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# One-Step Tape Casting of Composites via Slurry on Fiber

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# One-Step Tape Casting of Composites via Slurry on Fiber

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## Executive Summary

A major contributor to the continued advancement of aircraft engine performance is the development of less dense and stronger engine materials. Metal matrix composites (MMC) are being considered by many for jet engine applications in an effort to make improvements in these areas. Tape casting in particular is being developed in an effort to develop MMC's. The objectives of this paper are: to present a one step tape casting process and a supporting computer code; to assess the flexibility and controllability of the one step process; and to compare this one step technique to other tape casting and composite production techniques. The process is termed "one-step" because the green tape containing the fibers and matrix is made in one casting - where the binder and matrix are laid down on the fibers in one step. The general tape casting process consists of winding the fiber on a rotating drum; mixing the solvent, copolymer binder, and powder; casting the slurry onto the fibers using the dozer blade set at a specific height above the drum; casting flat matrix-only tapes; and stacking, and hot pressing.

In Tcast2 - the FORTRAN program developed to assist in tape casting - the fiber architecture in the final composite is expressed as and controlled through $\alpha$, the ratio of the drum fiber spacing to the fiber spacing between the plies. Fiber spacing through the width is controlled through the winding of the fiber on the drum. The fiber spacing through the thickness (between plies) is controlled by the dozer blade height and slurry characteristics. Easy control of fiber volume fraction and architecture through drum winding and blade height are the primary result of this work. When a new fiber and powder system is being tape cast for the first time, the first preparatory step is characterization of the green tape which results from the casting of the binder-solvent-powder mixture. With a particular fiber and slurry chosen, and the resulting green tape characterized, the Tcast2 program can be used to determine the dozer blade heights and fiber spacings needed to produce whatever fiber architecture and fraction fiber desired in the composite.

A wide variety of fibers have been successfully used to make tape cast composites at NASA Glenn. Initial studies were made using 0.005 inch diameter W fiber. Various castings have been made with the carbon core, carbon coated, silicon carbide fibers SCS-6 and SCS-9. Saphikon single crystal aluminum oxide (0.00478 inch diameter) fibers as well as the very fine tow fibers (Nextel 610) have been used in tape casting. Tow fibers are run through a powder slurry as they are wound on a drum.

Many different alloys and powder size distributions have been examined. Composites have been made using -140 mesh Ti-24Al-11Nb (at%), -100 mesh Ti-6Al-4V, -325 mesh Fe-24Cr-4Al-1Y (wt%), and -325 mesh $MoSi_2$-30$Si_3N_4$ (vol%) powders, as well as others. Since blade heights are typically in the range of about 0.01 to 0.04 inches, -70 mesh is expected to be the coarsest powder reasonable for tape casting.

Different binders were examined, and qualitatively assessed on the basis of tape strength, ductility, warpage, and ease of mixing - casting - and removal from the substrate; other aspects of the binders quantitatively examined were burn-out cleanliness and temperatures, and toxicity (of binder and solvents, based on literature data). The binders examined were each cast on a variety of substrates. Thermal Gravimetric Analysis (TGA) in vacuum was performed on all of the binder mixes. Ultimately, the copolymer mix of $PIB_{low}$ and $PMMA_{high}$ was chosen and used in subsequent tape casting.

The ultimate room temperature tensile strength and strain at failure of the 4-ply, 0˚ tape cast, Ti-24Al-11Nb/SCS-6, 32vol% fiber composite were: $\sigma_{UTS}$ = 215 ksi (1477 MPa), and $\varepsilon_f$ = 1.15%. The results of the room temperature 0 - 120 ksi, $\nu$ = 0.3 Hz fatigue test on the 4-ply Ti-24Al-11Nb/SCS-6, 32vol% fiber tape cast composite were: Number of cycles to failure, $N_f$ = 202,205 and maximum strain on the 100th cycle, $\Delta\varepsilon$ = 0.43%), which indicates the room temperature fatigue properties to be good and similar to above average Ti-24Al-11Nb composites made using other techniques.Though the above mechanical property tests are not exhaustive, they support the feasibility of tape casting.

# Introduction

A major contributor to the continued advancement of aircraft engine performance is the development of less dense and stronger engine materials. Density, creep and tensile strength, toughness and high temperature durability are all of concern; metal matrix composites (MMC) are being considered for jet engine applications in an effort to make improvements in these properties. For such parts as fan blades - continuous fiber MMC's have received much interest.[1,2,3] In continuous fiber composites, fiber lengths are of the same order as the part, and usually placed in specific directions relative to the geometry of the part and the stresses expected. Processing steps commonly include the consolidation of a multi-ply layup of fiber and matrix materials. Several processing techniques, such as foil-fiber-foil,[4] arc spray,[5] powder cloth,[6] and tape casting,[7] have been developed, and have successfully produced quality continuous fiber MMC test plates. Each of these techniques has different advantages and disadvantages.[3] Tape casting in particular is being developed in an effort to improve composite quality, and decrease costs as compared to the other techniques.[7,8,9] The objectives of this paper are: to present a one step tape casting process and a supporting computer code; to assess the flexibility and controllability of the one step process; and to compare this one step technique to other tape casting and composite production techniques.

Efforts are in place at several companies to produce titanium matrix composites suitable for use near the cooler, front end of jet engines. Fan blades and fan frame applications are two possibilities for use of titanium matrix composites. Many of the fabrication techniques, such as hot isostatic pressing and hot pressing, for the production of basic air foil shapes from multi-ply layups are similar and have been presented elsewhere,[8,10] and will not be discussed in detail here. Several variants of tape casting have been developed including the following. Edd and Niemann (Alcoa Laboratories and McDonnell Douglas respectively) have presented what will be referred to here as a two-step tape casting procedure where a matrix-only green tape is made separate from a fiber only tape.[7,8] In this two-step process binders are mixed with matrix powder and cast to make the green matrix tape. Then fiber is wound on a drum and binder applied onto the fibers to make the fiber mat. Spear (Atlantic Research Corp.) is examining a similar two-step tape casting technique.[11] Textron Systems Division (formerly Textron Specialty Materials) has developed a technique to produce green fiber-matrix-binder mats by which matrix-only and binder only tapes are made separately and then combined in a continuous fashion with fibers fed through a spacer (or creeling device); this might be viewed as a three step process where two castings are made and then the fibers sandwiched between them. Chesnutt, et al., have shown excellent results using a hybrid approach which combines foil/fiber/foil and tape casting techniques.[12] At the NASA Glenn Research Center we have been developing and using a "one-step" tape casting process in which the green tape is made in one step by casting the powder slurry directly onto the fibers - the results and details of this process are presented in this paper. In particular, a FORTRAN computer program which assists tape casting will be presented. Though the details of a similar "one-step" process, referred to as "Tape-Cast Powder Monotape," have not been found in the literature, the process of casting a powder slurry onto fibers was used in a collaboration among Pratt & Whitney, Atlantic Research Corp., and Rockwell International Science Center (with credit for initial development of the process given to the Alcoa Technical Center under the auspices of the National Aerospace Plane Materials and Structures Augmentation Program).[13,14] Figure 1 shows a schematic of the one-step tape casting process of winding the fiber on a rotating drum; mixing the solvent, copolymer binder, and powder; casting the slurry onto the fibers using the dozer blade set at a specific height above the drum; casting flat matrix-only tapes; and stacking, and hot pressing.

# Development of Tape Casting Procedures

## *Fibers and Drum Winding*

Fiber locations in the composite are determined largely by the initial winding on the drum; the drum spacing is maintained through subsequent steps which will be discussed later. A wide variety of fibers have been successfully used to make tape cast composites at NASA Glenn. Initial studies were made using 0.005 inch diameter W fiber. Various castings were made with the carbon core, carbon coated, silicon carbide fibers SCS-6 and SCS-9. Saphikon single crystal aluminum oxide (0.00478 inch diameter) fibers as well as the very fine tow fibers (Nextel 610 and Hi-Nicalon) have been used in tape casting. All fibers are wound on a smooth drum prior to casting. In all cases the drum is clad with release paper prior to winding. Tow fibers (strings of approximately 500 filaments) are spread out, hand-wound in large groups on a drum and infiltrated with matrix slurry.[15,16] We have not found a fiber which could not be used in tape casting.

Specific fiber spacings on the drum are computer controlled (except in the case of tow fibers) by accurately controlling the movement of the feed arm across the drum and the drum rotation. The consistency of fiber spacing in the end composites is first dependent upon the quality of the winding. A poor winding - one in which the interfiber spacing varies too much, or results in fibers touching while on the drum, will result in poor fiber spacing in the composite. A key advantage of casting the powder slurry directly onto the fibers is that there is much less movement of fibers during consolidation and thus better fiber spacing control within the plies and better fiber row retention. What is meant by row retention is that fibers within each ply are nicely lined up in a straight line, as they were on the drum. Fibers were typically wound at a drum rotation rate of 15 rpm; on a 16 inch diameter drum, this translates to a rate of 63 feet of fiber laid down on the drum per minute. The average fiber spacing was found to be accurate to about ±2.5%, with the average fiber spacing being consistently on the low side. For example, the requested value of 124 fibers/inch resulted in an actual, measured value of 121 fib/in. Preparation of the fiber wound drum is a time consuming step in the production of the green tapes. Improvements made here, such as increasing the rotation rate used to wind the fiber on the drum, will have relatively large economic utility. All windings for $0^o$ composites were made two inches wide for the production of 2 x 6 inch, four ply plates. It takes about 17 min. to wind the drum at 124 fibers/inch, two inches wide. Prior to winding the drum however, the drum must be cleaned, clad with release paper, and the fiber attached to it; this takes about 10 min. For the 90 degree composite, a casting six inches wide was made.

