

N84 10158

LESSONS LEARNED FROM THE DEVELOPMENT AND
MANUFACTURE OF CERAMIC REUSABLE SURFACE INSULATION
MATERIALS FOR THE SPACE SHUTTLE ORBITERS

Ronald P. Banas, Donald R. Elgin, Edward R. Cordia,
Kenneth N. Nickel, Edward P. Gzowski, and Lawrence Aguilar
Lockheed Missiles & Space Company, Inc.
Sunnyvale, California

SUMMARY

Three ceramic, reusable surface insulation materials and two borosilicate glass coatings were used in the fabrication of tiles for the Space Shuttle orbiters. Approximately 77,000 tiles have been made from these materials for the first three orbiters, Columbia, Challenger and Discovery. Lessons learned in the development, scale-up to production and manufacturing phases of these materials will benefit future production of ceramic reusable surface insulation materials.

INTRODUCTION

The landing of Columbia after STS-5 on 11 November 1982 demonstrated the reality of a truly "reusable" thermal protection system. The concept of a non-ablating, rigid, reusable, ceramic insulation material was identified by a Lockheed patent disclosure in December 1960. It was recommended as a TPS for Lifting Reentry Vehicles by Lockheed in 1964 (ref. 1) and was pursued as a low level research and development effort during the early 1960's. A concentrated development effort was started in 1968 (refs. 2, 3, 4 and 5) to parallel the NASA Phase B studies that defined some early Space Shuttle configurations (ref. 6). Many lessons were learned during each phase of the evolution from laboratory development to an initial production facility in 1971 (refs. 7 and 8), and finally to the full production facility (refs. 9 and 10), which produced a shipset of tiles for the orbiter Columbia.

Lessons learned during the development and scale-up to production of three rigid, ceramic, Reusable Surface Insulation (RSI) materials and two borosilicate glass coatings will be discussed. However, the main emphasis will be on the significant lessons learned from the following manufacturing phases in the full production facility:

1. Processing of raw materials into tile blanks and coating slurries
2. Programming and machining of tiles using numerical controlled milling machines
3. Preparing and spraying tiles with the two coatings
4. Controlling material shrinkage during the high-temperature (2100-2275°F) coating glazing cycles

5. Measuring the tiles before coating and after coating glazing
6. Loading tiles into polyurethane array frames, shimming the tiles to the proper tile-to-tile gap width and machining the inner-mold-line of all tiles in an array

The RSI materials include LI-900 (Lockheed insulation at a density of 9 lb/ft³), an all-silica material developed by Lockheed Missiles & Space Company Inc. (LMSC) in 1972. A predecessor, LI-1500, (a 15 lb/ft³ density all-silica material) was developed by LMSC in 1962 (ref. 1). It was the lowest weight prime material for Lockheed's reusable lifting reentry vehicle studies (ref. 11) from 1962 until 1971 when LI-900 was developed (ref. 12). LI-2200, a 22 lb/ft³ density all-silica material, was patented by NASA ARC (ref. 13) and scaled up to production by LMSC in 1977. FRCI-12 (Fibrous Refractory Composite Insulation at a density of 12 lb/ft³) is a composite fiber RSI material. During design, development, test, and evaluation of Columbia, the need for improved thermal protection tiles was recognized. Stronger, less dense tiles more resistant to impact damage were desired. A ceramic tile material with these characteristics, in addition to the other required properties, was invented by the NASA ARC (ref. 14) and scaled up to production size billets by LMSC (ref. 15). This material, FRCI, composed of a blend of silica fibers and aluminum borosilicate fibers, is an outgrowth of LI-900 and LI-2200 technologies and basic research of high temperature materials.

The two borosilicate coatings are Class 1 and Class 2. The Class 1 (0036C) coating (ref. 16) is white and has a ratio of solar absorptance to total hemispherical emittance between 0.2 and 0.4 from -170°F to 135°F, and an emittance \geq 0.7 at 1200°F. The Class 2 or Reaction Cured Glass (RCG) coating (ref. 17) is black, and has a total hemispherical emittance \geq 0.8 at 2300°F and a ratio of solar absorptance to total hemispherical emittance between 0.7 and 1.1 from 170°F to 250°F. It is used on LI-900 tiles that experience surface temperatures from 1200°F to 2300°F and all LI-2200 and FRCI-12 tiles.

LI-900 was scaled up to production in 1975 and the first production billet for Columbia was fabricated in September 1976. LI-2200 was implemented as a pilot plant operation to produce about 100 tiles per orbiter in October 1977. After the final tile deliveries were made for Columbia and Challenger, about 3000 tiles per orbiter were made from LI-2200.

The pilot plant operation for FRCI-12 started in January 1979 under a contract from NASA ARC (ref. 15). Facility modification and the scale-up to production billet sizes started in October 1979. The first FRCI-12 production billet for Discovery (OV-103) was produced in October 1981. About 2700 FRCI-12 tiles are scheduled for installation on Discovery and Atlantis (OV-104). The processing parameters involved in the production of these materials are described in reference 18.

LIST OF SYMBOLS AND ABBREVIATIONS

APT	Applied Programmed Tool, a computer language used to drive numerically controlled milling machines
ARC	Ares Research Center
ATA	Array tile assembly, which consists of a polyurethane array frame loaded with tiles. Tile IML's are cut in the frame. The ATA is used as a shipping container and for tile installation on the orbiter.
Billet	A finished piece of LI-900, LI-2200 or FRCI-12
Breather Area	The uncoated area on the sides of the tile that starts at the coating terminator line and extends to the tile IML. The breather area allows air to vent out during ascent to preclude a loss of coating.
CADAM	Lockheed Computer Aided Design and Manufacturing system
CAD/CAM	Computer Aided Design/Computer Aided Manufacturing
CATIA	A Computer-Graphics Aided Three-Dimensional Interactive Application system developed by Dassault Aircraft in France
Class 1 Coating	White coating used for temperatures of 1200°F or less
Class 2 Coating	Black RCG coating used for temperatures between 1200° and 2300°F
Class 1 Tile	LI-900 covered on the OML and sides with a white borosilicate coating
Class 2 Tile	LI-900 covered on the OML and side with a black borosilicate coating (Class 2 coating)
Class 4 Tile	LI-2200 covered on the OML and sides with a black borosilicate coating (Class 2 coating)
Dry Density	The density of an LI-900, LI-2200 or FRCI-12 billet prior to exposure to the sintering cycle
FRCI	Fibrous Refractory Composite Insulation

FRCI-12	A rigid, composite fiber insulation made of 78% silica fibers, 22% Nextel fibers with 3% by weight of silicon carbide at an average density of 12.5 lb/ft ³
FRSI	Felt Reusable Surface Insulation
GHP	Guarded Hot Plate
IML	Inner Mold Line
IP	In-plane direction which is perpendicular to the through-the-thickness direction
LI-2200	A rigid, all-silica fibrous insulation with about 2% by weight of silicon carbide at an average density of 22 lb/ft ³
LI-900	A rigid, all-silica fibrous insulation with an average density of 8.75 lb/ft ³
LMSC	Lockheed Missiles & Space Co. Inc.
MD	Master Dimension
MDI	Master Dimensions Intersections
Mylars	Tile section cuts put on flexible heavy gauge plastic by Rockwell or LMSC and used by LMSC inspectors to measure the sides of complex tiles
NC	Numerical Control
Nested Tile	A tile that is individually measured for plan-form dimensions and has its IML cut while being held in a polyurethane nest
Nextel 312 [®]	Aluminum borosilicate fiber; a product of Minnesota Mining and Manufacturing Co.
OML	Outer Mold Line; experiences aerodynamic heating during ascent and reentry
0036B	The original Class 1 coating; a dual layer coating consisting of porous optically adjusted subcoat and fused glass topcoat
0036C	The present Class 1 coating; a single layer system that meets the optical property requirements
0050	The original Class 2 coating; a fused silica subcoat and a topcoat of 7930 frit at 8% B ₂ O ₃ with a silicon carbide emittance agent

PTX Lot	A blend of 20 Manville silica fiber lots
RCC	Reinforced Carbon Carbon
RCG	Reaction Cured Glass (the Class 2 coating)
RSI	Reusable Surface Insulation
STS	Space Transportation System
Terminator or Witness Line	The line that is put on the sides of most tiles to define the extent of the coating down the sides
TPS	Thermal Protection System
TTT	Through-the-thickness direction; also, the pressing direction during the casting operation

RSI LOCATIONS ON COLUMBIA

Over 30,500 RSI tiles were installed on Columbia by Rockwell. About 18,500 of the 23,400 tiles made by LMSC were HRSI, which is either LI-900 or LI-2200 with the Class 2 coating. The remaining 6000 tiles were LRSI, which is LI-900 with the Class 1 coating. The locations of the HRSI and LRSI tiles are shown in figure 1 along with the location of the RCC and FRSI. More details on the composition of these materials and the installation procedures used for all Orbiter TPS materials can be found in references 19 and 20.

LI-900 PROCESS DESCRIPTION

Materials

The principal component in LI-900 is all-amorphous silica fibers with an average diameter of 1.2 to 1.4 microns and lengths to 1/4 inch. During development, a major goal was to obtain a stable material that resists devitrification at elevated temperatures. This was accomplished in an extensive development program with the fiber supplier, Manville Corporation. The final product, Q-fiber, is amorphous silica with greater than 99.7 percent purity. These fibers retain their amorphous structure when exposed to a temperature environment of 2500°F for extended periods. The LI-900 system contains a colloidal silica binder that requires extensive treatment to obtain the purity required for high-temperature morphological stability.

Material Pretreatment

During the development of LI-900, certain pretreatment procedures were developed to improve uniformity and processability of the constituent materials. Maintaining uniform shrinkage characteristics was difficult early in the development of the process. This was overcome by heat-treating the fiber before processing it into billets. In addition, unfiberized glass called "shot", if not removed, causes high density, devitrified inclusions in the sintered material. To eliminate the "shot", the fiber is slurried with deionized water and passed through a hydro-cyclone cleaner (fig. 2). The cleaned fiber slurry is transferred into a centrifugal extractor to remove excess water and to form a fiber "cake", in preparation for final drying. Also, silica fiber lots received from Manville exhibit variable fiber characteristics that cause variations in billet densities. A blend of 20 Manville lots, called a PTX lot, was developed to reduce this variability (fig. 3).

LI-900 Fabrication

The LI-900 process flow is shown in figure 4. LI-900 billets are cast in two sizes, 15 x 15 x 6.5 inches and 10 x 20 x 7.3 inches. The operation is performed in an automated casting line. Prewighed amounts of fiber are loaded into twenty-six hoppers on a carousel that automatically positions and empties the hoppers sequentially. Originally, 4.9 lbs of fiber were used for each casting. Later development resulted in a change to 5.2 lbs of fiber along with

a reduced water to fiber ratio (fig. 5). These changes, plus others to be described later, resulted in improved density distribution within the billets. The fiber and a pre-determined quantity of water are combined in a tank containing a low shear mixer that uniformly disperses the fiber with minimum chopping. At the conclusion of the timed mix cycle, the slurry is automatically transferred into a casting mold positioned directly below the mixing tank.

Entrapped air bubbles are removed from the slurry prior to compressing the billet to its final cast size. This is accomplished by a combination of vibration and stirring. Care must be exercised during this operation to maintain a homogeneous dispersion of the fiber. Water is removed and the casting is compressed to a specified height in the volume adjustment operation. Concurrently, a vacuum is applied to the bottom of the mold to remove a specified quantity of water. At this stage, the standard billet contains 5.2 lbs fiber and approximately 24 lbs of water. The next step in the casting operation is the dispersion of a colloidal silica binder in the compressed casting at the weight adjustment station. The binder solution components are automatically mixed and dispensed through metering pumps. The solution is dumped on top of the compressed billet and a vacuum is applied to the bottom of the casting mold. The residual water in the billet is displaced by the binder that is dispersed throughout the casting to a specified solids concentration. Upon removal from the mold, the wet billet is weighed to provide a check that all steps of the casting operation were performed correctly. The current method, described above, is an improvement over the original method which excluded the void reduction and vacuum water withdrawal (fig. 6).

Since maintaining a uniform distribution of the colloidal silica binder in the casting is important to maintain uniform physical properties, a gelling agent is used to set the binder and prevent it from migrating during drying. The billets are dried using either a conventional oven or a microwave dryer. They are weighed after drying to assure that the specified amount of water is removed prior to sintering. Originally, the castings received a first and second sintering with an additional binder addition between sinterings (fig. 5). Later development resulted in a single sintering combined with the change from 4.9 to 5.2 lbs of fiber. The result is an improvement in billet density distribution and an increase in yield.

An additional improvement in average billet density was obtained with the implementation of a fiber compact shrinkage test. A correlation between billet density and sintering schedule was developed for each PTX lot (fig. 7). This allows adjustment of the sintering schedule to accommodate the PTX lot shrinkage characteristics which influence the billet density.

Originally, the dried castings were sintered in specially designed 3-zone tunnel kilns at a peak temperature of 2350°F. These kilns were used from 1975 to 1982. Early in 1982, the sintering operations were transferred to elevator kilns. Six side heating is utilized in these kilns compared to five side heating in the previous kilns (fig. 6). This improves the strength distribution within the billets. The sintering schedule is adjusted to produce billets with an average density of 8.8 lb/ft³ by adjusting the sintering time to accommodate the PTX lot shrinkage variations.

LI-2200 PROCESS DESCRIPTION

Materials

LI-2200 is composed of amorphous silica (Q-fiber) and a small amount of silicon carbide powder which provides additional thermal protection if the material is exposed to excessive temperatures due to coating loss at the outer surface. With LI-2200, the fiber heat-treatment is omitted, and only the hydro-cyclone cleaning is performed. Until January 1981, the fiber cleaning was performed by an air bubbling procedure which was less efficient and less reproducible than the present procedure.

LI-2200 Fabrication

The LI-2200 process flow is shown in figure 8. The billets are cast in a specially designed, manually operated casting tower in a 14.4 x 14.4 x 8 inch size. The mixing process differs from LI-900 in that the preweighed fibers are combined with water, SiC, and ammonium hydroxide into a V-blender equipped with an intensifier bar. Since LI-2200 requires a significantly higher casting density than LI-900, the slurry requires some chopping action to obtain the necessary fiber packing. After the blended slurry is transferred into the casting tower and sealed, void elimination is accomplished by applying a high vacuum to the slurry prior to billet formation. The billet is formed by removing part of the water by gravity drain, compressing the slurry to a final thickness, and then extracting additional water with vacuum.

The billets are dried in a batch oven at 450°F for 16 hours. The dry density of the LI-2200 is approximately 13 lb/ft³. Originally, this was the final operation before sintering. However, the billets sometimes exhibited cracks after sintering. An additional drying at 1000°F for 12 hours was developed and implemented in September 1981 to eliminate this problem.

