Potential Failure Mechanisms For C/C-SiC Components

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Baseline properties for the "4-D" C/C-SiC under study may or may not be robust enough to withstand the forces imparted in the valve environment during the burn cycle. That is not the topic of this discussion. Let us examine more closely, using the HT-7 pintle failure, potential root causes and likely mechanisms pertaining just to the C/C-SiC material, disregarding all aspects associated with motor/valve dynamics including external forces which may or may not have been imparted to the pintle body during the burn cycle. The latter perspective is left to the propulsion/mechanical engineers and participating 'rocket scientists'. It is postulated here that the primary weaknesses of this particular material system and hence the most likely factors indirectly leading to its failure are associated with . . . (1) the machining process (or perhaps the *particular* machining process applied to the articles), (2) an unduely high level of closed porosity, and (3) the inevitably low continuous fiber volume in the z direction.

Evidence that the machining process damaged the pintle article is given by the image taken of the HT-7 shaft-to-head fracture surface (so called failure #2) showing that the seal coat material (a slurry of SMP-10 preceramic polymer, fine SiC particles and surfactants), *which was applied after the primary and most aggressive machining operations*, penetrated into the interlayer spacing between u-v-w layers. This image was given in ATK's Failure Investigation Report as well as in the more recent paper generated by the author. It is duplicated here again for reference in Figure 1 below. The region of slurry penetration is indicated by the red arrows of item 'A'.

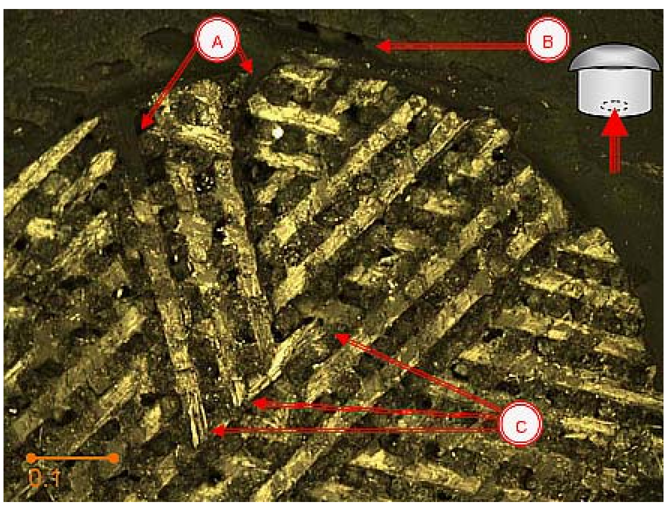


Figure 1. Image of one of the fracture surfaces in the second failure near the shaft-to-head interface

This penetration had to occur in a separated or highly weakened u-v interlayer region and not just within the existing composite porosity. Furthermore, the fact that the seal material is visibly apparent on the post-fractured surface strongly implies that the seepage contributed to the fracture, specifically during initiation of the fracture process. Machining is common practice for most composite structures and some of the more modern methods include machining to size and dimension via water-jet, laser and precision CNC. However, many ceramic materials are still machined the old fashioned way via grinding, sanding and sawing. Due to their brittle nature, ceramic materials are particularly vulnerable to machining damage. With any approach however, careful planning must be utilized in order to minimize machining damage (ideally, the goal should be to prevent damage, but this is usually not possible). As a consequence, a 'zone' of residual microcracks and/or weakened interfaces are often generated throughout the perphery of machined articles and has been observed in a variety of composite systems. It is especially systemic in composite systems containing brittle ceramic matrices. Figure 2 gives an example of the extent of damage which often results from typical CNC machining practices for a glass fiber-reinforced composite.

