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High temperature tensile properties of 2D cross-ply carbon-carbon composites

S.R. Dhakate[1*], T. Aoki[2], T. Ogasawara[2]

[1]*Carbon Technology Unit, Engineering Materials Division, National Physical Laboratory, Dr. K.S. Krishnan Marg, New Delhi 110012, India*

[2]*Advanced Composites Evaluation Technology Centre, Japan Aerospace Exploration Agency, Ohsawa, Mitaka, Tokyo 181-0015, Japan*

[*]Corresponding author. Tel: (+91) 11 45608257; Fax: (+91) 11 45609310; E-mail: dhakate@mail.nplindia.ernet.in

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ABSTRACT

High temperature tensile properties of 2D carbon-carbon composite made from high strength T700 carbon fibers were evaluated at different temperatures. Carbon-carbon composites were heat treated at different temperatures i.e., 750, 1000, 1500, 2000, 2500 °C and their tensile properties were measured at room temperature and at different high temperatures. It is observed that, maximum value of tensile strength at room temperature is of composite heat treated at 1500 °C thereafter strength decreases with increasing processing temperature up to 2500 °C. The decreases in strength are related to degradation of fiber properties in composites and in-situ damage. On the other hand, tensile strength is higher at high temperature compared to room temperature. It increases progressively with increasing the test temperature up to 2000 °C. Thereafter, strength decreases and ultimate value of tensile strength is less than that of the room temperature value of 2500 °C heat treated composites. Increase in strength up to 1500 °C is due to the improvement in fiber-matrix interactions, matrix properties due to relaxation of thermally induced stresses during high temperature test. Above 1500 °C enhancement in tensile strength is due to the enhancement in strength of carbon fibers and due to the creep deformation. Decrease in strength at measurement temperature 2500 °C is due to the additional in-situ degradation of fiber properties during high temperature test.

Keywords: Carbon/carbon composites; high temperature; tensile properties.

Sanjay R Dhakate is scientist working at National Physical Laboratory, New Delhi (India) from 1992. He did his Ph.D. from Delhi university. His research interests mainly on carbon materials i.e. Carbon fibers, Carbon –Carbon composites, Carbon nanotubes, graphene synthesis by exfoliation of graphite, Nanoribbons by unzipping CNTs, continuous polymer and carbon nanofibers by electrospinning, Development of carbon based PEM fuel cell components. He is a receipt of JSPS and JICA fellowship. Recently he has been selected for Advanced Materials Letters Scientist award 2010.

Toshio Ogasawara is a senior researcher of Japan Aerospace Exploration Agency (JAXA). He received his B.S (1985), M.S. (1987), and Dr. Eng. (1994) in materials science from Tokyo Institute of Technology. He worked at Nissan Motor Co. Ltd., from 1987 to 1998, and joined JAXA (former National Aerospace Laboratory of Japan) in 1998. He has worked for research and development of high température polymer matrix composites, carbon / carbon composites, ceramic matrix composites, and carbon nanotube based composites.

Takuya Aoki is a senior researcher of Japan Aerospace Exploration Agency (JAXA). He received his B.S (1996), M.S. (1998), and Dr. Eng. (2001) in materials science from Tokyo University of Science. He joined JAXA (former National Aerospace Laboratory of Japan) in 2001. He has worked for research and development of carbon/carbon composites, ceramic matrix composites, and ultra-high temperature ceramics.

Introduction

Carbon/carbon (C/C) composites are light weight material possess excellent thermo-mechanical properties at room temperature and at high temperatures. Thus C/C composites have been considered as the most promising material for high temperature structural applications, such as thermal protection system in space vehicles, heat resistant components in rocket nozzles, plasma-facing surface and diverter plates of the tokomak fusion reactor etc. [**1-4**]. The performance of C/C composite is known to depend on the types of carbon fibers, matrix precursors, weave geometry (1D, 2D or 3D), nature of bonding between the