The LI-2200 is sintered in elevator kilns. The sintering schedule is similar to that used for LI-900, except that the peak temperature is 2420°F. The soak time at peak temperature is adjusted to maintain final densities within a ± 2 lb/ft³ density range. Originally, the soak times were based on fiber chemistry. A more accurate method, based on fiber compact shrinkage, was developed and implemented in June 1981.

FRCT-12 PROCESS DESCRIPTION

Materials

FRCT-12 is a composite fiber material containing amorphous silica (Q-fiber) and aluminum borosilicate fibers (Nextel 312[®]), a product of Minnesota Mining and Manufacturing Company) in a fused fiber matrix. Silicon carbide powder is added for additional thermal protection as it is in LI-2200. The bulk silica and Nextel fibers are heat treated at 2200°F and 2000°F respectively to stabilize and standardize fiber properties. Hydro-cyclone cleaning is performed on the silica fibers to remove particulate contaminants, followed by drying.

Fabrication

The FRCI-12 process flow is shown in figure 9. Silica and Nextel fibers are intermixed and cast into billets using a multi-stage, wet-slurry blending process and automated casting equipment. Castings are dried using a combination of microwave and convection-air ovens to achieve optimum drying rates. Dry castings are sintered in elevator kilns using microprocessor controllers to achieve uniform, repeatable heating to the optimum sintering temperature (approximately 2400°F). The optimum sintering temperature varies as a function of FRCI composition and desired final density.

FRCI can be fabricated with a range of compositions and densities to allow tailoring the material to a specific application. An FRCI formulation with a density of 12 pounds per cubic foot and a silica to Nextel fiber ratio of 78/22, identified as FRCI-12, was developed to replace LI-2200. Other FRCI materials with densities of 8 lb/ft³ and 20 lb/ft³ have been produced. FRCI-12 tiles were substituted for approximately 2764 LI-2200 tiles on the third orbiter, Discovery. Substituting FRCI-12 for LI-2200 saves approximately 870 pounds of weight per orbiter due to the lower density of FRCI-12. Also the tensile strength design value is increased by 50 percent, and the susceptibility to coating impact damage is reduced by eliminating residual tensile strain in the coating due to a better match in coefficient of thermal expansion between FRCI and tile coating materials.

Development of full-scale manufacturing processes for FRCI-12 required considerable effort by many individuals within NASA, Rockwell International, and LMSC from October 1979 through October 1981. Several important lessons were learned during this development about the inter-relationships between processing and fundamental material properties. The development effort was complicated by the requirement to produce a tile material to meet all the existing requirements of the baseline material, while also providing improvements of significant importance to warrant replacement of proven materials.

The first significant FRCI problem was encountered during initial scale-up work on the NAS2-10134 contract. Nextel fibers did not readily disperse when blended with silica fibers in the full-scale mixing equipment. Clumps of undispersed Nextel fibers, which varied from 1/8 to 1/2 inches in length and were present in the sintered FRCI-12 material, caused unacceptable coating discontinuities on finished tiles. The original laboratory method called for wetting the Nextel fibers prior to introduction into a small lab-scale V-blender containing silica fibers. This lab-scale equipment and procedure produced an acceptable mixture of the two fibers in lab-size castings. However, when the same procedure was used in full-scale production equipment, the Nextel fibers did not disperse and Nextel clumps occurred in the finished material. An interim dispersion method, only marginally acceptable, was devised for the pilot plant operation conducted under NAS2-10134. Nextel fiber was preblended in water in a lab-size V-blender to break up Nextel clumps, mixed with an equal amount of silica fiber in the same blender to prevent reaggregation of the Nextel into clumps, and then blended with the remaining silica fiber in the full-scale blender to produce a slurry with suitable characteristics for casting. This interim dispersion method reduced the number and size of clumps in the finished material, but the method was not considered suitable for full-scale production due to the need for considerable coating touch-up. A high-speed, high-shear blender was substituted

for the small V-blender for preblending operations in the final production process. As shown in figure 10, use of the high-shear mixer for preblending Nextel fibers totally eliminated Nextel clumps in finished tiles (ref. 21).

The second significant problem was encountered during characterization testing of the pilot-plant material. The apparent¹ thermal conductivity of the pilot-plant FRCI-12 was higher than the LI-900 base line value. Rockwell's criterion for substitution of any material for LI-900 or LI-2200 was that the thermal response must be equal to or lower than that of LI-900. A guarded hot plate (GHP) apparatus is normally used to characterize the thermal conductivity of a material. Measurements can be obtained over a wide range of temperatures and pressures to establish design values. However, the $\pm 18\%$ uncertainty band associated with GHP data makes comparative measurements on different specimens, with minor variations in thermal conductivity, uncertain. Comparative measurements are more easily accommodated with the instrumented tile method (ref. 23) shown in figure 11. Tiles fabricated from different materials can be tested side-by-side in a radiant heating environment at reduced pressures. Either steady-state or transient heating conditions can be simulated. This method yields more reliable comparative results than the GHP method which is limited to testing one material at a time. Apparent thermal conductivity values for laboratory FRCI-12 were much lower than the pilot-plant FRCI-12, but still higher than LI-900, indicating that some key parameter(s) must have been inadvertently varied during scale-up. An investigation of the effects of various compositional and processing variables on FRCI-12 properties showed that apparent thermal conductivity can be affected by several factors, the most important being density change during the billet sintering cycle (ref. 24). Pilot-plant FRCI-12 experienced a density change of 5.5 lb/ft^3 during sintering, whereas laboratory material experienced a change of 4 lb/ft^3 . Full-scale production FRCI-12, which has acceptable apparent thermal conductivity (ref. 25), experiences only a 2 lb/ft^3 density change. Reducing the density change during sintering was accomplished by use of the high dry density concept, producing castings with increased fiber content (i.e. more fibers per unit volume) and sintering at a lower temperature for a shorter time (fig. 12). Reducing the time/temperature profile during sintering caused a reduction in average tensile strength of the material compared with pilot-plant material. However, use of six-sided heating in the kiln in place of the five-sided heating resulted in a more uniform strength distribution within the billets and provided design tensile strength values nearly equal to the pilot plant values by lowering the deviation.

Another significant development problem was encountered when attempts to achieve FRCI-8 (8 lb/ft^3) thermal conductivity equivalent to LI-900 were unsuccessful. Minimizing the change in density during sintering was not sufficient to produce FRCI-8 with acceptable thermal conductivity. A combination with other, less significant, thermal conductivity "drivers" was necessary. Experiments showed that reducing the Nextel fiber concentration in the material formulation and reducing the size of the silicon carbide particles in the material, provided the additional reduction in thermal conductivity that was required (ref. 26). Pilot-plant FRCI-8 experienced a change in density during sintering of 2.5 lb/ft^3 , has a silica to Nextel fiber ratio of 78/22, and contained 320 mesh silicon carbide particles. Full-scale FRCI-8, which has acceptable

1 For porous materials, the term apparent thermal conductivity is used to denote heat transfer within the material by solid conduction, gas conduction and radiation (ref. 22).

thermal conductivity, experiences only a 1.5 lb/ft³ change in density, has a silica to Nextel fiber ratio of 85/15, and contains 600 mesh silicon carbide particles. Reducing the change in density during sintering was accomplished by producing castings with higher dry density and sintering at a lower temperature for a shorter time (fig. 13). Reducing the time/temperature profile during sintering and reducing the concentration of Nextel fibers in the formulation resulted in lower average strength for the full-scale material compared with the pilot-plant FRCI-8. However, use of six side heating in the kiln instead of five side heating, and increasing the heat-up rate to the sintering temperature (to minimize shrinkage during heat-up and maximize time above the critical fiber bonding temperature of 2350°F) resulted in a more uniform strength distribution within the billets and provided design allowable strength values nearly equal to pilot plant values.

TILE PROCESS FLOW

The sequence of operations performed after the insulation material is cut into cubes is shown in figure 14. After the tiles are machined, they are heat cleaned to remove organic contaminants, masked to allow an uncoated breather space area along the lower perimeter adjacent to the IML, sprayed with Class 1 or Class 2 coating and sintered at 2100 to 2250°F in an Ipsen tunnel-hearth roller kiln. After vacuum waterproofing with a methyl trimethoxy silane (Dow Corning DC 6070), the tile identification number is painted on the OML and the tiles are checked dimensionally as required prior to the IML cuts.

The tile IML cut is performed either individually, which is called a "nested" tile, or on a group of tiles simultaneously in an array frame, which is known as an ATA.

The dimensions of a nested tile are checked as required prior to the IML cut. The dimensions of the ATA tiles are checked by their ability to fit into a premeasured polyurethane array frame with the required tile-to-tile gaps, which are generally 0.045 ±0.016 inch on the lower wings and fuselage and 0.055 ±0.016 inch on the upper wings, fuselage and vertical fin.

ENGINEERING

Engineering Data Flow

Engineering data, which is used to define the tile and array frame geometries, is received from Rockwell in the form of engineering assembly drawings, tile bounding plane data and inner/outer mold line data (fig. 15). Only 115 of the 23,400 tiles that Lockheed made for OV-102 are defined by conventional engineering drawings. The mold line data can be represented by points (X, Y, Z coordinates) and corresponding normal vectors (MDI data), and recorded on a magnetic tape and/or contained as surface definitions described in the Master Dimensions Specification Book, Document No. MD-V70, Rockwell International. The MDI data are transformed from an orbiter coordinate system to a local tile/array coordinate system that is compatible with the APT language. These transformed data are stored in a geometry file that is accessed by the NC programmer for use in preparing the part program to machine tiles.

A tile machining drawing is used to determine the proper tile shrinkage compensation factor (see Tile Measurement and Shimming Methods section) to include in the tile part program.

The geometry file, which contains the tile boundary planes and the OML and IML surfaces, is also used to write the NC part program to machine the array frame. LMSC fabricated 739 array frames for Columbia. Product Assurance Inspection Standards are also prepared for use in inspection of the tiles on the Cordax measuring machines.

Master Dimension Refinements

Almost 18,500 of the 23,400 tiles LMSC made for OV-102 were MDI tiles, which are defined in a grid of X, Y, Z coordinates and corresponding normal vectors. The remaining tiles are the more complex MD tiles, which have their geometries defined in the Master Dimensions Specifications book. Substantial refinements have occurred in the procedures used to define the 4900 MD tiles. Originally, hand calculators or personal computers were used to calculate points to approximate complex surfaces, tile and array corner points, Product Assurance Inspection Standards, and points to check the accuracy of inspection aids (mylars) furnished by Rockwell (table I). Surfaces which could not be analytically defined were approximated by calculating points and passing a curve through these points.

Software, in the form of APT and FORTRAN computer programs, is now being used to calculate tile and array corner points and to provide accurate blank sizes. Additional software and a CAD/CAM system are both used to develop and check the accuracy of complex surfaces. This same CAD/CAM system is used to provide mylars to check hard-to-inspect tiles, reducing the time required for tile inspection. The accuracy of certain Rockwell furnished mylars is checked at LMSC using the CAD/CAM system if a discrepancy is noted during the inspection process.

Mylars were seldom used for Columbia tiles. However, for Challenger and Discovery, mylars were used extensively for hard-to-measure tiles. For example, 123 complex hinge cover tiles, which contain conical surfaces, ruled surfaces and through holes, were recently delivered for Discovery ahead of schedule. This success was due to a joint LMSC-RI effort to make 28 mylars that were used to inspect these tiles.

NC Programming Refinements

Initially, an attempt was made to automate tile programming by using a cut package to program each family of tiles (table II). Each minor difference in tile geometry required a different cut package which was inefficient and often difficult to use. Limited knowledge of the unusual and complex surfaces involved made the programming task very difficult and time consuming. Programmers experienced many failures before being able to visualize a tile, working from just the master dimensions data. It required 90 NC programmers working for ten months, using more than 160 hours per week of Univac 1108 computer time. An interactive graphics system was not available to program tiles.

Tiles with planar sides were programmed as planar surfaces. This resulted in corner shrinkage when the tile coating was glazed (see Tile Shrinkage section). One cutting tool was used to machine most tiles. Since no coating terminator line was machined on complex tiles, problems were encountered when these tiles were masked for spraying and a high rejection occurred for tiles with insufficient breather area (fig. 16). The use of different fixtures to locate different size blanks on the three Danley Corp. NC machines caused a relatively high percentage of tiles to be scrapped due to operator error in locating the blank.

Programming and tile machining are now more efficient due to knowledge and experiences gained over the life of the program. About 6000 tool tries were required to develop the proper part programs for the tiles on Columbia. About 1400 tool tries were required in connection with design changes on Challenger and about 600 tool tries as a result of design changes were required on Discovery. Planar sides are now programmed as cylinders with 900-inch radii to reduce tile corner shrinkage. About 30 electroplated diamond tools ranging in diameter from 1/8 to 2 inches were designed during the first year of production and are now used to reduce machining time. A coating terminator line is now added to most complex tiles to facilitate coating spraying and reduce the number of tiles scrapped or reworked due to incorrect breather area. The same fixture is also used on the NC mills to locate all blanks, regardless of size. This change has greatly decreased the number of tiles scrapped because of operator error in locating the blank on the bed of the NC machine.

Interactive Graphics

The recent use of an interactive graphics system (CATIA) to program the redesign of specific complex tiles has demonstrated that this method of programming reduces the number of tool tries required before an acceptable part can be made. The NC programmer has the ability to replay the cutter motion on the graphics system terminal and correct any errors observed prior to machining the first tool try.

The expanded use of an interactive graphics system for the redesign of the more complex HRSI tiles would markedly reduce both the cost and time required to manufacture these tiles (fig. 17). An example of how this system would work follows. A three dimensional engineering model of a given tile is constructed on the system. The tile model can be rotated on the terminal scope, showing all facets and all surface intersections. The model is then accessed, the cutter motion is added, the cutter motion is replayed to check for and correct errors, and then a tape is produced and sent to the machine shop for a tool try. Any errors encountered during the tool try can be easily corrected using the same engineering model. However, as the NC programmer becomes more proficient with the 3-D model, this sequence should minimize the number of tool tries. In addition, Product Assurance can access the same engineering model and extract the attributes necessary to inspect the tile.

BOROSILICATE COATINGS

Class 1 Coating

Development of the Class 1 (white) coating was a significant challenge

because of the stringent optical property and weight requirements. The optical property requirements are a ratio of solar absorptance to total hemispherical emittance between 0.2 and 0.4, to achieve low temperatures while on orbit, and an emittance of 0.7 at 1200°F to allow maximum re-radiation of the convective heating energy during reentry (fig. 18). An intensive development program was successful in producing a dual layer coating that was started in production in October 1977. This coating (0036B) consisted of a fused, water-impervious topcoat of clear glass plus zinc oxide, over a porous subcoat that contained aluminum oxide for high reflectance and silicon carbide for high emittance. The subcoat required drying at 1300°F prior to spraying the topcoat. After both layers were applied, glazing at 2100°F was required to produce a water-impervious, dual layer coating.