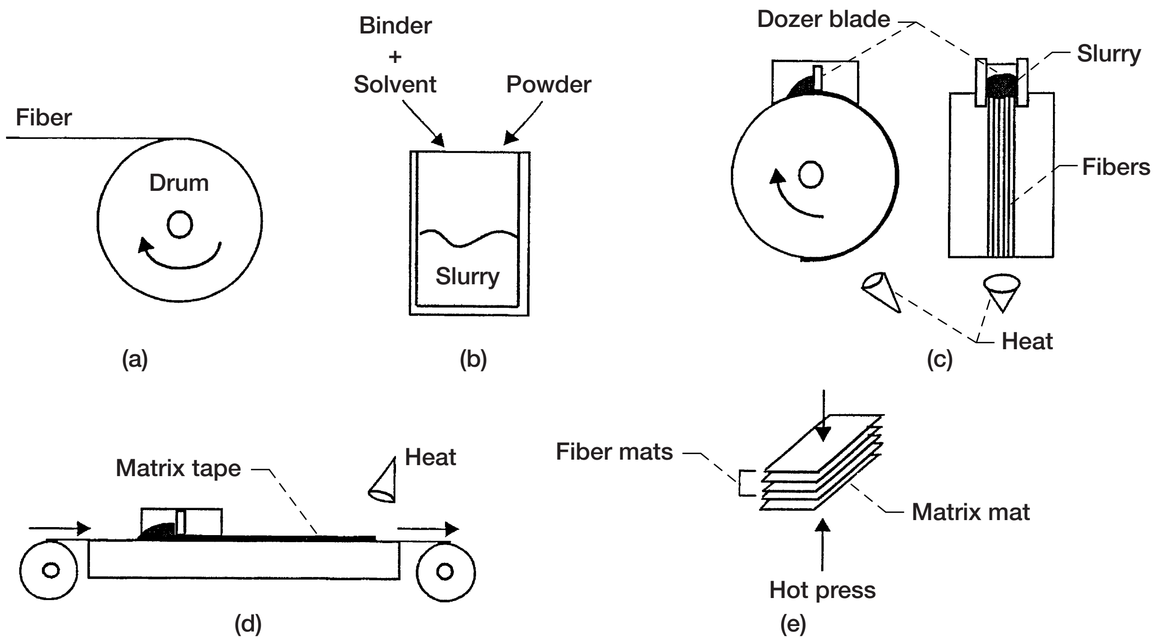



Figure 1.—Schematic of "one-step" tape casting process. (a) winding the fibers, we use about 15 rpm on a 16 inch diameter drum; (b) mixing solvent (toluene), copolymer binder (PIB and PMMA), and powder; (c) two views of the casting of the powder slurry onto the fiber wound drum and heat to quicken curing of the fiber/matrix mats, we let the tape cure for about 1 hour without heat; (d) flat continuous casting of the matrix-only end plies on release paper, we do this in small batches using the excess slurry from the drum casting; (e) stacking of the fiber/matrix mats with a matrix-only end ply to cover the exposed fibers, and vacuum hot pressing to give the fully dense composite.

Since fiber spacings have been excellent and row retention nearly perfect in finished tape cast composites, it is believed that the fiber spacing on the drum is locked in and retained through consolidation due to significant penetration of the powder between the fibers during slurry casting. This superior fiber spacing and row retention yields greater flexibility by enabling the volume fraction of fiber to be more accurately controlled by both drum fiber spacing and fiber row spacing. A good winding and excellent retention of that spacing enables the fiber volume fraction to be raised further, as compared to other processes, without the fear of problems such as fibers touching. And since the architecture can be controlled accurately (drum fiber spacing and fiber row spacing) coarser powders may be possible because wider fiber spacings can be used while maintaining high fiber volume fractions. As can be seen in Fig. 4 of reference 3, other processes such as foil-fiber-foil, plasma-spray, and powder cloth may rely on high within ply (drum) fiber spacings to achieve the desired volume fraction fiber - resulting in a large number of fibers touching and a non-square architecture. Though the degree of any degradation (or enhancement) of properties due to a non-square architecture is not clear - fibers touching has been found to degrade composite properties through fiber damage. Arc-spraying has been found to damage the fibers.[5,17] Tape casting is considered to be more gentle on fibers than powder cloth (because several of the processing steps are similar and there is less movement of fiber and powder during consolidation) - and powder cloth has been found to not damage fibers.[18]

In Tcast2 - the FORTRAN program developed to assist in tape casting - the fiber architecture is expressed as the ratio, $\alpha$, of the drum fiber spacing, fs, to the fiber spacing between the rows (through the composite thickness); both in units of fiber per inch. For a ratio of $\alpha = 1$ the architecture will be square, thus the fiber spacing will be the same through the width of the composite and its thickness. The density and diameter of the fiber are needed to accurately tape cast composites and are a required inputs to Tcast2. In situations were fiber diameter varies significantly (such as in the case of the 0.00478 inch dia. Saphikon alumina fibers mentioned above which varied by about +/- 0.001 inch) a representative average diameter is needed. Some densities of fibers are given in Table 1.

Table 1 Densities and approximate diameters of various fibers used in Tape Casting.

| | SCS-6 | SCS-9 | Saphikon, $Al_2O_3$ | W, W218 | Hi-Nicalon |
|---|---|---|---|---|---|
| Density, g/cc | 3.00 | 2.8 | 3.97 | 19.3 | 2.3 |
| Diameter, inches | 0.00566 | 0.0031 | 0.00478 | 0.004 | $5.5\text{-}7.9\text{x}10^{-4}$ |
| Diameter, microns | 144 | 79 | 121 | 102 | 14-20 |

## *Powders*

Several different alloys and powder size distributions have been examined. Composites have been successfully made using -140 mesh Ti-24Al-11Nb (at%), -100 mesh Ti-6Al-4V, -325 mesh Fe-24Cr-4Al-1Y (wt%), and -325 mesh $MoSi_2$-$30Si_3N_4$ (vol%) powders, as well as other powders examined which produced good green tapes. We have not found a powder which could not be used in tape casting - though tape casting has definite constraints on the maximum powder size.

The maximum powder size must be less than the dozer blade height; since blade heights are typically in the range of about 0.01 to 0.04 inches (250 - 1000 $\mu$m) -70 mesh is expected to be the coarsest powder reasonable for tape casting. It is economically most desirable to use very coarse powder because it is the least expensive; coarser powders have lower surface area and are therefore cleaner, and thus pose less risk of significant contamination of the composite from impurities such as oxygen and carbon. From a processing point of view however, it is desirable to have the powder size maximum be less than or equal to the average edge-to-edge fiber spacing; thus promoting better filling of the spaces between the fibers, and enabling consolidation of the green tapes with a minimum of powder and fiber movement. Minimizing powder and fiber movement during consolidation, and locking in the drum fiber spacing with good filling, results in a more gentle consolidation, and helps to insure good fiber spacing and properties in the final composite. For four ply composites using SCS-6 fiber at 35 vol% and a square architecture, the edge-to-edge fiber spacing is about.0.0026 inches; thus use of a powder sieved to -230 mesh would insure nearly all the powder to be less than the average edge-to-edge fiber spacing. Though good green tapes have been made using -100 mesh powder - our experience indicates that good filling of the spaces between the fibers is aided by a significant amount of fines (-325 mesh) in the powder. Though using a coarse size distribution such as -80 to +200 mesh may result in usable tapes and acceptable composites, retention of fine powders (using -100 mesh for example) will result in a more aesthetically pleasing tape and less movement of fiber and powder during consolidation. Table 2 lists some powders and their ideal densities. The density of the matrix is a required input in the tape casting program, Tcast2.

Table 2 Densities in g/cm$^3$ of various matrix alloys, in weight percent unless marked; density variations expected to be about +/- 1%; *at%.

| | Ti6Al4V | Ti24Al11Nb* | Ti21Al23Nb* | Fe24Cr4Al1Y | $MoSi_2$30vol%$Si_3N_4$ |
|---|---|---|---|---|---|
| Density | 4.43 | 4.5 | 5.1 | 7.2 | 4.92 |

## *Binders and Slurry Development*

Table 3 lists the different binders examined and the tape casting recipe developed. Initial binder selection was based on previous experience with the powder cloth[6] process and the literature.[7] Factors most important to final binder selection were tape strength, burn-out cleanliness, and toxicity. Many matrix-only, flat castings were made and qualitatively assessed on the basis of tape strength, ductility, warpage, and ease of mixing - casting - and removal from the substrate; other aspects of the binders quantitatively examined were burn-out cleanliness and temperatures, and toxicity (of binder and solvents using literature data). The binders examined were each cast on a variety of substrates including stainless steel, glass, Teflon, and a heavy, glossy release paper. Thermal Gravimetric Analysis (TGA) in vacuum was performed on the binder mixes. TGA included measurement of burn-out temperatures, mass of residual ash, and visual inspection of the TGA crucible. The crucibles were alumina, the heating rate was 20 K/min and the initial weight of cured binder samples was 25 g. While testing the different binders, materials were mixed in glass beakers, pored in front of a dozer blade and cast on flat substrates by pulling the dozer by hand at a rate of about 6 in/s. Green tape properties and TGA results are presented later

When modifying the recipes in Table 3 through use of other powders, it is recommended that the weight of powder used be scaled such that both volume and surface area of powder in the slurry are considered; the volume being considered through the matrix density and the surface area of the powder by its average particle size (diameter). For example, when using -325 mesh Fe based powders and the copolymer mix of $PIB_{low}$ and $PMMA_{high}$ the weight percent of Fe powder recommended in the slurry is 82.5%. When switching to a -100 mesh Ti based powder, a ratio of the matrix densities will help maintain a similar volume of powder in the slurry (82.5%$\rho_{Ti}/\rho_{Fe}$%), and a ratio of spherical surface area to volume ratios will help maintain constant powder surface area (82.5%$d_{Ti}/d_{Fe}$). The density ratio, about 0.6, shows that less powder (by weight) is needed to make a similar volume of powder in the slurry; but the ratio of the surface area to volume ratios of the powders, estimated to be about 2, indicates that since the Ti powder is coarser a higher weight fraction powder in the slurry can be tolerated while still coating the particles. The Ti and Fe matrix densities being represented by $\rho_{Ti}$ and $\rho_{Fe}$ respectively. Since the surface area to volume ratio of a sphere is 3/radius, the ratio of the surface area to volume ratios of the two spheres can be represented by their diameters, where in this case $d_{Ti}$ and $d_{Fe}$ are the average particle diameters of the -100 mesh and -325 mesh powders respectively.

Slurries were prepared by first dissolving the binder in the solvent. This sometimes took several hours. After a good binder solvent mixture was made - the powder was added to it and mixed by hand using a spatula just before tape casting. Mixing was fast enough to overcome particle settling and slow enough to avoid the production of too many bubbles in the slurry. Ball milling, to mix the powder and dissolved binders, was not necessary. Large quantities of the binder solvent mixture can be made in advance and stored if precautions are made to prevent the loss of solvent from the mix; we have had success using glass containers with Teflon-lined screw-on caps.