fiber and the matrix (fiber-matrix interface) and processing conditions [**5, 6**]. In order to understand the mechanical properties of C/C composites; several studies have been carried out by researcher and it is available in open literature particularly on 2D-C/C composites [**7-17**]. But on the other hand, very limited information available is on the high temperature mechanical properties of C/C composites [**18-23**] because of the sensitivity of this work. The reports available on the high temperature properties are contradictory in regards of temperature dependence tensile strength or flexural strength of C/C composites. Some studies reported that, mechanical properties of C/C composites are not depends on the test temperature [**19-21**]. In other studies reported that, strength of C/C composite increases with test temperature [**22-24**]. However, in case of polycrystalline graphite, tensile strength is enhanced by de-gassing up to 1500 °C [**24**] and above 2000 °C is due to the creep deformation [**25**]. It is also reported that in case of C/C composites strength increases at high temperature up to 1500 °C is due to the degassing of absorbed water and above 1500 °C is due to the creep deformation [**22**]. In some studies, it is also reported that de-gas treatment enhanced the interlaminar shear strength of C/C composites [**26, 27**] but it does not influence strength of the composites.

In the present investigation systematic approach is adapted to study the effect of high temperature on tensile properties of high strength carbon fiber composites. The C/C composites are heat treated at different temperatures and characterized for tensile properties at room and at different high temperatures to understand the effect of test temperature on properties of C/C composites. It is found that, tensile strength increases with increasing test temperature up to 2000 °C compared to room temperature properties. Thereafter, strength decreases and ultimate value of tensile strength is less than that of the room temperature value of 2500 °C heat treated composite. Above 1500 °C enhancement in tensile strength is due to the enhancement in strength of carbon fibers as a cause of creep deformation. Decrease in strength at measurement temperature 2500 °C is due to the additional in-situ degradation of fiber properties during high temperature test.

Experimental

In the present study, two types of 2D cross-ply (0°/90°) commercially available C/C composites were used (Supplied by Across company Ltd., Japan). These composites were made from poyacrylonitrile (PAN) based T700 high strength carbon fibers as reinforcements and phenolic resin as a matrix precursor. Composites were heat treated at 750, 1000, 1500, 2000, 2500 and 2800 °C in inert atmosphere. Tensile strength of composites heat treated at different temperatures (750, 1000, 1500, 2000 2500 and 2800 °C) were measured at room temperature on servo-hydraulic testing machine (Model 8502, Instron Corporation, USA). The tensile test specimens were machined from composites plate such that loading direction is parallel to 0° oriented fiber plies. The tensile specimen with gauge length of 30 mm, thickness ~ 3 to 3.5 mm and width 6 mm and overall length of specimen was 180 mm. The tension was applied at constant speed 0.5 mm/min. The dog bone types test specimens gripping region was protected by thick paper tabs and strain was determined by strain gage. The high temperature tensile test was conducted at on individual composites heat treatment temperature (HTT) i.e. composites heat treated at 750 °C, there tensile test is carried out at 750 °C and so on. Tensile test of composites at high temperature (after machining of desire specimen geometry, **Fig. 1** was measured at on screw-driven Instron universal testing machine (model 8502) equipped with a furnace having high temperature capability up to 1500 °C under vacuum. The strain during the tensile test at high temperature was determined using an electro optical non contacting extensometer 200X (Zimmer Germany). Above 1500 °C high temperature tensile test carried out at Institute of Space and Astronautical Science (JAXA), Kanagawa.