The extent and depth of the damage zone are expected to be notably higher for brittle ceramic matrix composites. Machining of surfaces in which the plies are parallel to the surface are less severe since interlaminar damage is minimized (interlayer in our case). However, surfaces in which the plies (or layers) are perpendicular to the surface are highly susceptible to interlaminar/interlayer fraying-type damage which permeates into the substrate body and comprizes the damage zone. This zone of weakened planes and separated layers is a result of mechanical interlayer damage which markedly reduces the fiber-to-matrix bonding properties in the damage zone. The effects are often manifested as latent debonds or micro-separations between weakened planes (layers) which fail at a later time when the appropriate forces are applied and/or they become permeable to the outside as was the case for the second HT-7 failure shown in Figure 1. Again, these descriptions apply to layered or laminated composites in general but they are particularly relevant to composites containing brittle matrices.

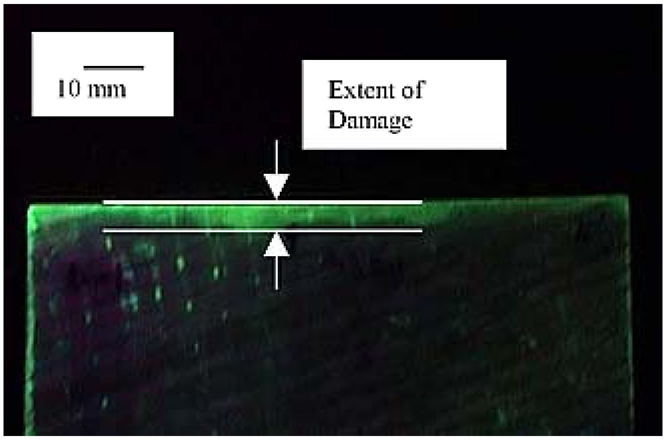


Figure 2. Machined surface after dye penetrant test on a glass fiber composite. Taken from, "Secondary Processing of Polymer Matrix Composites"; Inderdeep Singh, Debasis Nayak, Naresh Bhatnagar; Department of Mechanical Engineering; Indian Institute of Technology Delhi, New Delhi, India-110016.

Additional evidence of machining damage can be seen in other shaft cross-sectional views which reveal *z* bundles machined through to varying degrees. The extent of this damage on the HT-7 pintle has been well documented and visually confirmed in previous communications. There is no denying it, machined *z* bundles along the *z* surface of the article (the shaft direction) represent weak areas which are prone to failure. The more tow that has been machined away, the more weakened is the *z* directional reinforcement . . . and the *z* bundles are supposed to provide the composite's primary tensile strength component along the shaft direction. The nearest *z* bundle to one that has been completely removed is at least 0.05" away. Surfaces in which the *z* reinforcement has either been drastically reduced or completely machined away represent some of the weakest regions in this particular C/C-SiC system. There is evidence that areas exist on the machined pintle shaft in which two or more adjacent circumferential tows have been completely machined through. Without a doubt, these are extremely weak areas. Couple this with weakened *u*-*v*-*w* planes as iterated above and the most prominent failure initiation sites along the circumferemtial shaft surface can easily be identified (before the seal coat is even applied). Some of these details are illustrated in Figure 3 below.