In mid 1978, effort was directed toward combining the dual layers while retaining both the optical properties and the water imperviousness. A single layer coating (0036C) was successfully developed, qualified and implemented into production in early 1978. During this period, the maximum coating weight requirement was increased from 0.09 to 0.12 lb/ft² to alleviate a coating cracking problem which was unavoidable with a 0.008 inch thick coating. This coating was successfully applied to about 5700 tiles for orbiter 102. For Challenger, OV-099, the maximum coating weight requirement was increased to 0.17 lb/ft². While in production for OV-102, a major water imperviousness problem affected the Class 1 coating. A three month investigation revealed that the cause was large frit particle size that precluded complete fusion (ref. 27). A complete particle size distribution requirement was determined and imposed on the frit supplier, Corning Glass Works. Particle size controls were also instituted for the coating slurry (fig. 19) to assure complete fusion during the 2100°F coating glazing cycle.

Class 2 Coating

The Class 2 (Gray) coating (0050), developed in 1974 (fig. 20), was a dual layer system that contained Corning 7930 frit with a boron content of 3% and a silicon carbide emittance agent. Because of a high coating residual tensile strain (values of 200-300 microinches per inch), crack propagation was not inhibited. This coating was replaced in June 1976 with a NASA ARC-patented Reaction Cured Glass (RCG) coating. The RCG coating is a single layer system that meets all the optical property requirements and also has lower coating residual tensile strains. It was implemented into production in May 1976 and was used on 195 tiles that were installed on the lower mid-fuselage of the Enterprise, which was used for all the subsonic aerodynamic tests at the NASA Dryden Flight Research Center. Subsequently (ref. 28), the RCG coating was shown to be susceptible to both coating impact damage and crack propagation. However, this susceptibility is probably common to any thin glass coating; RCG is a single layer coating system that was easy to scale up to a production operation and which proved to be repairable when damage occurred.

The Class 2 frit used in the RCG coating also encountered a particle size anomaly in January 1976 when the coating process was transferred to LMSC from NASA ARC (ref. 29). Examples of coating anomalies that are caused by too many fines (particle size less than 1 micron) are shown on the left side of figure 19. Within 3 months after the implementation of both the Class 1 and Class 2 coatings into the production facility, both frits and slurries were controlled by full particle size distribution requirements (ref. 30).

Universal Patch

Rigidized fibrous insulation is subject to casting voids and to scratches and gouges during handling. The initial method for filling these voids was to fill them with cured silica slip for Class 1 coated tiles and with RCG coating for Class 2 coated tiles. This process required multiple fill and drying cycles. Also, there was concern that the dense fills could vibrate loose, and further enlarge the voids.

A universal patch material was developed that has a density approximately equal to that of the tile, is applicable to both silica and FRCI tiles, is compatible with the coating glazing cycle, is capable of repairing tile edges and corners, and is easily and rapidly applied. Existing, approved Shuttle materials which are used to compound the patch material are silica fibers, colloidal silica, acrylate solution, deionized water, and a combination of fuchsin and methylene blue dyes. The slurry is simply placed into a void at twice the void volume, and flattened with a teflon spatula. After patching, the tile is dried at 1200°F for 8 minutes, and is then ready for coating. Full patch cure occurs during coating glazing. Excellent bonding of patch to tile has been demonstrated, and no crystallization occurs from exposure to a temperature of 2300°F for 15 hours. The scrap rate for damaged tiles was significantly reduced after this procedure was introduced into production.

Tile Coating Application

Class 1 and Class 2 borosilicate glass coatings are applied to tile blanks by spray application of a slurry. Tile blanks are set up on holding fixtures, masked to provide a breather space near the IML surface, and patched as necessary to cover surface deformities. Class 1 tiles are seal-coated with a suspension of colloidal silica in water and dried prior to application of the coating. Class 2 tiles are wetted with alcohol prior to application of the coating.

The amount of slurry applied to each tile is controlled by maintaining slurry viscosity, line pressure, and the number of coats within predetermined limits. The coating weight is determined "wet" and a conversion factor is applied to calculate "dry" weight. Coating weights are between 0.07 and 0.17 lb/ft² for Class 1 tiles and between 0.09 and 0.17 lb/ft² for Class 2 tiles. The corresponding coating thicknesses are 5 to 15 mils for Class 1 tiles and 8 to 15 mils for Class 2 tiles.

Several significant problems with the coating process were encountered during production of tiles for Columbia. One problem involved robot sprayers which were initially used in 1977 to coat the less complex tiles. The first 3000 to 4000 tiles, primarily Class 2, that were coated using the robots had excessively high reject and/or rework rates for coating deficiencies such as runs, non-uniformity and excess or insufficient coating thickness. Extensive experimentation with adjustments and programming of the robots indicated that the following problems could not be resolved with the existent capabilities of these first generation robots:

1. They were unable to accommodate minor variations in viscosity typically encountered with production batches of slurry.

2. There was no capability to change the speed of the robot from that used during the programming (teaching) phase.
3. Robots used twice as much slurry as manual spraying (i.e. only 45 tiles per 5 gallon batch were sprayed compared to as many as 80-100 tiles per batch for manual spraying).
4. The cassette tapes used to control the robots were not interchangeable between robots so each robot had to be taught (programmed) individually.
5. Dirt on the tape heads caused unplanned and uncontrollable motion in the robots.
6. There was no feedback loop in the system during the production spraying phase that could change the speed or the rate at which the slurry was being sprayed.

It was found that experienced coating technicians could accommodate the variations in tile geometry and slurry viscosity and provide a yield in excess of 95% for this operation (fig. 21). Use of robots was discontinued for spraying production tiles in March 1979. Current, more sophisticated robots, with advanced technology such as control by floppy disk or computer, and active feedback loops, could probably handle the mechanical problems. However, the ability to distinguish subtle changes in slurry viscosity and apply in-process corrections still appears to be handled best by skilled operators. A qualified sprayer can adjust the spraying speed to overcome any subtle changes in viscosity and can also touch up the tile as required at any time in the spraying sequence.

Another significant problem was high rejection rate for coating weight discrepancies. Coating weights were initially determined after glazing. Excess or insufficient coating caused the tile to be scrapped. A method of determining the coating weight while still "wet" was developed. The "wet weight" is determined by the operator and corrections are made if necessary before the coating dries. Underweight tiles receive an extra coat of slurry and overweight tiles are stripped and recoated (fig. 2). Another problem was changes in slurry viscosity with time. Slurry viscosity degraded with time to the point where it was too "thin" to be sprayed without running and sagging. Investigation showed that trace amounts of iron contamination were introduced by processing equipment at the frit manufacturer's production facility. The iron oxidized with time in the made-up slurry at LMSC, destabilizing the particle suspension. A heat treating procedure at 1000°F was used initially to oxidize the iron before making the coating slurries. Later, the source of the iron contamination at the manufacturer was identified and eliminated.

Another significant problem was wide variation in tile shrinkage rate and degree of coating fusion. Investigation showed that a narrow and repeatable temperature range is required to provide the desired dimensional tolerances and uniform degree of fusion. The degree of fusion not only affects appearance, but also water imperviousness. The original glazing kilns (stage tunnel kilns) were not capable of holding the desired temperature tolerances. Tunnel hearth roller kilns (Ipsen Inc.) were obtained that maintain temperatures within $\pm 10^\circ\text{F}$ between 2100 and 2300°F. Tile glazing problems were virtually eliminated by using these kilns.

Tile Waterproofing

Initially, the tiles were rendered hydrophobic (waterproof) by immersion in a hexamethyl - disilazane silicone/freon solution. Tile weight gain was about 1.0 percent. Several hundred grams of freon solvent evaporated from each tile during thermal exposure. The process provided good water imperviousness, but left a dark, carbonaceous residue when heated to between 800° and 1000°F, resulting in an increase in the ratio of solar absorptance to hemispherical emittance to above the 0.4 specification limit.

The present method involves release of a trimethoxysilane vapor into a vacuum chamber containing the tiles. The tile weight gain is only 0.1 percent. Also, the material sublimes in a char-free condition, and no change in optical properties is encountered.

Tile Shrinkage

During the development of LI-900, dimensional control had not been identified as a problem since little was known about tile gap heating, and the tile dimensional tolerances were not defined. Tile shrinkage and warpage were not fully understood and the significant factors that influence tile behavior during the glazing cycle were not known.

In 1976 a series of test programs led to the realization that tile shrinkage could be correlated to a single factor, tile thickness, if all other parameters were held constant. This knowledge was aided by the utilization of precision Bendix Cordax machines, which provide accurate, repeatable measurement data for each tile. This enabled Lockheed engineers to develop a clear and complete picture of tile dimensional changes during glazing.

The initial results showed that tile length or width changes were related to the glazed tile thickness. A best-fit, least-squares logarithmic equation was developed using the available data. Reference 31 describes the details of this activity.

A plot of the basic offset equation, which reflects the use of the Richmond III glass melt, is shown in figure 23. Notice that the curve crosses the dashed line of zero shrinkage at a sintered tile thickness of 2.1 inch. This means that "thin" tiles experience a net shrinkage, while thick tiles "grow" due to addition of the coating on the sides.

The basic offset equation was used for most of the 15,000 NC tile part programs that Lockheed wrote for OV-102. These programs are relatively expensive software and are not easily changed. After a new glass melt, Waterville 1, was put into use, it was discovered that tiles made from the new glass melt fibers did not shrink the same as tiles from the original Richmond III glass melt. Therefore, another test program was conducted and a new offset equation was developed.

A plot of this new equation is shown in figure 23. Waterville 1 tiles shrink more than Richmond III tiles. The difference is significant when compared to the allowable side tolerance of ± 0.008 inch. Since it was not cost-effective to revise the NC part programs to correct for the increased shrinkage

of the new melt, it was decided to continue using the existing NC software and to apply an offset correction at the time the tile is machined. Figure 23 shows the offset for a given glazed tile thickness. A table of tile machining offsets was developed and implemented by entering a letter code on the IBM travel card that accompanies each tile. New software was written which allowed the NC machine to "read" the letter code and apply the corresponding offset to each tile side during machining.

As new glass melts were introduced into the manufacturing process, test programs were conducted to develop the appropriate offset tables (ref. 31). To date, seven melts have been used for all orbiters and an eighth melt is being processed. Approximately 110,000 tiles have been made from these melts to date.

TPS tiles were originally machined with planar vertical sides. Subsequent shrinkage investigations revealed that special offsets were necessary to control shrinkage during coating glazing (fig. 24). These special offsets are classified into three categories: planar, which is the type of shrinkage discussed above, side-slope and radius-type compensation. The planar type shrinkage, which is the largest of the three, accommodates planform shrinkage during the glazing cycle. The side-slope and radius compensations are smaller in magnitude and accommodate distortion shrinkages. The side-slope distortion occurs because the OML edge shrinks more than the base of the tile. Radius compensation is necessary because the corners shrink more than the middle side of the tile. All adjustments are made in an equal and opposite sense and constitute typical adjustments made to NC machine part programs.

The solution to the side slope distortion problem was simple and economical. Since the original part programs used the coating terminator line as the drive path, a conically shaped tool having the same diameter at the tip as the cylindrical tool was designed (fig. 26). The cone angle was designed to give optimum offset to a majority of tiles that were manufactured by LMSC. With the same diameter at the tip as the cylindrical tool, the same part programs could be used with no changes.

The radius type compensation is made by simply machining a cylinder through a point at the center of the tile side. This results in more material left at the corners to accommodate the "pillow" type distortion.

During the early stages of Columbia tile delivery, simple (square-flat) tiles were manufactured. When more complicated tiles were fabricated, dimensional problems occurred. Tiles whose sides were not parallel or normal to each other (i.e., tiles with a wrap-around OML) had a high dimensional rejection rate. An investigation revealed that the shrinkage normal to the in-plane direction, which is defined as the through-the-thickness direction, is about three times larger than the in-plane shrinkage (fig. 27). The in-plane direction usually denotes a plane that is parallel to the orbiter surface. The shrinkage and distortion of these surfaces follow a complex relationship. Since the numerically controlled machines have limitations in application of automatic offsets to only the in-plane direction, which usually lies parallel to the machine bed, no letter offset method exists to adjust the part programs. As a result, a fixed offset is used in the NC part program for through-the-thickness shrinkage corrections. For example, all eleven cove tiles receive a fixed through-the-thickness offset for a specific silica glass melt. Wraparound and step tiles are treated in a similar manner (fig. 27).

Tile Measurement and Shimming Methods

Most of the OV-102 tiles had to be measured and verified for planform dimensional conformance prior to loading into array frames to machine the IML (fig. 14). Consequently, an automated system to measure tiles was implemented. Two Cordax measuring devices were programmed to automatically summon the inspection standards from a host computer, locate the tile on the machine bed and automatically determine the acceptability of the tile by using a series of 6 to 12 predetermined touch points on the tile sides, and 5 touch points on the OML surface.

The average time to measure a tile using a Cordax machine is 5 to 15 minutes. Another device, the "maxi-measure" (fig. 28) was developed to reduce the load on the Cordax measurement machines. This device consists of two parallel plates that measure the maximum dimension of tiles with parallel sides. The average measurement time for the "maxi-measure" device is less than one minute.

After about 70% of the tiles were fabricated for OV-102 the "load-and-go" concept was implemented to reduce the time required to dimensionally inspect tiles and to increase the rate of ATA deliveries to Rockwell. As shown in figure 29, the concept consists of an initial measurement of the array frames with aluminum templates or by probe on a large bed NC mill. The tiles are then loaded into the frame and shimmed to the proper gaps. If the proper tile-to-tile gaps are obtained, the IML's of all tiles in the array are cut as a group on one of the two large bed NC mills. The ATA's are then shipped to Rockwell. The advantages of the "load-and-go" concept are:

1. The number of tiles that must be checked for planform dimensions is greatly reduced.
2. Tile planform dimensional outages greater than ± 0.015 inch per side are allowed but the proper tile-to-tile gaps are maintained, and the overall array dimensions are to print.
3. ATA's that did not shim to the minimum tile-to-tile gap were reworked by refiring an entire row of oversized tiles to reduce their planform dimensions (Fig. 30).
4. If the ATA cannot be shimmed to the maximum tile-to-tile gap selected, tiles are remade to allow the ATA to shim properly.