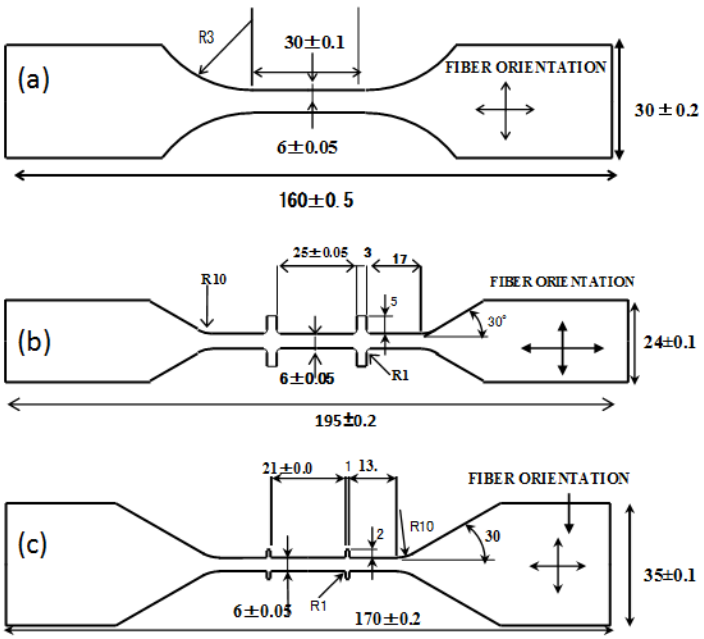

Fig. 1. Tensile test specimen geometry in different dimension (a) for room temperature test, (b) high temperature up to 1500 °C, and (c) high temperature up to 2500 °C.

Tensile test was carried on screw-driven universal testing machine (AG-10G, Shimadzu Corporation, Japan) equipped with high temperature facility up to 3000 °C in inert atmosphere. The strain during the tensile test at elevated temperature was measured using an optical extension device (Type NCE Shimazdu Corporation, Japan) by tracking the 2 × 2 mm target of the specimens. The test specimens were loaded via a polycrystalline graphite test fixture and whole specimen was set in a uniform heating zone of the furnace. The temperature of the specimen was measured using a thermocouple by setting near the gauge section of specimen up to a temperature 1500 °C and using optical pyrometer above temperature 1500 °C. The test specimen was heated by constant heating rate of 10°C/min up to test temperature. After maintaining 30 min at test temperature, tensile test was conducted at constant cross head speed 0.5 mm/min. After tensile test, fracture surface of specimens were observed by field emission scanning electron microscope (SEM, modal S-

4700, Hitachi Ltd, Japan). To know the stretching effect under applied stress on the crystalline parameters of composites before and after a high temperature tensile test, C/C composites specimen was characterized by XRD with a Cu-Kα radiation under condition of 40 KV and 200 mA and scan speed 0.2/min (modal RINT2500, Ringaku Co.; Tokyo, Japan). **Fig. 1** shows the dog bone type tensile test specimen geometry in different dimensions (a) room temperature test specimen (b) high temperature test specimen up to 1500°C and (c) high temperature test specimen up to 2500°C.

Results and discussion

Tensile strength with measurement temperature

Fig. 2 shows the variation in tensile strength with processing HTT of C/C composites after measurement at room temperature and at different high temperatures. At pre-carbonized stage (HTT 750 °C) tensile strength at room temperature is in the order of 165-170 MPa and on further heat treatment (on 1000 °C) strength decreases and it is 125-130 MPa. The decrease in strength is related to increase in stress concentration centre due to the shrinkage of carbon matrix because up to 750°C shrinkage of the matrix is not completed [**28**]. Also the decrease in strength is due to the conversion of the matrix from relatively compliant polymer to a low strain to failure carbon, since the matrix is well bonded and brittle in nature after heat treatment to 1000 °C, a flaw or crack initiated in the matrix can propagate through the fibers, resulting more or less catastrophic failure because of cracks are highly concentrated by stresses [**29**]. With increasing the HTT up to 1500 °C strength increases and it is maximum at 1500 °C. The maximum value of strength and strain at 1500 °C is might be due to the both fiber and carbon matrix experience same temperature in composites. As a result composites fracture at strain level at which both fiber and matrix reaches the maximum obtainable stress and strain [**30**]. After 1500 °C, with increasing HTT, tensile strength slightly decreases up to HTT 2500 °C. Degradation of tensile strength after 1500 °C is related to degradation of fiber properties in composites because the fibers used are only heat treated to 1500 °C. Above this temperature, fiber morphology changes and stresses acting on fiber-matrix interface damages fibers in the composite. It is well known that, high strength carbon fiber makes strong interaction with the phenolic resin derived carbon matrix [**31**]. In this case during tensile test, 0° oriented fiber under tensile stress and fibers transversely under compressive stress. Because of strong fiber-matrix interaction with increasing tensile stresses, transversely bonded fibers are under stretch which resulted in to the damages of fibers. As shown in **Fig. 3**, in 1500 °C heat treated composites, fiber morphology does not change only change is observed in the matrix phase. Above 1500 °C, matrix as well as the fiber morphology changes in the composites. The fiber cross section is distorted and fiber diameter is also decreasing as a consequence degradation of fiber properties [**32**]. This phenomenon increases further with increase in HTT.

