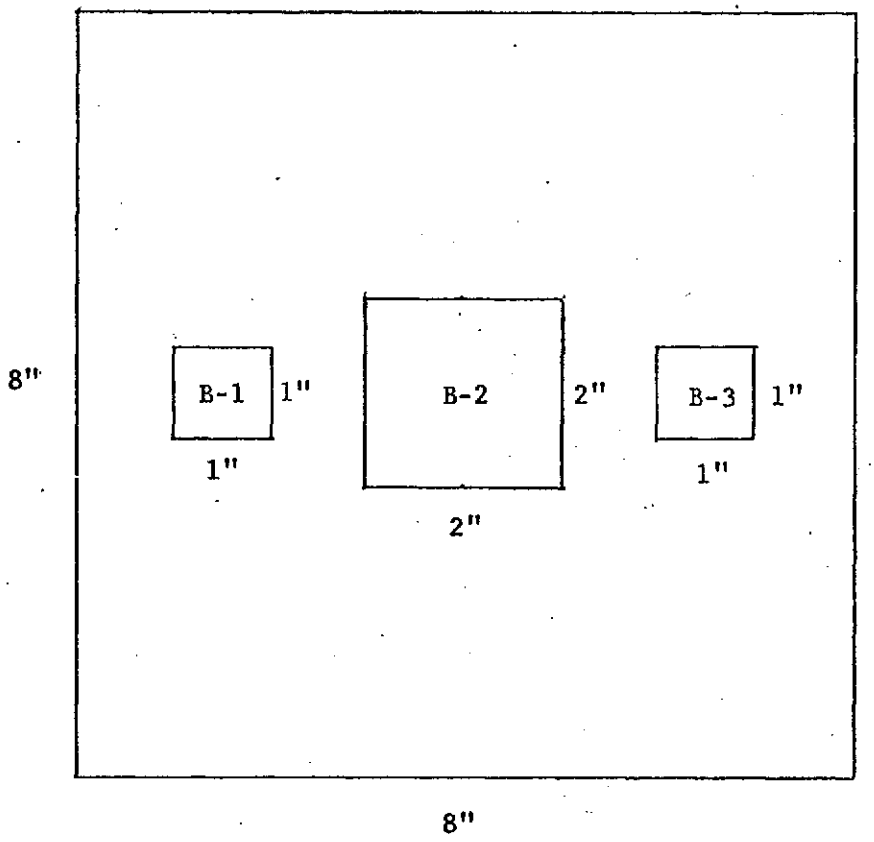

Figure 2-15. Back Side Thermocouple Placement, All Structures

Figure 2-16. Thermocouple Placement, Foam Filled Panels

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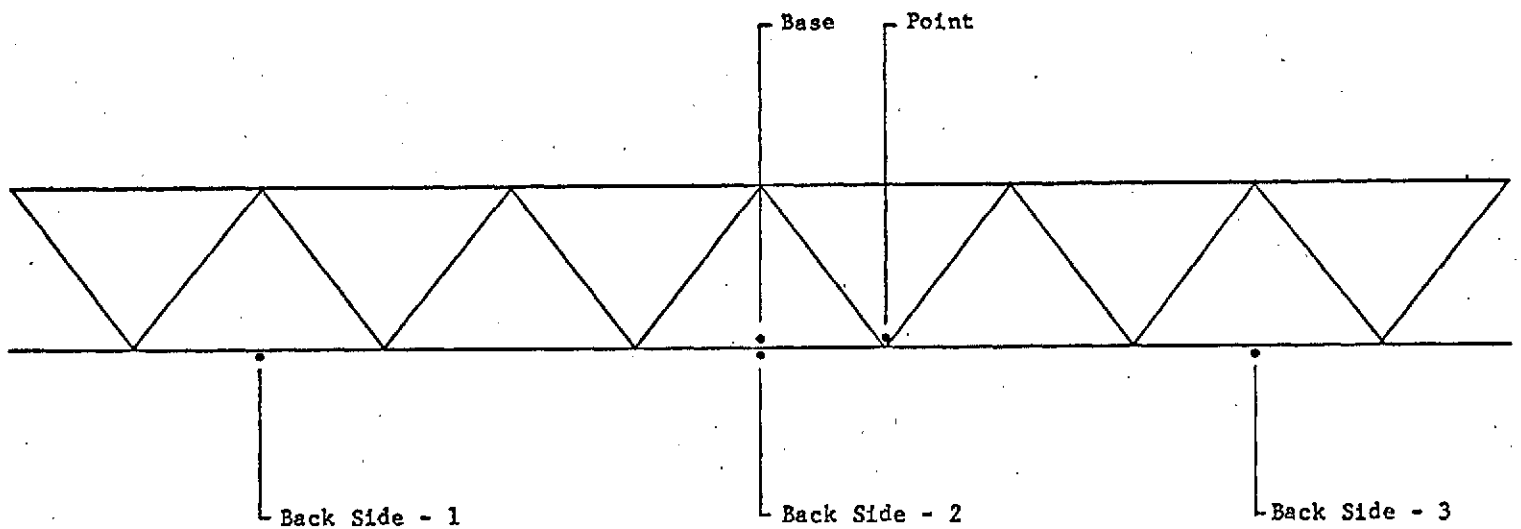

Figure 2-17. Thermocouple Placement, Unfilled Panels

All thermocouple leads were bonded in place using Briskeat ceramic adhesive, and cured 2 hours at 339°K (150°F), with the exception of those placed directly into the PBI foam. The latter were held in place by simple mechanical friction. A 5.08 cm x 5.08 cm (2 in. x 2 in.) aluminum patch was bonded over the center back side thermocouple, while a 2.54 cm x 2.54 cm (1 in. x 1 in.) patch was bonded on all other external thermocouples.

An additional panel, no. 13, was fabricated and prepared for testing. This panel was based on a simple skinned sandwich approach; no truss structure was used. A 96 kg/m^3 (6 lb/ft^3) PBI foam core was used in conjunction with 1-ply, 181 glass reinforced PBI skins. The skins and foam were precured to 645°K (700°F). PBI adhesive was applied as a powder, and the structure was cocured to 728°K (850°F) using the PBI cure cycle. Thermocouples were attached in the same manner as for the truss structure. Their location is shown in Figure 2-18. This panel was fabricated to determine whether a simplified structural sandwich would exhibit adequate performance. Such a structure would be much simpler and cheaper to fabricate than woven or stitched truss panels, while exhibiting good mechanical properties. The skinned foam panel no. 13 appeared stiff, strong, and of good quality. In addition, a smooth cosmetic surface was achieved, which would prove extremely difficult to accomplish with a stitched truss structure. The question to be answered was whether this panel would debond when tested in the T3 tester.

2.4.1.2 Thermal Testing. The panels were taken to NASA-Ames for testing by Ames technicians. Both WRD and NASA-Ames personnel observed the tests. It was decided to test one each of the six panel types and the skinned foam experimental specimen. The remaining panels, also one each of the six types, were retained by NASA-Ames for later testing in an aluminum-backed configuration.

Table 2-3 gives a description of the panels along with qualitative comments and observations of those tested. Figures 2-19, 2-20, and 2-21 compare performance of foam filled vs. empty structures, while Figures 2-22 and 2-23 compare performance of various structure types. Figures 2-24 through 2-30 show the performance of the individual panels tested.

A number of conclusions can be drawn from these initial tests:

(1) The performance of unfilled structures was inadequate. Even in the best case of PBI/glass, the back side temperature was too high, i.e. near 700°K (800°F).

(2) Kevlar reinforced Kerimid 601 is not suitable for these applications. The structures burned vigorously under the test conditions.

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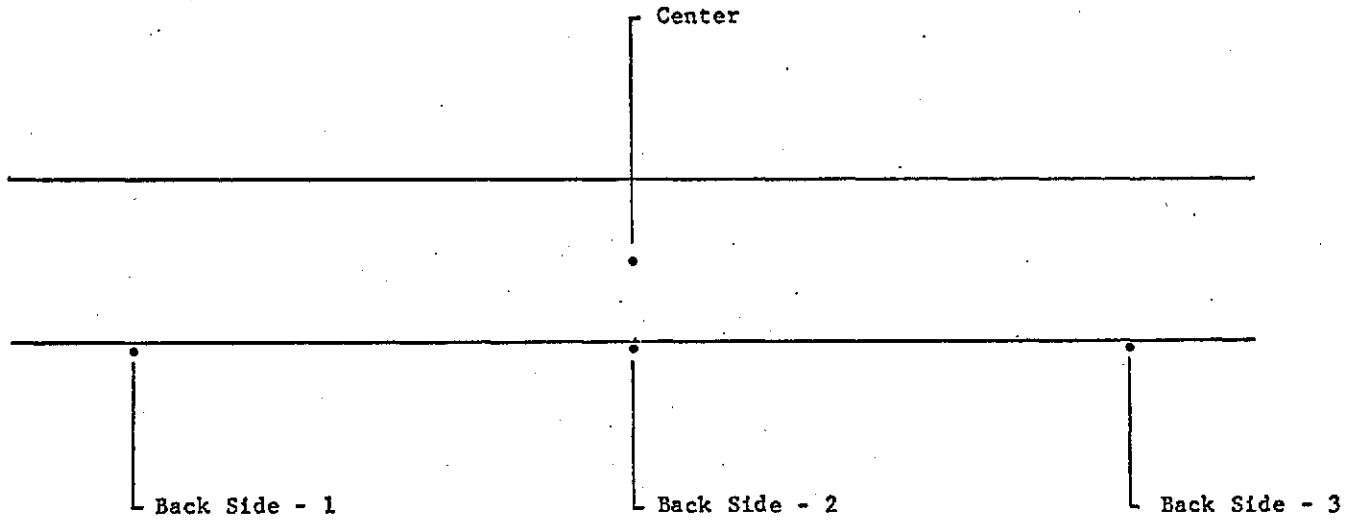

Figure 2-18. Thermocouple Placement, Skinned Sandwich

TABLE 2-3

T3 BURN TESTS

Sample Description/Composition	Panel No.	Comments
PBI/Glass, unfilled	1	Almost no smoke or distortion the entire run. Holds together very well, with the usual burn-away of resin off the center of the fire face. (Note: No. 3 thermocouple was replaced by a contact pyrometer on the back side.)
PBI/Glass, PBI foam	6	Little smoke, moderate distortion (significantly less than Kerimid), good stability to the end of the run. The center of the fire face is burned clean of resin.
Kerimid/Glass, unfilled	9	Severe smoking, the specimen warps but stays together for the test.
Kerimid/Glass, PBI foam	12	Some warping (normal), resin burned clean from glass at the end of 10 minutes, virgin foam left at the end of the 10-min. test.
Kerimid/Kevlar, unfilled	4	Splits, smokes, then ignites. The face is gone and ignition of back occurs in 20 seconds. Complete burn-through and consumption in 1 minute.
Kerimid/Kevlar, PBI foam	8	Smokes, ignites at fire face in 50 seconds. Face burns away, but structure stays intact. Some fissuring of foam, but no burn-through in 10 minutes.
PBI Skin, 6 lb/ft^3 PBI core, no truss	13	Little smoking or initial surface effects, good dimensional stability, good integrity the entire 10-min. run. Resin cleaned from the center of the fire face by the end of the run. (Note: No. 13 is a PBI/181 glass skin, 1 in., 6 lb/ft^3 PBI core, no truss, no stitching, bonded with PBI adhesive.)

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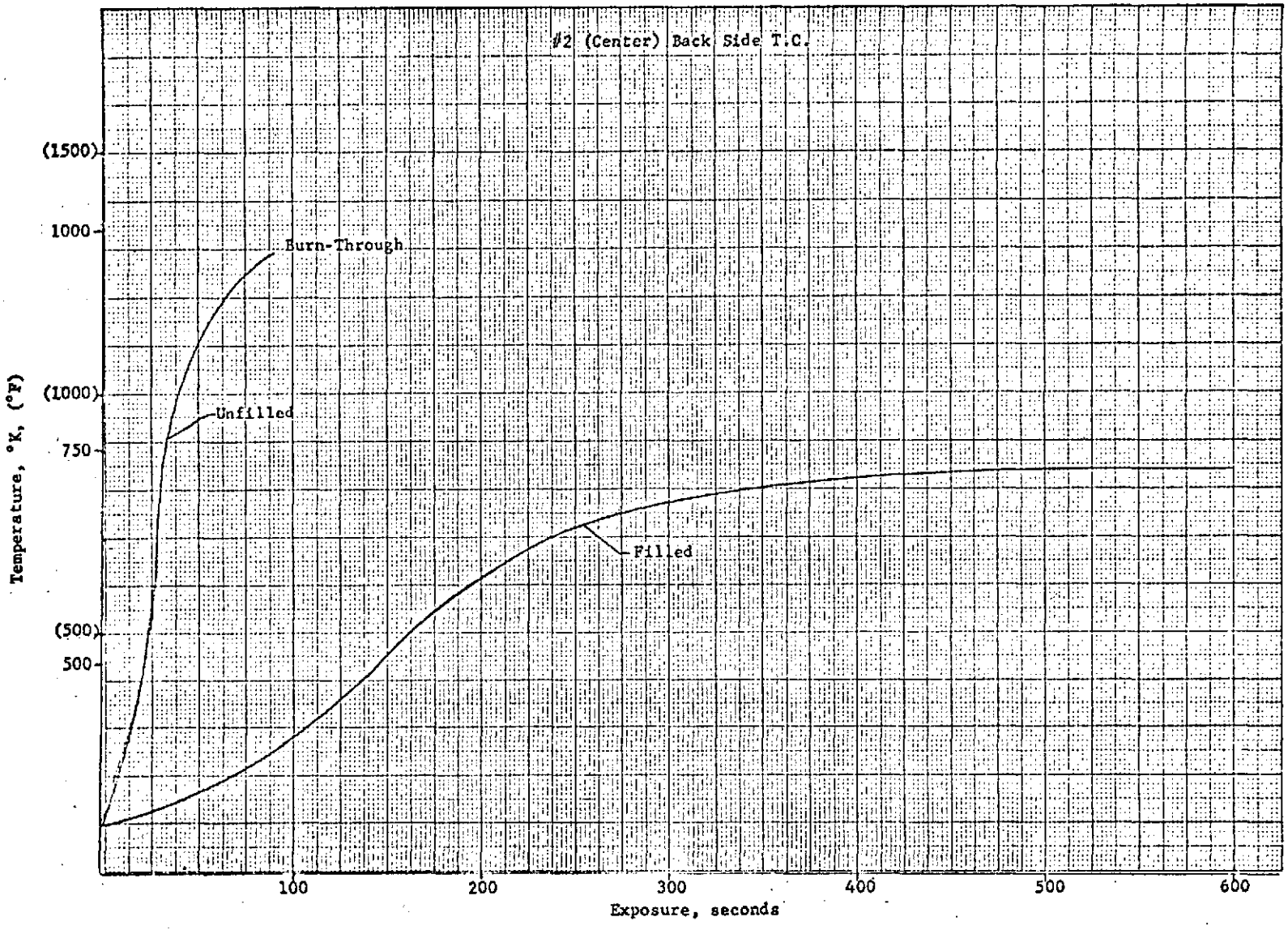