For OV-102, about 3600 tiles were shipped to Rockwell in 170 ATA's using the "load-and-go" concept. For OV-099, which was the first shipset to use the "load-and-go" concept for all ATA's, 3,200 tiles were shipped as nested tiles and 20,500 tiles were loaded into 745 ATA's under the "load-and-go" concept. For Orbiters 103 and 104, which have about 18,200 LMSC tiles, about 2,100 tiles are nested and 15,800 tiles will be loaded into 535 arrays under the "load-and-go" concept.

Material Physical Properties

LMSC has had responsibility for material characterization tests of all the ceramic RSI materials. Rockwell has had responsibility for performing the systems tests on all RSI materials including RCC and FRSI. Figure 31 shows typical average room temperature physical properties developed by LMSC for

LI-900, LI-2200 and FRCI-12. More detailed data along with values at both elevated and cryogenic temperatures can be obtained in reference 19.

CONCLUDING REMARKS

This paper has presented a multitude of lessons learned in the development and scale-up to production of three ceramic RSI materials and two borosilicate coatings that were used in the manufacture of tiles for the first three orbiters: Columbia (late 1976 to early 1979), Challenger (April 1979 to March 1982), and Discovery (March 1982 to present). These improved methods, which are summarized in Table III, are presently being used in the fabrication of tiles for Atlantis, the fourth orbiter.

The effectiveness of the lessons learned is revealed in the overall tile yields: 48% for about 23,400 tiles for Columbia, 81% for about 23,800 tiles for Challenger and 88% for about 18,200 tiles for Discovery. With the deletion of certain planform measurements of nested tiles on 1 February 1983 as a result of an expanded process control program, the overall yield on Atlantis tiles is expected to be about 90%. While some of the increase in yield can be attributed to modified requirements, the majority of the yield increase is due to the improved methods discussed herein, primarily the addition of coating terminator lines on most tiles, the addition of homing devices on the NC mills, the reliance on real time process control for coating weight, and the "load-and-go" concept.

Experience since the start of production in October 1976 has shown that ceramic fiber reusable surface insulations still retain some "art" in their fabrication processes as opposed to all "science". Consequently, making a consistent, repeatable product requires good process control and all changes to the process must be thoroughly evaluated prior to implementation and tightly controlled after implementation.

Another lesson that has been illustrated through the development of LI-900 (ref. 12), LI-2200 (ref. 13) and FRCI-12 (ref. 15) is that the RSI materials can be "tailored" to the application as with fiber-reinforced composites. This "tailoring" is also evidenced in the recent advanced studies of FRCI using ratios of Nextel to silica of up to 80/20 (ref. 32). Hence, these families of RSI materials offer the designer a very flexible design concept.

Finally, if LMSC were to introduce a new, man-rated ceramic RSI material into production for an advanced Shuttle or Orbital Transfer Vehicle, the minimum changes that would be introduced are:

- o Vacuum degassing of casting slurries
- o Addition of silicon carbide particles to the billets for emittance retention of the RSI in the event of coating loss during entry
- o Use of an interactive graphics system like CATIA or CADAM for the design of tile geometries and to provide an automated method to write NC part programs
- o Implementation of real-time process control in critical manufacturing areas

- o Consideration of different coating and tile concepts if rewaterproofing is required after every flight (i.e., tiles with larger planform dimensions and fewer if any material shrinkage problems in the absence of a coating glazing cycle)

REFERENCES

1. Thermal Protection Concepts for Lifting Entry Vehicles. Lockheed Missiles & Space Co. Report 6-62-64-5, May 5, 1964.
2. Hammitt, R. L.: Lightweight Insulation, LI-15, Test Summary. Lockheed Missiles and Space Co., LMSC-685434, Code 9999, Apr. 26, 1968.
3. Rusert, E. L.: Development of a Rigidized Surface Insulative Thermal Protection System for Shuttle Orbiter, Final Report. NASA CR-11-973, Feb. 15, 1971.
4. Banas, R. P.; Kural, M. H.; Deruntz, J. A.; Burns, A. B.; Chinn, A. J.; Lambert, R.; Ritz, J. P., Vanwest, B.; and Woneis, J. T.: Space Shuttle Thermal Protection System Development. Lockheed Report LMSC-D152738, Lockheed Missiles and Space Co., Space Systems Division, Jan. 17, 1972.
5. LI-1500 Heat Shield, Independent Development Program C548. Lockheed Missiles and Space Co. Report LMSC-D153908, Dec. 24, 1971.
6. Space Shuttle Concepts. Final Report for NAS8-26362, Lockheed Missiles and Space Co. Report LMSC-D153024, Mar. 15, 1972.
7. Development and Design Application of Rigidized Surface Insulation Thermal Protection Systems. Lockheed Missiles and Space Co. Report LMSC-D282673, Dec. 30, 1972.
8. A Proposal for Space Shuttle Orbiter High-Temperature Reusable Surface Insulation. Lockheed Missiles and Space Company Report LMSC-D336595, Mar. 14, 1973.
9. Forsberg, K.: Producing the High-Temperature Reusable Surface Insulation for the Thermal Protection System of the Space Shuttle. Presented at XIVe Congress International Aeronautique, Paris, France, June 1979.
10. Burns, A. Bruce and McCarter, C. R.: Manufacture of Reusable Surface Insulation (RSI) for the Space Shuttle Orbiter. Presented at the 1980 SAE Aerospace Congress & Exposition, Oct. 15, 1980.
11. Banas, R. P.; Dolton, T. A.; Houston, S. J.; and Wilson, R. G.: Lifting Entry Vehicle Thermal Protection Review. Proceedings of Thermodynamics and Thermophysics of Space Flight. Palo Alto, California, 1970, pp. 239-276.
12. Improvement of Reusable Surface Insulation Material. Final Report for NAS9-12137. Lockheed Missiles and Space Co. Report LMSC-D266204, Mar. 1, 1972.
13. Goldstein, H. E., Smith, Marnell and Leiser, Daniel: Silica Reusable Surface Insulation. United States Patent 3,952,083, Apr. 20, 1976.
14. Leiser, Daniel B., Goldstein, Howard, E. and Smith, Marnell: Fibrous Refractory Composite Insulation. United States Patent 4,148,962, Apr. 10, 1979 (FRCI-12).

15. Banas, R. P. and Cordia E. R.: Advanced High-Temperature Insulation Material For Reentry Heat Shield Applications. Presented at 4th Annual Conference On Composites and Advanced Materials, Ceramic - Metal Systems Division - American Ceramic Society, Jan. 1980.
16. Wheeler, W. H., Garofalini, S. H. and Beasley, R. M.: Development of an Unusual Coating System for the Space Shuttle Orbiters. Paper presented at American Ceramic Society Meeting in Cincinnati, Ohio, May 4, 1976.
17. Goldstein, H. E., Leiser, D. B. and Katvala, V. W.: Reaction Cured Glass and Glass Coatings. United States Patent No. 4,093,771, June 6, 1978.
18. Banas, R. P., Gzowski, E. R. and Larsen, W. T.: Processing Aspects of the Space Shuttle Orbiter's Ceramic Reusable Surface Insulation. Proceedings of the 7th Conference on Composites and Advanced Materials, Ceramic-Metal Systems Division - American Ceramic Society, Cocoa Beach, Fla. Jan. 16-21, 1983.
19. Korb, L. J. and Clancy, H. M.: Shuttle Orbiter Thermal Protection System: A Material And Structural Overview. Presented at the 26th National Symposium Society for the Advancement of Materials and Processes Engineering, Paper No. STS 81-0219, Apr. 28-30 1981.
20. Dotts, R. L., Battley, H. H., Hughes, J. T. and Neuenschwander, W. E.: Space Shuttle Orbiter - Reusable Surface Insulation (RSI) Flight Performance. AIAA paper No. 82-0788-CP, May 1982.
21. Holmquist, G. R. and Cordia E. R.: Advances in Reusable Surface Insulation for Space Shuttle Application. MATERIALS 1980 - Proceedings of the 12th National SAMPE Technical Conference, Volume 12. Society for Advancement of Materials and Processes Engineering, Oct 1980.
22. Banas, R. P. and Cunningham Jr., G. R.: Determination of Effective Thermal Conductivity for the Space Shuttle Orbiter's Reusable Surface Insulation (RSI). AIAA Paper No. 74-730, Thermophysics & Heat Transfer Conference, Boston, Ma., July 1974.
23. Williams, S. D. and Curry, D. M.: Nonlinear Least Squares - An Aid to Thermal Property Determination. NASA TM X-58092, June 1972.
24. Holmquist G. R., Cordia E. R. and Tomer, R. S.: Effects of Composition and Processing on Thermal Performance of a Rigidized Fibrous Ceramics Insulation Material. Ceramic Engineering and Science Proceedings - 5th Annual Conference on Composites and Advanced Materials, Volume 2, American Ceramic Society, July 1981.
25. Elgin, D. and Schirle, J.: Thermal Performance of a Fibrous Refractory Composite Insulation (FRCI). Presented at 5th Annual Conference on Composites and Advanced Materials. American Ceramic Society. Merritt Island, Florida. Jan. 18-22, 1981.

26. Tomer, R. S. and Cordia, E. R.: Development of an Improved Lightweight Insulation Material for the Space Shuttle Orbiter's Thermal Protection System, Ceramic Engineering and Science Proceedings - 6th Annual Conference on Composites and Advanced Materials, Volume 3, American Ceramic Society, pp. 601-611, Sept. 1982.
27. Tanabe, T. M. Izu, Y. D.: Influence of Particle Size Distribution on Ceramic Coating Characteristics. Presented at 32nd Pacific Coast Regional Meeting of American Ceramic Society, Seattle, Washington, Oct. 24-26, 1979.
28. Dotts, R. L., Smith, J. A., and Tillian, D. J.: Space Shuttle Orbiter Reusable Surface Insulation Flight Results. Shuttle Performance: Lessons Learned, NASA CP-2283, Part 2, 1983, pp. 949-966.
29. Creedon, J. F., Banas, R. P. and Stern, P.: Results of Lockheed Missiles & Space Co. Studies of NASA/ARC Reaction Cured Glass Coating for the Space Shuttle Orbiter. Presented at American Ceramic Society's 29th Pacific Coast Meeting under Ceramic-Metal Systems Division, San Francisco, Ca., Nov. 2, 1976.
30. Nakano, H. N. and Izu, Y. D.: Significance of Particle Size Distribution on Refractory Frit Sintering. Presented at 12th Annual Meeting of Fine Particle Society - Powder and Bulk Solids Conference, Rosemont, Illinois, May 12-14, 1981.
31. Fitchett, B. T.: Dimensional Control of Space Shuttle Tiles during Manufacture. Proceedings of the 7th Conference on Composites and Advanced Materials, Ceramic - Metal Systems Division, American Ceramic Society, Cocoa Beach, Fla., Jan. 16-21, 1983.
32. Leiser, D. L., Smith, M., Stewart, D. and Goldstein, H.: Thermal and Mechanical Properties of Advanced High-Temperature Ceramic Composite Insulation. Proceedings of the 7th Conference on Composites and Advanced Materials, Ceramic - Metal Systems Division - American Ceramic Society, Cocoa Beach, Fla., Jan. 16-21, 1983.

TABLE I.- MASTER DIMENSIONS REFINEMENTS

ORIGINAL METHOD	IMPROVED METHOD
<ul style="list-style-type: none"> • HAND CALCULATION OF TILE CORNER POINTS & PA STANDARDS. 	<ul style="list-style-type: none"> • N/D DEVELOPED SOFTWARE TO CALCULATE CORNER POINTS & PA STANDARDS.
<ul style="list-style-type: none"> • HAND CALCULATION OF BLANK SIZES. 	<ul style="list-style-type: none"> • SOFTWARE DEVELOPED TO PROVIDE BLANK SIZES.
<ul style="list-style-type: none"> • APPROXIMATION OF COMPLEX SURFACES. 	<ul style="list-style-type: none"> • CAD/CAM DEVELOPMENT & CHECK OF COMPLEX SURFACES.
<ul style="list-style-type: none"> • PRODUCT ASSURANCE POINTS FOR HARD-TO-INSPECT TILES. 	<ul style="list-style-type: none"> • CAD/CAM DEVELOPED MYLARS TO INSPECT COMPLEX TILES.
<ul style="list-style-type: none"> • HAND CALCULATION OF POINTS TO CHECK ROCKWELL MYLARS. 	<ul style="list-style-type: none"> • CAD/CAM CHECK OF ROCKWELL MYLARS.

TABLE II.- NC PROGRAMMING REFINEMENTS

ORIGINAL METHOD	IMPROVED METHOD
<ul style="list-style-type: none"> • CRUDE CUT PACKAGES TO PROGRAM TILE FAMILIES. 	<ul style="list-style-type: none"> • OPTIMIZED CUT PACKAGES TO FACILITATE PROGRAMMING & REDUCE COMPUTER RUN TIME.
<ul style="list-style-type: none"> • ONE INCH DIAMETER CUTTER USED FOR MOST MACHINING. 	<ul style="list-style-type: none"> • ABOUT 30 CUTTER GEOMETRIES WERE USED TO REDUCE MACHINE TIME
<ul style="list-style-type: none"> • NO COATING LINE ON COMPLEX TILES. 	<ul style="list-style-type: none"> • COATING TERMINATOR LINE ADDED TO COMPLEX TILES REDUCED SCRAP.
<ul style="list-style-type: none"> • SEPARATE FIXTURES FOR DIFFERENT BLANK SIZES. 	<ul style="list-style-type: none"> • SAME FIXTURE FOR ALL BLANKS REDUCED SCRAP.
<ul style="list-style-type: none"> • PLANAR SIDES PROGRAMMED AS PLANAR SURFACES. 	<ul style="list-style-type: none"> • PLANAR SIDES PROGRAMMED AS CYLINDERS TO REDUCE CORNER SHRINKAGE.
<ul style="list-style-type: none"> • LIMITED KNOWLEDGE OF COMPLEX SURFACES. 	<ul style="list-style-type: none"> • EXPERIENCE REDUCED PROGRAMMING & MACHINING TIME.
<ul style="list-style-type: none"> • LIMITED USE OF CAD/CAM SYSTEM TO PROGRAM TILES. 	<ul style="list-style-type: none"> • CAD/CAM SYSTEM SHOULD REDUCE TOOL TRIES.
<ul style="list-style-type: none"> • NO CHECK FOR REFERENCE POINT 	<ul style="list-style-type: none"> • HOMING DEVICES INSTALLED ON N/C MILLS ASSURE PROPER REFERENCE POINT