The tensile strength at high temperature is higher than the room temperature strength and with increasing the measurement temperature, tensile strength increases progressively up to 2000 °C and above 2000 °C it decreases. The value of tensile strength is 195, 135, 235, 262 and 164 MPa for measurement temperature 750, 1000, 1500, 2000 and 2500 °C which is higher by 20, 6, 14 and 30% and lower by 20% at 2500 °C than that of the room temperature tensile strength. From the measurement temperature 750 °C the strength increases moderately up to 1500 °C. After measurement temperature 1500 °C, strength increase suddenly at measurement temperature 2000 °C and thereafter it decreases in the same manner at measurement temperature 2500 °C.

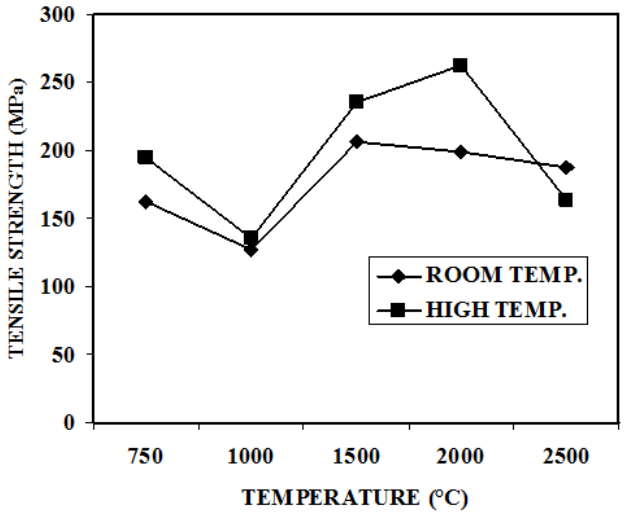

Fig. 2. Variation in tensile strength with processing HTT after measurement at room temperature and high temperatures.

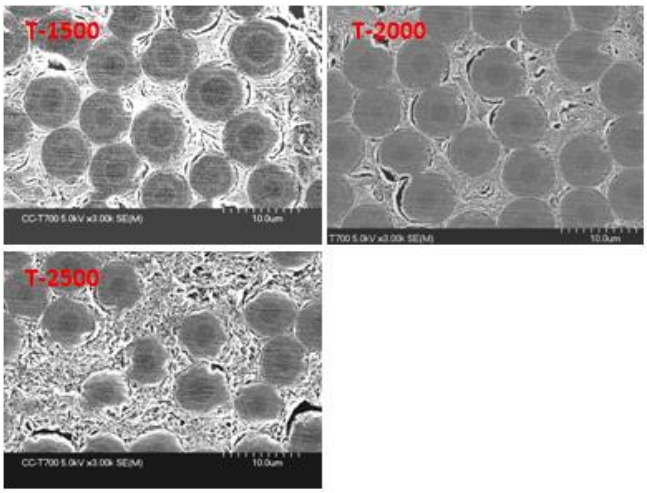

Fig. 3. SEM micrographs of C/C composites after HTT 1500, 2000 and 2500 °C.

The possible reason in the enhancement of tensile strength during high temperature measurement are discussed as below:

i. Due to the thermal mismatch in the coefficient of thermal expansion (CTE) between the fibers (fiber axis and transverse direction of fiber axis), between the fiber and matrix, thermal stresses induced in C/C composites at the end of cooling from high temperature processing. The CTE of the PAN based T700 carbon fiber -0.7×10^{-6}/K

(parallel to fiber axis) and of matrix ~ 3.0 × 10^{-6}/K [**3**], due to the large different between matrix CTE and axial CTE of the fiber, tensile stresses will be exerted on fiber-matrix interface and in matrix. This resulted in to the thermal stress cracks and annular gaps between the fiber and matrix in composite. As explain above, in these composites fiber-matrix interactions are strong even though due to the thermal contraction between fiber and matrix, some annular gaps between fiber and matrix observed. Also due to the shrinkage of matrix during heat treatment and CTE mismatch within the matrix phase, shrinkage crack and thermal stress cracks are in the matrix phase. The thermal stress cracks and annular gaps between the fiber and matrix closed up due to release of thermal stresses at high temperature heating during high temperature tensile properties measurement [**33**]. This resulted into the improvement in the fiber- matrix interactions [**34**]. The properties of matrix in the composites are also enhanced due to the release of thermal stresses from the matrix as well. As a consequence improves the ability of matrix to distribute applied stresses more evenly during high temperature measurement. This resulted into both the fibers and matrix fracture at a maximum obtainable stresses in one plane.