Figure 2-19. Kerimid/Kevlar, Filled vs. Unfilled

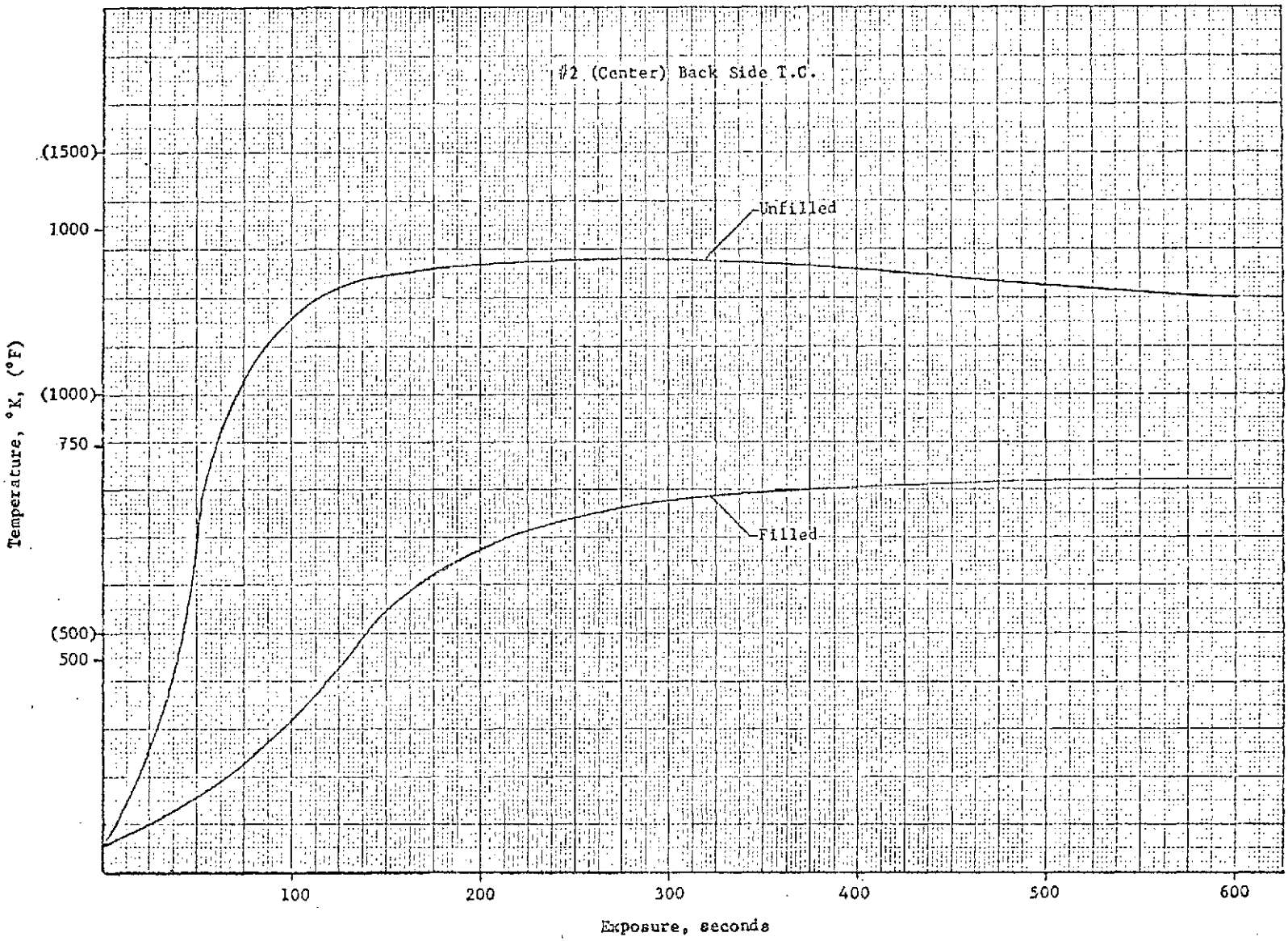

Figure 2-20. Kerimid/Glass, Filled vs. Unfilled

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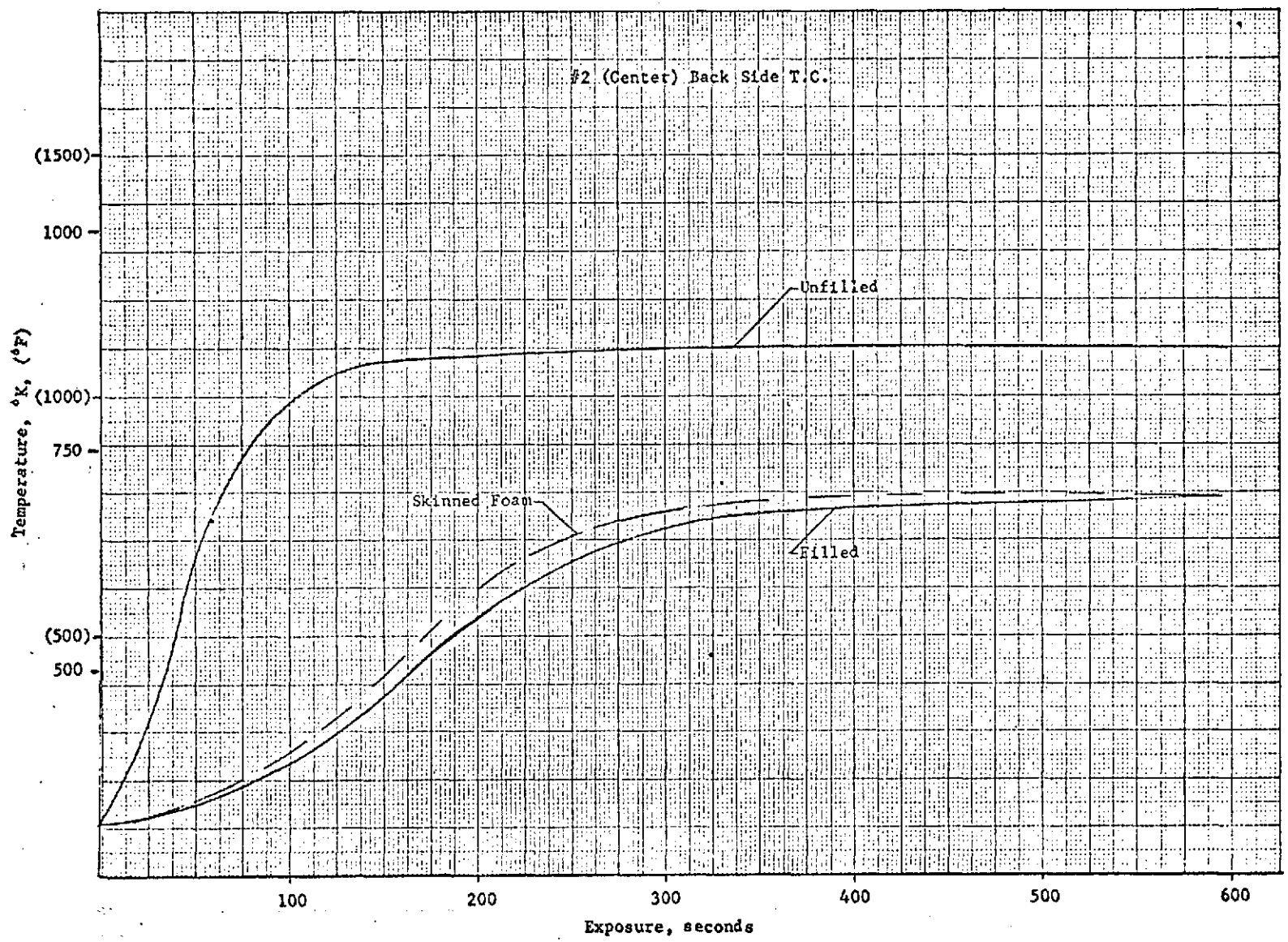