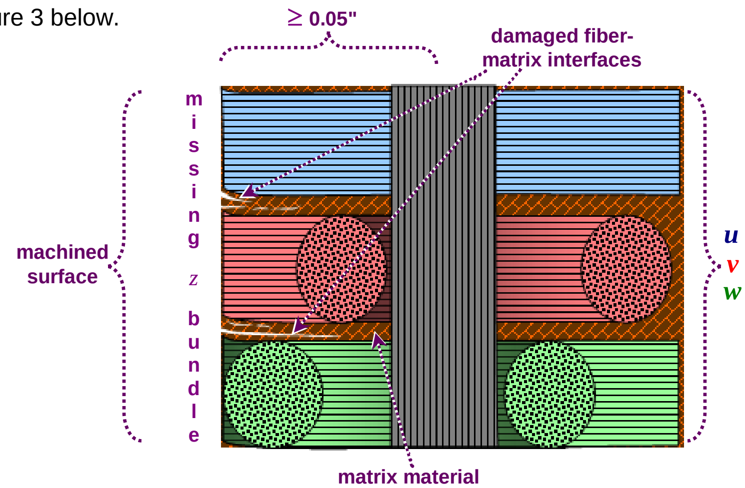
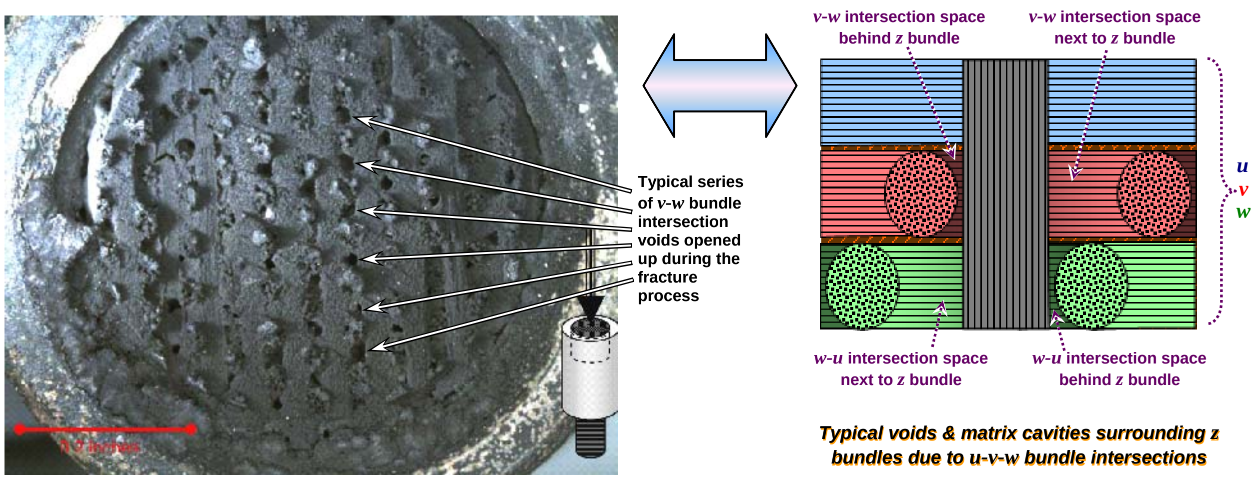


Figure 3. Illustration of likely *u*-*v*-*w* interlayer and z bundle damage generated as a result of machining operations.

Evidence for the existence of undesirably high levels of localized porosity, voids and cavities that were inadvertantly closed off, sealed or otherwise impervious to the densification fluids is provided again, by previous studies evaluating the failed HT-7 failure. Specifically, the fracture surface on the thread side of failure #1 reveals this localized porosity quite well. This image is duplicated in Figure 4a and contains an excellent represenation of the spaces/voids associated with *u*-*v*-*w* fiber bundle intersections as illustrated in Figure 4b (a ‘*u*+90’ direction exaggerated for detail). If the top layer of bundles shown in 4a is designated as the *v* layer then the apparent ‘holes’ must represent the void spaces under the *u* layer at *v*-*w* bundle intersections as illustrated 4b . . . except the void spaces in the actual HT-7 fracture surface are apparently absent of matrix material. In actuality, some of these holes are due to fiber (bundle) pull-out which will be addressed later.



(a) View looking down on the *x*-*y* plane

(b) View looking down on the *y*-*z* plane

Figure 4. (a) Thread side of the first HT-7 failure showing apparent layer of porosity/voids present at the fracture interface due to unfilled *v*-*w* bundle intersections; (b) Illustration of preform structure showing typical spaces and cavities associated with *v*-w bundle intersections.