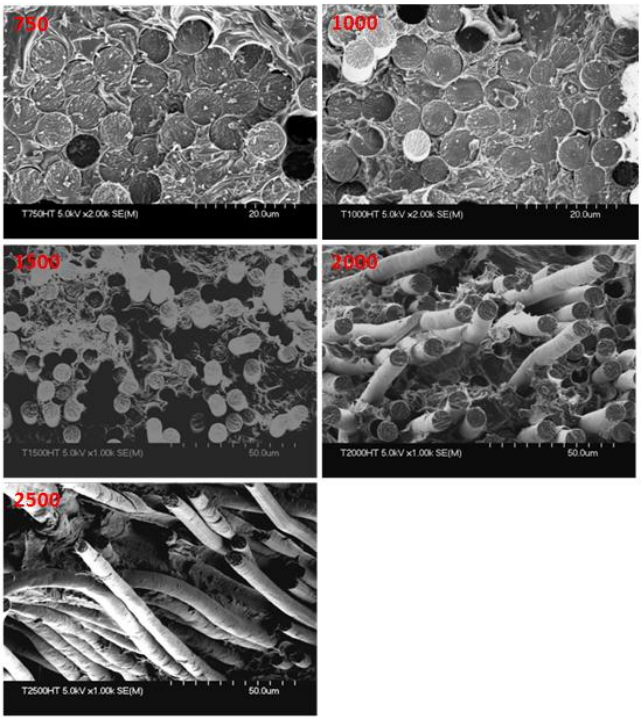

Fig. 4. Fracture faces of C/C composites after high temperature tensile test at 750, 1000, 1500, 2000 and 2500 °C.

ii. According to the conventional theory of composites, required strong bond strength between fiber and matrix to permit efficient stress transfer between the two components of the composites. When stress is applied, e.g. in tension, the matrix can stretch and support the fibers such that their full mechanical properties can be realized. But in brittle matrix composites, due to the strong fiber-matrix interactions, the whole composites fail in a catastrophically brittle manner and the fibers will not achieve their full reinforcing potential. Therefore, tensile strength of C/C composites is improved by decreasing the bonding strength along the fiber-matrix interface. In the present study as discussed above, tensile strength increases up to measurement temperature 2000 °C. As shown in **Fig. 4**, fracture surfaces of composites after high temperature tensile test at different temperatures, it is clearly evident both the fiber and matrix is in close contact, also matrix adhered on fiber surface and fracture in one plane up to 1500 °C. This implies that during the high temperature test, fiber-matrix interaction are going to stronger than the fiber-matrix interactions at room temperature test which yielding low tensile elongation shown in **Fig. 5**. The improvements in the fiber-matrix interactions during high temperature measurements are responsible in the enhancement of strength. It is also observed from the SEM micrographs, after high temperature measurement, annular gaps between the fibers and matrix is due to the restoration of thermal stresses again on cooling from measurement temperature.

iii. The high temperature tensile strength of carbon fibers is carried out by only few researchers [**22, 35-37**]. The tensile strength of PAN based carbon fibers at room temperature deceases after HTT more than 1500 °C [**3**]. But on the other hand, tensile strength of carbon fiber during high temperature measurement slightly increases from its room temperature tensile strength up to measurement temperature 2000 °C [**37**]. On contrary, the fracture strain of the fibers increases with increasing the test temperature and young's modulus decreases with slight increase in tensile strength. This implies that fibers are tougher and to have high fracture strain at elevated temperature. During the tensile test at temperature 2000 °C, 0° oriented fibers are under stretching and as a result graphitic layer developed and aligned parallel to fiber axis and also the internal stress relaxation occurred which enhanced the tensile strength. This similar phenomenon expected to occur in matrix. The fracture strain enhancement is one of the parameter in increasing the tensile strength of C/C composites at measurement temperature 2000 °C is due to the creep deformation.