Figure 2-21. PBI/Glass, Filled vs. Unfilled

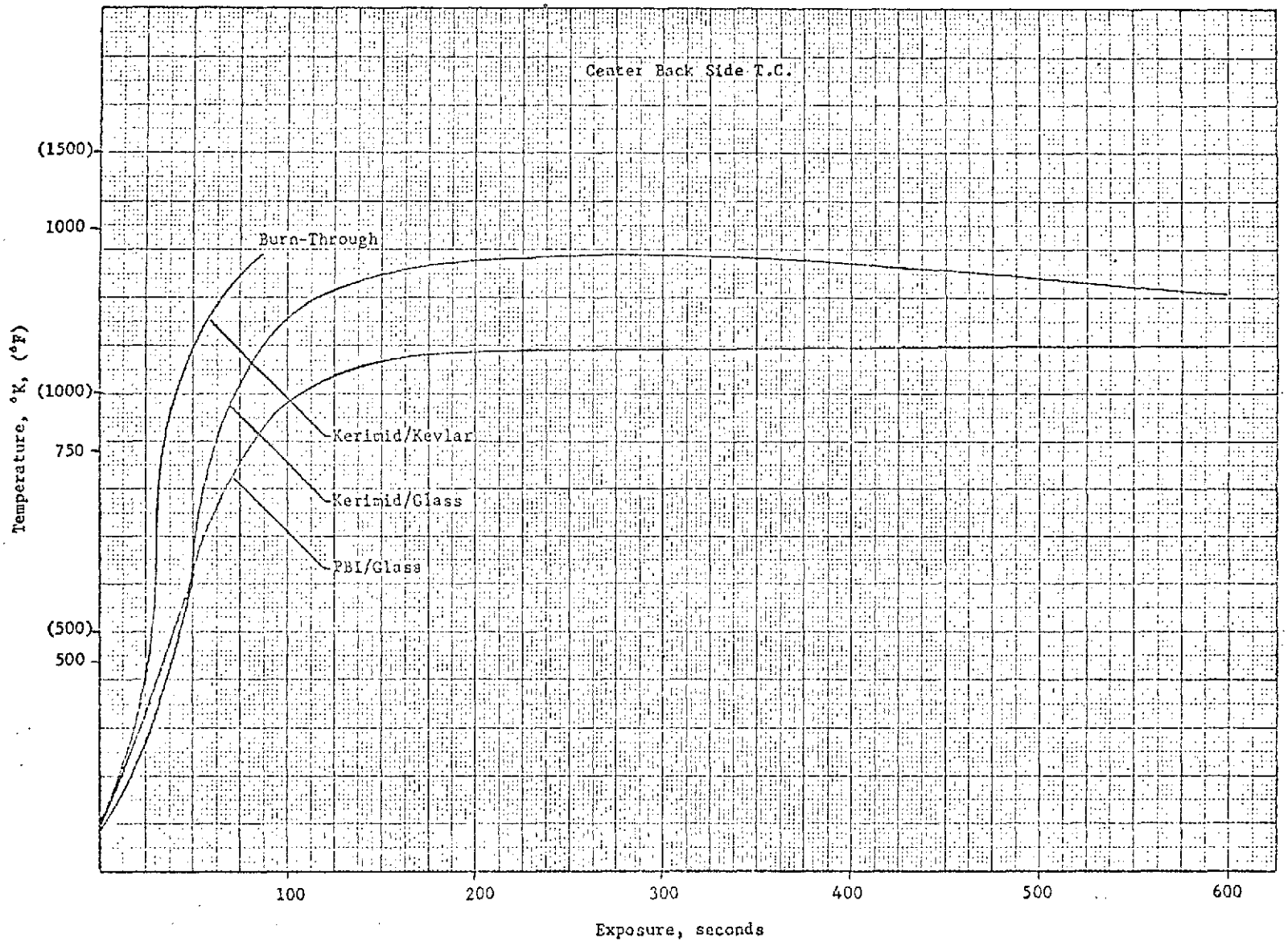

Figure 2-22. Performance of Unfilled Structures

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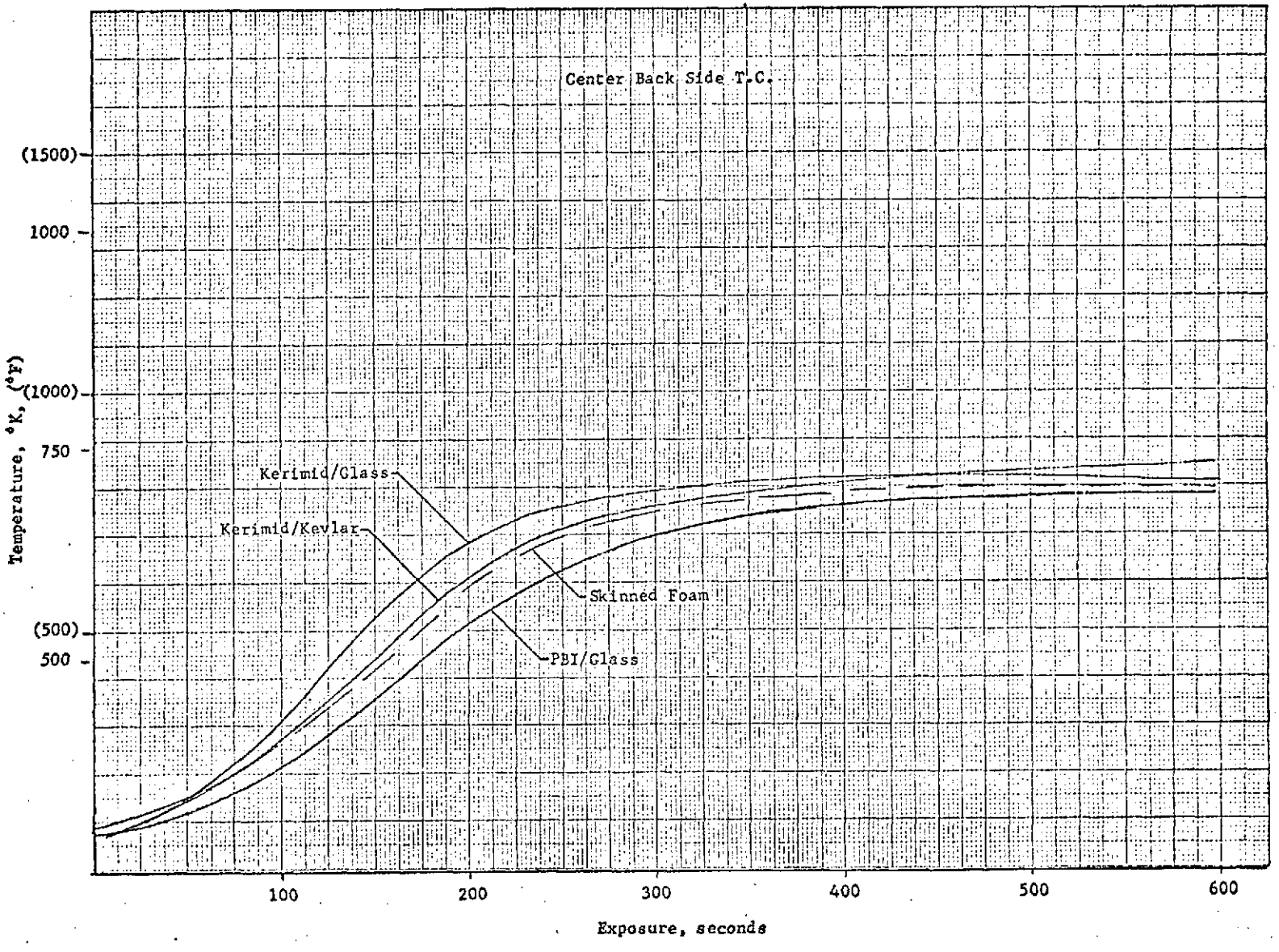

Figure 2-23. Performance of Filled Structures

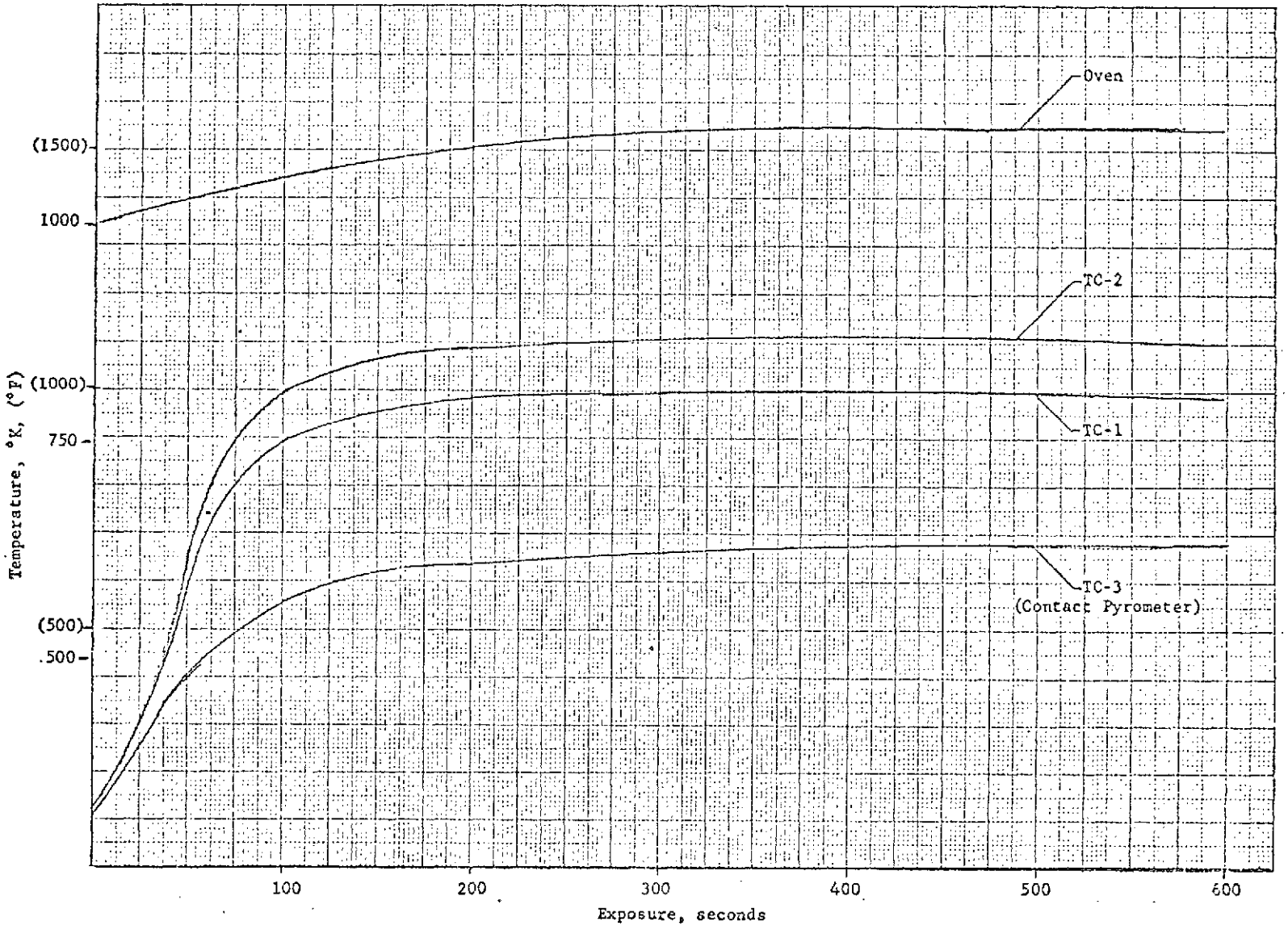

Figure 2-24. Panel 1, Unfilled PBI/Glass, Back Side Thermocouples vs. Time

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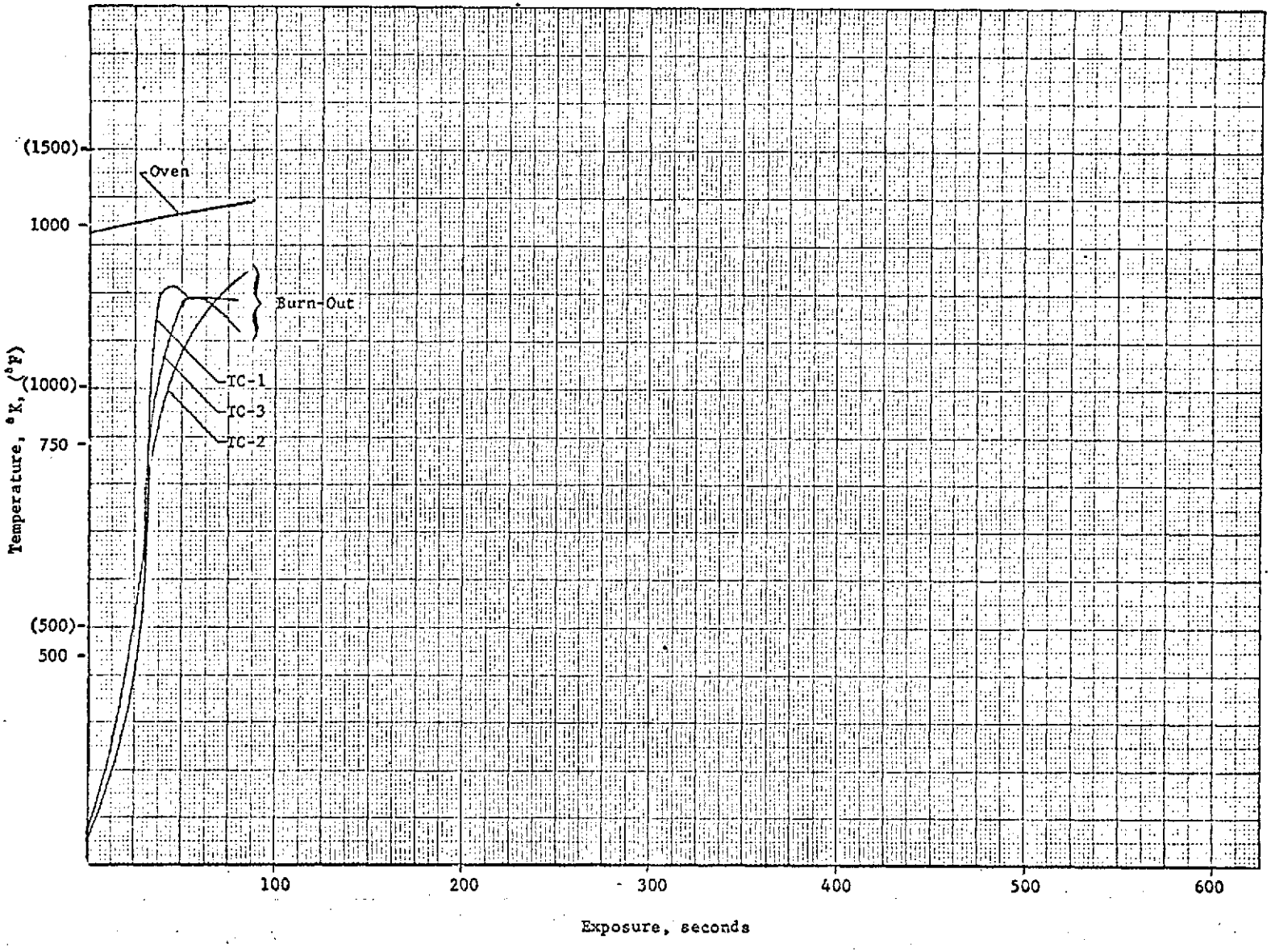