While this level of porosity is surely not characteristic of the entire composite structure, it is, at the very least, prevalent in localized regions of the material . . . it was definitely present at this fracture interface as indicated in 4a. It is entirely possible that additional layers of voids are present but unseen just below this layer. It is important to realize that these voids were not generated during the burn cycle or the fracture process, rather, they were opened up during these processes and were actually formed during the composite manufacturing (densification) process. They are believed to be part of the ‘closed’ porosity portion of the total porosity/void volume fraction which were unveiled during the fracture sequence. In addition, they are considered to be responsible, in part, to the root cause of the fracture, particularly why the failure occurred at this particular planar region. These voids are probably localized to this region (further evidence for localized density/porosity variation in this material will be given shortly in Figure 5). If such porosity were present in all or most of the *u*-*v*-*w* layers, the total porosity of the composite would be on the order 40-50%, and much of this would be permeable to the Archimedes porosity/density test fluid resulting in a measured open porosity fraction significantly greater than the reported value of ~13%. The probability that this porosity was localized to this region is a reflection of the material variability. There are likely other areas or planar sections containing similar arrays of voids which are vacant of matrix material, perhaps those associated with *w*-*u* bundle intersections (under *v* layers) and *u*-*v* intersections (under *w* layers). Certain NDE methods may detect these voids to a degree, perhaps x-ray or high resolution ultrasonics. In any case however, these highly porous layers cannot be ignored since they represent weakened mechanical properties (they are present from the core of the substrate all the way to the surface) and they can definitely play a major role in subsequent fractures of the article.

The exact cause for the excessive level of voids generated in Figure 4a cannot be ascertained at this time. However, it is important to realize that these regions or layers of formerly closed porosity/voids were produced during the first or second SMP-10 densification cycle and became permanently sealed off (impervious) from that point on . . . the densification process was apparently not aggressive enough to open these voids up. A preliminary review of the manufacturing parameters and conditions used to produce this billet has already been issued with the conclusion that too few 3000°F heat treatment steps were applied to effectively open up and interconnect all the available the pores and void spaces. It is also suspected that mediocre impregnation techniques may have been employed.

Variability and density/porosity defects can also be imparted prior to or during the preform rigidization process causing localized bundle structural distortions or deformations which alter the porosity/void characteristics in the affected regions. Some of these voids may become impervious to densification fluids, creating additional closed porosity . . . the affected deformation region in the preform may itself represent weakened structure. Thus, potential weaving distortions and obvious preform deformations are apparent sources of material property variation. This is illustrated in Figure 5 which shows a cross-sectional view of a deformed region in the shaft side of the first fracture (a cross-sectional view of the mating face to that given in Figure 4a). Again, this image was evaluated in a previous report and is rehashed here to examine more closely the nature of variability introduced into the dry preform before any C/C or SiC densification cycles were applied. Red indications are remnants from ATK's analysis. The view looks down into the *x*-*z* plane (that is, a '*u*+90' direction, similar to that given in Figure 4b). White arrows indicate an array of filled or partially filled voids/cavities associated with *v*-*w* bundle intersections which have been exposed due to an apparent deformation in the preform (prior to densification processing). The deformation seems to have been imparted in a direction coming from the bottom right region of the image causing them to become visible in this view (*v*-*w* intersection cavities are most apparent when looking in a '*u*' direction, that is, on to the *x*-*z* plane). The deformation may have twisted the preform or otherwise opened up the affected *v*-*w* cavities by altering their volumes and shapes accordingly.