iv. But on the other hand, at high temperature 2500 °C tensile strength is decrease. During HTT the graphitization of carbon material initiated at above 2000°C [**38**] and with increasing the temperature degree of graphitization increases and as a result relaxation of stresses [**18**]. In the present case tensile strength at test temperature 2500 °C decreases. This decrease is because of (a) the fibers used in composites are heat treated at 1500 °C only and as cause in-situ fiber strength degradation in composites during HTT of composites. (b) The fibers in composites under tensile stress during high temperature test at 2500 °C. These fiber elongated under applied stress parallel to fiber direction and transversely fibers bonded with matrix strongly as discussed above. With increasing stress transversely bonded fibers are under stretch as a result damage of fibers and degradation of composite properties. (c) Under tensile stress at high temperature elongation of fibers resulted into decrease in fiber diameter and decrease in the strength of fibers as well. As a result decreases in interlayer spacing of these composites due to orientation of graphitic layer parallel to

fiber axis (**Fig. 7**). It is also reported; tensile strength of PAN based carbon fibers above measurement temperature 2000°C decreases [**37**]. As seen from the fracture surface of composites after high temperature measurement 2500 °C, composites fracture by creep deformation but tensile strength decreases.

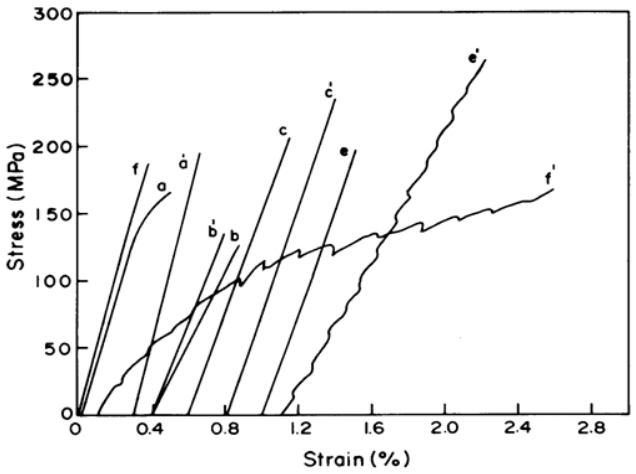

Fig. 5. Stress-strain curve of different heat treated composite tested at room (a) 750 °C, (b) 1000 °C, (c) 1500 °C, (d) 2000 °C, (e) 2500 °C and after high temperature test at temperature (a′) 750 °C, (b′) 1000 °C, (c′) 1500 °C, (d′) 2000 °C, (e′) 2500 °C.

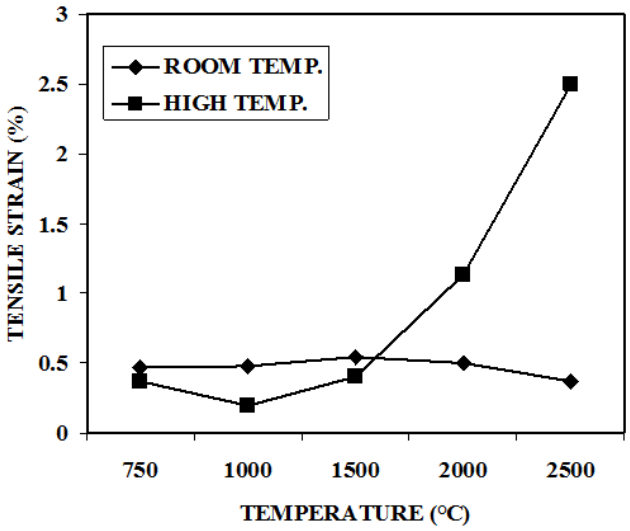

Fig. 6. Variation in tensile strain with processing HTT after measurement at room temperature and high temperatures.