TABLE III.- A SUMMARY OF LESSONS LEARNED

ORIGINAL PAGE IS
OF POOR QUALITY

1. FIBER PREPARATION METHODS
 - o A 20 blend of Hanville silica fibers provides better material uniformity.
 - o Use of a hydro-cyclone removes unwanted glass shot from the silica fiber lots.
 - o Dual stage agitation of Nextel fibers eliminates fiber clumping in FRCI-12 billets.
2. BILLET CASTING AND SINTERING METHODS
 - o Precursor silica fiber lot contact tests allow better prediction of production billet sintering requirements.
 - o Elimination of an intermediate billet sintering cycle for LI-900 was successfully accomplished.
 - o Implementation of void reduction and vacuum assisted casting procedures minimize voids in LI-900 billets.
 - o Implementation of six-side heating for LI-900 and FRCI-12 during the sintering cycle improved the strength distribution within billets.
 - o Vacuum degassing of slurries for LI-2200 and FRCI-12 prior to casting eliminates voids in the billets.
 - o The high dry-density concept for FRCI-12 and FRCI-8 was successfully used to tailor the apparent thermal conductivity.
3. ENGINEERING DATA AND NUMERICAL CONTROL PROGRAMMING REFINEMENTS
 - o Experience with the Lat1 has led to many refinements in the tile part programs that have made tile fabrication more efficient.
 - o Addition of various software tools reduced the requirement to hand calculate various master dimension surfaces.
 - o The use of interactive graphics methods for tile design and NC part programming would reduce the number of tool trials and improve efficiency.
 - o Use of CAPAM to generate mylars for use as tile dimensional inspection tools eliminates the need to supply computerized tile dimensional data for hard-to-measure tiles.
4. TILE FABRICATION
 - o The addition of homing devices on the NC mill provided assurance that tiles were being cut correctly.
 - o Design changes to the cutting tool improved the consistency of the tile-to-tile gaps in the delivered tiles.
 - o An understanding of fiber shrinkage characteristics for LI-900, LI-2200 and FRCI-12 led to the implementation of tile machining offsets that resulted in tiles that meet the dimensional requirements.
 - o Implementation of letter offsets on the IBM tile travel cards with appropriate changes in the NC hardware and software provided an efficient method to modify tile machining offsets.
 - o Recognition of the anisotropic shrinkage characteristics of LI-900, LI-2200 and FRCI-12 further improved the tile dimensional yield.
5. COATINGS AND THEIR APPLICATION
 - o Use of a universal patch compound prior to coating spraying eliminated irregular surfaces and craters in the glazed coatings.
 - o Man is a more efficient tile sprayer than robots.
 - o Control of particle size for glass frits and enamel slurries eliminated most coating anomalies.
 - o Real time process control of coating weight in the manufacturing area eliminated tiles scrapped for coating weight.
 - o The propensity for cracks in the Class 1 coating was removed by increasing the maximum coating weight to 0.17 lb/ft², making it consistent with the Class 2 coating.
6. TILE MEASUREMENT AND GRINDING
 - o Introduction of the "maxi-measure" and Cordax apparatus yielded accurate dimensional data.
 - o The "Load-and-Go" concept minimized tiles being scrapped for platform dimensional anomalies, while preserving dimensional metro. at the army level.
 - o Introduction of a second glazing operation, to unsize tiles, eliminated tiles scrapped for an over-size condition.
 - o Use of mylars for complex or hard-to-measure tiles for Challenger greatly improved the determination of acceptability compared to the "ship-and-fit" criterion used for Columbia's complex tiles.

- TOTAL RSi CERAMIC TILES - 30,812 (LMSC 23,400)
- REINFORCED CARBON/CARBON (RCC) (44 PANELS/NOSE CAP)
- FELT REUSABLE SURFACE INSULATION (FRSi) (3,581 FT²)

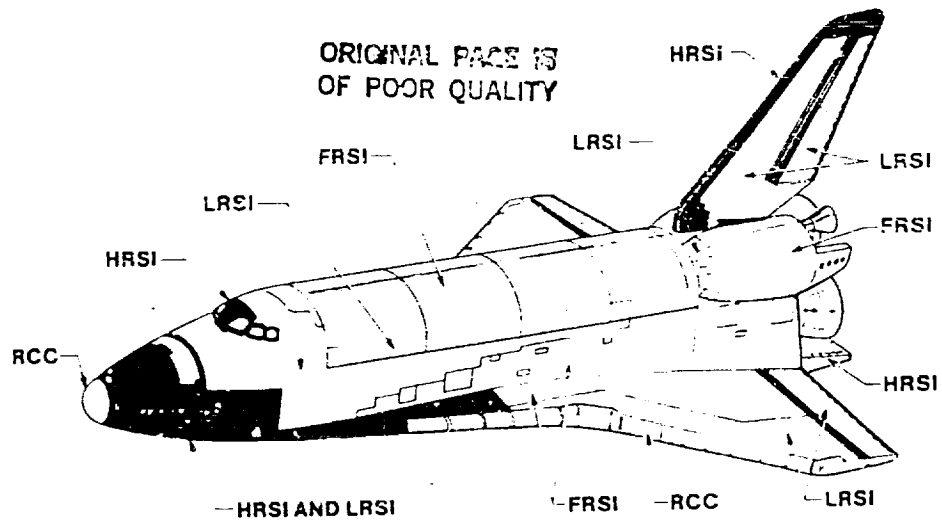


Figure 1.- TPS locations on Columbia (OV-102).

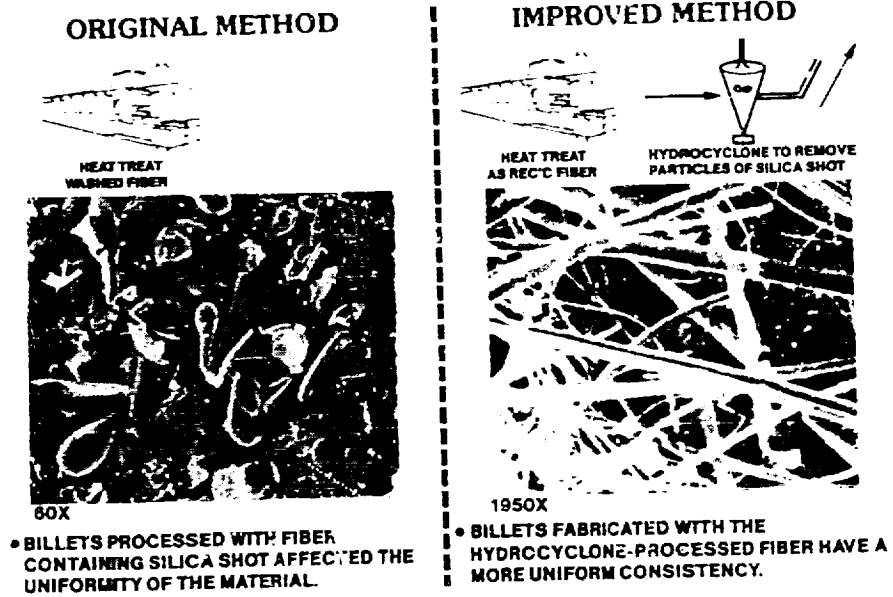


Figure 2.- Pretreatment of silica fibers for LI-900.

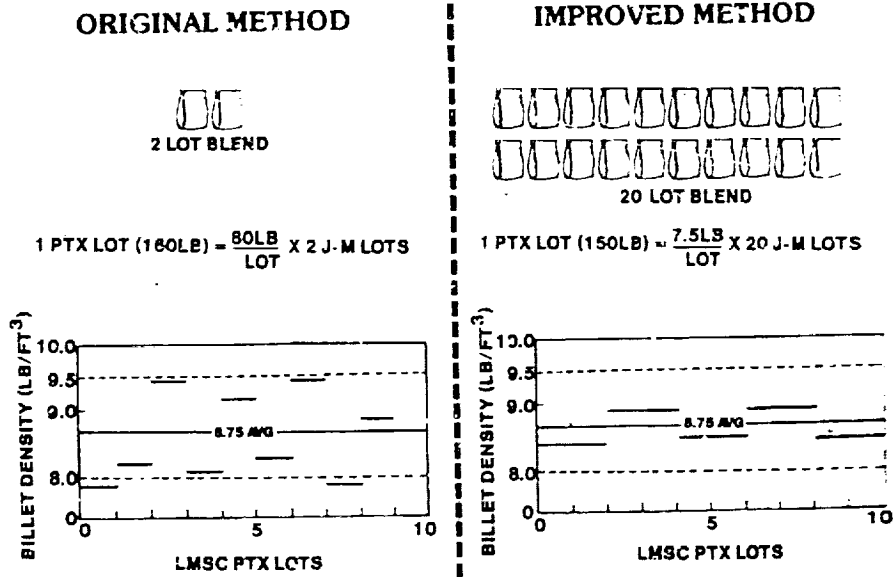


Figure 3.- Blending J-M fiber lots for LI-900.

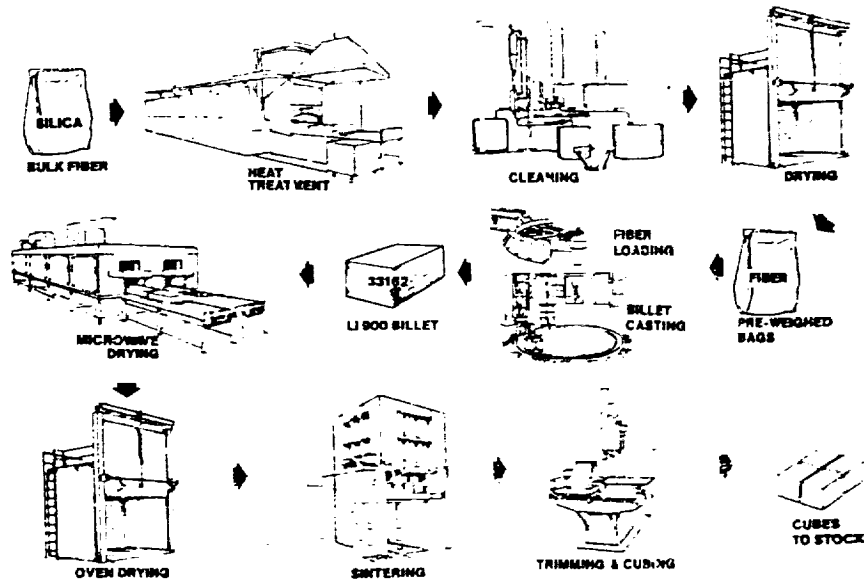


Figure 4.- LI-900 process flow.

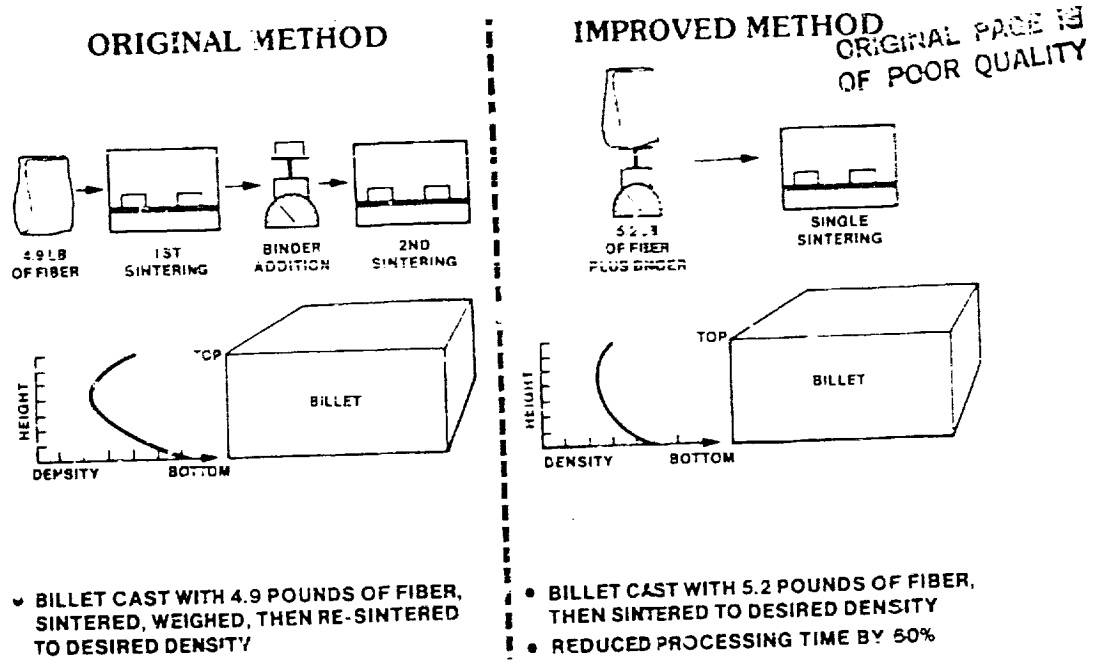


Figure 5 - Changes in LI-900 casting and sintering procedures.

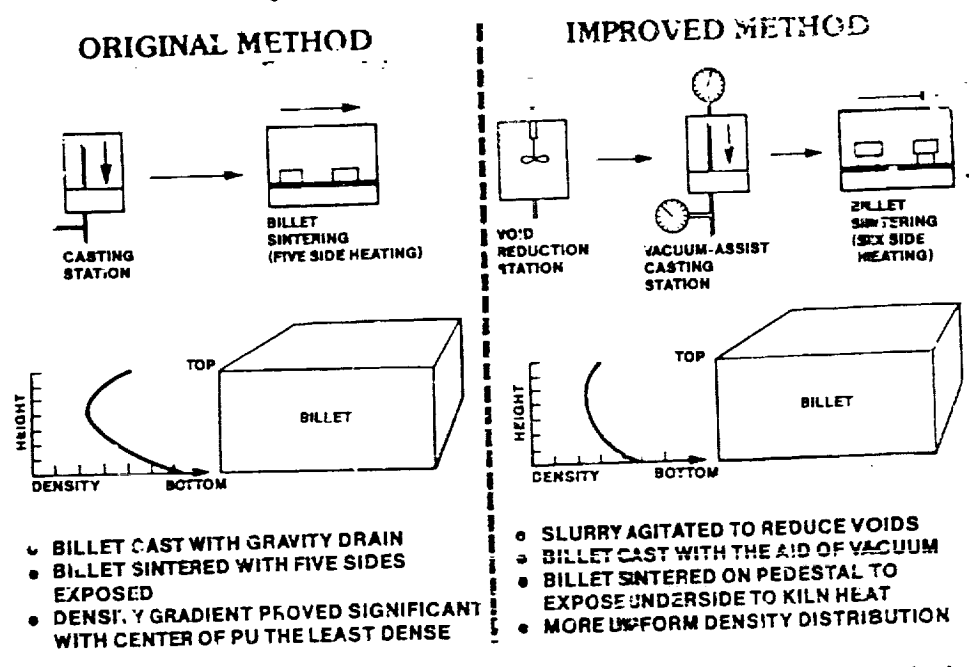
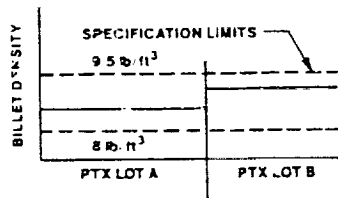
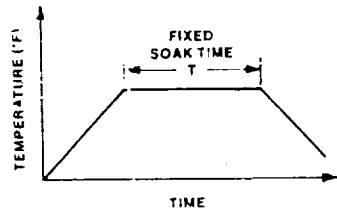


Figure 6.- LI-900 void reduction and vacuum assisted casting methods.