Figure 2-25. Panel 4, Kerimid/Kevlar, Unfilled, Back Side Thermocouples vs. Time

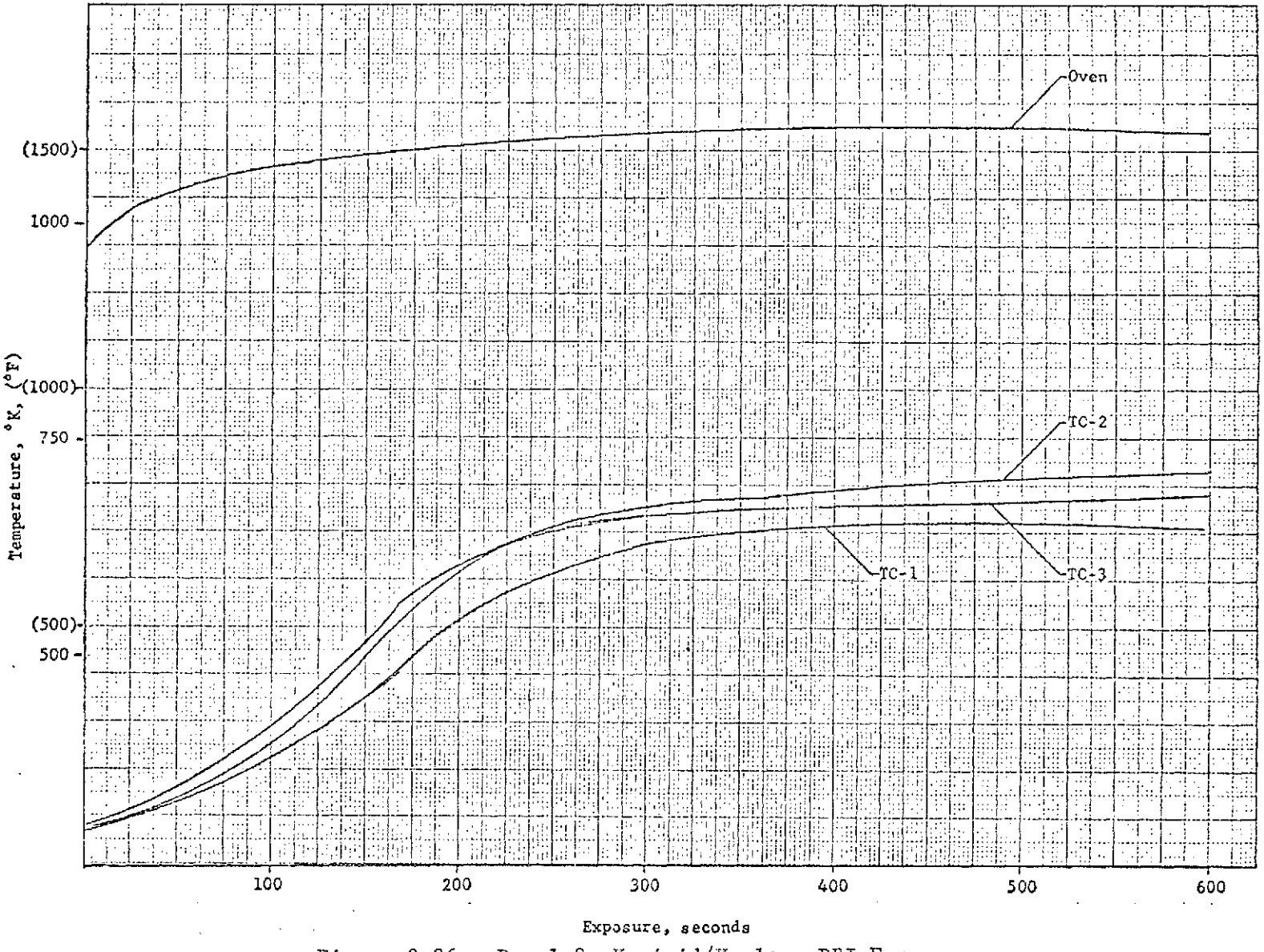

Figure 2-26. Panel 8, Kerimid/Kevlar, PBI Foam, Back Side Thermocouples vs. Time

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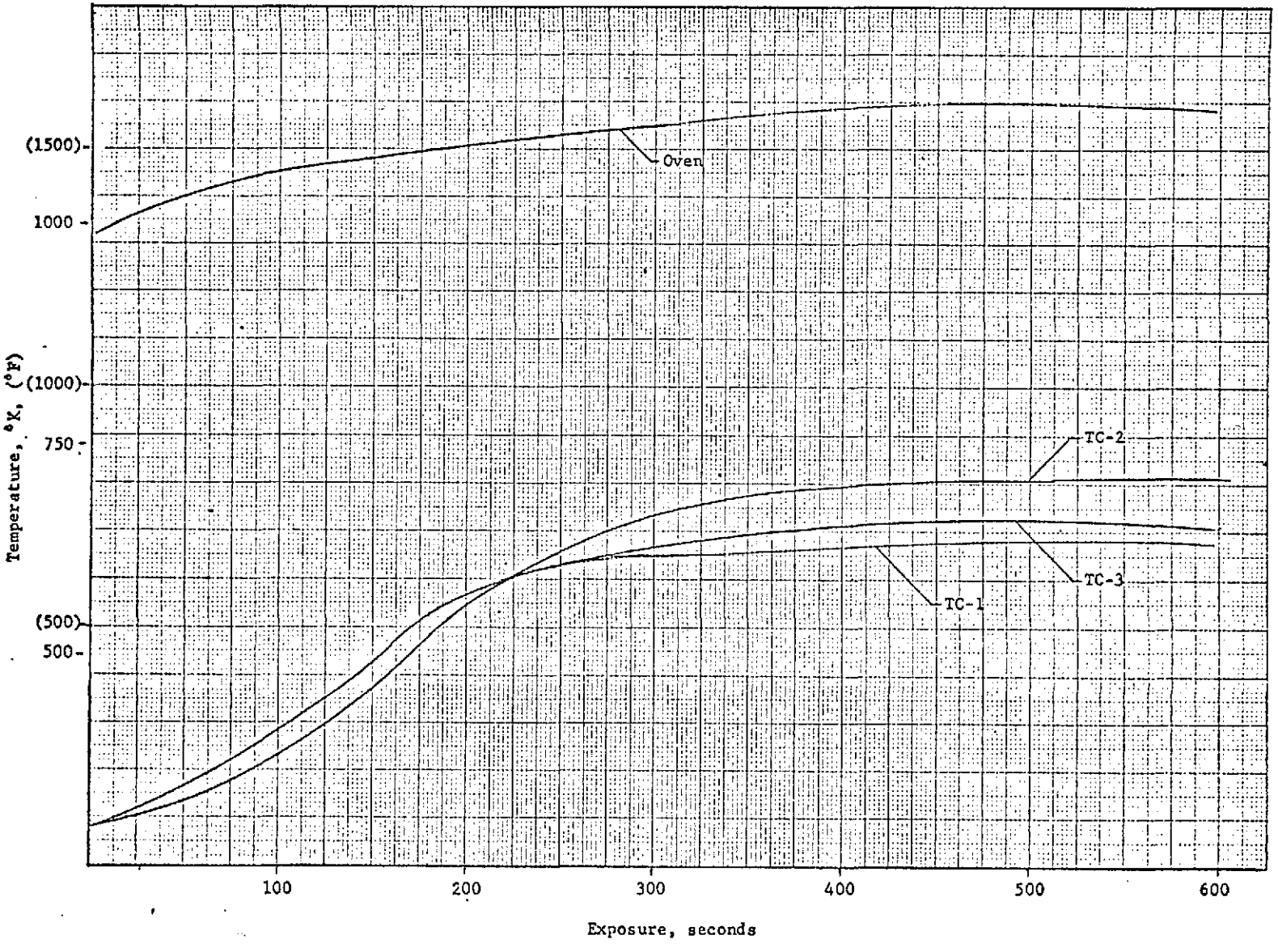