Fracture behavior with measurement temperature

Fig. 5 shows variation in tensile strain with processing HTT of C/C composites after measurement at room temperature and high temperatures. It is observed that, tensile strain varies as like the tensile strength during room temperature test of composites. This indicates that in-situ retention or degradation of fibers properties also influence the failure strain of ultimate composites. The fiber property more or less does not change up to its processing temperature (fiber HTT 1500 °C) in composites. Some change occurs is due to the fiber-matrix interactions and stresses acting on interface. After HTT 750 °C tensile strain decreases, this is related to matrix shrinkage and decrease in the density of composites. Thereafter maximum strain at on 1500 °C and after that strain continuously decreases up to 2500 °C. The decrease is related to in-situ degradation of fiber properties and change in microstructure of composites. In these composite due strong fiber-matrix interaction both matrix and the fiber undergoes through stress graphitization process which resulted into decreases in interlayer spacing and increases in modulus of composites [**Fig. 6** and **7**]. On the other hand, during high temperature tensile test, trend of tensile strain is different, tensile strain decreases up to 1000 °C and above 1000 °C it increases up to 2500 °C continuously. But strain after high temperature test at 1500 °C is less than that of room temperature tensile strain of composites heat treated at same temperature. Decrease in strain is related to change in mode of fracture behavior due to change in bonding nature between fiber and matrix and laminates to laminates (between 0° and 90°oriented fibers) as discussed above.

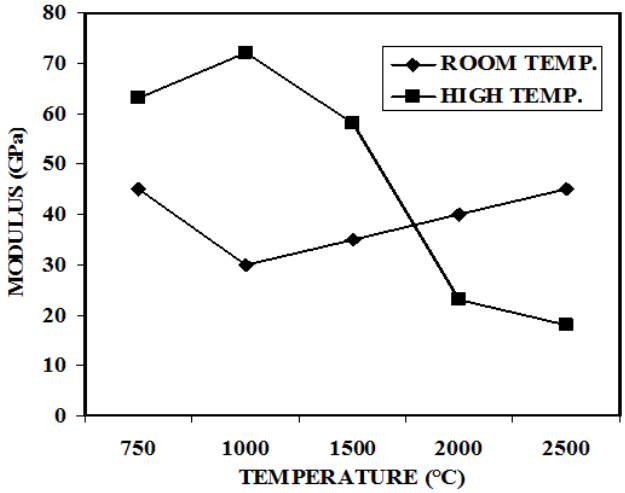

Fig. 7. Variation in tensile modulus with processing HTT after measurement at room temperature and high temperatures.

As a result during fracture of composites, cracks required higher stress to propagate straight by decreasing the strain because these cracks are not saturated by the stresses (cracks are going to closed during high temperature measurement). This is evidence from the SEM micrographs shown in **Fig. 4**. In case of composites tested at high temperature 750, 1000 and 1500 °C, the composites fracture in one plane as a result flat smooth fracture surface where the fiber and matrix well bonded. However, above test temperature 1500 °C, the tensile strain suddenly increases. The increase is related to elongation of fibers from matrix phase in composites during high temperature test. During high temperature test, the fiber morphology changes as well as fiber diameter decreases and due to the graphitization, fiber-matrix interactions are going to weekend to certain extends. Therefore, during high temperature test, elongation takes place at applied stress. This resulted in to the increases in strain with increasing the test temperature. In **Fig. 4**, fracture surface of composites tested at 2000 °C, composites fracture by elongation of fibers from matrix i.e. with applied stress at high temperature strain also increases nonlinearly (fiber pullout).

Further with increasing the test temperature, extent of fiber elongation increases (**Fig. 4**, fracture surface of 2500 °C tested composites) and as a result increases in strain with applied stress. On contrary crystalline strain between graphitic layers decreases due to the stress graphitization of fibers and the matrix under applied stress during high temperature test and as result decreases in interlayer spacing. This phenomenon is higher with increasing the test temperature (**Fig. 7**). Also the composites heat treated twice on same temperature (2000 °C) no significant change in interlayer spacing of composites observed (in **Fig. 7** as HTT 2nd).

Tensile modulus with measurement temperatures

Fig. 8 shows the variation in tensile modulus with processing HTT of C/C composites after measurement at room temperature and at high temperatures. Initially at room temperature modulus is in the order of 45-50 GPa of 750 °C heat treated composites and it decreases further on heat treatment at 1000 °C. Above 1000 °C the modulus increases continuously up to 2500 °C and ultimate value is same as that of 750 °C heat treated composites. The increase in modulus is related to the change in microstructure of composite. In other way, increase in HTT of composites, decrease in interlayer spacing and tensile strain above 1500 °C. The strain decreases when the material behavior changes from hard to soft by graphitization process.