Ultimately, as will be discussed later, the copolymer mix of $PIB_{low}$ and $PMMA_{high}$ was chosen and used in subsequent tape casting because it had the best combination of green tape properties and burn-out cleanliness. The solvent used with this PIB, PMMA mix is toluene, which is one of the more benign industrial solvents. Wet chemical analysis was preformed on a FeCrAlY/W fiber composite to determine carbon and oxygen pickup during processing; the results of which are presented in the results section, as are the results of binder properties, burnout (TGA) and green tape properties

Table 3 Binders examined, all percents are weight percents based on -325 mesh FeNiCrAl powder.

| |
|---|
| Methocel™,* 20-214. A cellulose based binder soluble in water with unique gelling capabilities. To prevent premature gelling and promote dispersion of the powder - ingredients are mixed separately and heated. A = [mix and heat to 50 ˚C 11.4% water, 1.14% glycerin, 0.43% polyglycol, then add 57.1% powder], B = [heat to 90 ˚C 28.5% water, add and mix 1.43% Methocel]. Combined A and B, mix, and cool to about 18 ˚C. |
| NeoCryl, B-700. From ICI Resins US, this methacrylate polymer has what is referred to as a glass transition temperature of 48 ˚C. Dissolve 4.3% NeoCryl in 9.4% 2-ethoxyethyl acetate, add 86.3% powder and mix. |
| NeoCryl, A-614. This binder comes in solution and has a glass transition temperature of -10 ˚C. Mix 88% powder in 12% NeoCryl A-614 solution. |
| PIB, polyisobutylene, a synthetic rubber available in a variety of average molecular weights. M.W. $4.2x10^5$: dissolve 1.7% $PIB_{low}$ in 9.5% toluene, and mix with 88.8% powder. M.W. $1.2x10^6$: dissolve 2% $PIB_{med}$ in 21% toluene, and mix with 76% powder. M.W. $4.7x10^6$: dissolve 0.5% $PIB_{high}$ in 17% toluene, and mix with 82.5% powder. |
| PMMA, poly(methyl methacrylate), a plastic available in a variety of weight-average molecular weights. M.W. $1.5x10^4$: dissolve 6% $PMMA_{low}$ in 10% toluene, and mix with 84% powder. M.W. $1.2x10^5$: dissolve 4% $PMMA_{med}$ in 12% toluene, and mix with 84% powder. M.W. $9.96x10^5$: dissolve 4.5% $PMMA_{high}$ in 18% toluene, and mix with 77.5% powder. |
| PVP, polyvinylpyrrolidone, a water soluble binder. Dissolve 3.7% PVP in 17% ethanol, and mix with 79.3% powder. |
| Copolymer mix of $PIB_{low}$ and $PMMA_{high}$. Dissolve 1.3% $PIB_{low}$ and 1.2% $PMMA_{high}$ in 15% toluene, and mix with 82.5% powder. In use with Ti base powders we dissolve 2.2% $PIB_{low}$ and 1.7% $PMMA_{high}$ in 21% toluene, and mix with 75% Ti alloy powder. |

*Trademark of The Dow Chemical Company

## *Tape Casting Development and Procedures*

The goal of this paper is to present efficient tape casting methods by which the fiber spacing, through both the width and thickness of sheet composites, can be independently controlled; thus both fiber volume fraction and cross-section architecture are controlled. Fiber spacing through the width is easily specified through the winding of the fiber on the drum. The fiber spacing through the thickness (between plies) is controlled by the dozer blade height and the characteristics of the slurry and subsequent consolidation processing. Easy control of fiber volume fraction and architecture through drum winding and blade height are presented below and are the primary result of this work.

When a new fiber and powder system is being tape cast, the first step is characterization of the green tape which results from the casting of the binder-solvent-powder mixture. Characterization of the green tape includes experimentally measuring the matrix-only (fiber free) green tape density, $\rho_g$, and the thickness shrinkage, S, from the wet to green state of the tape. The shrinkage, S, is defined as

$$S = \frac{(BH - T_g)}{BH} \tag{1}$$

where BH is the dozer blade height above the substrate, and $T_g$ is the thickness of the cured, matrix-only, green tape. Initial estimates of $\rho_g$ and S are made by tape casting a matrix-only tape on a flat surface (or on the tape casting drum). These preliminary estimates and measurements must however be checked and corrected using measurements from a tape casting on a fiber wound drum. The density and shrinkage of the matrix-only green tape is determined from the tape containing fibers through rule of mixture calculations as follows:

$$\rho_g = \frac{(\rho_{fg} - V_{fg}(\rho_f))}{1 - V_{fg}} \tag{2}$$

and since there is no shrinkage in the fibers,

$$S = \frac{S_f}{(1 - V_{fg})} \tag{3}$$

where $V_{fg}$ is the volume fraction of fibers in the green tape, $\rho_f$ is the density of the fiber, $\rho_{fg}$ is the density of the green tape with fibers, and $S_f$ is the thickness shrinkage of the fibrous green tape,

$$S_f = \frac{(BH_f - T_{fg})}{BH_f} \tag{4}$$

where $T_{fg}$ is the thickness of the green tape with fibers and $BH_f$ is the blade height used to cast it. The fiber volume fraction in the green tape is calculated using the measured thickness $T_{fg}$, the drum fiber spacing fs (in units of fibers/inch) and the fiber diameter d:

$$V_{fg} = \frac{\pi d^2 (fs)}{4T_{fg}} \quad . \tag{5}$$

Now with the density of the matrix-only green tape $\rho_g$, and tape shrinkage S, the blade heights for different drum fiber spacings and composite architectures can be determined by the Tcast2 program. Figure 2 is a schematic of cross-sections and is included to help clarify some of the variables.

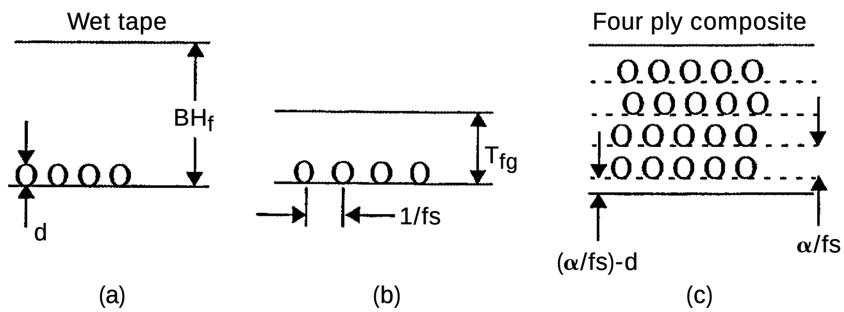



Figure 2.—Cross-section schematics of (a) the wet tape after tape casting but before curing showing the dozer blade height $BH_f$ and the fiber diameter d; (b) the green tape after curing showing fiber spacing as the reciprocal of the number of fibers per inch 1/fs, and the thickness of the green tape $T_{fg}$; and (c) a four ply composite plate showing the thickness of matrix on the outer surface ($\alpha$/fs)-d, and the fiber spacing between fiber rows $\alpha$/fs.

In subsequent efforts using Tcast2 to solve equations (2) through (5) we see an additional independent equation is needed since there are five unknown variables ($\rho_{fg}$, $V_{fg}$, $S_f$, $BH_f$, and $T_{fg}$) and only four equations. Since volume changes throughout processing of the sheet or plate are accomplished through changes in thickness only, a relation among composite density $\rho_c$, green tape density, drum fiber spacing, cross-section architecture, and green tape thickness can be developed. The architecture is brought in as a variable, $\alpha$, in the dense ply thickness; $\alpha$ is the ratio of the drum fiber spacing to the fiber spacing desired between the plies. In a square architecture, the thickness of a single fully dense ply is equal to the reciprocal of the drum fiber spacing (fs, in units of fibers/inch). The thickness of the green ply with fibers can thus be expressed as,

$$T_{fg} = \left(\frac{\rho_c}{\rho_{fg}}\right)\left(\frac{\alpha}{fs}\right) \quad . \tag{6}$$

The fully dense composite density $\rho_c$, and fiber volume fraction, $V_{fc}$, can be found directly from $\alpha$, fs, and d,

$$V_{fc} = \frac{\pi d^2 fs(p)}{4\left(\frac{\alpha(p+1)}{fs} - d\right)} \text{ , and} \tag{7}$$

$$\rho_c = \rho_f V_{fc} + \rho_m (1 - V_{fc}) \ , \tag{8}$$

where p is the number of (fibrous) plies. An assumption used in (7) and (8) is that the thickness of matrix on the outer surfaces of the composite is equal to the distance between the fiber edges through the thickness (that is, equal to ($\alpha$/fs) - d).

With a particular fiber and slurry chosen, and the resulting green tape characterized, the Tcast2 program can be used to determine the dozer blade heights needed for different situations. Table 4 outlines the input and output of the code. The Tcast2 code and an example of it in use are included in the Appendix.

Table 4 Outline of input and output of the tape casting helper Tcast2 program.

| Inputs | Outputs |
|---|---|
| Variable | Composite fiber volume fraction, $V_{fc}$ |
| Drum fiber spacing, fib/in, fs | Dozer blade height for fiber mat, $BH_f$ |
| Number of plies, p | Dozer blade height for matrix-only end ply, $BH_e$ |
| Architecture ratio, $\alpha$ | Composite density, $\rho_c$ |
| Constant | Thickness of green mat with fibers, $T_{fg}$ |
| Matrix density, g/cc, $\rho_{m,}$ | Thickness of green matrix-only end ply, $T_{eg}$ |
| Fiber density, g/cc, $\rho_f$ | Thickness of final composite, $T_c$ |
| Fiber diameter, inches, d | |
| Wet to Green Shrinkage, S | |
| Fiber free Green Tape Density, $\rho_g$ | |

With the slurry prepared, drum wound, and blade height set, castings were made under a fume hood by pouring the slurry in front of the dozer blade and rotating the drum at a rate of 2 rpm. The drum circumference was 50 inches - thus the casting rate was 8.3 ft/min. Rotation of the drum continued during the room temperature curing of the tape. No attempts have been made to accelerate the 15 to 30 min. curing time typically used. Wet tapes were cast two inches wide and green tapes trimmed to 1.937 inch. Of the 50 inch drum circumference, about two inches are lost in the start up and take off of the dozer blade. Thus there is about 4% of the fiber lost and 7% of the powder. The tape is cut from the drum with a razor blade and then cut with a shear to 1.937 x 6 inch sheets.

The excess slurry from the drum casting was immediately used to cast matrix-only tapes on a flat, release paper clad substrate. Figure 1(d) shows how the matrix-only tapes might be made in a continuous process. One of these matrix-only mats were then used to cover the exposed fibers on the end ply (see Figures 1(e) and 2(c)). As part of my examination of surface porosity and embrittlement, an extra matrix-only ply was added to the outer surface of the top and bottom plies. This extra cladding could then been ground off if desired.

### *Consolidation of Green Tapes and Mechanical Testing*

After layup of the matrix-only and four fibrous plies, the green tapes were vacuum hot pressed (VHP). Pressing conditions, several of which are listed in Table 5, varied depending on the matrix. Most materials, including Ti based composites, are hot pressed between sheets of Mo foil. The Mo foil is etched off using nitric acid after pressing. Vacuum hot pressing produced fully dense composite plates from which dog bone samples were machined for mechanical testing using either EDM or water jet cutting. Table 6 summarizes the tape cast plates analyzed. Figure 3 shows the details of the dog bones made for mechanical testing. In the case of Hi-Nicalon/$MoSi_2$-$Si_3N_4$ composites, hot pressing was done in two steps: first at 2100 ˚F, 20 ksi for 2 hours, then at 2550 ˚F, 10ksi for 2 hours.