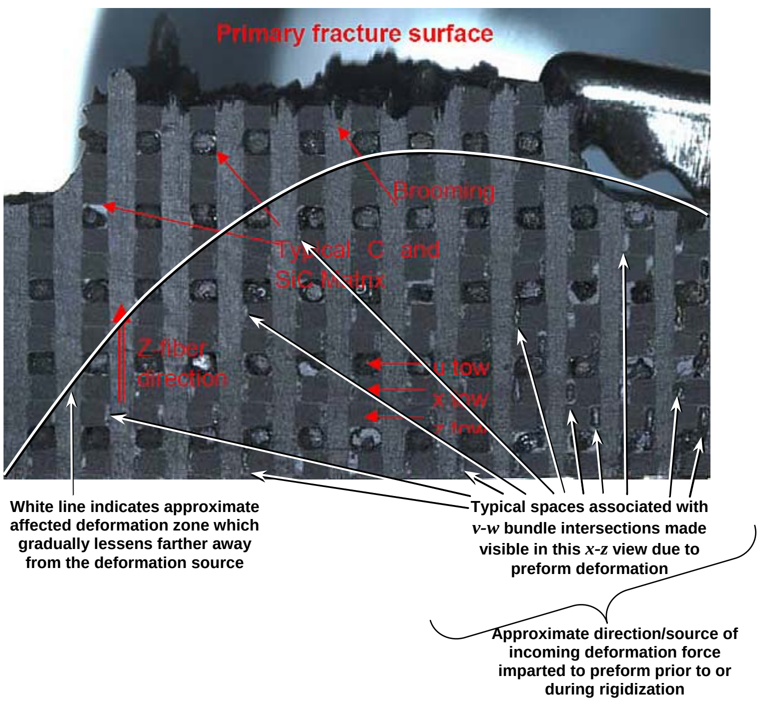


Figure 5. Cross-sectional view of the shaft side fracture surface looking down onto the x-z plane. Emphasized here are details associated with an apparent deformation that the dry preform experienced before or during the rigidizatiuon process.

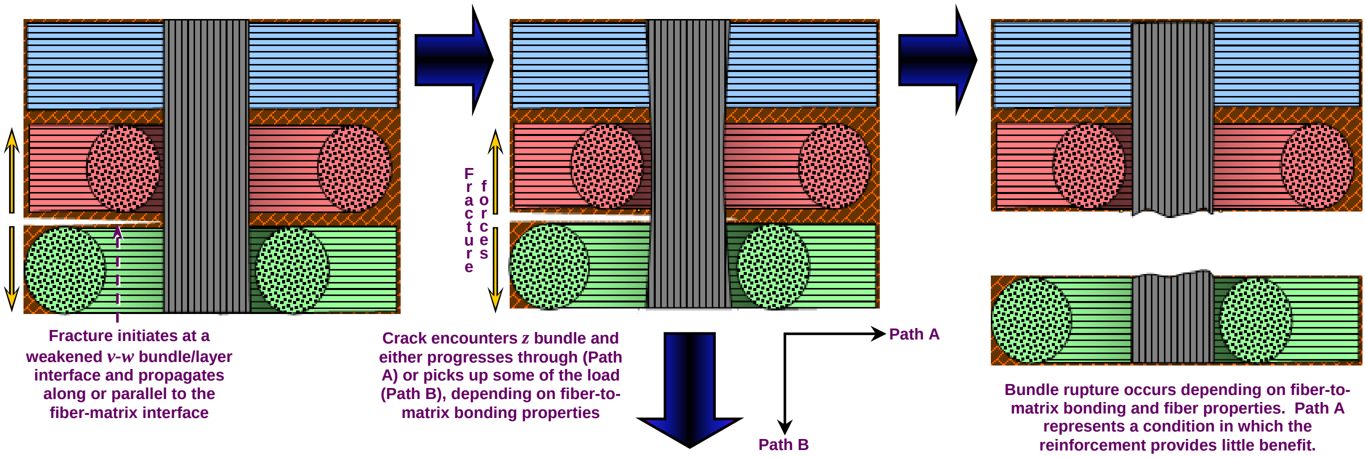
Average spacing between *z* fiber bundles in a given row is about 0.05". Out-of-row nearest neighbor distances are even greater. This relatively high spacing is a reflection of the low fiber volume fraction inherent in this particular composite system. Consequently, the wide spacing between *z* bundles transforms into an effectively low fiber volume fraction in the *z* direction. In polymer matrix composites (PMC's), fiber volume = mechanical strength. In ceramic matrix composites (CMC's), this rule may not be as relevant but still plays a dominant role in specific directions and in particular composite designs. For the C/C-SiC CMC system under study, the *z* bundles supposedly provide the primary tensile capabilites along the shaft dimension (overall however, the presence of the fiber fraction in CMC's reduces their brittleness and adds flexibility to the composite, a property quite lacking in monolithic ceramics).