ORIGINAL PAGE 13
OF POOR QUALITY

ORIGINAL METHOD



IMPROVED METHOD

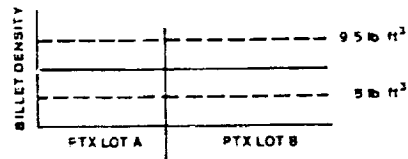
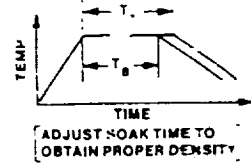
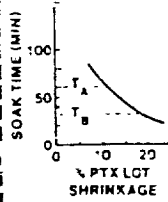
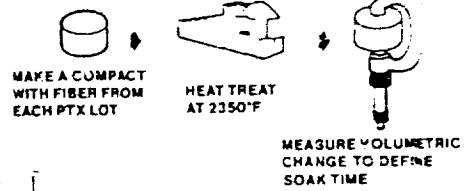


Figure 7.- Silica fiber compact test for LI-900.

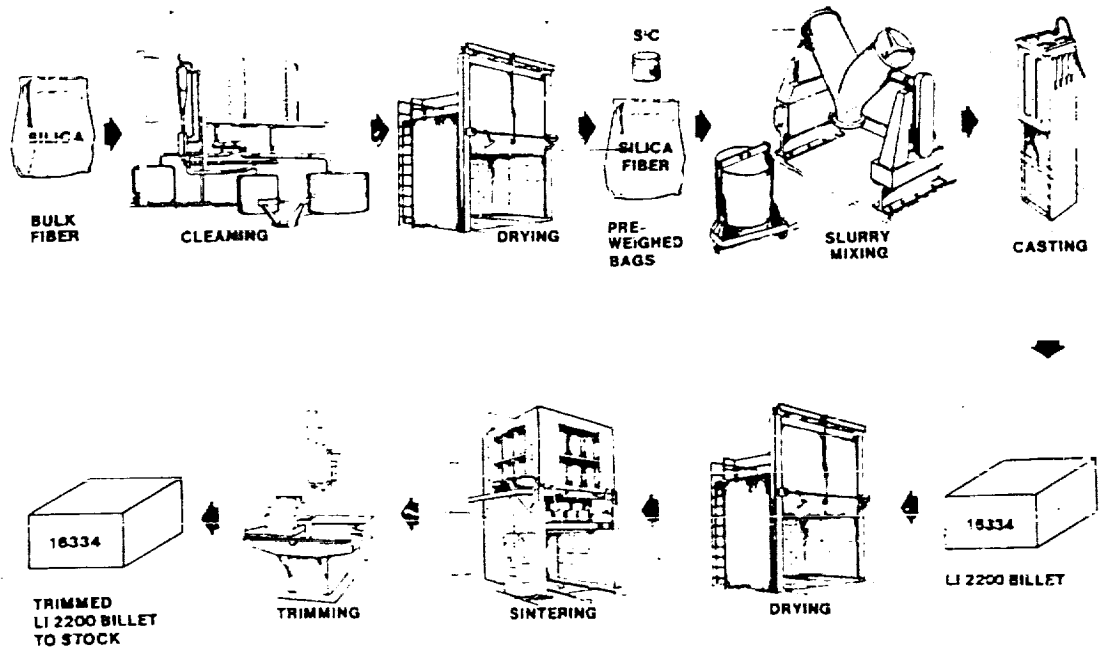


Figure 8.- LI-2200 process flow.

ORIGINAL PAGE IS
OF POOR QUALITY

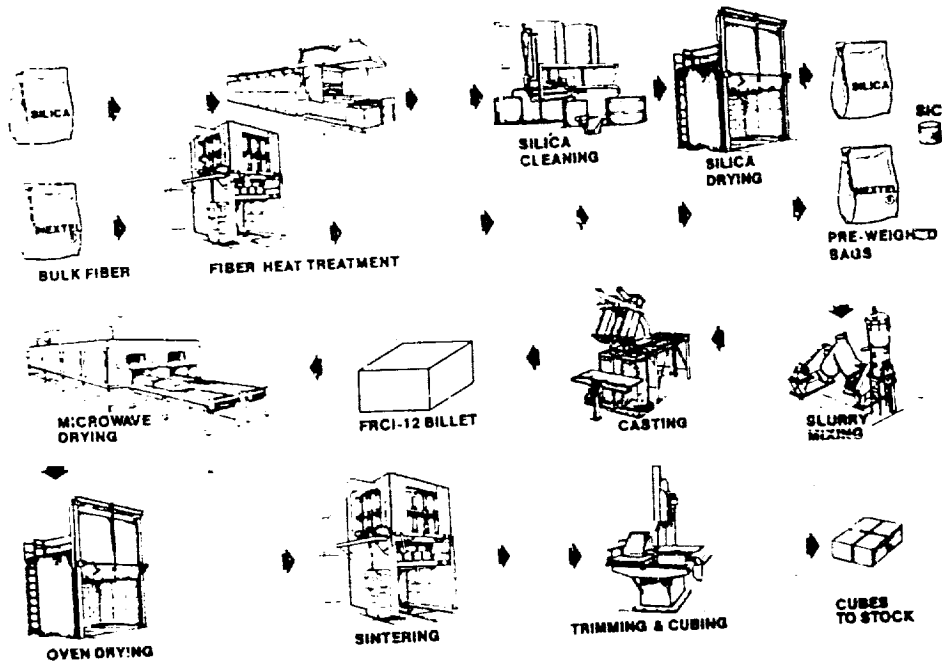


Figure 9.- FRCI-12 process flow.

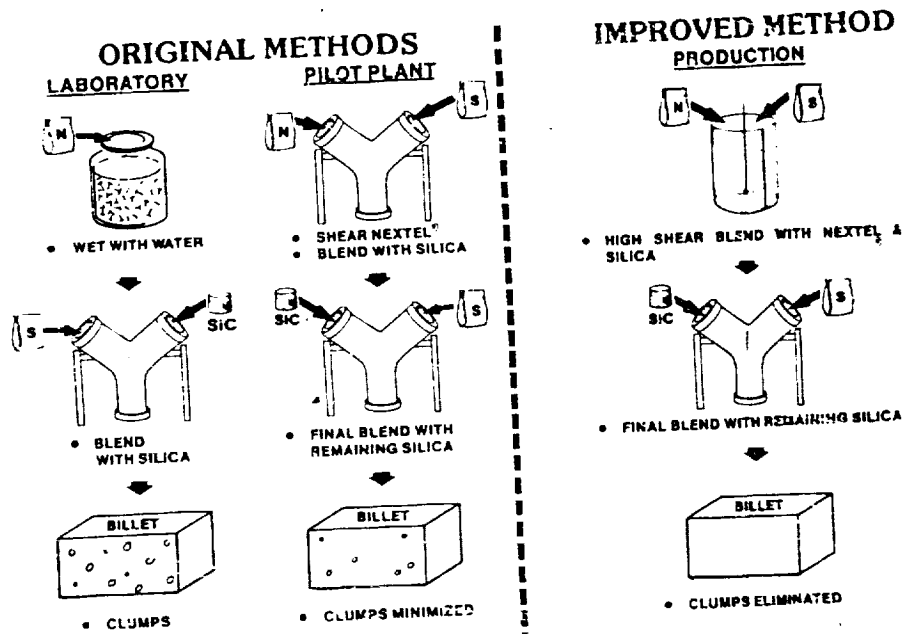


Figure 10.- Nextel dispersion methods for FRCI-12.

ORIGINAL PAGE IS
OF POOR QUALITY

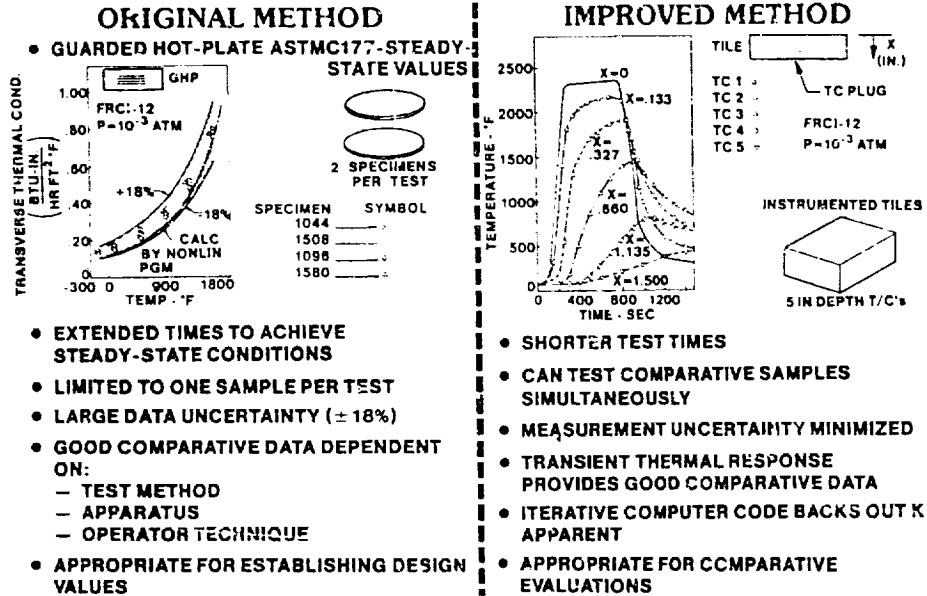


Figure 11.- Thermal performance evaluation methods.

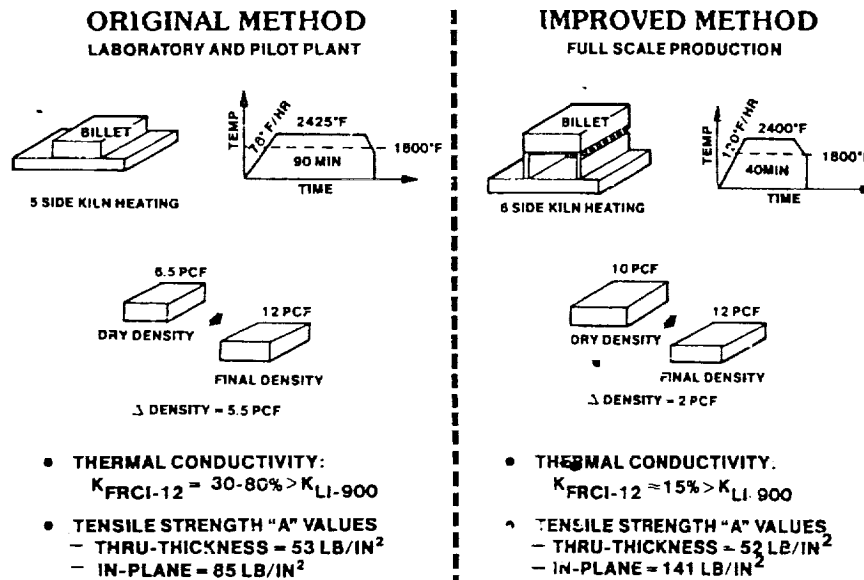
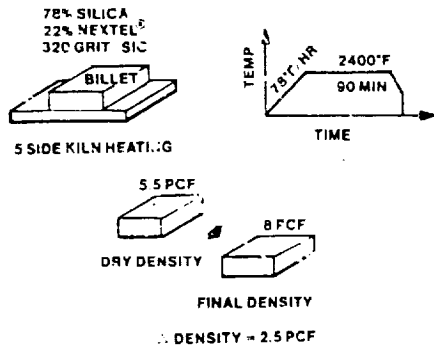


Figure 12.- High dry density concept for FRCI-12.

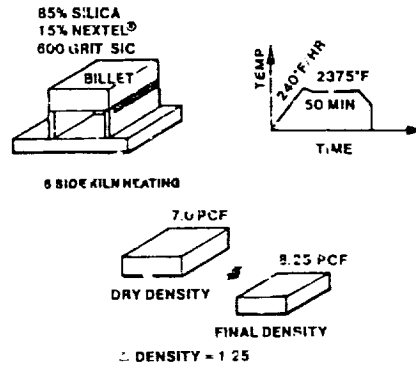
ORIGINAL PAGE 13
OF POOR QUALITY

ORIGINAL METHOD
LABORATORY AND PILOT PLANT



- THERMAL CONDUCTIVITY
 $K_{FRCI-8} = 25-55\% > K_{LI-900}$
- TENSILE STRENGTH "A" VALUE
THRU-THICKNESS = 22 LB/IN²

IMPROVED METHOD
FULL SCALE EQUIPMENT



- THERMAL CONDUCTIVITY
 $K_{FRCI-8} \approx K_{LI-900}$
- TENSILE STRENGTH "A" VALUE
THRU-THICKNESS = 26 LB/IN²

Figure 13.- FRCI-8 high dry density concept.

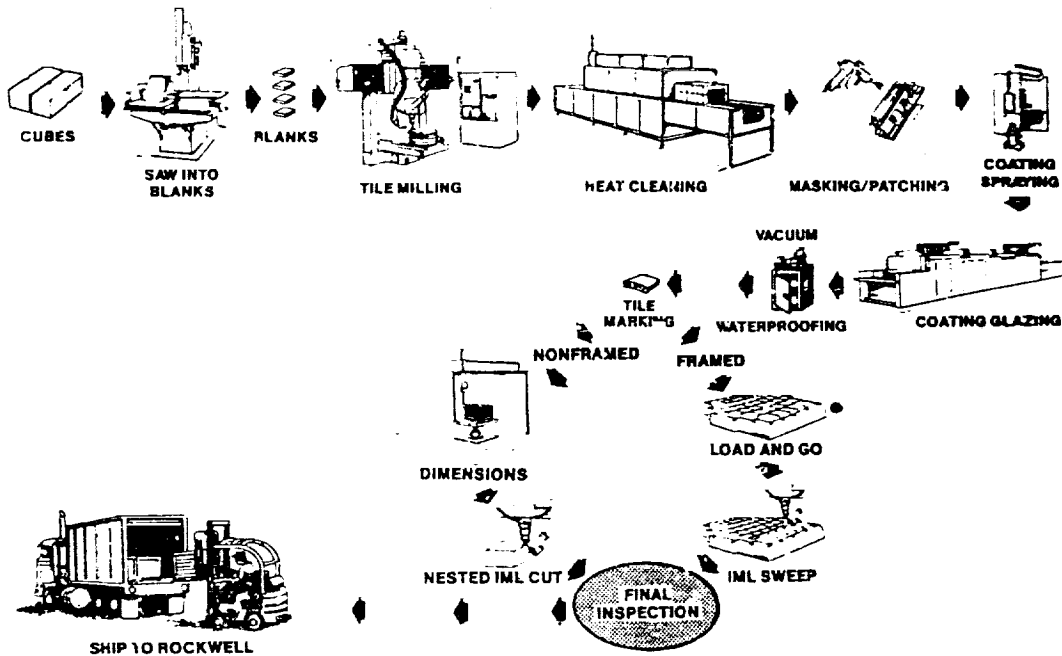


Figure 14.- The tile process flow.