Table 5 Vacuum Hot Pressing conditions used to produce fully dense 4 ply composite plates. Plates were approximately 2” x 6” by 0.037” thick. Alloys in wt% or marked *at%.

| Matrix and fiber | | Conditions | |
|---|---|---|---|
| | Time, min. | Pressure, ksi | Temp. ˚F |
| Ti-6Al-4V/SCS-6 | 120. | 15 | 1650 |
| Ti-24Al-11Nb*/SCS-6 | 60 | 18 | 1900 |
| Ti-21Al-23Nb*/SCS-6 | 60 | 15 | 2000 |
| Fe-24Cr-4Al-1Y/SCS-6 | 60 | 15 | 1950 |

Table 6 Tape Cast composites presented, all were 4-ply, and used SCS-6 fiber.

| Sample | Alloy | Powder size | Fiber spacing fibers/inch | Volume % fiber | Orientation |
|---|---|---|---|---|---|
| 32vol% | Ti-24Al-11Nb | -200 mesh | 120 | 32 | $0^o$ |
| 35vol% | Ti-21Al-23Nb | -140 +325 | 121 | 35 | $0^o$ |
| HDH* | Ti-6Al-4V | -170 +325 | 122 | 26 | $0^o$ |
| Coarse HDH | Ti-6Al-4V | -80 +200 | 122 | 25 | $0^o$ |
| 45vol% | Ti-24Al-11Nb | -100 mesh | 133 | 45 | $0^o$ |
| $90^o$ | Ti-24Al-11Nb | -140 mesh | 120 | 32 | $90^o$ |

*Hydride dehydried

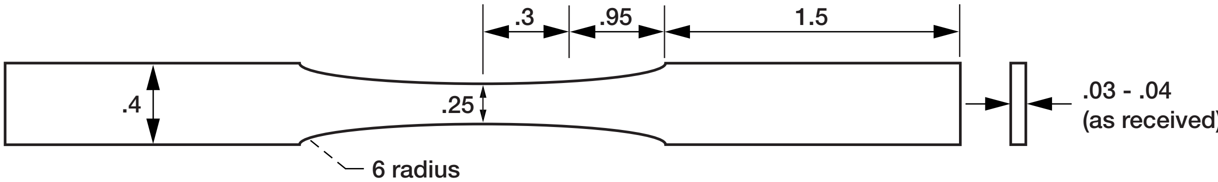


Figure 3.—Dogbone specimen used for tensile and fatigue tests, in units of inches.

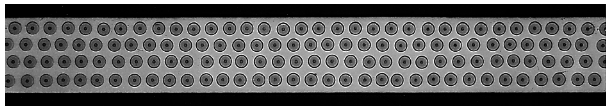
Room temperature fatigue and tensile tests were done on a 4-ply, 0˚ tape cast Ti-24Al-11Nb/SCS-6 composites, with fibers wound at 120 fib/in and a fiber volume fraction of 32%. Extra matrix-only plies were added to the exterior - thus making the matrix a little thicker (thicker than $\alpha$/fs -d) on the outer surface of the plate, and intentionally making $V_{fc}$ a little lower than that given by equation (7). Stress during fatigue was from 0 to 120 ksi (825 MPa) at 0.3 Hz. Room temperature tensile tests were done on two HDH samples and two HDH samples heat treated at 800 $^{o}$F for 300 hours.

# Results and Discussion

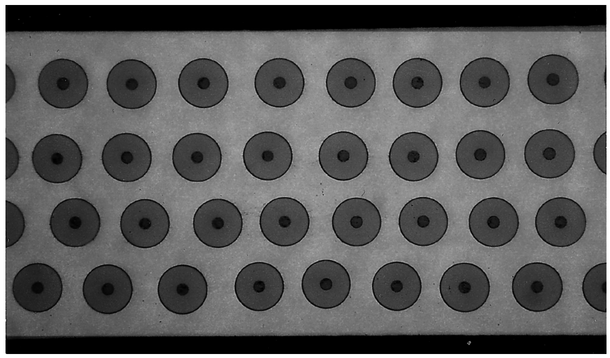
## *Microstructure and Fiber Spacing*

As can be seen in the micrographs shown in Figures 4 through 8, and later in the fiber spacing histograms, tape casting produces composites that are nearly free of touching fibers. The superior ability of tape casting to maintain and control within ply fiber spacing and the spacing between plies enables composites of relatively high volume fraction fiber to be made, such as that shown in Figure 7 at 45vol% fiber. Fiber volume fraction was determined using the actual average fiber diameter, fiber spacing and composite plate thickness, such that Vol. Frac. Fiber = $\pi d^2$fs/thickness. Figure 4 shows the cross section of sample 35vol% (see Table 5) which is characteristic of structures resulting from tape casting; as indicated in Table 5, this 4-ply, 0$^{o}$, 35 vol% fiber composite was made using Ti-21Al-23Nb, -140 +325 powder and SCS-6 fiber at 121 fibers/inch. Figure 5 shows sample HDH, a 4 ply, 26 vol% fiber composite plate made using Ti-6Al-4V powder provided by Concurrent Technology Corp.; this -170 +325 powder (-80 +200 for Coarse HDH) is known as hydride-dehydried (HDH) due to the novel and inexpensive techniques used to manufacture it. HDH powder is very irregular and nonspherical. Figures 6 through 8 show sections transverse to the fibers in samples Coarse HDH, 45vol%, and 90$^{o}$.

For comparison, Figures 9, 10 and 11 show typical within-ply fiber spacing histograms for composites fabricated by the powder cloth, foil-fiber-foil, and low-pressure plasma spray techniques respectively, all taken from reference 3. The spacing resulting from powder cloth and foil-fiber-foil techniques is poor, with a large portion of the fibers touching. Plasma spraying resulted in fewer fibers touching, and a rather wide distribution of fiber spacings, indicating poor control over the within-ply fiber spacing.



(a)



(b)

Figure 4a and b.—Cross-section of 0.036 inch thick 35 vol% fiber tape cast composite, 0°, Ti-21Al-23Nb, -140 + 325 powder and SCS-6 fiber at 121 fibers/inch.

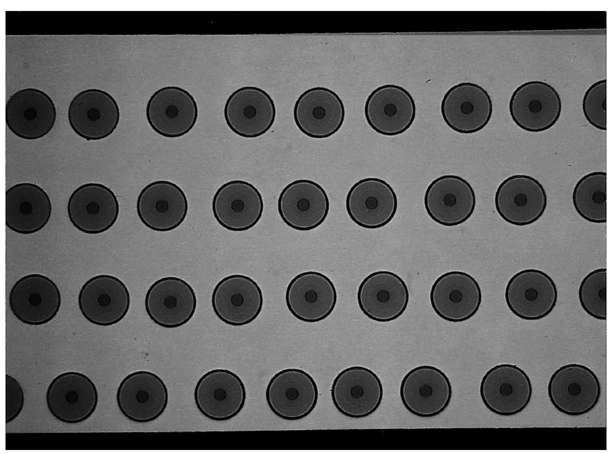


Figure 5.—Cross-section of 0.046 inch thick 26 vol% fiber tape cast composite made using HDH, -170 + 325 mesh powder, Ti-6Al-4V.

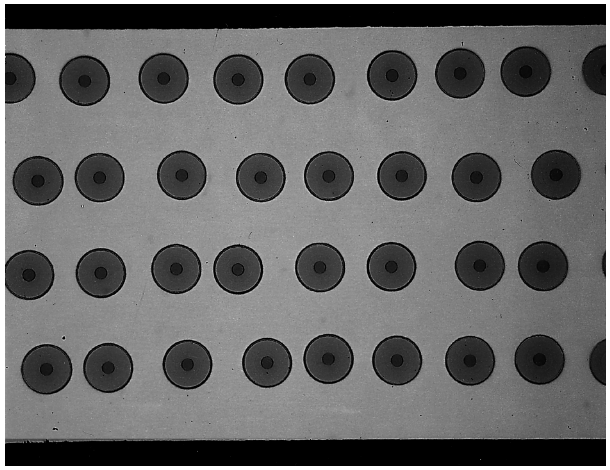


Figure 6.—Cross-section of 0.048 inch thick Coarse HDH tape cast composite, 25 vol% fiber, 0°, Ti-6Al-4V, -80 + 200 mesh powder, 122 fibers/inch.

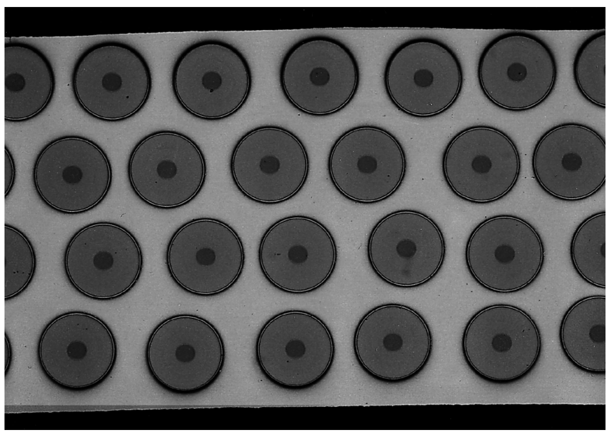


Figure 7.—Cross-section of 0.028 inch thick 45 vol% fiber, 0° tape cast composite, Ti-24Al-11Nb, -100 mesh powder, 133 fibers/inch.

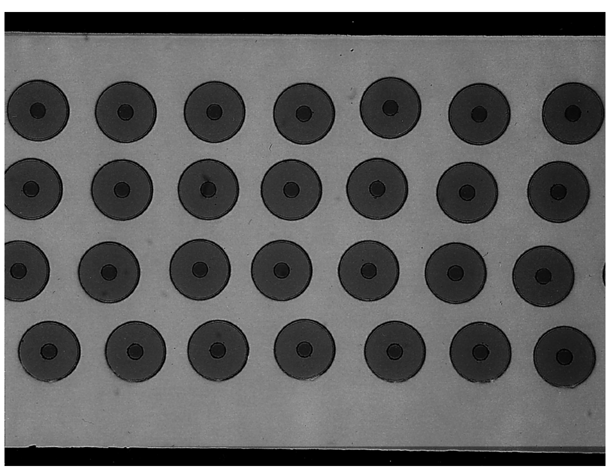


Figure 8.—Longitudinal section of 0.039 inch thick 90° tape cast composite, Ti-24Al-11Nb, -140 mesh, 120 fibers/inch, 32 vol% fiber.

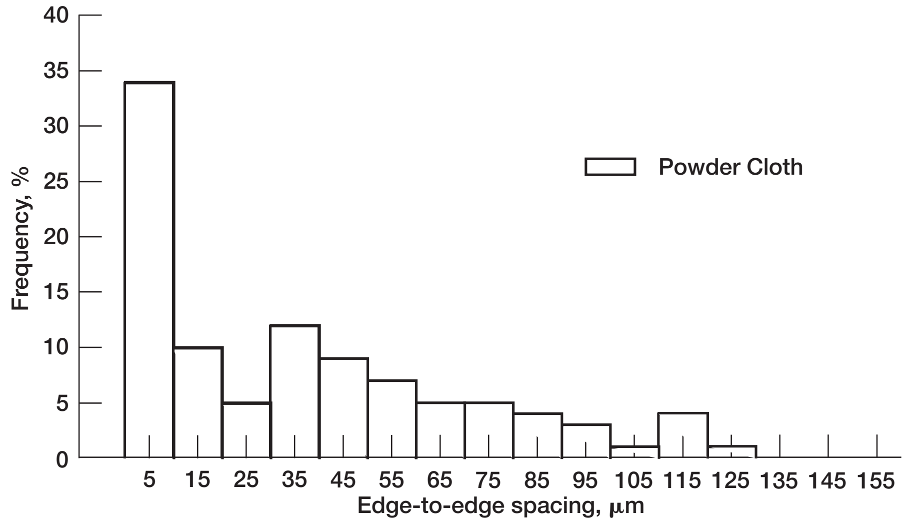



Figure 9.—Histogram of typical within-ply SCS-6 fiber spacing in an approximately 28 vol% fiber composite made using the Powder Cloth technique, indicating large numbers of fibers touching[3].