Figure 2-27. Panel 6, PBI/Glass, PBI Foam, Back Side Thermocouples vs. Time

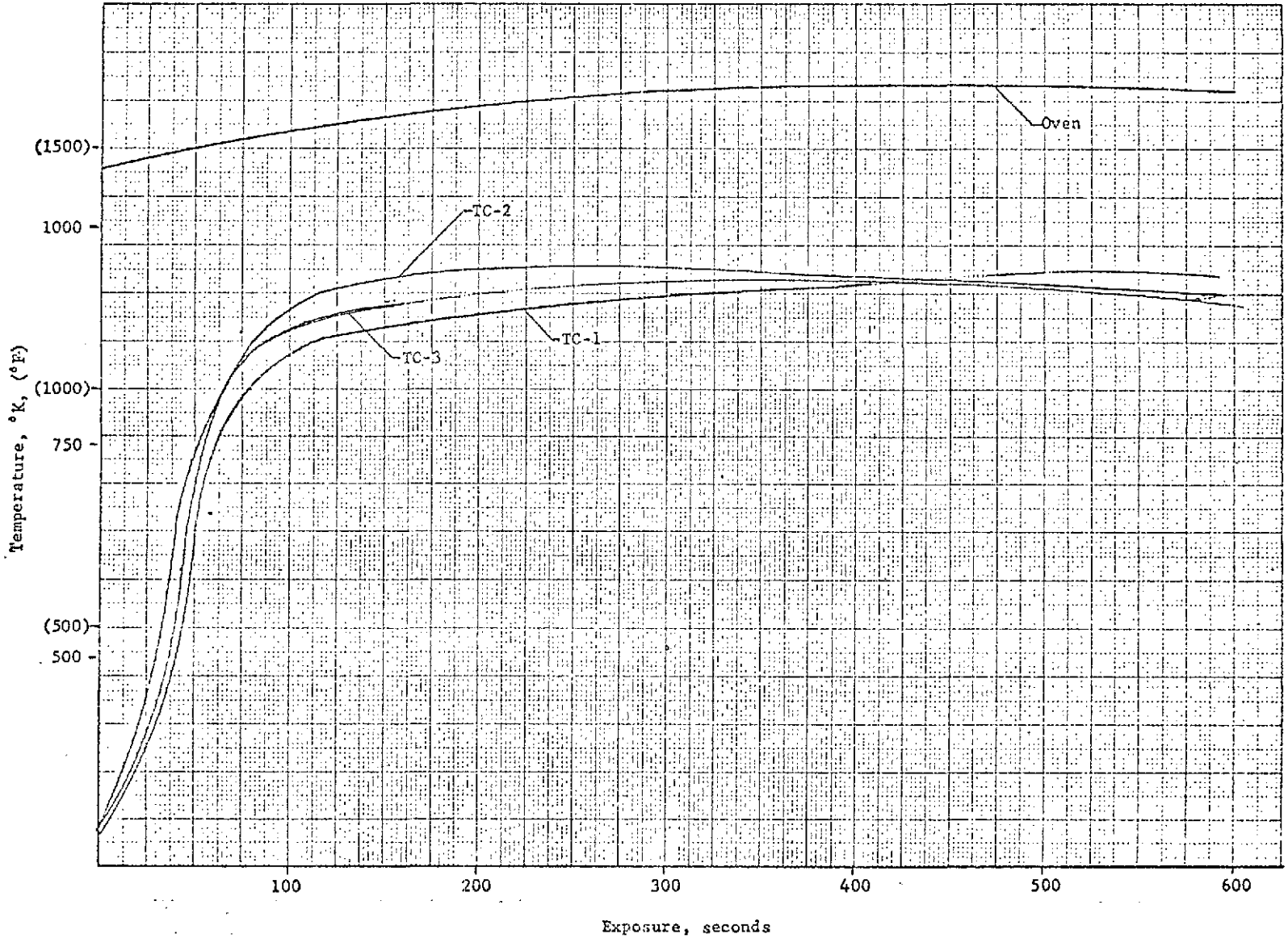

Figure 2-28. Panel 9, Kerimid/Glass, Unfilled, Back Side Thermocouples vs. Time

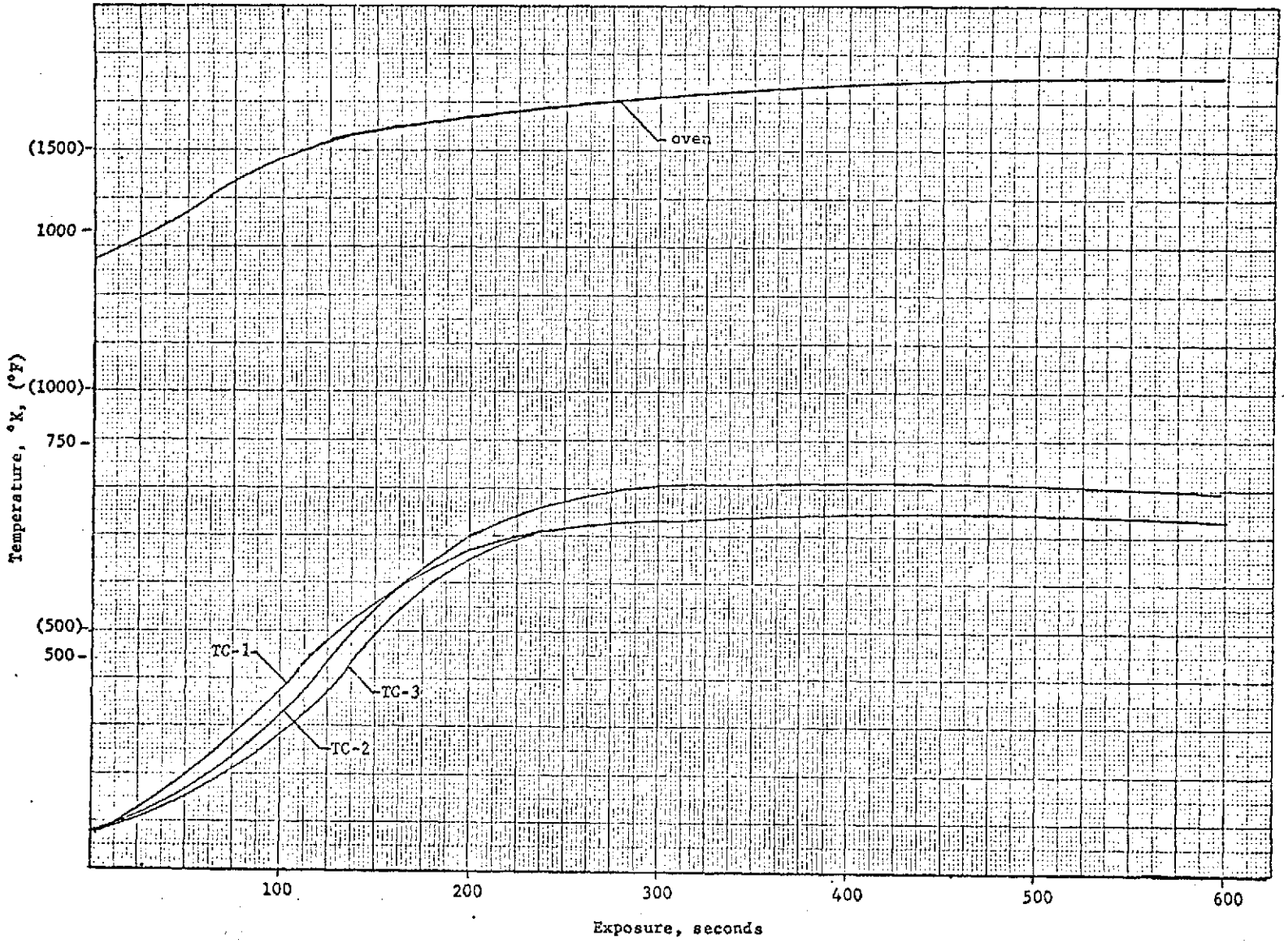

Figure 2-29. Panel 12, Kerimid/Glass, PBI Foam, Back Side Thermocouples vs. Time

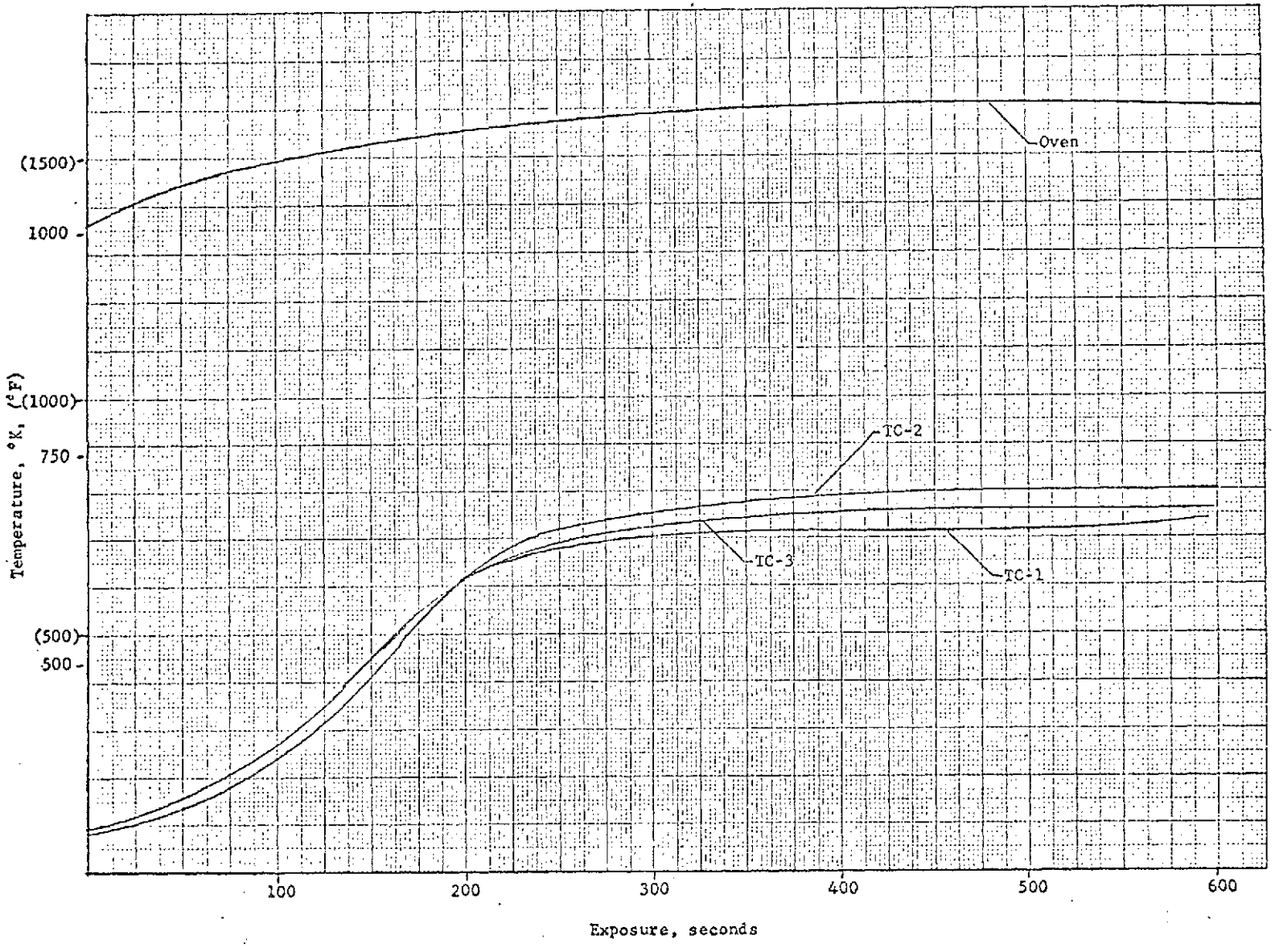

Figure 2-30. Panel 13, PBI Skin, No Truss, PBI Foam, Back Side Thermocouples vs. Time

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(3) Although the all-PBI structures are definitely superior in performance, a PBI foam filled Kerimid 601/glass panel may provide good performance at a lower price.

(4) The skinned panel approach is definitely viable and performed as well as the best truss structure panel. This concept should certainly be developed further.

2.4.2 Physical and Mechanical Properties

The density of the thirteen panel types was previously given in Table 2-2. Simple weighing and measuring techniques were used. Trimmed sections of the various panels were used to determine resin content of the various truss and skin materials. The Kevlar reinforced material had to be evaluated by weight calculations, as any known method that attached the Kerimid matrix also attacked the Kevlar reinforcing fibers.

An attempt was made to determine the flexural strength and modulus of the various structures. This proved to be impractical, due to the unusual geometry of the truss structure. The panels would buckle and deform, but not fail. Figures 2-31 through 2-34 illustrate typical deformation encountered in testing. The difficulty arises in that the size of the geometric details in the specimen is very large when compared to the dimensions of the testing fixtures. This made it impossible to run meaningful flexural test data on specimens of moderate size. WRD has developed an attractive approach for a meaningful test of larger panels that is beyond the scope of the present program. This approach is attached as Appendix B.

2.5 Second Generation Structure Fabrication

A discussion was held between WRD and NASA-Ames personnel at the conclusion of the initial set of thermal tests. It was decided that the set of panels shown in Table 2-4 would be fabricated.

For Items A, B, and C, NASA-Ames was sent a 15.24 by 15.24 cm (6 by 6 in.) and a 30.5 by 30.5 cm (12 by 12 in.) piece of each type for their testing and evaluation. The rest of the panels were used for mechanical property data. For Items D, E, and F, one panel each was retained by WRD for mechanical property testing, while the remaining panels were sent to NASA-Ames for evaluation.

The truss structures were fabricated with no difficulty, using procedures and cure cycles described in Section 2-3. However, two significant problems were encountered and overcome.

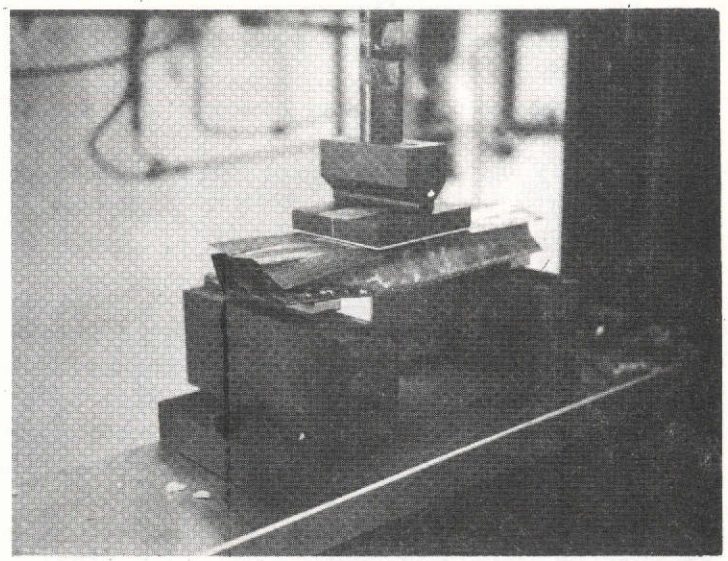

Figure 2-31. Longitudinal Flexure, Unfilled

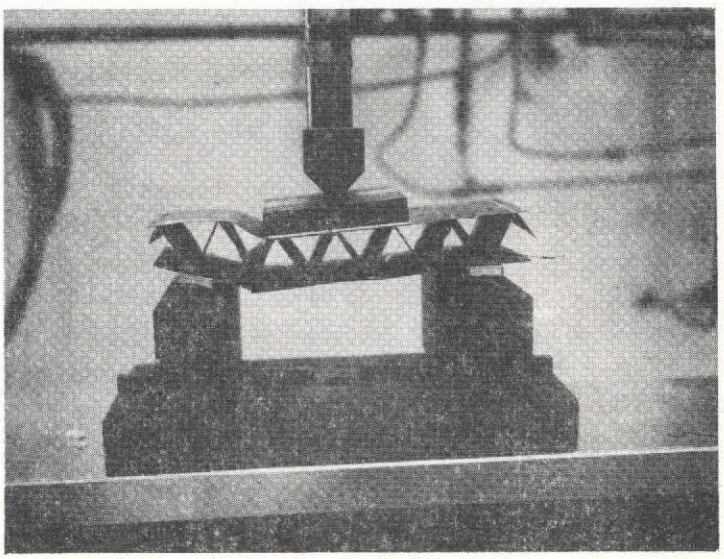

Figure 2-32. Transverse Flexure, Unfilled

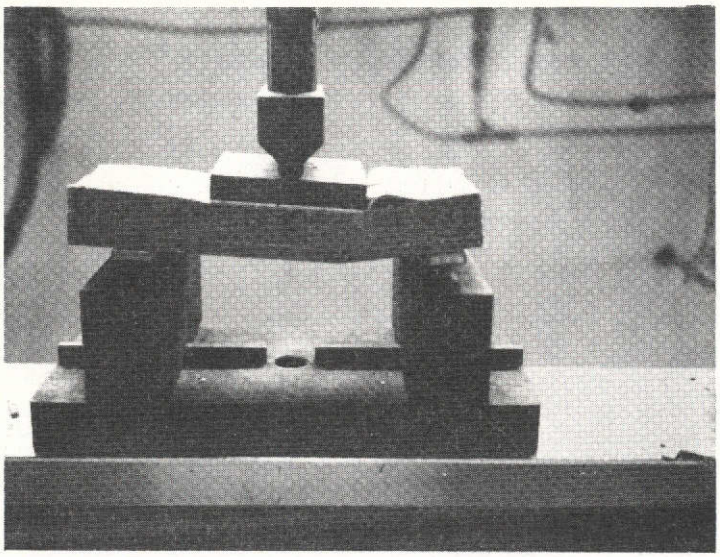

Figure 2-33. Longitudinal Flexure, Filled

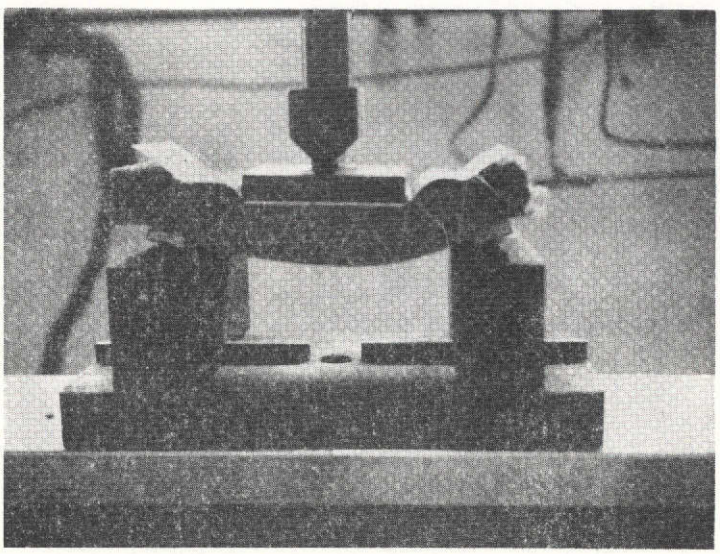

Figure 2-34. Transverse Flexure, Filled

TABLE 2-4

SECOND GENERATION PANELS

Type	Truss	Number of Panels
(A) PBI/181 Glass 2.54 cm (1 in.) thick	with foam no foam	2 2
(B) Kerimid 601/Glass 2.54 cm (1 in.) thick	with foam no foam	2 2
(C) Kerimid 601/Kevlar 49 2.54 cm (1 in.) thick	with foam	2
(D) 1 Ply PBI/Glass Skin 32 kg/m^3 (2 lb/ft^3) foam core (PBI), no truss, 2.54 cm (1 in.) thick		3
(E) 1 Ply Kerimid 601/Glass Skin 32 kg/m^3 (2 lb/ft^3) foam core (PBI), no truss, 2.54 cm (1 in.) thick		3
(F) 1 Ply PBI/Glass Skin 96 kg/m^3 (6 lb/ft^3) foam core (PBI), no truss, .635 cm (.125 in.) thick		2
	Total	18

(1) Uneven Foam Quality. Some significant variation in foam quality (i.e., porosity and density) was encountered throughout the program. Although the problem was not severe, it was certainly not desirable to have a sizeable scrap rate. The difficulty was identified as originating from heat distortion in the foam mold during cure. This allowed molten PBI to melt and "puddle" before foaming, with the result that variations in density and porosity occurred. A larger, stiffened tool (in this case, utilizing a large press and picture frame structure as the tool) resolved the problem.

(2) Skinned Panel Fabrication. When the glass/PBI skins were bonded to the 32 kg/m^3 (2 lb/ft^3) foam, a number of difficulties were encountered. When cured at 728°K (850°F), the foam distorted under pressure and unsatisfactory panels resulted. When a lower bonding temperature was used, the adhesive did not cure to a high molecular weight, and the resulting bond was weak and brittle. Local and massive delaminations were encountered.

It was therefore determined that the full 728°K (850°F) cure was needed for good bonding of the skins to the foam. New 48 kg/m^3 (3 lb/ft^3) PBI foam was made, but it also distorted under light pressure. Analysis revealed that significant phenol remained in the foam, even after a 728°K (850°F) hold of 4 hours was added to the foam cure cycle. The phenol acts as a plasticizing agent and causes a thermoplastic yielding to occur in the foam. This phenol had been driven out when small, thin foam sections had been fabricated in earlier work. The recent work involved 10 cm (4 in.) or thicker foam slabs as would be used in production of large numbers of foam parts. Here, the phenol did not readily escape during a standard PBI cure cycle.