It has already been shown that the transfer of fiber tensile strength to the composite level is extremely inefficient in the z direction (about 20% what it should be). This result was based on C/C-SiC billet test data supplied by FMI, which was probably acquired on samples using slow loading rates. For CMC's, the exact level of strength measured is directly dependent on the stress loading rate chosen by the test engineers (high rates give higher mechanical strengths and vise versa). At moderate to high fiber volume levels, this effect can be greatly enhanced by facilitating delayed failure of the composite due to toughening phenomena. At lower fiber volume levels however, the toughening effect diminishes and the failure process becomes more like that of the monolithic ceramic.

The effects of machining, closed porosity and low fiber volume are suspected of being primary drivers associated with the *root cause* of the HT-7 failure (considering only the C/C-SiC material, not the motor/value system). The relative influence of these three factors on previous HT failures may or may not have played prominent roles since the capabilites of both C-SiC and C/C-SiC pintle articles, which seemingly passed previous tests, could have been borderline to begin with. One thing is certain however . . . All three of these factors are variable throughout the current C/C-SiC material. The machining forces vary according to the surface contour requirements as well as the specific (localized) material properties encountered as the machining tool/process shapes the article.

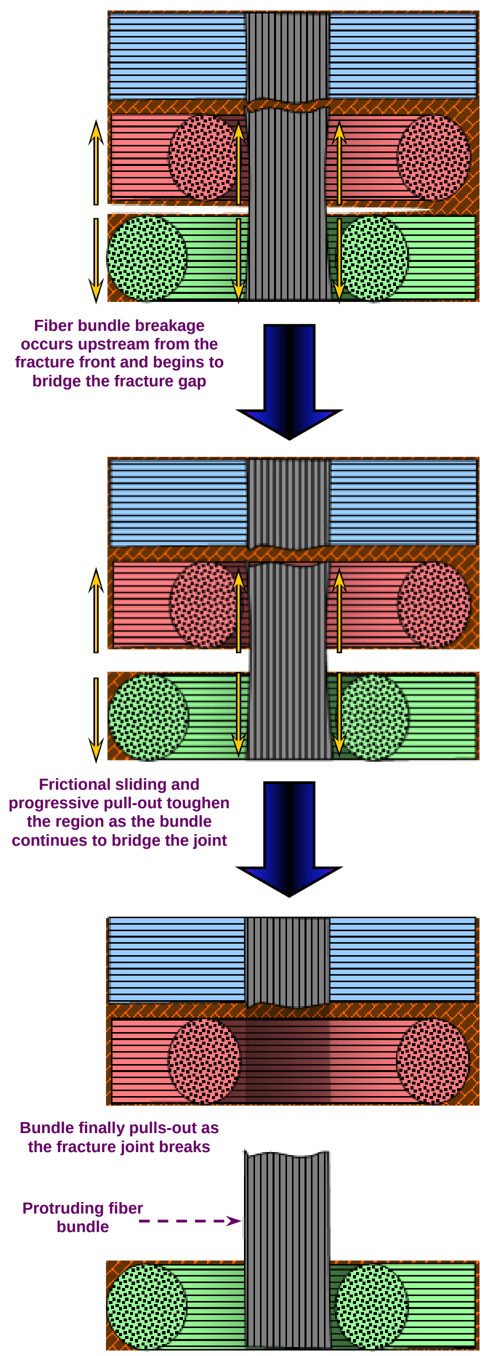
The total 'porosity' of a composite, which includes micro-, meso- and macro-pores, voids, cavities, tunnels, fiber-to-matrix debonds, interbundle separations and interlayer delaminations (if they are present), is almost always related to aspects of the manufacturing process and/or attributes of the material design/concept. *Localized* collections of voids, pores and cavities typically have a direct connection to the specific manufacturing processes employed and/or attributes associated with the particular material design under consideration. Random events can often be attributed to anomalies generated during specific steps in the process flow of the given billet or article which were defective for some reason or another, while recurring instances of high volume localized porosity is an indication that the basic manufacturing approach (and/or material design) is either inadequate or is not optimized for the material system in work. Many of these kinds of defects are extremely hard to detect and predict which is the primary reason NDE methods are used throughout the composites industry. For this particular C/C-SiC material system, it is believed that localized regions containing high levels of pores, voids and cavities that are closed, sealed off or impermeable (namely those at u-v-w bundle intersections) can be attributed, for the most part, to less-than-ideal impregnation/densification processes and techniques.