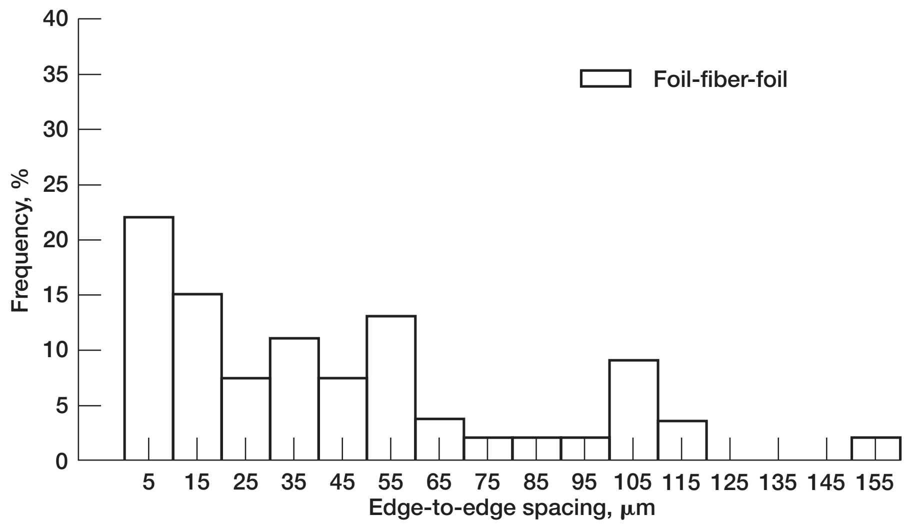



Figure 10.—Histogram of typical within-ply SCS-6 fiber spacing in an approximately 31 vol% fiber-foil-fiber composite, showing large numbers of fibers touching[3].

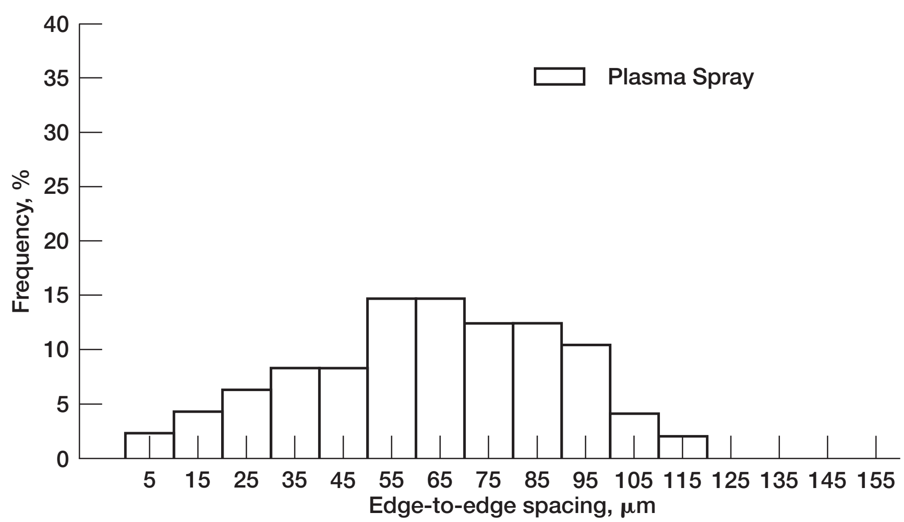



Figure 11.—Histogram of typical within-ply SCS-6 fiber spacing in an approximately 28 vol% fiber composite made using low-pressure Plasma Spray, showing some fibers touching[3].

Figures 12 through 16 show the fiber spacing histograms for the tape cast composites. Figure 12 shows the edge-to-edge fiber spacing to be narrowly grouped around the theoretical ideal (1/fs - d) of 66 μm for the 35vol% composite, with 84% of the fibers spaced within 15 μm of the theoretical ideal. The composite made by plasma spraying, in Fig. 11, has at best 42% of the fibers within 15 μm of the spacing intended. The spacing shown in Fig. 15 for this very high volume fraction fiber composite is not as good as in the other tape cast composites, but is still better than the other fabrication techniques represented in Figure 9-11. Improvements could be made in similar high volume fraction composites by decreasing the between ply spacing (thereby enabling the within row spacing to be increased), and by double casting. In double casting the slurry pile in front of the blade is maintained while the drum goes two times around, so after the tape is made (but is still wet) the slurry pile and dozer blade pass over it again - working the powders deeper into the spaces between the fibers. Double casting was used to make the six inch wide $90^{o}$ composite, Fig. 16, resulting in excellent spacing.

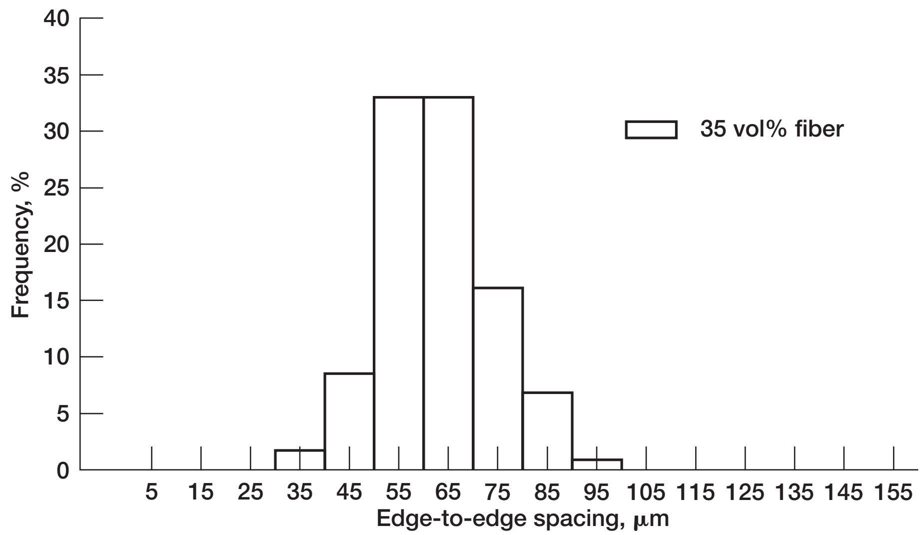



Figure 12.—Histogram of SCS-6 within-ply fiber spacing in the 35 vol% fiber, tape cast composite (see Table 5).

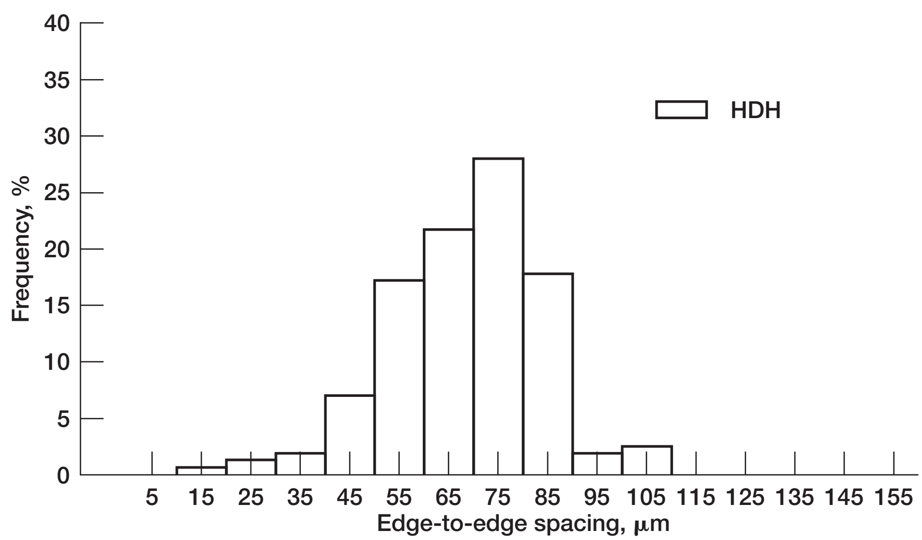



Figure 13.—Histogram of SCS-6 within-ply fiber spacing in the HDH, tape cast composite, 26 vol% fiber (see Table 5).

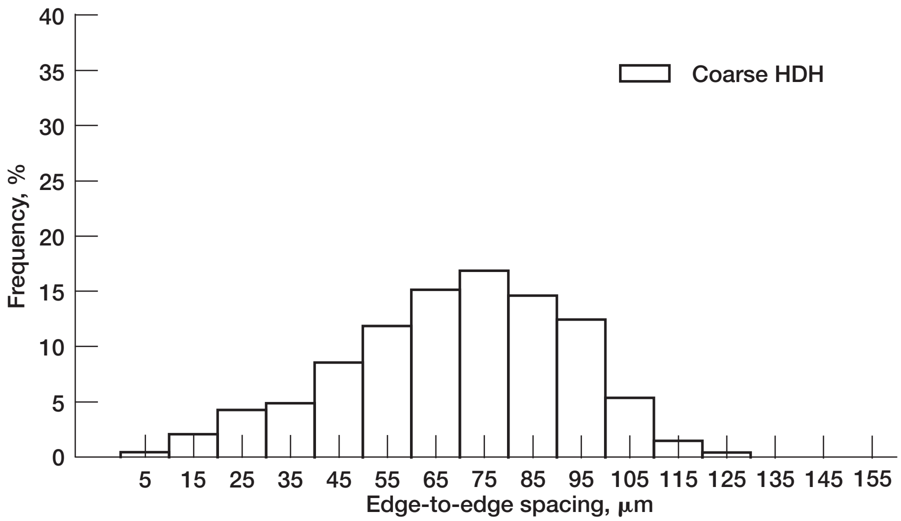



Figure 14.—Histogram of SCS-6 within-ply fiber spacing in the coarse HDH, tape cast composite, 25 vol% fiber (see Table 5).