The solution to the problem was to apply a vacuum to the foam blocks during the final extended high temperature cure. This effectively removes the phenol from the foam. The resulting PBI is readily bonded to the glass/PBI skins without thermoplastic distortion under light pressure.

The 48 kg/m^3 (3 lb/ft^3) foam proved significantly superior to the 32 kg/m^3 (2 lb/ft^3) foam in strength and uniformity and was therefore used in fabricating the panels for the program.

2.6 Second Generation Structure Testing

2.6.1 Fire Resistance Testing

As previously mentioned, panels were sent to NASA-Ames for a series of evaluations, i.e., T-3 testing, smoke generation, and toxicity tests.

The toxicity tests were not run during the course of the program, but the other two series of evaluations were carried out by NASA-Ames personnel. Table 2-5 summarizes the results of the smoke generation tests. All WRD panels far outperformed a commercial type aircraft panel. However, the all-PBI structures were outstanding, and significantly superior to all other candidate systems.

There was no apparent difference between the performance of the truss structure versus the skinned foam configuration.

Figures 2-35, 2-36, and 2-37 summarize results obtained by NASA-Ames during T3 flame testing of the new panel concept. A constant heat flux of 11.4 - 12.5x10^4 watt/n^2 (10 - 11 BTU/ft^2/sec) as measured before and after testing with a calorimeter was maintained throughout the test. From the results it may be concluded that the performance of the 0.64 cm (1/4 in.) thick panels was unacceptable and approximately equivalent to that of the unfilled truss structure. The all-PBI resin sandwich again shows performance significantly superior to that displayed by a panel with Kerimid 601 matrix surface skins. All panels were thermocoupled in the same manner as the first generation panels described earlier.

2.6.2 Mechanical Property Testing

Triplicate flatwise tension and flatwise compression tests were run on all structure types at room temperature and 335°K (160°F). Table 2-6 summarizes this data. The results are the average of three determinations.

Again, as with flexural strength tests, significant problems were encountered in obtaining meaningful data. The tensile tests were fairly straightforward. With a low density foam, low flatwise tension results were expected. There is little material on the foam surface for the adhesive to adhere to. Little significant difference was seen in the strength of the various structures.

With flatwise compression tests, it is difficult to determine what the results mean. The light foam is quite resiliant and does not "fail", as would a rigid foam. The load simply builds under compression as the foam block is compressed 25%, 50%, or more. The "break" or failure point was taken as a discontinuity in the stress/strain curve, indicating a

TABLE 2-5

SMOKE DENSITY RESULTS

Sample	Specimen Weight		% Loss	Test Condition	D_s 90 sec.	D_s 2 min.	D_s 4 min.	D_s max	Time D_m	T_e	Area Density
	Before	After									
Commercial type aircraft panel	10.53 g	8.16 g	22.5	flame	49.71	50.18	53.20	61.81	12 min.	30 sec.	.570 (1 in.)
	10.53 g	9.52 g	9.6	no flame	12.00	13.88	15.70	26.48		4 min.	
Kerimid 601/PBI, no truss core	10.7365 g	10.2381 g	4.64	flame	4.46	4.46	5.40	5.72	6' 30"	∞	.458 (1 in.)
	10.1671	10.0142	1.50	no flame	.28	.28	.57	1.15	9 min.	∞	
Kerimid 601/PBI, truss			7.86	flame	.28	.56	1.13	1.68	9 min.	∞	.540
			1.06	no flame	0	0	0	0	∞	∞	
PBI/181/PBI, truss			2.18	flame	0	0	0	0	∞	∞	.687
			3.15	no flame	0	0	0	0	∞	∞	
Kerimid 601/Kevlar/ PBI foam, truss			6.64	flame	.28	.28	.28	2.24	16 min.	∞	.671
			.91	no flame	0	0	0	0	∞	∞	
PBI/PBI foam, no truss				flame	0	0	0	.28	10 min.	∞	.566
				no flame	0	0	0	0	∞	∞	
Kerimid 601/181, truss				flame	2.24	2.79	3.33	8.01	11 min.	∞	.417
				no flame	0	0	0	0	∞	∞	
PBI/118, truss				flame	0	0	0	0	∞	∞	.364
				no flame	0	0	.28	.28	4 min.	∞	

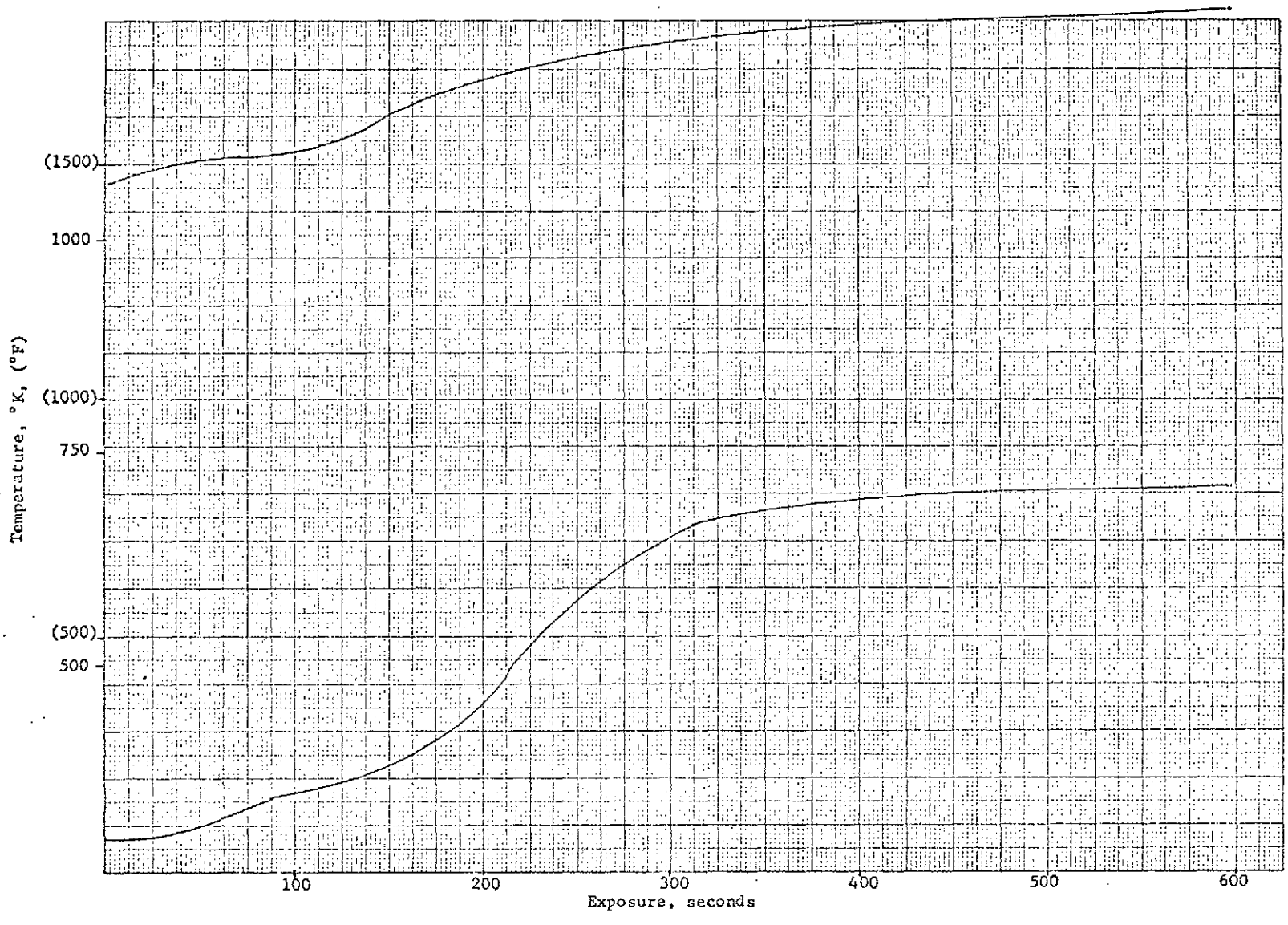

Figure 2-35. NASA Data, A11-PBI 48 kg/m^3, 2.54 cm thick (3 lb/ft^3, 1 in. thick) Foam

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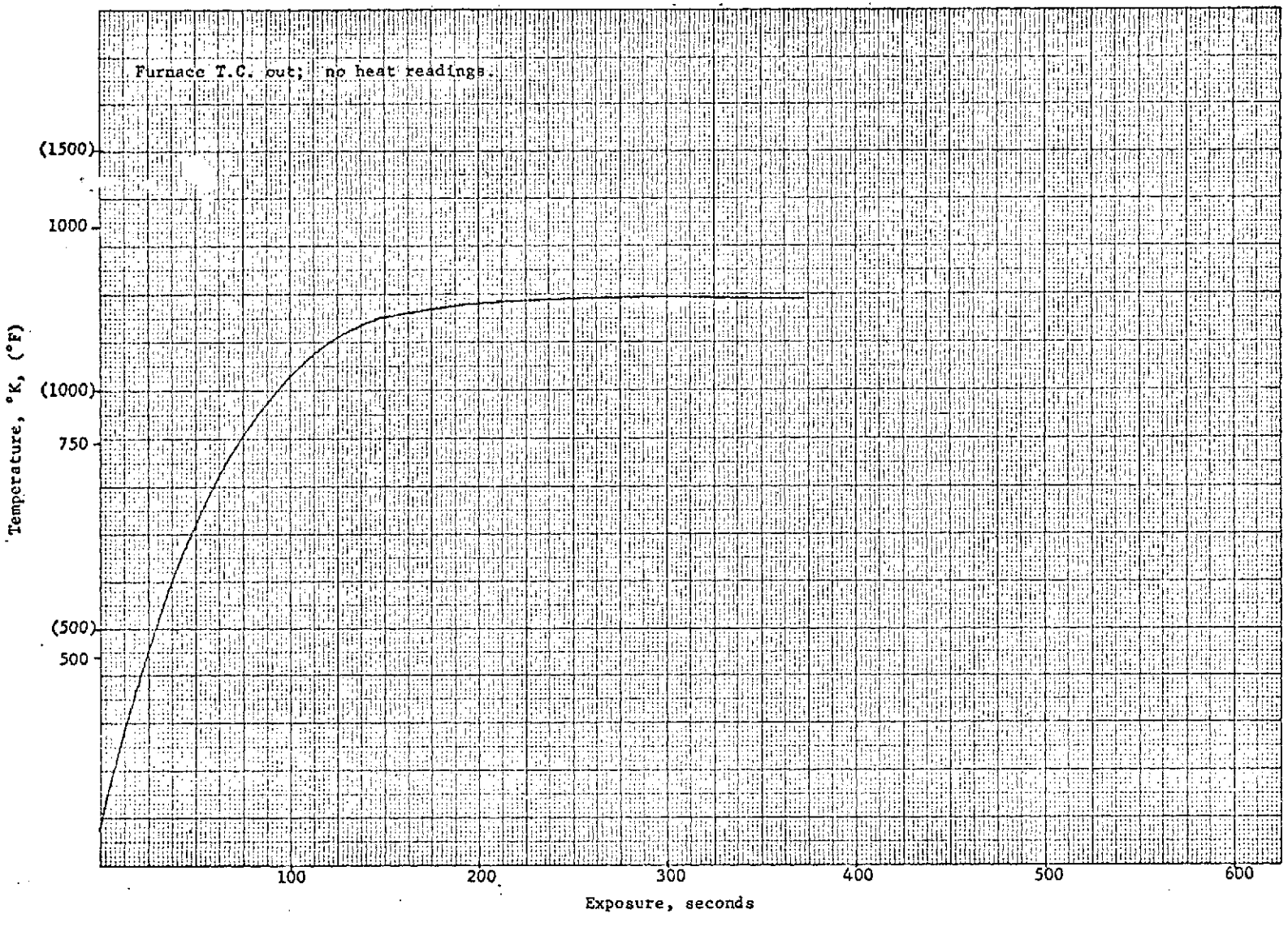


Figure 2-36. NASA Data, All-PBI 96 kg/m^3, 0.64 cm thick (6 lb/ft^3, 1/4 in. thick) Foam Sandwich