The *failure mechanism* for both PMC's and CMC's involves at least six factors or processes . . . (1) matrix cracking, (2) delamination (or 'delayering' in our case), (3) fiber breakage, (4) fiber-to-matrix debonding, (5) fiber (bundle) bridging, and (6) fiber (bundle) pull-out. Each of these factors facilitates the failure process by absorbing fracture energy in their own unique manner. Which particular factors dominate a given failure depends on the matrix type and the loading rate. Matrix cracking and delamination are primary mechanisms associated with the failure of PMC's subjected to slow loading rates or low velocity impacts. However, the dominating mechanisms leading to energy absorption in CMC's during high stress rates and high velocity impacts may involve a sequence of events on the order of . . . (1) fiber-to-matrix debonding, (2) fiber (bundle) bridging, (3) fiber (bundle) pull-out, and finally (4) fiber (bundle) breakage. Here, the energy absorption mechanism is analogous to one involving quasi-static propagation of a crack perpendicular to the fiber (bundle) direction. Most importantly, this scenario is associated with bridging of the crack by partially debonded fiber bundles which are in the path of the propagating crack. This effect tends to 'delay' the failure, and the mechanism in itself has a unique effect on the fracture toughness of the composite due to the process of progressive debonding and pull-out of the bridging fiber bundles. For the C/C-SiC system, the following scenario might be considered . . .



The fiber-to-matrix interface in composite materials consists of both chemical bonding and physical interactions (which includes mechanical interlocking). In PMC's, both chemical and physical binding are quite pronounced. But in CMC's (including C/C-SiC), physical/mechanical interactions constitute the most likely bonding mechanism dominating fiber-matrix interfaces throughout most regions of the composite.

Now in the C/C-SiC material, the fracture begins with a crack in the brittle matrix along the weakened fiber-to-matrix interface. When the crack encounters a fiber bundle, two events are possible. Path A: If fiber-to-matrix bonding interactions are exceptionally strong (i.e... high levels of both chemical and mechanical bonding), the crack will fracture the bundle and continue to grow across the fracture surface. In this scenario, the fibrous reinforcement provides no benefit to the composite (in this region). A similar conclusion could be drawn if the fiber-to-matrix bonding was too weak . . . the reinforcement would provide little or no benefit since the composite would experience excessive fiber pull-out and minimal strengthening effect from the reinforcement. In PMC's, the matrix is elastic which mitigates fiber rupture and provides for an exceptionally robust structural composite system.