However, modulus of composites after high temperature tensile measurement is higher, which is up to the test temperature 1700 °C after that modulus decreases and ultimate value is less than that of room temperature test value of composites heat treated at same temperature. The increase in modulus is related to change in the fracture behavior of composites at high temperature as discussed above. Above measurement temperature 1500 °C, value of tensile modulus is less than that of room temperature test value and modulus is decrease suddenly after 1500 °C. This decrease in modulus is related to increase in strain to failure of composites. This is due to the composite fracture by creep deformation phenomena. As observed in earlier study [**39**], high strength carbon fiber phenolic resin derived carbon matrix composites gives the columnar type microstructure where some region of fibers are still bonded strongly with carbon matrix after heat treatment more than 2000 °C. Therefore, during high temperature tensile test at applied stress, instead of increasing the stress -strain linearly, it increases nonlinearly. At applied stress up to certain limit, strain also increases linearly after that at constant strain, stress increases which required to break the fiber-matrix bond and this process is continues till the ultimate fracture. Further on high temperature test at 2500 °C, composite fracture strain increases with decrease in fracture stress and modulus is decrease. This suggest that above 2000 °C composites fracture purely by creep deformation phenomena and both strength and modulus decreases. However, composite fracture by creep deformation phenomena resulted in to the enhancement in the strength of composites [**22**]. But in the present investigation both the strength and modulus decreases because of fiber used are not heat treated more than 1500 °C.

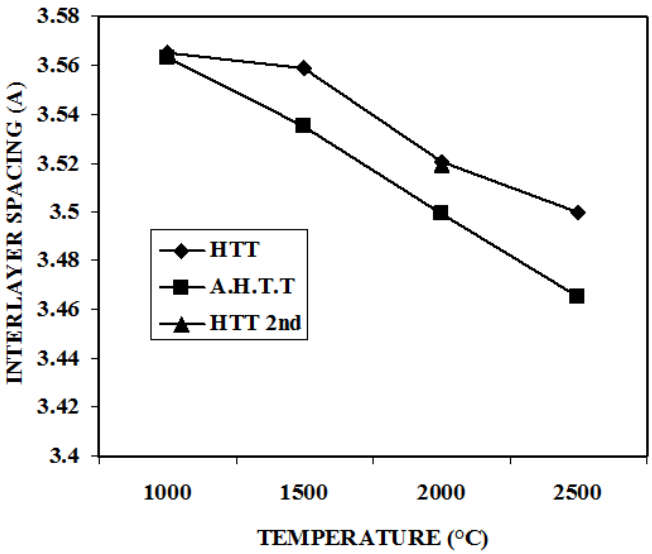

Fig. 8. Variation in interlayer spacing of C/C composites with processing HTT and after high temperatures tensile (A.H.T.T.) measurement.

Conclusion

It is observed that the maximum value of tensile strength at room temperature of C/C composite heat treated at 1500 °C. This suggest that the maximum obtainable strength of composites depends upon the HTT of fibers, above the HTT of fibers, degradation of composites properties is due to in-situ degradation of fiber properties in composite. On the other hand, at high temperature, tensile strength is higher and it increases progressively with increasing the test temperature up to 2000 °C, after that tensile strength decreases. The governing factors in the enhancement of tensile strength at high temperature are

1. The improvement in fiber-matrix interaction due to release of thermally induced stresses with increasing measurement temperature up to 1500 °C.
2. Relaxation of thermally induced stresses improves the properties of matrix and ability of matrix to distribute stresses more evenly.
3. Above 1500 °C increases in strength is due to enhancement of tensile strength of carbon fibers during measurement temperature at 2000 °C.
4. Creep deformation in the C/C composites observed at above high temperature measurement 2000 °C is also possible reason in the enhancement of strength at 2000 °C.
5. Decreases in high temperature strength of C/C composites at 2500 °C are due to degradation of fiber properties.

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