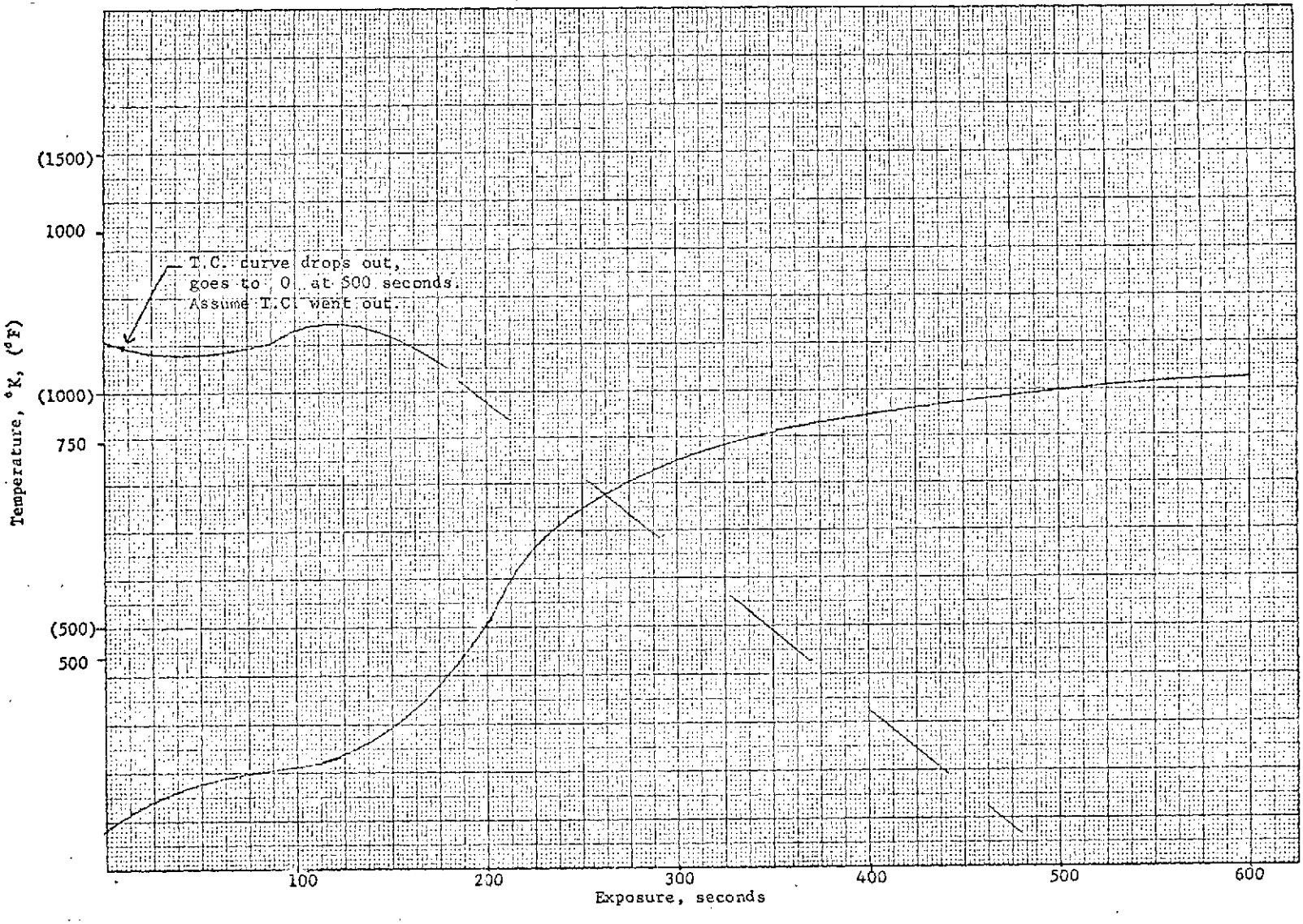


Figure 2-37. NASA Data, K-601/Glass Skin 48 kg/m^3, 2.54 cm thick (3 lb/ft^3, 1 in. thick) PBI Core Sandwich

TABLE 2-6

MECHANICAL PROPERTIES

Sample Description	Flatwise Tension KN/m² (lb/in²)		Flatwise Compression KN/m² (lb/in²)	
	RT	335°K [160°F]	RT	335°K [160°F]
(A) PBI/181 Glass, foam filled, 2.54 cm (1 in.) thick	106 (15.4)	92 (13.4)	231 (33.5)	233 (33.8)
Same, unfilled	142 (20.6)	119 (17.2)	61 (8.9)	53 (7.7)
(B) Kerimid 601/Glass, foam filled, 2.54 cm (1 in.) thick	138 (20.0)	79 (11.5)	361 (52.3)	297 (43.1)
Same, unfilled	75 (10.9)	148 (21.5)	43 (6.2)	40 (5.8)
(C) Kerimid 601/Kevlar 49, foam filled, 2.54 cm (1 in.) thick	170 (24.6)	110 (16.0)	303 (44.0)	258 (37.4)
(D) 1 Ply PBI/Glass Skin, 48 kg/m³ (3 lb/ft³) foam core (PBI), no truss, 2.54 cm (1 in.) thick	233 (33.8)	274 (39.8)	858 (124.4)*	654 (94.8)*
(E) 1 Ply Kerimid 601/Glass Skin, 48 kg/m³ (3 lb/ft³) foam core (PBI), no truss, 2.54 cm (1 in.) thick	76 (11.0)	74 (10.8)	80 (11.6)*	84 (12.2)*
(F) 1 Ply PBI/Glass Skin, 96 kg/m³ (6 lb/ft³) foam core (PBI), no truss, .635 cm (.125 in.) thick	181 (26.3)	227 (32.9)	n.a.*	n.a.*

* Difficult or impossible to find a "break" point. Specimen compresses as load builds; returns to initial condition when load is removed. See discussion in text.

failure somewhere in the structure. The specimen remained intact and could still be loaded to a significantly higher level. When the compressive stress was removed, the specimen returned to normal dimensions with only minor signs of failure.

In the case of the thin sandwich (Structure E), no discontinuity could be found as the specimen compressed, so no "break" point could be recorded. These anomalies should be taken into account when considering the data obtained. Direct comparisons of these results to data generated on rigid conventional structures (i.e., honeycomb sandwich, rigid foam, etc.) are misleading and may not be valid. Again, the necessity of carefully designing meaningful tests for these complex sandwich/foam/truss type structures cannot be overemphasized. Unfortunately, elaborate and/or expensive test specimens and fixtures were beyond the scope of this program.

3. CONCLUSIONS AND RECOMMENDATIONS

All structures tested showed some degree of flame resistance under the test conditions of this program. Kevlar 49 reinforcement is least desirable for such applications as it burns vigorously in the T3 testing environment. The performance of unfilled truss structures and of 0.64 cm (1/4 in.) thick sandwich is not adequate. The skinned sandwich/PBI foam core panels did not debond or delaminate under T3 test conditions and proved the most satisfactory concept for a number of reasons.

(1) The skinned sandwich panels are much simpler to fabricate and therefore significantly less expensive than truss structure.

(2) Aircraft manufacturers are familiar with the procedures required for the fabrication of such structure.

(3) Smooth, cosmetic surfaces can be readily achieved with the skinned foam panel concept. Such surfaces cannot be easily produced on truss structure panels.

(4) The skinned panels are far more familiar to aircraft manufacturers and would probably be accepted and utilized much more readily than would the unfamiliar truss structure panels.

(5) The truss structures show essentially no performance advantages over the skinned foam panels.

The performance of the glass/PBI skin/PBI core structures was quite superior to that achieved with glass/Kerimid 601 skin/PBI core panels. A large advantage in smoke generation for the PBI matrix was observed, as well as superior performance in T3 testing.

It is therefore recommended that the 181 E glass/PBI skin/PBI foam sandwich structure be selected for further development work aimed at the evaluation and production of highly fire resistant aircraft panels. The concept shows superb performance, relatively low production costs, and moderate material costs in volume. WRD believes it is highly probable that the goal of a structure capable of protecting human life for at least 10 minutes in a fuel fire condition can be achieved using the approach developed in this work.

It is also recommended that more sophisticated, comprehensive mechanical property testing be included as a vital part of any new effort in this area.

APPENDIX A

PROCEDURE FOR STITCHING TRUSS STRUCTURES

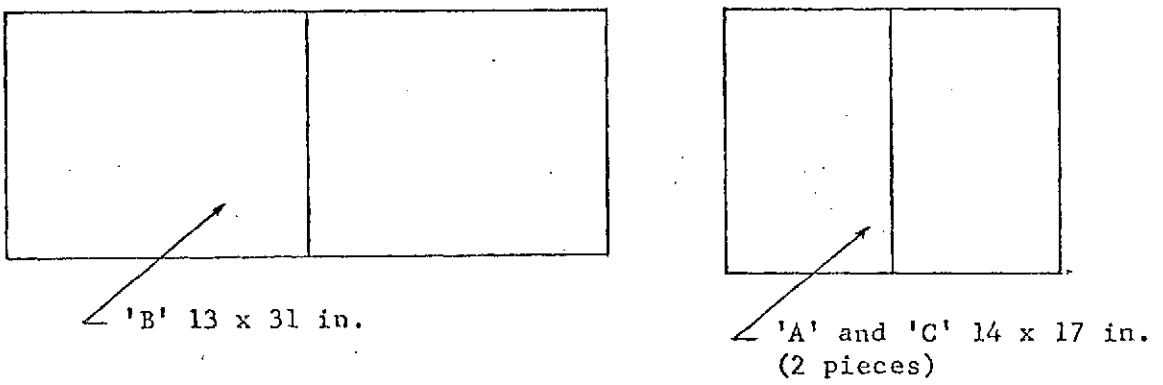
The procedure for the fabrication of the stitched truss structures for fire protection panels follows.

PREPREG PROCESSING FOR FIRE PROTECTION PANELS, 12 IN. x 12 IN. FINISH DIMENSIONS

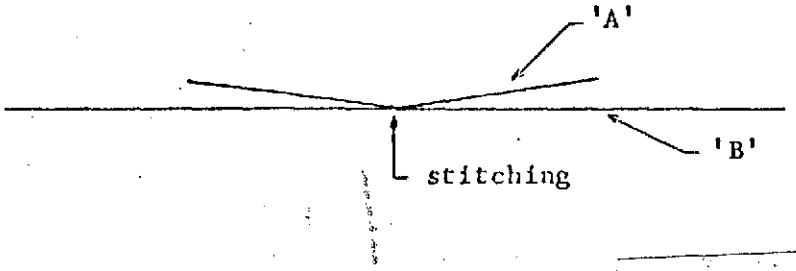
1. Cut two pieces prepreg, 14 in. x 17 in., 'A' and 'C'.

2. Cut one piece prepreg, 13 in. x 31 in., 'B'.

3. Scribe center line on all three pieces.



4. Place 'B' over 'A' and match center lines, and arrange so that approximately 0.5 in. of 'A' extends beyond each edge of 'B'. Staple in place and stitch the two pieces together, being sure the stitching line is straight and ⊥ to the long sides of prepreg. Tie each end of threads and remove staples



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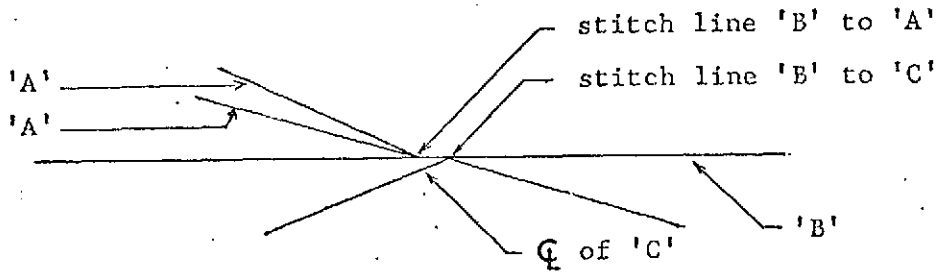
5. Fold back one side of 'A' to expose seam. Place form tool on material against seam and scribe a line along opposite edge of form tool.

6. Scribe a line approximately midway between the seam line and the line scribed in Step 5.

7. Place the stitched assembly over 'C' and line up the center line of 'C' with the line scribed in Step 6. Each edge of 'C' should extend beyond edges of 'B'.

8. In this position staple 'C' to 'B' and stitch together along line on 'B' in Step 5. Tie each end of threads and remove staples.



These two seams now form one side of triangular cell.

9. Lay out assembly on flat surface with 'C' down flat on surface.

10. Fold back both sides of 'A' and the seam 'B' to 'A' so that the seam 'B' to 'C' is exposed.

11. Place one edge of form tool against the exposed seam and scribe on 'C' along the opposite flat side of form tool.

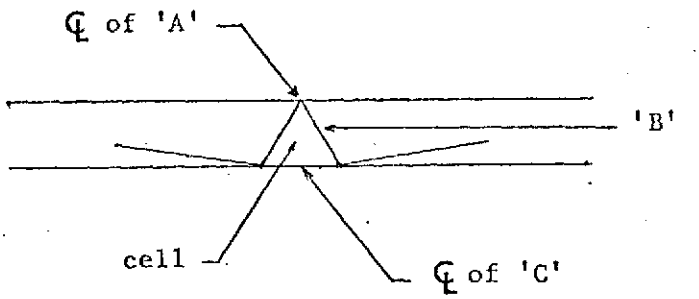
12. Keeping unit in same position, drop 'B' over 'C', and keep piece 'A' folded so that the seam 'B' to 'A' is exposed.

13. Place one edge of form tool against this exposed seam and scribe on 'B' a line along the opposite flat side of form tool.

14. Line up these two scribe lines (Steps 11 and 13) and staple into place.

15. Stitch along this line. This stitch completes all three sides for the initial cell.

16. Before tying the thread ends, be sure form tool will fit through the cell. If not, remove last set of stitches and adjust stitching line to accommodate form tool. Tie ends of thread and remove staples.



NOTES:

It must be borne in mind that Steps 4 through 16 are critical. The straightness of the seams and the parallelism of the seams 'B' to 'C' set the pattern for the entire assembly.

To continue the stitching process, the unit is laid out next with 'A' side down and scribe lines made on 'A' and 'B', next on 'B' and 'C', etc.

Each time a cell is completed, the form tool fitting should be checked before tying the thread ends.

Periodical measurements should be taken to assure uniform distances between seams. If these distances vary greatly, wrinkling and/or stretching will occur when completed assembly is cured.

APPENDIX B

TESTING OF FOAM COMPOSITE STRUCTURES

Whittaker CORPORATION

TO: Milan Maximovich
LOCATION:

DATE: 25 July 1974
SDE-74-67

FROM: K. R. Berg
LOCATION:

SUBJECT: Testing Foam-Composite Structure
MJO 4521-001

Flexural tests were conducted on the finished panels using a 3"x8" specimen. The standard test method that is called out for honeycomb structure (flexural testing) is a four point loading beam test. The test apparatus consisted of a flexural fixture having a lower support span of 6" and a load nose span of 2". The specimen was loaded with 3/4" wide pads at all of the contact points.

