INFLUENCE OF NEEDLED FELT C/C PROCESSING ON FRICTION PERFORMANCE

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Introduction

The aircraft industry now consumes the largest share of C/C composites produced. These materials are used as brake discs owing to outstanding thermal properties and low weight. In the past, a variety of different fiber architectures and matrix precursors were used for their production. Today, the market is decidedly shifting towards composites formed from needled felt architectures with a matrix derived entirely from CVI. These materials allow for considerable flexibility in design of product properties through adjustment of processing parameters such as needling configuration and heat treatment procedures. One result of these adjustments is an influence in friction performance.

Structural rearrangement of carbon atoms during heat treatment results in crystallographic ordering, the extent of which depends upon the organic precursor and carbonization (or deposition) conditions and is accompanied by changes in physical, mechanical, thermal, electrical and chemical properties [1]. The increased order that comes about with high temperature heat treatment (HHT) greatly influences thermal conductivity or diffusivity [2]. This is primarily due to increased mean free path as lamellae straighten, crystallites grow, and defect state minimized [3]. Thus, graphitizable carbons will undergo tremendous increases in thermal conductivity as temperatures of processing increase [4]. For single crystal graphite, thermal conductivity along the base plane is 200 times greater than perpendicular to it. Values as high as 4180 W/mK have been reported [5].

The thermal transport of a C/C composite is strongly influenced by fiber arrangement [6]. Values for thermal conductivity are generally several times greater within the plane of the fibers compared to the transverse direction (typical composites use a 2-D architecture). Use of 3-D fiber structures can greatly reduce this anisotropy. It is often considered that the composite thermal transport is limited by the value of the fibers. However, when a CVI matrix is used a substantial increase in conductivity can result in the direction of the fibers. This is a result of the high degree of order attainable in CVI carbons and the fact that the matrix will align itself with the fiber axis during deposition. Thus, the composite may have values higher than that of the fibers. The CVI matrix can also greatly increase conductivity transverse to the fiber direction as a result of the degree of order present. With appropriate architecture and matrix selection a C/C approaching isotropic thermal conductivity may be produced.

It has been shown that needled felt C/C composites can be produced with varying through thickness thermal diffusivity by altering the z-fiber (fibers in the through-thickness direction) content of a needled felt preform [7]. This influences friction performance through a wide range of stop conditions. Higher z-fiber content reduced surface temperatures and gave greater wear resistance to the composite. This also affects the onset of desorption transitions.

This paper reports on the friction performance of needled felt composites that have been graphitized after CVI densification. Details regarding the materials used are published elsewhere [7]. Since the matrix comprised ~70% of the material it strongly influenced the composite properties. Friction performances of various needled felt C/C composites with and without matrix graphitization are compared to demonstrate the effect of processing influences.

Experimental

Needled felt C/C composite preforms containing different amounts of z-fibers were densified by CVI. One set of composites was tested directly while another set was graphitized before testing. The resulting material was prepared for powder x-ray diffraction (XRD) analysis using standard techniques. Samples for thermal diffusivity measurements were taken from various portions of the C/C
discs such that measurement of through thickness (z-direction) and in-plane (xy-direction) thermal diffusivity could be performed. Samples were measured at temperatures from ambient to ~850°C using the flash laser diffusivity technique.

Sub-scale dynamometer brake tests of the needled felt C/C composites were performed under simulated taxi and normal landing conditions. Cold taxi stops were performed with repeats after discs had cooled to 30°C. Hot taxi stops were repeated immediately so that disc temperatures were raised to >60°C between stops. This caused an increase in maximum stop temperatures. In all tests retarding torque was held constant. The needled felt C/C composites were labeled A and B corresponding to z-fiber contents of approximately 5% and 15%, respectively, of the total fiber volume fraction.

Results and Discussion

Graphitization heat treatment resulted in increased crystalline order as indicated in Figs. 1 and 2 where XRD results of the C/C composite before and after HHT are shown. From this data, average interlayer spacing (d_{002}) was determined to have decreased from 3.380Å to 3.366Å as a result of HHT. Crystallite size is found to increase, from ~122Å to 245Å for Lc. Increased layer stacking order is indicated in Fig. 2 by the splitting of the diffuse peak at ~44° into two distinct peaks from the (100) and (101) planes. As a result of comparing to data obtained from just the graphitized carbon fibers, such layer ordering can be attributed to the CVI carbon phase. Moreover, a phase with the potential for superior thermal transport was created by HHT.