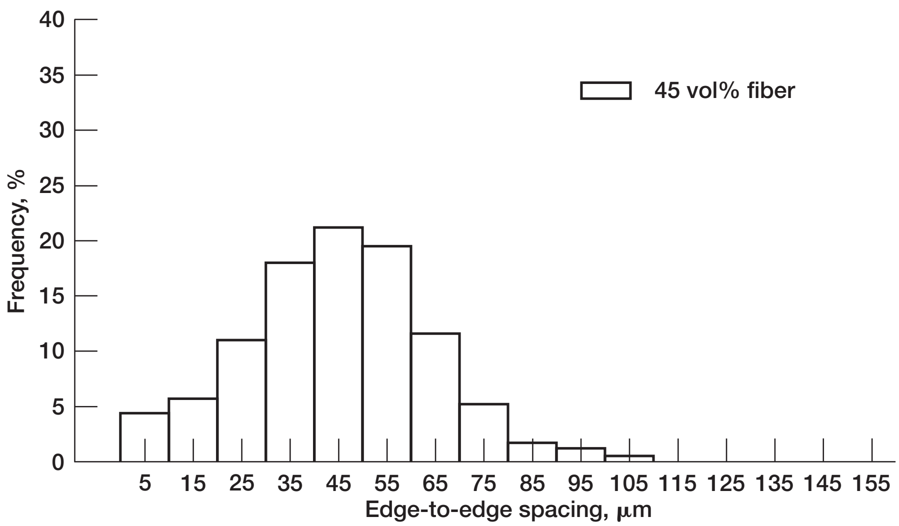



Figure 15.—Histogram of SCS-6 within-ply fiber spacing in the 45 vol% fiber, tape cast composite (see Table 5).

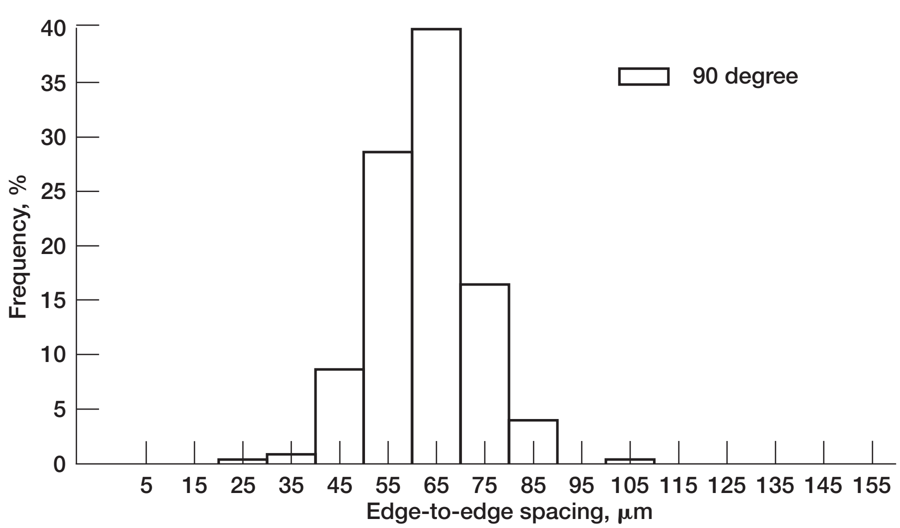



Figure 16.—Histogram of SCS-6 within-ply fiber spacing in the 90 degree, tape cast composite, 32 vol% fiber (see Table 5).

## *Mechanical Properties*

Tension and fatigue properties were measured using samples cut from the 32vol% four-ply SCS-6/Ti-24Al-11Nb plate (Table 5). The goal of these tests was to indicate whether panel quality was similar to that obtained from other fabrication techniques. The room temperature ultimate tensile strength and strain at failure of the 4-ply Ti-24Al-11Nb/SCS-6, 32vol% fiber tape cast composite were:

$\sigma_{UTS}$ = 215 ksi (1477 MPa), and $\varepsilon_f$ = 1.15%,

which indicates the quality of the tensile properties to be good and similar to above average Ti-24Al-11Nb composites made using powder cloth,[18,19] plasma spray,[20,21] and foil-fiber-foil[22,23,24] techniques. For example, in reference 19 the best SiC/Ti-24Al-11Nb powder cloth sample had a room temperature tensile strength of $\sigma_{UTS}$ = 217 ksi (1496 MPa), and $\varepsilon_f$ = 0.97%; the average stress and strain of the, 31 to 36 fiber volume percent, powder cloth samples being $\sigma_{UTS}$ = 179 ksi (1232 MPa), and $\varepsilon_f$ = 0.77%.[19] The results of the room temperature 0 - 120 ksi, $\nu$ = 0.3 Hz fatigue test on the 4-ply Ti-24Al-11Nb/SCS-6, 32vol% fiber tape cast composite were:

Number of cycles to failure, $N_f$ = 202,205 and

Maximum strain on the 100th cycle, $\Delta\varepsilon$ = 0.43%),

which indicates the room temperature fatigue properties to be good and similar to above average Ti-24Al-11Nb composites made using other techniques. Hall et al.[23] showed $N_f \cong$ 100,000 in Ti-24Al-11Nb/SCS-6 foil-fiber-foil composites of about 39 vol.% fiber, in load control at 1034 MPa (150 ksi), R = 0.1, $\nu$ = 0.33 Hz. Larsen[24] similarly showed $N_f \cong$ 100,000 in Ti-24Al-11Nb/SCS-6 foil-fiber-foil composites of about 35% fiber, in a stress range of 825 MPa (120 ksi), $\nu$ = 0.33 Hz.

Using the fiber elastic modulus from Brindley, et al., of 400 GPa, the modulus at the elastic limit of matrix-only Ti-24Al-11Nb of 108 GPa[19], and rule-of-mixture calculation, the ideal modulus (near the elastic limit) of the Ti-24Al-11Nb/SCS-6, 32vol% fiber tape cast composite was 201.4 GPa. At similar strain (0.43%) this tape cast composite had a modulus of 192 GPa, which is 95% of the rule-of-mixture value.

Though the above mechanical property tests are not exhaustive, they support the feasibility of tape casting.

## *Tape Casting Process Feasibility Comparisons*

Comparison of the “one-step” tape casting process detailed here with other processes is difficult due to the lack of published data. However, my best attempt will be made on the basis of what I have found in the literature, personal communications, and a per square foot basis of 4-ply 33 vol% fiber composite of square architecture using 0.00566 inch dia. SCS-6 fiber. At this fiber diameter and desired volume percent fiber, the fiber spacing is 119 fiber/inch.

Even though drum wound tape casting is considered a batch process - it is still considered to be economically feasible because of its: fast casting rate, excellent fiber spacing and architecture control, low labor costs, and low waste of fiber and powder. Also, the process of casting the slurry directly onto the fibers results in a simpler process with fewer steps.

The fiber winding rate on our 50 inch circumference drum is 15 rpm, equal to 63 ft/min. Little effort has been invested in maximizing this rate - faster wind rates are believed feasible. At this rotation and fiber spacing, fiber sheets are laid down at the rate of 0.044 $ft^2$/min (or 22.7 min/

$ft^2$). To put the slurry down on the fibers, a casting rate of 6 rpm was used. At 2 inches wide this is a casting rate of 4.2 $ft^2$/min. However, we have also made castings 6 inches wide, showing the feasibility of a casting rate of 12.6 $ft^2$/min (or 0.0794 min/ $ft^2$). Curing time for the wet tape is generally independent of the length and width. We let our tapes cure under hood ventilation conditions without heat for about 20 min. No attempts have been made to accelerate curing - thus for comparison purposes only (based on a 12 inch wide by 100 inch long casting curing in 20 min) the curing rate will be assumed to be 0.4 $ft^2$/min (or 2.5 min/ $ft^2$) for all applicable cases. Thus, the total time to make the Tape Cast green tape is approximately (22.7 + 0.079 + 2.5 min/ $ft^2$) 25.3 min/ $ft^2$ (or 0.04 $ft^2$/min).

The creeling techniques used at Textron Specialty Materials[25] can be used to make composite green tapes from the integration of matrix-only green tape - binder only tape - and fibers. A rough estimate of the productivity of this technique based on 1994 procedures follows. First matrix-only and binder only tapes are “tape cast” without fibers onto release paper. The casting rate for these matrix and binder tapes is believed to be limited by the curing time, since the tape is cast and must dry before it reaches the roll-up spool. By contrast, in tape casting on a drum, the entire tape is curing at about the same time. Assuming it takes 10 min. to cure, a 1 ft. wide tape on a drying table 10 ft. long would result in the production rate 1 $ft^2$/min. (or 1 min/$ft^2$) for the matrix-only and binder only tapes, each. In creeling, each fiber is fed through a spacer and into the sandwich area between the matrix and binder tapes. Each fiber is fed from a spool hung on a large rack. For a 1 inch wide tape, at 119 fibers/inch, 119 fibers are fed one at a time through spacers, from 119 spools all hanging on the spool rack and feeding into the casting area at the same time. It was estimated that it takes about 1 minute to take a fiber from the spool and feed it into the sandwich area through the spacers. Thus about 119 min/inch to feed the fibers in. The casting process is continuous once all fibers are fed in, and done in 500 ft. lots. Thus to get the process started represents a rate of 0.34 $ft^2$/min (or 2.86 min/$ft^2$). Now the matrix and binder tapes are fed at a rate of 0.5 ft/min with the fibers sandwiched between them with some heat. Assuming a width of 1 ft., this represents a rate of 0.5 $ft^2$/min (or 2 min/$ft^2$). Thus the total time to make the green tape by creeling is about (2 x 1 + 2.86 + 2 min/$ft^2$) 8 min/$ft^2$(or 0.125 $ft^2$/min). Which is a production rate about 3 times faster than drum wound tape casting as done at NASA Glenn.

Considering the accuracy of these production estimates the values for drum wound tape casting seem plausible, in particular since about 90% of the time in the process is used winding the fiber on the drum. Simple and immediate improvements may be achieved by increasing the rate at which fiber is put on the drum, and by having several drums working at the same time. There may be labor advantages to tape casting also. Tape casting requires one person for the entire process. And that person does not even need to be there during the most time consuming step, fiber winding. Thus, in labor, tape casting takes less than about 0.4 hr/$ft^2$ for production of the green tape. Creeling takes 3 or 4 people during the creeling and at least one during the making of matrix and binder only tapes. Thus, in labor, creeling takes about (2 tapes @ 1min/$ft^2$ x 1 person + 4.86 min/$ft^2$ x 4 people) 20 min./$ft^2$, or 0.33 hr/$ft^2$ for the production of the green tape. Thus these estimates of production rate in terms of man hours for tape casting (0.4 hr/$ft^2$) and creeling (0.33 hr/$ft^2$) are comparable.

## *Binders*

The physical characteristics of the matrix-only castings using the different binder recipes developed are listed in Table 7. Due to glue-like bonding, tapes made with PIB could not be removed from the substrate, thus castings were made on thin Mylar films, the Mylar then becoming part of the green tape. Textron uses heat to help with removal of tapes using PIB from the substrate. Undesirable were NeoCryl B-700 and PMMA due to their strength and warpage. It was found that mixing PIB and PMMA resulted in a strong, adequately flexible tape that released easily from the substrate.