The test proved to be unsatisfactory due to the geometry of the internal structure. Early skin buckling occurred due to the cell size, and crushing occurred at the load points.

As a result of this test a modification was made to the upper load nose. The 2" span was replaced with a 3"x3" load pad, still using the 6" span for the lower support. This improved the situation to a certain degree as it enabled the load to be applied over approximately three of the triangular structures. Even with this modification, it is concluded that a structure of this configuration should not be tested in this manner. A comparison based on this test with results obtained from honeycomb structures tested in accordance with the standard test method is not therefore recommended.

WRD would recommend an alternate test. A test apparatus would be built that would accommodate this type of structure and which could be used on honeycomb structure of an equivalent nature. This would allow comparison between the two structures and provide reliable data on the foam-composite structure.

The test apparatus (see Figure 1) would consist of a rigid (1" alum.) mounting plate which would be covered with a rubber diaphragm. The test panel would be mounted on the rubber diaphragm and secured around the edges. Air pressure would then be applied to the opposite side of the diaphragm causing the specimen to be loaded uniformly over a large area. The load would be transferred uniformly from the skin surface into the internal structure allowing it be loaded structurally similar to typical applications. This would result in reliable data. Deflection measurements would be taken as the air pressure was increased giving a direct relationship between load and deflection. The ultimate failure load of the panel would also be determined.

K. R. Berg

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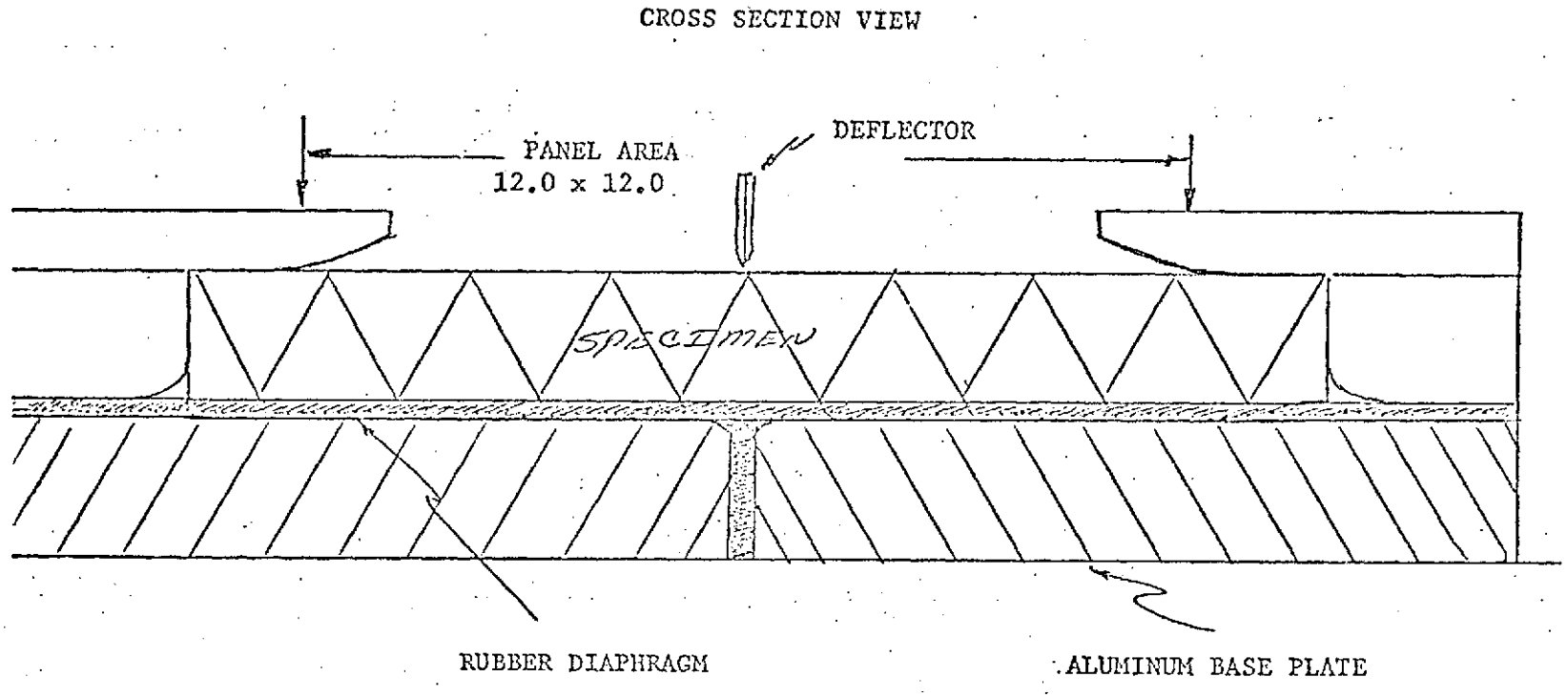

FIGURE 1. PANEL TEST METHOD.

APPENDIX C

WHITTAKER RESEARCH AND DEVELOPMENT PRELIMINARY PROCESS SPECIFICATION,
FABRICATING COMPOSITE SANDWICHES WITH PBI
SYNTACTIC FOAM CORE AND PBI/GLASS SKINS

SPECIFICATION

TYPE: PROCESS-PRELIMINARY

SPECIFICATION NO.: 2013

TITLE: FABRICATING COMPOSITE SANDWICHES WITH PBI
SYNTACTIC FOAM CORE AND PBI/GLASS SKINS

PREPARED BY: R. REED
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REVISIONS

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1.0 SCOPE

1.1 The objective of this specification is to establish the general processing requirements for fabricating composite sandwiches utilizing PBI syntactic foam core and PBI/glass skins.

1.2 This specification is applicable to all work accomplished by Whittaker Research & Development and any and all sub-contractors thereof unless specifically stated otherwise. This specification establishes the minimum requirements for utilizing the subject materials and no effort has been made to detail specific "hardware" requirements.

2.0 APPLICABLE DOCUMENTS

2.1 Mil-Specifications

2.2 NRD Specifications

2.2.1 NRD 2004, Barrier Material for Imidite Laminates

2.2.2 NRD 1004, Imidite 1850 Acceptance Specification

2.3 Commercial Specifications

2.4 Other

3.0 MATERIALS

3.1 Imidite foam compound SA & PC

3.2 Imidite 1850

3.3 TFE 30/112 barrier material

3.4 Miscellaneous materials

3.4.1 Release agents

3.4.2 Bagging materials

4.0 EQUIPMENT

4.1 Oven capable of operation at 850°F

4.2 Vacuum pump

4.3 Heated platen press - 650°F (optional)

4.4 Autoclave (optional)

5.0 QUALITY ASSURANCE

5.1 All materials shall be qualified for shop use per the applicable acceptance specification.

5.2 The following sections of the Narmco Quality Assurance manual are applicable:

QAM 61.33
61.35
61.44
61.52
61.54

6.0 GENERAL

6.1 Safety

6.1.1 There are no special safety requirements applicable to this specification.

6.2 Storage

6.2.1 All raw materials shall be stored in their original containers and shall be protected from contamination.

6.2.2 All applicable materials shall be stored at room temperature.

7.0 PROCEDURE

7.1 Preforming

7.1.1 Prepare a suitable flat preform mold to contain the PBI syntactic foam powder. The flat molds can be of any suitable material such as aluminum, plastic (epoxy, etc.) plaster or wood.

7.1.2 Place a film of suitable release material (PVA, cellophane, FEP) in bottom of mold.

7.1.3 Charge mold with calculated weight (density x volume x 1.15 = weight of foam powder) of foam powder. Screed level and cover with release film.

7.1.4 The charged mold can then be either vacuum bagged for oven cure or placed in a heated platen press for the following cure:

7.1.4.1 Raise temperature uniformily to 250°F ± 10°F (foam temperature) and apply 15 psig (28 in-hg). Hold at temperature for 30 ± 5 minutes. Cool to 150°F or less under pressure before removing from mold.

7.2 Preform contouring

7.2.1 If the shape of the finished article is other than flat with uniform thickness then a contouring operation must be accomplished.

7.2.2 The foam preform sheets can be contoured by any of several methods - depending upon shape and facilities available.

7.2.3 Place the preformed sheet in either a 300°F ± 10°F oven or between the platens of a press regulated at 300°F ± 10°F Hold 10 minutes or until preform softens. Remove preform and drape over a heated mandrel or form into cavity as required.

NOTE: This operation may be accomplished utilizing matched tooling.

7.2.4 Vacuum bag shaped preforms to tool utilizing a 400°F service bagging material such as Capran(R)80, a high temperature nylon, or other suitable material. Pull minimum (1/2 - 1 in.Hg) vacuum only to seat bag. Insert mandrel in oven regulated at 350°F ± 10°F and allow mandrel and foam to stabilize at oven temperature. Pull maximum vacuum to conform foam to mandrel shape and allow to cool to 150°F or less under maximum vacuum. Remove foam from mandrel.

7.2.5 Note: The mandrel or tool should be covered with a suitable release agent or film. Typical of these are:

7.2.5.1 Release agents

TFE 30 - sintered to tool
Vydax - sintered to tool
Frecote

7.2.5.2 Films

3M's TB5 Glass/TFE
FEP
Capran 80

7.3 Inner Skin Layup

7.3.1 Tool preparation

7.3.1.1 Tool should be cleaned with steel wool and solvent.

7.3.1.2 Coat tool with silicone resin DC 20 or equivalent and bake per manufacturer's recommendations. Remove excess silicone by abrading with fine steel wool.

7.3.1.3 Coat the tool surface with a release agent such as TFE 30, Vydax or Frecote. Note: TFE 30 must be sintered into a continuous film by heating the tool surface to 800°F. This can be accomplished by utilizing a propane torch.

7.3.2 Cut the required number of Imidite 1850 prepreg plies per drawing - note warp direction.

7.3.3 Utilize heat guns and irons regulated to 500°F ± 20°F (approx.) and heat tack prepreg to tool or mandrel.

7.3.4 Cover prepreg with 1 ply of perforated cellophane and 1 - 3 plies of glass breather. Bag with PVA or similar material.

7.3.5 Pull maximum vacuum and place in an oven. Raise temperature to 300°F, 2 - 3°F per min. Reduce temperature as soon as 300°F is reached. Allow to cool to room temperature under maximum vacuum.

7.3.6 Remove bag, breather and cellophane and lightly sand to remove resin rich spots.

7.4 Inner Skin-Foam Sub-Assembly

7.4.1 Prefit foam core sections to inner skin. Interference butt joints should be made if possible. If excessive gaps do exist an adhesive tape (Imidite 2801 on 112/112 scrim) should be interposed at the foam core butt joints.

7.4.2 Cover the layup with 2 plies TFE 30/112 barrier material and 3 - 4 plies of 1500 type glass breather.

7.4.3 Vacuum bag with 5 mil thick soft aluminum foil utilizing Dow-Corning silicone sealant 93-046 and primer QA-2-1011 as required to form and/or seal the bag.

7.4.4 Place in an oven and draw maximum vacuum. Cure to the following schedule:

a. Utilize a uniform heating rate of 1 - 3±F/min.

b. Raise temperature to 650 - 700°F.

c. Hold @ 650 - 700°F for 120 ± 10 min.

d. Cool under vacuum to 150°F or less.

7.4.5 Remove bag, breather and barrier.

7.5 Machining

7.5.1 The skin-core sub-assembly can be machined to the desired physical configuration by conventional means such as sanding, grinding and routing.

7.6 Outer Skin Layup

7.6.1 Repeat steps 7.3.2 and 7.3.3 except the material is tacked to the core.

7.6.2 Cover layup with 2 plies TFE 30/112 barrier material and 3 - 4 plies of 1500 type glass breather.

7.6.3 Bag per 7.4.3

7.6.4 Cure per 7.4.4

7.6.5 Remove bag, breather and barrier and remove part from tool.

7.7 Postcure

7.7.1 Post curing to be accomplished in an inert atmosphere such as nitrogen or argon.

7.7.2 Place the part in a suitable chamber - one that will maintain an inert gaseous envelope around the part and postcure as follows:

a. Utilize a 1/2 - 1°F/min heating rate.

b. Raise temperature to 400°F and hold 2 hours.

c. Raise temperature to 500°F and hold 2 hours.

d. Raise temperature to 600°F and hold 2 hours.

e. Raise temperature to 650°F and hole 2 hours.

f. Raise temperature to 700°F and hold 24 hours.

7.7.2 (cont'd)

g. Raise temperature to 750°F and hold 24 hours.

h. Raise temperature to 800°F and hold 8 hours.

i. Raise temperature to 850°F and hold 8 hours.

j. Cool under inert atmosphere to 400°F or less.

7.8 Part Finish

7.8.1 For certain application it may be found advantageous to seal the part against moisture absorbtion and to enhance its high temperature oxidation stability. Dow-Corning DC-7146 silicone resin has been found excellent for this purpose.

7.9 Process Variations

7.9.1 The processes described herein are general in nature and should not be construed as the only acceptable ones.

7.9.2 Typical variations to the processes described herein and known to produce good quality composites are:

7.9.2.1 The foam core sections may require a complete cure (to 600°F) and postcure (to 850°F) after forming to shape to eliminate shrinkage prior to prefitting.

7.9.2.2 Preformed core may be cured directly to previously cured PBI/glass skins and effect a good bond.

7.9.2.3 PBI glass prepreg may be cured directly to previously cured and postcured foam core and effect a good bond.

7.9.2.4 The skin-core bonding-cure cycles may require pressures in excess of that obtainable with a vacuum bag type cure. This is generally dependent upon skin thickness, prefit accuracy and general complexity of the layup. Maximum vacuum and 15-20 PSIG autoclave pressure has been adequate for assemblies fabricated to date.