![Figure 1. XRD pattern for needled felt C/C before and after HHT.](image1)

Thermal diffusivity increased as a result of HHT. Figure 3 gives results for the through thickness values obtained from the B-type needled felt (~15% z-fiber content). Room temperature values were found to increase by a factor of 12 compared to before HHT. Diffusivity decreased with increasing measurement temperature, though the high temperature values for the HHT C/C is more than twice as high as the room temperature values obtained before HHT. The samples used were obtained from near the friction surface of the discs. When using samples from the disc interior higher values were obtained (0.85 cm²/s at room temperature). This is consistent with results obtained from the discs prior to HHT. It was determined that the needled felts used contained higher z-fiber content in the mid-plane of the discs as a result of the needling process used [8]. The higher z-fiber content results in greater values of through thickness thermal diffusivity.

![Figure 3. Through-thickness thermal diffusivity of needled felt C/C (~15% z-fiber) before and after HHT.](image3)

Diffusivity of the lower z-fiber content material (type A) was also increased by a factor of ~12 in the z-direction. Room temperature values averaged ~0.42 cm²/s and ~0.78 cm²/s for near surface and mid-plane specimens, respectively. In plane thermal diffusivity was also tremendously increased by HHT. The average room temperature values were 2.48 cm²/s and 2.27 cm²/s for A-type and B-type C/Cs, respectively. The slightly lower in-
plane value of the higher z-content disc is consistent with the trend before HHT and results from the lower content of xy-fibers. It must be emphasized that the CVI carbon forms about the fibers such that transport in the matrix is greatest in the direction of the fiber axis.

The values for thermal diffusivity are used to determine thermal conductivity by using specific heat values for graphitic material found in the literature [6] and assuming constant density. The expression: \( K = \alpha \rho C_p \) gives the relationship between thermal conductivity \( K \), thermal diffusivity \( \alpha \), density \( \rho \) and specific heat \( C_p \). These are given in Fig. 4 normalized by room temperature values. Through thickness thermal conductivity of 72 W/mK at 24°C is quite high for a C/C material. This reduces to ~40 W/mK at 827°C. An in-plane conductivity of ~280 W/mK is obtained for ambient temperatures. Values reported in the literature range from 10 W/mK to 171 W/mK for conductivity transverse to fibers, however little detail is given regarding C/C structure and heat treatment [6]. Reported in-plane values range from 100 W/mK to 350 W/mK for typical C/C composites. In-plane values for C/C produced from vapor grown fibers have been reported to have thermal conductivity as high as 564 W/mK. The values reported here fall within these reported ranges. Although measured through thickness values are at the middle of the range reported, it is considered to be high for composites suitable for brake use.

These transition data are presented in a different format in Fig. 6. Before HHT these needled felt C/C discs passed the transition at ~600 rpm. With the increased thermal diffusivity after HHT, and lower surface temperatures, the transition is not passed unless initial velocities are >1400 rpm. It was also often observed that when transition occurred during a stop, considerable noise was generated.

Figure 4. Through-thickness thermal properties of needled felt C/C (~15% z-fiber) after HHT. Room temperature values were used to normalize data as shown.

Stops were performed under a variety of conditions to determine the braking power required to generate a moisture desorption transition. After machining, the discs were broken in by conducting 56 hot taxi stops, shown in Fig. 5. Then initial stop velocity was increased from 546 rpm to 1192 rpm for three stops. Maximum near surface temperature rose to ~120°C but no transition occurred. Initial velocity was increased to 1418 rpm which did cause the transition to occur after ~3 seconds into the stop (#2). Average friction coefficient increased 500%. The discs were removed and inspected. After reinstalling three stops at 546 rpm were performed. Then a stop with 1638 rpm (#3), three at 546 rpm, one at 2184 rpm (#4), three at 546 rpm, another at 2184 rpm (#5), followed by three at 546 rpm. As can be seen by the friction coefficient, transition was passed above 1400 rpm. Stops less than 1400 rpm were below the transition.

Figure 5. Average friction coefficient of needled felt C/C (type A) after HHT under variety of stop conditions as indicated in the text. RH = 54%..

Figure 6. Average friction coefficient of needled felt C/C (type A) after HHT under variety of initial stop velocities. Stops 56-73 of data shown in Fig 5.