Table 7 Characteristics of matrix-only tape casting using -325 mesh FeCrAlY and a dozer blade 0.04 inches above the substrate. Refer to Table 3 for slurry recipe details. $[\rho_g] = g/cm^3$, [S] = %

| Binder | Properties | Substrate/removal | Shrinkage, S | Green density, $\rho_g$ |
|---|---|---|---|---|
| Methocel | Strong, flexible, like paper. | S. Steel, glass Teflon/ very easy | 75. | 4. |
| NeoCryl, B-700 | Brittle, warped | Teflon/easy | 47. | 4.5 |
| NeoCryl, A-614 | Strong, flexible | Teflon/ very easy | 37 | 4.1 |
| $PIB_{low}$ $\rho = 0.92 g/cm^3$ | Strong, flexible | on Mylar film/ not removed | 38 | 4.1 |
| $PMMA_{high}$ | Weak, brittle, warped | Teflon/ very easy | 52 | 4.1 |
| PVP | Strong, flexible | Teflon/ easy | 70 | 4.2 |
| Copolymer, PIB + PMMA | Strong, flexible | Teflon/ very easy | 50 | 4. |

Table 8 lists the results of the Thermal Gravimetric Analysis. Although some of the TGA data related to cleanliness are qualitative, and contamination specifications were not available, it is known that oxygen content in the matrix can significantly reduce matrix strain at failure.[19] To prevent undesirable contamination of the composite, only clean “burning” binders are desired; thus Methocel, NeoCryl A-614, $PIB_{low}$ on Mylar, and PVP were judged unacceptable. The copolymer mixture of $PIB_{low}$ + $PMMA_{high}$ gave the best combination of physical properties and cleanliness and was thus the binder used to tape cast composites for mechanical testing. Burnout is also complete below 455 $^{o}$C, the temperature titanium oxide is absorbed into the matrix.[12] Others use PIB and have shown it to be clean when used with metal powders.[7,12]

Oxygen and carbon pickup in a FeCrAlY/W fiber composite were examined using a double dose of the PIB + PMMA binder in the green mats. All of the O and C were assumed to be in the matrix, and weight percents calculated on the basis of the matrix-only (with the mass of the fibers removed). The virgin powder had 0.18 and 0.011 wt% O and C respectively - while the matrix, after vacuum hot pressing, had 0.128 and 0.027 wt% O and C respectively in the matrix. It is clear that oxygen is not a problem - since O is lower in the composite than in the virgin powder. Carbon pickup of 0.016% C (160ppm) is also low and compares well with other clean processes noted in the literature.[6*,8]

*It is believed, due to prior use of the convention, that the % C contamination noted in Table IV of reference 6 for W/NiAl is on the basis of the composite mass. If the C is assumed to be in the matrix there is about 0.017% C for W/NiAl (pickup of 150ppm C).

Table 8 Results of Thermal Gravimetric Analysis of binders. See Table 3 for recipes used. No metal powder was used in the TGA tests.

| Binder | Residual mass, mg | Crucible condition | Burnout temperatures, ˚C |
|---|---|---|---|
| Methocel | 0.4 | Poor, black, dirty | single mass loss peak at 350 |
| NeoCryl, B-700 | 0.1 | Excellent | mass loss peaks at 125, 280, 350 |
| NeoCryl, A-614 | 0.2 | Fair, black | last mass loss at 350 to 400 |
| $PIB_{low}$ | 0.0 | Excellent | last mass loss at 365 |
| Mylar | 1.7 | Poor, black, dirty | single mass loss peak at 420 |
| $PMMA_{high}$ | 0.2 | Very good | mass loss peaks at 125, 260, 340 |
| Copolymer, $PIB_l + PMMA_h$ | 0.0 | Excellent | mass loss peaks at 130, 265, 365 |
| PVP | 0.27 | Poor, black | single mass loss peak at 430 |

The toxicological properties of PIB and PMMA have not been thoroughly investigated. PIB was developed as a synthetic rubber, and PMMA is, among other uses, a paint additive. Many here at NASA Glenn have handled these materials with no known negative health effects. These materials however should never be inhaled or ingested. Contact with bare skin should be avoided. As with most polymers, these materials are suspected carcinogens to tissues around an implant sites where ever retention may occur, such as in the skin, lungs, or organs.[26] The disposal of PIB and PMMA should be by mixing with a combustible solvent and burning in a chemical incinerator with afterburner and scrubber. The toxicity of toluene, the solvent used with PIB and PMMA, can be summarized as moderate through oral, inhalation and intraperitoneal routes and low via absorption through the skin.[26] Acute poisoning, resulting from exposures to high concentrations of toluene vapors are rare. Commercial grades of toluene usually contain small amounts of the poisonous and accumulative benzene as an impurity. With proper venting, toluene does not pose a toxicological problem. Similarly, no outstanding toxicology problems were found for any of the other binders.

The copolymer mixture of $PIB_{low}$ and $PMMA_{high}$ listed in Table 3 was the best binder. $PIB_{low}$ was chosen over the two higher molecular weight PIB's available because $PIB_{low}$ took less time to dissolve in the solvent and formed a more stable binder/solvent mix. Matrix-only tapes made with PIB are extremely flexible with nearly no stiffness; resulting in a cloth analogous to silk, with no strength in compression, like a chain. Tapes made with $PIB_{low}$ by itself could not be removed without damage from the substrate. $PMMA_{high}$ was chosen over the two lower molecular weight PMMA's available because $PMMA_{high}$ made smoother and flatter matrix-only tapes. Tapes made with $PMMA_{high}$ alone were too weak and brittle. Mixing $PIB_{low}$ and $PMMA_{high}$ resulted in a tape with adequate strength and flexibility and good release properties from the substrate.

Improvements can be made to the Table 3 PIB + PMMA recipe. I believe that improvements in strength and flexibility can be achieved by increasing the amount of PIB and decreasing PMMA. These improvements may enable a decrease in the amount of binder used. The increased flexibility of a PIB based binder may also help with the production of more complex shapes. PMMA is added simply to improve the release properties of the PIB; other binders may also do this well. A group at Textron Specialty Materials uses a PIB-10%NeoCryl binder mix. Future efforts will attempt to improve the binder by increasing the PIB:PMMA ratio in the recipe.

The density of fiber free, matrix-only, tape cast green tapes, $\rho_g$, and the wet to green shrinkage, S, for different metal powders are listed in Table 9. The green tape densities and shrinkage data listed in Table 9 are averages of fiber free tape castings and of tapes with fibers cast on a drum (see eq. 2 and 3). Most castings were made with fibers using a blade height very close to that needed for a 35 vol% fiber composite (about 0.020” blade height). It is worth noting however that $\rho_g$ and S were found to vary slightly as a function of blade height. Other more significant threats to the accuracy of the blade height are: one of the dozer blade sides running over a fiber, and overlapping release paper. In developing a Tape Casting program however, it is recommended to quantify how $\rho_g$ and S vary with blade height. In castings made with FeCrAlY, $\rho_g$ and S were 5% lower than the average at the smallest blade heights (0.015”) and 5% larger at the larger blade heights (0.030”). Variation in blade height, from that determined from the mean, was found to be about 0.001 inch.

Table 9 Average fiber free green tape density, $\rho_g$, and wet to green shrinkage, S, for different metal powders, all using $PIB_{low}$ + $PMMA_{high}$ binder (recipe given in Table 3). All powders were spherical except for the Ti-6Al-4V, HDH powder; this hydride-dehydride powder was very rough, irregular, and nonspherical.

| Powder | $\rho_g$, g/cm$^3$ | Shrinkage, S, % |
|---|---|---|
| Ti-21Al-23Nb at%, -140 +325 mesh | 2.12 | 43 |
| Ti-24Al-11Nb at%, -140 and -200 mesh, | 2.02 | 42 |
| Ti-6Al-4V wt%, -100 mesh | 1.74 | 30 |
| Ti-6Al-4V, HDH -80, +200 and -170, +325 mesh | 1.6 | 18 |
| $MoSi_2$-30$Si_3N_4$ vol%, -325 mesh, | 1.1 | 46 |
| Fe-24Cr-4Al-1Y wt%, -325 mesh | 3.6 | 50 |
| Fe-25Ni-29Co-5.5Cr-0.5Al wt%, -325 mesh, $\rho_m$=8.25 | 3.2 | 62* |
| Fe-11.7Al-1.53V-0.29C-0.5Mn wt%, -325 mesh, $\rho_m$=6.7 | 3.1 | 61* |

*Not averaged, based on a single measurement.

# Conclusions

A process by which metal matrix composites can be made was presented. The process involves putting a powder slurry on fibers to make a precursor green tape. These green tapes are cut, stacked and hot pressed to form the fully dense composite. A computer program was presented which enables complete quantification and control of the process. Once some easily obtained properties of the slurry and its behavior are determined (such as the shrinkage from the wet to green state, and the density of the green tape) modification of the fiber spacing and blade height give the maker precise control of fiber volume fraction, and fiber architecture in the composite. The process was shown to be accurate and flexible through the production of a wide variety of volume fraction fiber composites made from a wide variety of fibers and powders. The most time consuming step of the tape casting process (other than hot pressing) was winding the fiber on the drum. The tape casting techniques developed resulted in high quality metal matrix composites, with ultimate tensile strength in the range of 215 ksi (1477 MPa), a strain at failure of 1.15%, and in fatigue at room temperature 0 - 120 ksi, $\nu = 0.3$ Hz, a 4-ply Ti-24Al-11Nb/SCS-6, 32vol% fiber tape cast composite lasted 202,205 cycles with a maximum strain on the 100th cycle of 0.43%.