Path B (the toughening process): If the extent and strength of fiber-to-matrix bonding is moderate, energy will be transferred into the formation of debonds perpendicular to the progressing crack (fiber-to-matrix debonds above and below the crack). In this case, the rate of progression of the fracture is temporarily 'delayed'. As the matrix on the other side of the bundle begins to crack, the bundle is still capable of transfering load across the crack surfaces in an effect referred to as 'fiber bridging'. When the bundle finally breaks (usually away from the fracture plane), the crack continues to advance but bridging allows the fiber bundle to continue carrying some of the load. Here, fiber-to-matrx frictional forces, continued debonding and bundle pull-out absorb more fracture energy which greatly enhances the toughness level of the composite. Thus, the CMC toughening mechanism begins with the fiber bundle bridging stage and continues to function beyond breakage until pull-out finally occurs.

A combination of apparent fiber bundle pull-out (moderate interface bonding) and crack propagation directly through the bundles (strong interface bonding) seems to be prevalent across the fracture surfaces of failure #1. However, the latter factor might exhibit a slight dominance (as indicated in Figure 5) implying that crack propagation directly through the bundle occurred in a substantial number of the crack-to-bundle encounters (i.e... Path A). This would support previous data and conclusions reflecting the impressive level of apparent fiber-to-matrix bonding throughout the body of the pintle. It also implies that the z fibrous reinforcement provided less-than-optimal benefit or advantage to the composite in this particular failure. And there is no reason to believe that this mechanism/path is localized just to this region of the material . . . It may be representative of the overall mechanical behavior relative to the reinforcement throughout the entire body. This would be indicative of the unduely low fiber volume fraction in the z direction (or spacing between bundles; both reflect the same property).

One of the most significant effects of the fibrous reinforcement in CMC's (and C/C-SiC) occurs in the post-cracking stage where the bundles 'bridge' the crack gaps and delay the failure process. Bridging of fiber bundles in the crack joint toughens the fractured interface by transfering the load directly to the reinforcement phase temporarily delaying the ultimate separation of the two surfaces. Delayed failure is consistent with loading rate since high stress rates (and impacts) are expected to give higher strength values than slow loading rates. This strength dependency on stress rate is also characteristic of unreinforced monolithic and glassy ceramics. The high impact strengths for ceramics used in armor applications are related to the delayed failure effect and its role in the toughening phenomena. However, increased composite toughness via fiber bundle bridging, pull-out and delayed bundle breakage usually comes at the expense of decreased ultimate strength levels.

A few words about modeling approaches. Most material testing and measurement programs are aimed at developing average, overall properties for the system under study, or they eventually end up with similar generalized averages after all the testing is done. Unfortunately, critical material properties vary form point-to-point in all composite systems. Also, too often, those seeking 'material properties' in order to build a model for a given composite system are merely seeking 'mechanical properties', and in some applications, a purely mechanical model may be adequate. However, for a reinforced composite system, the term 'material properties' must include, not only all the mechanical attributes (the various stresses, strains, modulii, toughness and fatigue characteristics), but also precise values for relevant physical properties such as the composite bulk density, true density, open porosity, closed porosity, fiber volume, matrix content, fiber content, matrix volume and matrix density, along with very accurate and confirmed values for each of the constituent densities and their porosities (for instance, filament and bundle porosities, a subtle but leading contributor to composite mechanical attributes, is rarely recognized by the mechanical model makers). For most advanced composite systems, any model that contains only mechanical attributes will not provide the total picture and in many cases, will be inadequate. A mechanical model simply cannot accurately describe all the expected behaviors and ramifications of system which has many contributing factors.

Isotropy/anisotropy: The a-SiC and β-SiC matrix phases are essentially isotropic in and within themselves while the carbon matrix phase may be considered partially isotropic within itself (however, a phase boundary between a-SiC and β-SiC domains is imminent . . . these two allotropes are not the same). On the other hand, the fiber phase is highly anisotropic within itself and most importantly, throughout the composite, and the composite network as a whole, is extremely anisotropic in all directions. It would go without saying, any model concept that neglects this high degree of anisotropy is wholly inadequate. The C/C-SiC system bears some similarities modern Portland cement formulations which contain fibers for toughening and structural benefits.