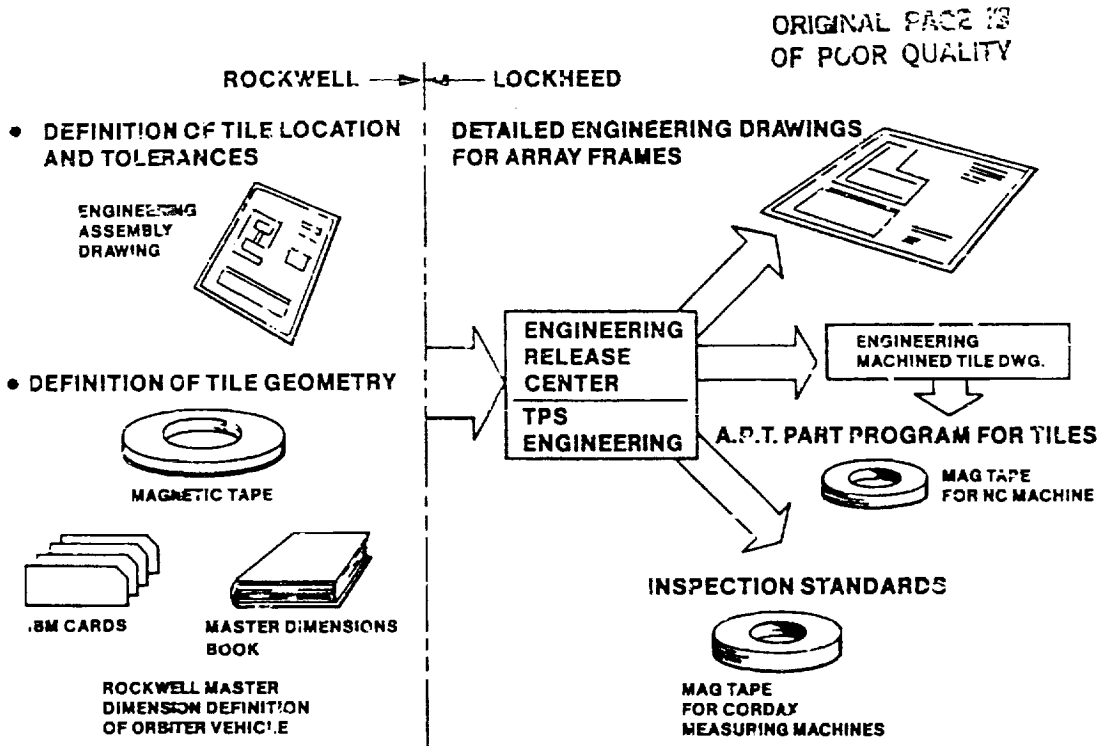


Figure 15.- The engineering data flow.

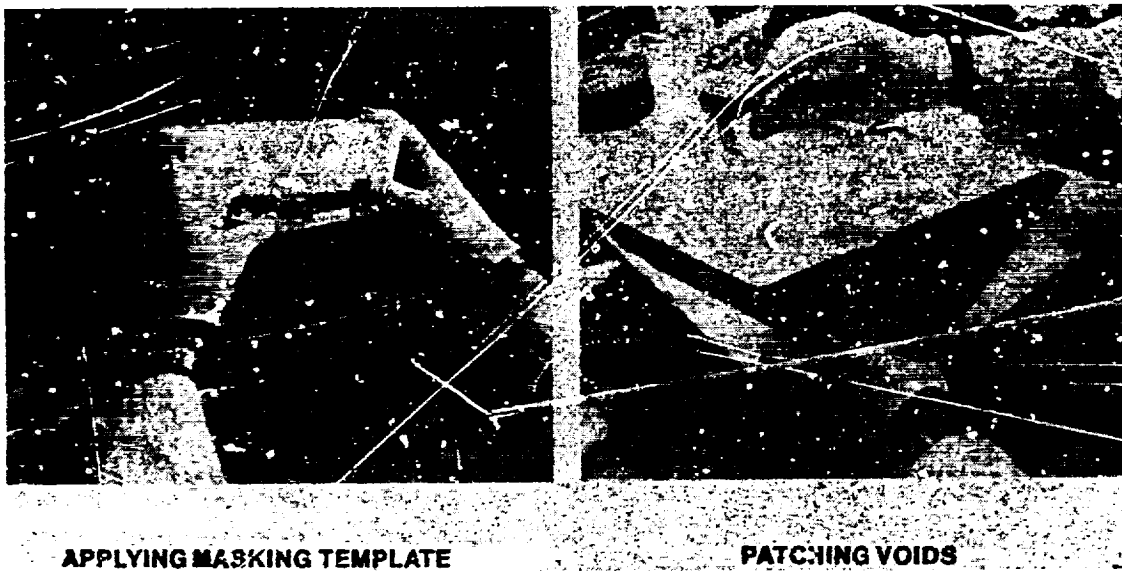


Figure 16.- Examples of complex tiles without coating terminator lines.

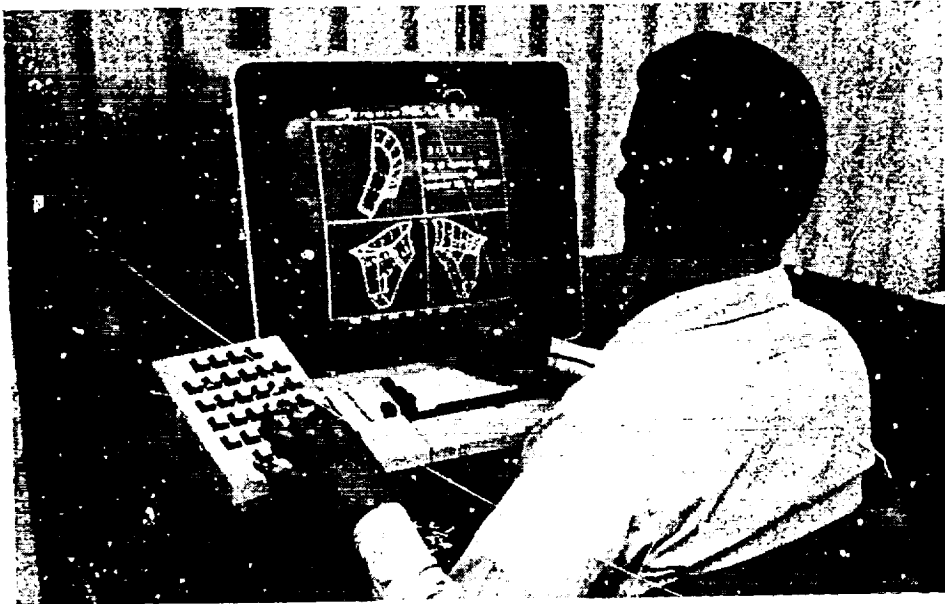
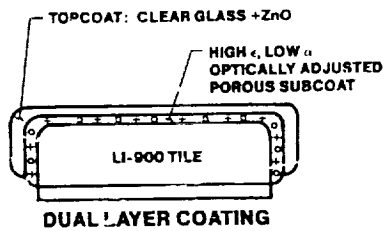


Figure 17.- An interactive graphics method showing a Shuttle infrared leeward temperature sensing tile.

ORIGINAL COATING



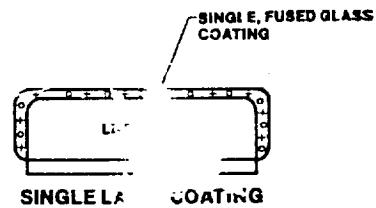
REQUIREMENTS:

- $0.2 < \alpha/\epsilon < 0.4$ - (135°F to + 250°F)
- $\epsilon \geq 0.8$ AT 1200°F
- WEIGHT ≤ 0.09 lb/ft²
- CRACK FREE AND WATER IMPERVIOUS

STATUS:

- PRODUCTION START IN OCT. 1977
- THIN COATING CRACKED EASILY

IMPROVED COATING



ADVANTAGES:

- ALL COMPONENTS IN ONE LAYER
- MINIMIZED WATER IMPERVIOUSNESS PROBLEM
- LESS RESIDUAL STRAIN

STATUS:

- USED ON COLUMBIA AT 0.12 lb/ft²
- USED ON CHALLENGER AT 0.17 lb/ft²

Figure 18.- Class I coating optimization.

ORIGINAL METHOD

• COATING ANOMALIES



"MUD CRACKING"

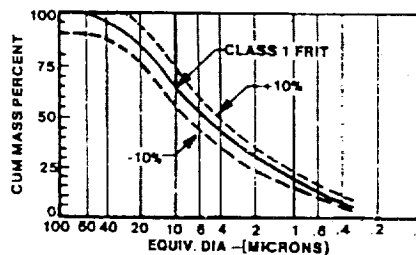
"ONION SKINNING"

STATUS:

- NO CLASS 1 FRIT SIZE CONTROL OTHER THAN 90% THROUGH 325 MESH AND A 50% POINT REQUIREMENT
- USE OF SLURRY IMMEDIATELY AFTER PREPARATION ELIMINATED VISCOSITY VARIATIONS
- NO CLASS 2 FRIT SIZE CONTROL OTHER THAN A 50% POINT REQUIREMENT

IMPROVED METHOD

• DEFINED FULL PARTICLE SIZE DISTRIBUTION FOR CL.1/CL. 2 FRITS AND SLURRIES

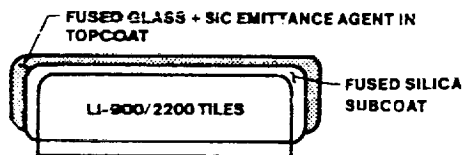


ADVANTAGES:

- SLURRY VISCOSITY CONTROL IS IMPROVED
- SPRAYING CHARACTERISTICS ARE MORE UNIFORM
- COATING CRACKS DURING GLAZING ARE ELIMINATED
- FUSION OF CLASS 1 AND CLASS 2 COATINGS IS ASSURED

Figure 19.- Effect of glass frit particle size effects on glazed coatings.

ORIGINAL COATING

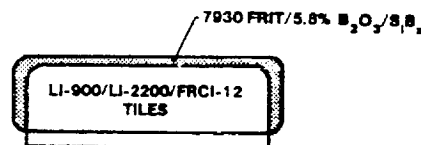


DUAL LAYER COATING (0050)

CHARACTERISTICS:

- EMITTANCE ≥ 0.8 AT 2300°F
- HIGH RESIDUAL TENSILE STRAINS (200-300 $\mu\epsilon$)
— HIGH CRACK PROPAGATION
- GLAZED AT 2500°F
- FOAMED DURING EXPOSURE TO PLASMA TESTS

IMPROVED COATING



SINGLE LAYER COATING (RCG)

CHARACTERISTICS:

- WEIGHT ≤ 0.17 lb/ft²
- GLAZED AT 2200°F
- EMITTANCE ≥ 0.8 AT 2300°F
- LOW RESIDUAL TENSILE STRAINS (50-100 $\mu\epsilon$)
— MINIMAL CRACK PROPAGATION
- REUSABLE TO 2300°F
- GOOD FOR SINGLE EXPOSURE TO 2700°F

Figure 20.- Class 2 coating optimization.

ORIGINAL PAGE #1
OF POOR QUALITY

ORIGINAL METHOD



- ROBOTS FOR SIMPLE TILES (5 COATED SURFACES)
- MAN FOR COMPLEX TILES (UP TO 20 SURFACES)

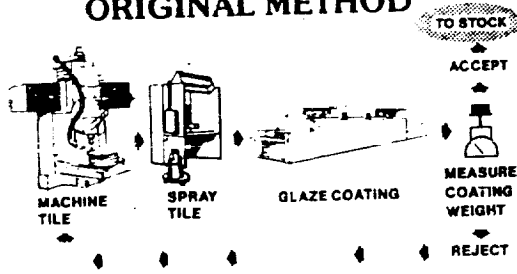
IMPROVED METHOD



- MAN IS THE MOST FLEXIBLE ROBOT FOR ALL TILES

Figure 21.- Methods of spraying class 1 and class 2 tiles.

ORIGINAL METHOD



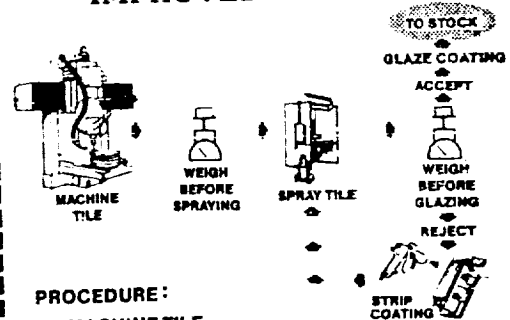
PROCEDURE:

- MACHINE TILE
- WEIGH BEFORE SPRAYING IN INSPECTION
- WEIGH AFTER COATING GLAZING IN INSPECTION
- SCRAP TILES FOR LOW OR HIGH COATING WEIGHT
- MACHINE NEW TILE

DISADVANTAGES:

- TILES SCRAPPED FOR HIGH/LOW COATING WEIGHT
- TIME LOST TO REMACHINE TILES

IMPROVED METHOD



PROCEDURE:

- MACHINE TILE
- WEIGH BEFORE SPRAYING IN MFG
- WEIGH BEFORE GLAZING IN MFG
- STRIP COATING FROM LOW/HIGH WT. TILES
- RESPRAY SAME TILE

ADVANTAGES:

- ELIMINATES:
 - TILES SCRAPPED FOR COATING WEIGHT
 - MACHINING NEW TILES
 - QUALITY ASSURANCE WEIGHT CHECKS
- SUBSTITUTES IN PROCESS COATING WEIGHT CHECK

Figure 22.- Methods to control coating weight.