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# Appendix A

The FORTRAN 77 program Tcast2 is designed to assist tape casting of a powder slurry onto fibers. The program can also be used for tape casting of fiber free green mats. The source code, Tcast2.f, was compiled using a Lahey FORTRAN compiler to produce an executable file. The executable file is Tcast2.exe. Any IBM compatible PC with an 80286 or better processor running DOS 3 can be used. What follows are the code prompts, an example of inputs and displayed output.

| | |
|---|---|
| D1 | This program estimates the fiber volume fraction of tape cast composites, and the dozer blade heights needed to produce them. It is assumed that a powder slurry will be cast onto regularly spaced fibers. The program will need the density of the fibers, the matrix density, and that of the green mat without fibers. The density of the green mat can be found by weighing and measuring a sample fiber free casting. The program will also request the percent shrinkage in thickness from the wet to the green state for the fiber free tape. This allows the program to estimate the dozer blade heights. This program can also help with the casting of fiber free mats. To use the code for a fiber free system - use a very, very small fiber diameter and a fiber spacing equal to the number of green plies used plus 1, divided by the final thickness wanted in the plate, that is, fs=(p+1)/Tc); with the drum: between ply fiber spacing ratio equal to 1. |
| D2 | Input the density of the fully dense matrix in g/cc. Enter zero to QUIT. |
| | 4.5 |
| D3 | Input the density of the fibers in g/cc. |
| | 3.00 |
| D4 | Input the diameter of the fibers in units of inches. |
| | 0.00568 |
| D5 | Input the fiber spacing in fibers/inch. |
| | 123 |
| D6 | Input the numbers of plies used in the composite, not including the fiber free end ply. |
| | 4 |
| D7 | Input the ratio of the drum fiber spacing (in fiber/in) to the desired spacing between the plies (in fib/in). |

| | |
|---|---|
| | 1 |
| D8 | The volume fraction fiber in the composite is, VF= 0.356494<br>The density of the composite is, Dc= 3.96526<br>Input the density of the green tape without fibers. |
| | 2.0 |
| D9 | The thickness of the green tape with fibers is,<br>Tgf= 3.69839E-02cm |
| D10 | Input the shrinkage expected for a fiber free ply from<br>the wet to green state, that is, the fraction decrease<br>in thickness between the wet as cast tape and after<br>the solvent has left the binder making the green tape.<br>With a dozer blade height of BH and a green fiber free<br>ply thickness of Tg, the shrinkage expected is,<br>S=(BH-Tg)/BH. |
| | 0.42 |
| D11 | The dozer blade height needed for the plies containing<br>fibers at 123.000 fiber/in is, BHf= 5.52081E-02cm<br>The blade height needed for the end ply is,<br>BHe= 2.41417E-02cm |
| D13 | Summarizing results:<br>Volume frac. fibers in the dense composite is<br>VF= 0.356494<br>Blade height w/ fibers = 2.17355E-02in<br>Blade height for end ply (no fibers) = 9.50462E-03in<br>Density of fully dense composite = 3.96526g/cc<br>Thickness of green tape w/ fibers,<br>Tgf= 1.45606E-02in<br>Thickness of green end ply, no fibers,<br>Tge= 5.51268E-03in<br>Thickness of the dense composite = 3.49704E-02in |
| D14 | Input the density of the fully dense matrix in g/cc.<br>Enter zero to QUIT. |

The first thing that comes up after the initiation of the executable file Tcast2.exe are the displays marked above as D1 and D2. In D1, fs is the fiber spacing in units of fibers per inch, p is the number of plies, and Tc is the thickness you wish the final plate to be. When considering a fiber free system, as when using the code to make fibrous composites, p does not include the needed matrix-only end ply. Thus when making a “4-ply” plate, with or without fiber, 5 plies are actually used. The drum: between ply fiber spacing ratio is $\alpha$, see (6).

# Appendix B

The FORTRAN Tcast2 code.

```
*      Program Tcast
       implicit real (j-n)
*      by
*      Henry C. de Groh III
*      NASA m.s.105-1
*      Cleveland OH 44135
*      (216) 433-5025
*      Last modified Sept. 26, 1994
       write(*,*)'This program estimates the fiber volume fraction'
       write(*,*)'of tape cast composites, and the dozer blade'
 10    write(*,*)'heights needed to produce them. It is assumed that'
 11    write(*,*)'a powder slurry will be cast onto regularly spaced'
       write(*,*)'fibers. The program will need the density of the'
       write(*,*)'fibers, the matrix density, and that of the green'
       write(*,*)'mat without fibers. The density of the green mat'
       write(*,*)'can be found by weighing and measuring a sample'
       write(*,*)'fiber free casting. The program will also request'
       write(*,*)'the percent shrinkage in thickness from the wet to'
       write(*,*)'the green state for the fiber free tape. This'
 20    write(*,*)'allows the program to estimate the dozer blade'
       write(*,*)'heights. This program can also help with the cast-'
       write(*,*)'ing of fiber free mats. To use the code for a'
       write(*,*)'fiber free system - use a very, very small fiber'
       write(*,*)'diameter and a fiber spacing equal to the'
       write(*,*)'number of green plies used plus 1, divided by the'
       write(*,*)'final thickness wanted in the plate, that is,'
       write(*,*)'fs=(p+1)/Tc; with the drum:between ply fiber'
       write(*,*)'spacing ratio equal to 1.'
       write(*,*)' '
*      Dm, density of the matrix
 23    Dm=0
*      Df, Density of the fiber
       Df=0
*      di, diameter of the fiber in inches
 25    di=0
*      d, fiber diameter in cm
       d=0
*      fsi, fiber spacing in fibers per inch
       fsi=0
*      fs, spacing in fibers/cm
       fs=0
*      ps, the inerply to interply fiber spacing ratio, drum fiber
*      spacing in fibers/in :to: fiber spacing through the thickness
       ps=0
*      p, number of plies in the composite
       p=0
*      Dg, density of the fiber free green tape
 30    Dg=0
*      VFg, volume fraction fiber in the green tape
```

```
      VFg=0
*     Dgf, density of the green tape with fibers
      Dgf=0
*     Tgf, thickness of the green ply with fibers
 33   Tgf=0
*     S, fractional shrinkage from wet to green for fiber free tapes
      S=0
*     Sc, fractional shrinkage from wet to green with fibers
      Sc=0
      write(*,*)'Input the density of the fully dense matrix in g/cc.'
      write(*,*)'Enter zero to QUIT.'
      write(*,*)' '
      read(*,*) Dm
      If(Dm.eq.0) go to 109
 40   write(*,*)'Input the density of the fibers in g/cc.'
      write(*,*)' '
      read(*,*) Df
      write(*,*)'Input the diameter of the fibers in units of inches.'
      write(*,*)' '
      read(*,*) di
      d=di*2.54
 45   write(*,*)'Input the fiber spacing in fibers/inch.'
      write(*,*)' '
      read(*,*) fsi
      fs=fsi/2.54
      write(*,*)'Input the numbers of plies used in the composite, not'
      write(*,*)'including the fiber free end ply.'
      write(*,*)' '
 50   read(*,*) p
      write(*,*)'Input the ratio of the drum fiber spacing'
      write(*,*)'(in fiber/in) to the desired spacing between'
      write(*,*)'the plies (in fib/in).'
      write(*,*)' '
      read(*,*) ps
*     VF is the volume fraction of fibers expected in the composite.
*     Note that the thickness of the dense plies containing fibers is ps/fs.
      VF=3.1416*d*d*p*fs/4.0/((p+1)*ps/fs-d)
      write(*,*)'The volume fraction fiber in the composite is, VF=',VF
*     Dc is the density of the matrix plus fiber composite.
 55   Dc=VF*Df+(1.0-VF)*Dm
      write(*,*)'The density of the composite is, Dc=',Dc
*     Dg is the desity of the fiber free green tape.
      write(*,*)'Input the density of the green tape without fibers.'
      write(*,*)' '
 60   read(*,*) Dg
*     The density of the green tape WITH the fibers will be needed for later
*     calculations,Dgf, but, this density will vary with fiber spacing. Thus
*     a system of two equations (with two unknowns) will be set up and solved.
*     VFg is the volume fraction of fibers in the green tape (single ply with
*65   fibers). In the first equation for VFg, Dg is used as a starting point,
*     later, Dg is replaced by Dgf, the density of the green tape with fibers.
      VFg=3.1416*d*d/4.0*fs*fs*Dg/Dc/ps
 68   a=VFg
      Dgf=Df*VFg+Dg*(1.0-VFg)
```

```
 70   VFg=3.1416*d*d/4.0*fs*fs*Dgf/Dc/ps
      If(abs(a-VFg) .GT. 0.00001) go to 68
*     Tgf is the thickness of the green ply with fibers.
      Tgf=Dc/Dgf/fs*ps
      write(*,*)'The thickness of the green tape with fibers is,'
      write(*,*)'Tgf=',Tgf,'cm'
 76   write(*,*)'Input the shrinkage expected for a fiber free ply from'
      write(*,*)'the wet to green state, that is, the fraction decrease'
      write(*,*)'in thickness between the wet as cast tape and after'
      write(*,*)'the solvent has left the binder making the green tape.'
 80   write(*,*)'With a dozer blade height of BH and a green fiber free'
      write(*,*)'ply thickness of Tg, the shrinkage expected is,'
      write(*,*)'S=(BH-Tg)/BH.'
      write(*,*)' '
      read(*,*) S
*     Sc is the thickness shrinkage expected in the ply with fibers.
      Sc=VFg*0.0+(1-VFg)*S
*     Sc also equals (BHf-Tgf)/BHf, where BHf is the blade height needed for
*87   the composite ply containing the fibers.
      BHf=Tgf/(1.0-Sc)
      write(*,*)'The dozer blade height needed for the plies containing'
 90   write(*,*)'fibers at',fsi,' fiber/in is, BHf=',BHf,'cm'
*     BHe is the blade height needed for the powder cloth end ply.
*     Te is the thickness of the fully dense end ply (no fibers).
*     Tge is the thickness of the green end ply.
      Te=ps/fs-d
      Tge=Te*Dm/Dg
 96   BHe=Tge/(1.0-S)
*     Tc is the thickness of the of the dense multiply composite.
      Tc=(ps/fs*p+Te)/2.54
      write(*,*)'The blade height needed for the end ply is,'
      write(*,*)'BHe=',BHe,'cm'
      write(*,*)' '
      write(*,*)'Summarizing results:'
      write(*,*)'Volume frac. fibers in the dense composite is'
      write(*,*)'VF=',VF
 100  BHfi=BHf/2.54
      write(*,*)'Blade height w/ fibers =',BHfi,'in'
      BHei=BHe/2.54
      write(*,*)'Blade height for end ply (no fibers) =',BHei,'in'
 104  write(*,*)'Density of fully dense composite =',Dc,'g/cc'
      Tgfi=Tgf/2.54
      write(*,*)'Thickness of green tape w/ fibers,'
      write(*,*)'Tgf=',Tgfi,'in'
      Tgei=Tge/2.54
      write(*,*)'Thickness of green end ply, no fibers,'
      write(*,*)'Tge=',Tgei,'in'
      write(*,*)'Thickness of the dense composite =',Tc,'in'
      write(*,*)' '
      go to 23
 109  stop
      end
```

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A process by which metal matrix composites can be made was presented. The process involves putting a powder slurry on fibers to make a precursor green tape. These green tapes are cut, stacked and hot pressed to form the fully dense composite. A computer program was presented which enables complete quantification and control of the process. Once some easily obtained properties of the slurry and its behavior are determined (such as the shrinkage from the wet to green state, and the density of the green tape) modification of the fiber spacing and blade height give the maker precise control of fiber volume fraction, and fiber architecture in the composite. The process was shown to be accurate and flexible through the production of a wide variety of volume fraction fiber composites made from a wide variety of fibers and powders. The most time consuming step of the tape casting process (other than hot pressing) was winding the fiber on the drum. The tape casting techniques developed resulted in high quality metal matrix composites, with ultimate tensile strength in the range of 215 ksi (1477 MPa), a strain at failure of 1.15 percent, and in fatigue at room temperature 0 to 120 ksi, n = 0.3 Hz, a 4-ply Ti-24Al-11Nb/SCS-6, 32 vol% fiber tape cast composite lasted 202,205 cycles with a maximum strain on the 100th cycle of 0.43 percent.

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