Stops were performed with the same discs under dry cold taxi conditions (2% relative humidity air). The appearance of the friction surface was gray and highly polished when viewed by eye. Under polarized light microscopy the original carbon structure was revealed, indicating little friction film had developed. The debris present mostly filled the small surface pores and cracks. An example is given in Fig. 7. The film that had formed under ambient humidity levels had been removed by stops in dry air. In addition, these conditions did not allow for substantial film build-up.
A similar condition was noted after normal landing stops (initial velocity 2944 rpm) were conducted under ambient humidity levels. In that case surface temperatures were high enough to desorb moisture and give performance above the transition. Under those conditions no substantial friction film had developed, and surfaces appeared similar to that shown in Fig. 7. The difference between the two sets of stops was that normal landing gave variable friction while dry cold taxi gave more stable μ from one stop to the next. This may be due to the lower velocities of cold taxi since vibration and misalignment, which are both system dependent, can cause variable friction at the higher speeds.

![Figure 7. Friction surface after dry cold taxi stops (above transition) showing polished surface with minimal friction film present.](image1)

After a run-in surface containing substantial film was forced through a transition (test shown in Fig. 5, #2) the discs were removed for inspection. What was a gray polished appearance had been altered to have dark black streaks or bands. Some regions appeared to be flaking off. Microscope examination revealed many blisters had developed in the friction film. This can be seen as regions in and out of focus in the micrograph of Fig. 8. Many blisters were fractured and flaking away from the underlying C/C. Many regions contained coarse, loose debris on the surface and some areas were stripped of film.

![Figure 8. Optical micrograph of disc A3 showing blister formation after transition stop.](image2)

It is generally accepted that desorption of moisture occurs at the transition. What is still not well established is the mechanism responsible for the increased friction. Furthermore, our studies indicate the transition is associated with the destruction of the friction film. It is likely that the loss of moisture changes inter-particle attraction forces, and alters the film properties and its bond to the C/C substrate. The fact that little film developed above the transition reinforces the theory. Blister formation may also be associated with the vibrations created when passing the transition.

The wear rates from these tests are shown in Fig. 9. The data indicate a reduction in wear rate occurs from HHT. The trend before heat treatment was for the hot taxi stops to give the greatest wear rate [7]. This was attributed to the higher μ that resulted from operation above the transition. The C/C after HHT did not pass the transition, and the wear rates did not change much from the cold taxi to hot taxi condition. Also, the trend of higher z-fiber content materials having greater wear resistance is not seen in the specimens after HHT. This may be due to the small wear rates and lack of precision in determining wear. Normal landing stops after HHT gave higher wear compared to taxi and can be attributed to the performance of C/C operating above the transition. Loss of friction film allows for greater wear of the surface owing to the loss of protection such a film provides.
Figure 9. Wear rates for dynamometer testing of needled felts before and after HHT. Specimens A and B tested under Cold Taxi (ACT, BCT), Hot Taxi (AHT, BHT) and Normal Landing (ANL) conditions.

The wear rates for the material tested in dry taxi conditions was the highest measured, 0.0002 gm/rot. This is seven times higher than normal landing stops that also performed above the transition. It is important to consider the mechanism responsible for such performance. When operating under ambient humidity levels there is a large amount of moisture adsorbed by the friction films and debris, as well as the bulk material. While temperatures rise during braking some moisture is desorbed and mu increases. However, enough moisture can be stored in the C/C composite to continually supply the surface with small amounts during sliding. This can act to reduce wear mechanisms. Such a moisture reservoir is lost when operating at very low relative humidity. After a period of dry operation very little moisture is left to create a wear reduction mechanism.

Conclusions

We have studied the affect of high temperature heat treatment of C/C composites and the subsequent influence on thermal transport properties. Heat treatment increased crystallite size and reduced interlayer spacing. This caused thermal diffusivity to increase by a factor of 12 at room temperature. The increased diffusivity resulted from the CVI matrix graphitization, which is preferentially aligned with the fibers.

The needled felt C/C materials tested after HHT gave significantly different friction performance compared to the same materials prior to HHT. Taxi stops produced a thick friction film that was not found on specimens before HHT. The brake power needed to generate a friction transition was greatly increased by HHT. Initial stop velocity of 1400 rpm was required compared to 600 rpm before HHT. This is due to increased thermal transport from the friction interface giving lower surface temperatures. Operation above transition caused substantial friction film removal and higher wear rates.

The occurrence of a transition during a stop caused blistering of friction film, and often resulted in system vibration. In addition to the above, wear rates were reduced by HHT.

References


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