ORIGINAL PAGE IS
OF POOR QUALITY

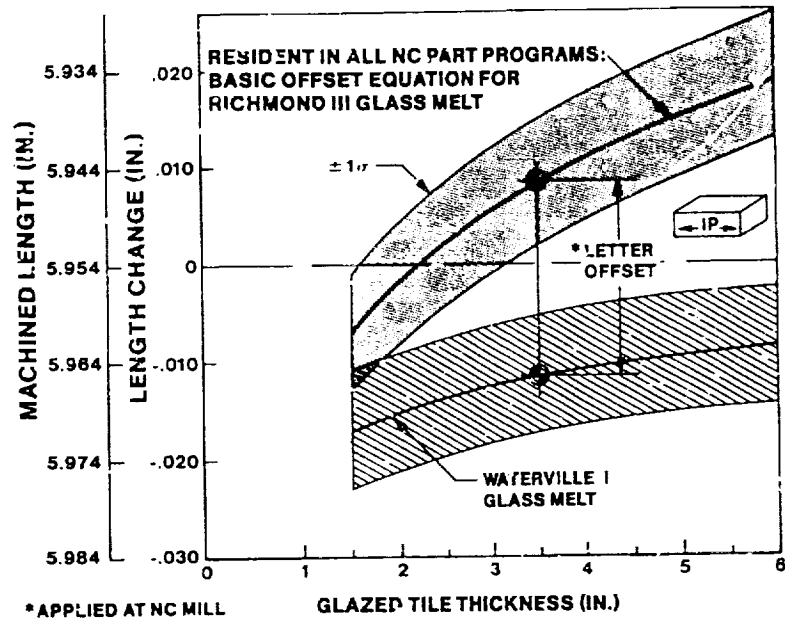


Figure 23.- In-plane machining offsets for LI-900.

- ALL ENGINEERING DEFINITION IS BASED ON COMPUTER (MASTER DIMENSION) DATA
- EACH TILE HAS UNIQUE PART PROGRAM FOR NC MACHINING
- ALL MANUFACTURING AND INSPECTION OPERATIONS ARE CONTROLLED BY ONE IBM CARD PER TILE
- DIMENSIONAL REQUIREMENT ± 0.016 -INCH (LENGTH & WIDTH) AND ± 0.010 -INCH (THICKNESS) FOR MOST TILES
- EACH TILE REQUIRES COMPENSATION FOR MATERIAL SHRINKAGE DURING COATING GLAZING
- SHRINKAGE VARIES WITH
 - GLASS MELT
 - TILE THICKNESS
 - TILE PLATFORM

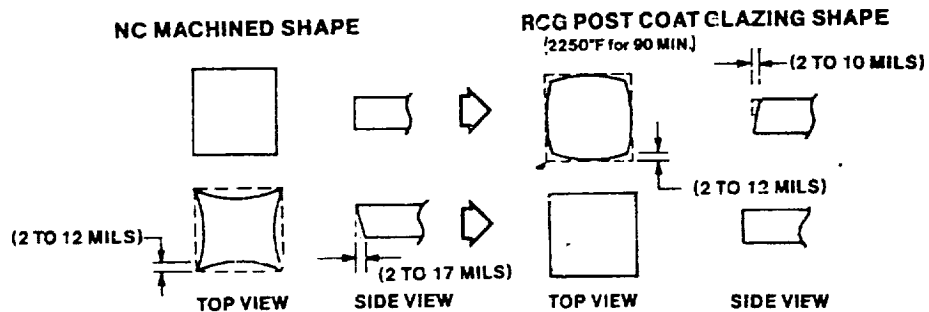


Figure 24.- Tile fabrication requirements.

ORIGINAL PAGE IS
OF POOR QUALITY

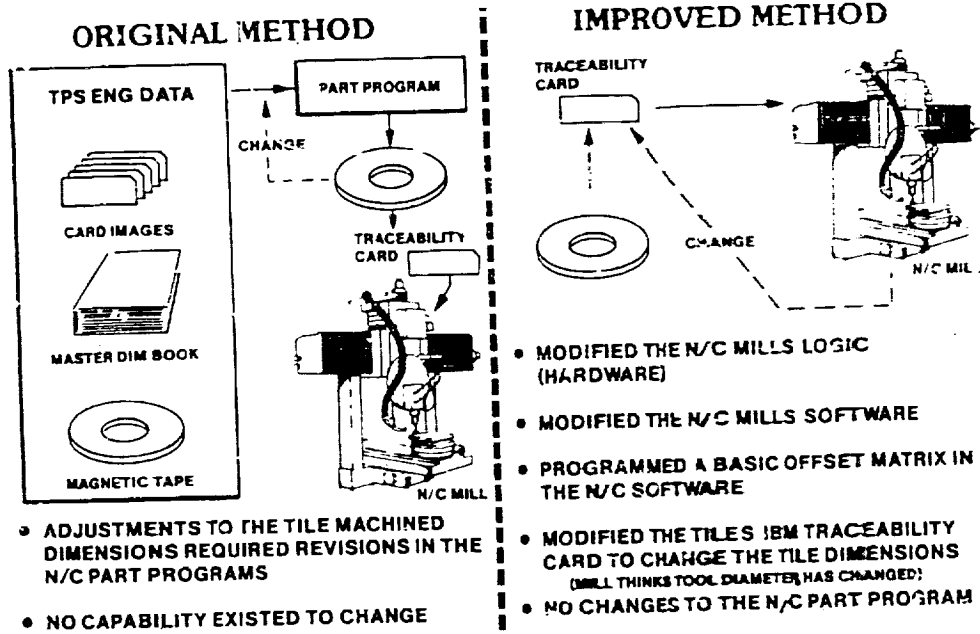


Figure 25.- Implementation aspects of tile machining letter offsets.

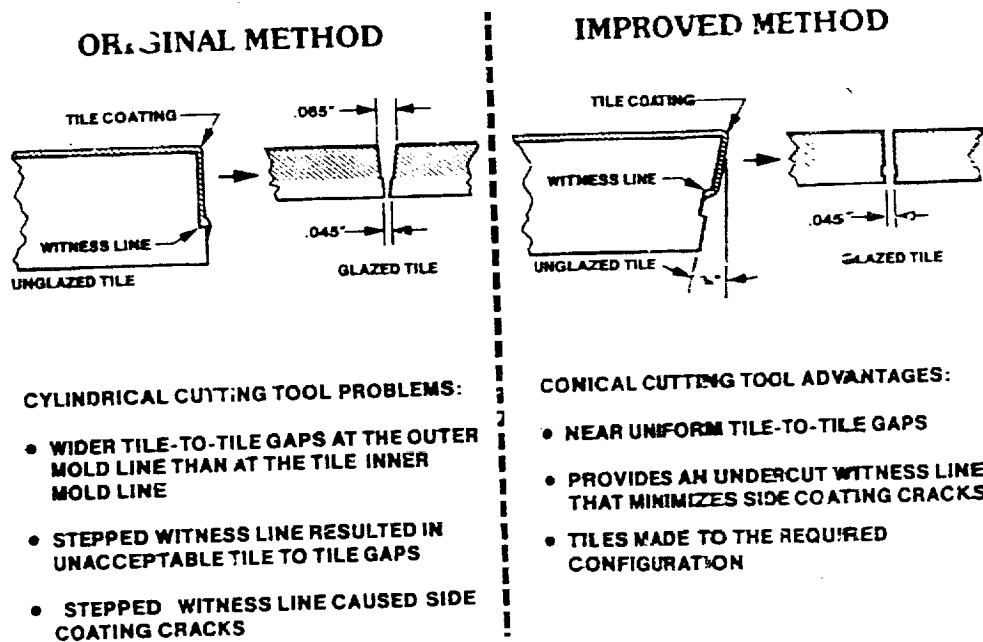
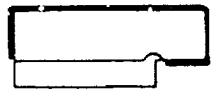


Figure 26.- Cutting tool configuration changes.

ORIGINAL PAGE 13
OF POOR QUALITY



STEP

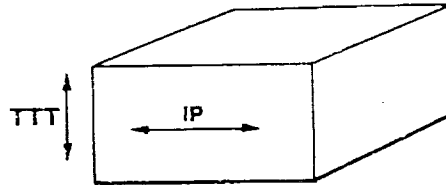
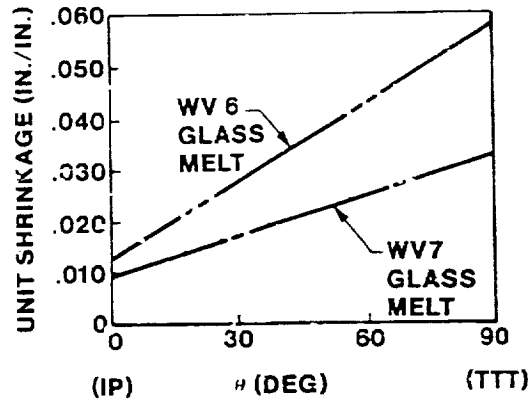


WRAPAROUND



ELEVON COVE

• ALL REQUIRE TTT SHRINKAGE COMPENSATION

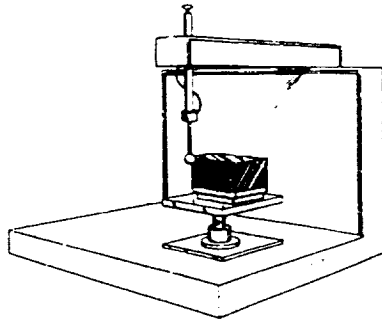


" measured from
in-plane direction

• ALL "FIXES" CONTAINED IN NC PART PROGRAMS

Figure 27.- LI-900 tile shrinkage in the through-the-thickness direction.

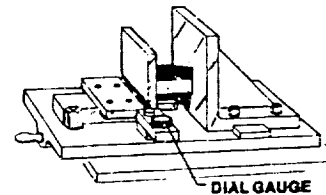
ORIGINAL METHOD



CORDAX 5000

- USED TO MEASURE ALL TILES BEFORE LOADING INTO ARRAY FRAMES
- ONLY DISCRETE POINTS ARE MEASURED ON TILE SIDE

IMPROVED METHOD



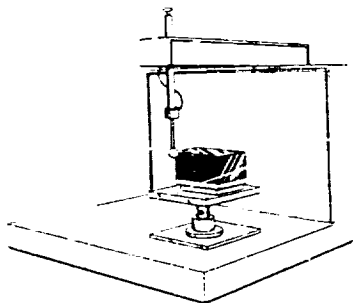
MAXI-MEASURE

- MEASURES THE SHIMMED DIMENSIONS
- ALLOWS MORE EFFICIENT MEASUREMENT OF TILES WITH PARALLEL SIDES
- CORDAX OR MYLARS ARE USED FOR TILES WITH NON-PARALLEL SIDES

Figure 28.- Tile measurement methods.

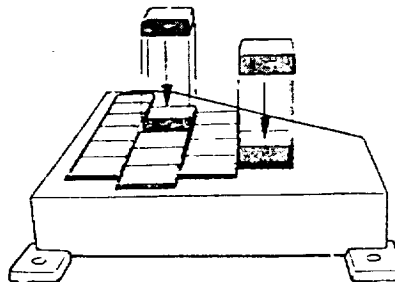
ORIGINAL PAGE IS
OF POOR QUALITY

ORIGINAL METHOD



- CORDAX 5000 MEASURING DEVICE WAS USED TO MEASURE ALL TILES BEFORE THEY WERE LOADED INTO ARRAY FRAMES. THIS MADE IT NECESSARY TO GENERATE STANDARDS THAT DEFINED THE CONFIGURATION OF EACH TILE.

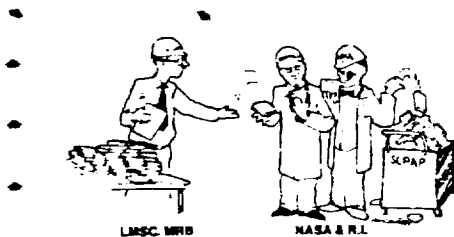
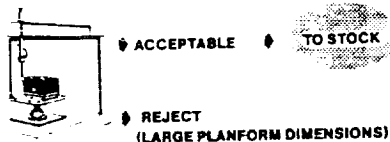
IMPROVED METHOD



- ALL TILES ARE LOADED INTO PREMEASURED ARRAY FRAMES. IF TILES LOAD TO SPECIFIED GAPS, THE ENTIRE FRAME IS ACCEPTED FOR SHIPMENT

Figure 29.- The load and go concept.

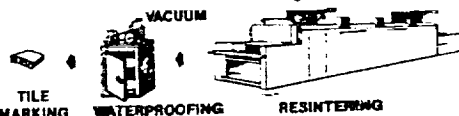
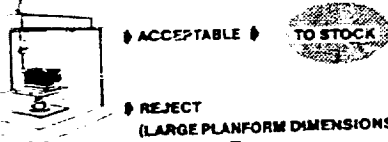
ORIGINAL METHOD



TILE REMAKE PROCEDURE:

- CHANGE TILE MACHINING OFFSET
- MACHINE REPLACEMENT TILE

IMPROVED METHOD



TILE REWORK PROCEDURE:

- RESINTER ORIGINAL TILE TO ACCEPTABLE DIMENSIONS

ADVANTAGES:

- NO TILE REMAKES REQUIRED
- FEWER SCRAPPED TILES
- MINIMUM IMPACT TO DELIVERY SCHEDULE

Figure 30.- A second sintering to shrink oversize tiles.

ORIGINAL PAGE 13
OF POOR QUALITY

DENSITY (lb/ft ³)	LI-900	LI-2200	FRCI-12
	8.3-9.5	20-24	11.9-13.5
TENSILE STRENGTH* (lb/in ²) THRU-THE-THICKNESS IN-PLANE	24	73	81
	67	180	257
COMPRESSIVE STRENGTH* (lb/in ²) THRU-THE-THICKNESS IN-PLANE	26	130	132
	76	230	265
THERMAL EXPANSION* (in/in - °F) THRU-THE-THICKNESS IN-PLANE	4 x 10 ⁻⁷	4 x 10 ⁻⁷	7 x 10 ⁻⁷
	4 x 10 ⁻⁷	4 x 10 ⁻⁷	7 x 10 ⁻⁷
APPARENT THERMAL CONDUCTIVITY* (BTU-in/ft ² hr - °F) THRU-THE-THICKNESS 70°F @ 10 ⁻¹ ATM 1000°F @ 10 ⁻⁴ ATM IN-PLANE 70°F @ 1 ATM 1000°F @ 1 ATM			
	0.10	0.22	0.13
	0.28	0.41	0.34
	0.44	0.73	0.53
	1.08	1.25	1.13
SPECIFIC HEAT* (BTU/lb - °F)	0.17	0.17	0.17

*AVERAGE VALUE

Figure 31.- Typical physical properties of LI-900, LI-2200 and FRCI-12.