

# The Influence of the Addition of Inorganic Compounds on the Matrix Microstructure and Shear Strength of C/C Composites

E.Yasuda, Y.Hotta, S.M.Park, J.Ariyoshi, T.Akatsu and Y.Tanabe

Research Laboratory of Engineering Materials,  
Tokyo Institute of Technology

## INTRODUCTION

The carbon fiber/carbon composites (C/Cs) have potential applications as structural materials for space and aeronautic industries because of light weight, heat resistance, excellent mechanical property etc. The C/Cs usually show an anisotropy in their strength, which is caused by the anisotropic orientation of carbon fiber(CF) and graphite nature. Thereby, C/Cs are designed and used to realize high strength considering its anisotropy and the condition of applied stress. Authors have already reported that the strength of UD-C/C extremely decreased by off-axial stress and that was controlled by shear fracture mode. Hence we have to increase inter laminar shear strength of UD-C/C. In this study, we manufactured C/Cs changing the process that is considered to give some influence on the shear strength, such as heat treatment temperature (HTT)[1][2], inorganic compounds (graphite, SiC and TiO<sub>2</sub> powder) addition to the matrix for the purpose of disturbing the orientation of graphite crystallites in the matrix. According to the results, we discussed the relations between the shear strength and microstructural feature of C/Cs such as the matrix texture.

## EXPERIMENTAL

The C/Cs were made from PAN derived CF, furan resin and inorganic compounds with the following processes. At first, we dispersed graphite powder (GP, mean dia;1.0 $\mu$ m), SiC (mean dia;0.27 $\mu$ m) or TiO<sub>2</sub> (mean dia;0.57 $\mu$ m) in furan resin by ball milling so as 5 weight percentages (concerning GP; 1% too) of inorganic compounds against hardened furan resin. CFRP was prepared by filament winding method with the furan resin containing inorganic particles. The CFRP was carbonized through the heat treatment at 1000°C for 2 hours (concerning GP;5 hour) in an argon gas flowing atmosphere followed by the heat treatment at 1300°C-2800°C in argon for 0.5 hour.

The shear strength of C/Cs was measured by compressive test with the double notched test pieces (2mmx11mmx50mm). The crosshead speed was 0.5mm/min. The shear strength was calculated from the maximum stress divided by the fractured area of the specimen.

In this study, we estimated the Lc and d<sub>002</sub> from the XRD measurement based on the JSPS method applying

Cu-K $\alpha$  and separating the (002) profile using the function of PersonVII with the method of least squares.

## RESULTS and DISCUSSION

### 1. The effect of graphite powder addition

Figure 1 shows the shear strength of C/C as a function of the amount of GP addition. The shear strength increased with the increase of GP addition until 1-2 weight percentage. When the GP amount was over 1-2 weight percentage, the shear strength slightly decreased. This behavior can be explained by the following reasons. The initial increase is caused by disorder of graphitic structural unit in C/C matrix. The decrease at higher amount of GP depends on the introduction of defect as GP itself.

### 2. The effect of SiC or TiO<sub>2</sub> powder addition

Figure 2 shows the shear strength of C/C with and without additives as a function of HTT. There is no obvious difference among them heat treated at below 2000°C. In the case of SiC powder addition, the shear strength was low compared to C/C without additives above 2500°C. Also, the shear strength of TiO<sub>2</sub> powder addition to C/C diminished above 2300°C. Lc of each C/Cs is plotted in figure 3 as a function of HTT. Lc of SiC-powder-added-C/C increased above 2500°C and TiO<sub>2</sub>-powder-added-C/C indicated the largest value of Lc above 2300°C. The increase of Lc with SiC or TiO<sub>2</sub> addition was caused by catalytic graphitization. In addition, we observed the macroscopic disordered structure of graphitic crystallite in the matrix due to inorganic powder addition with SEM.

According to the preliminary experiment of highly oriented pyrolytic graphite (HOPG, figure 2), well-graphitized graphitic structural unit in C/C matrix results lower shear strength than poorly graphitized matrix. Therefore, we have to consider that the decrement of the shear strength of SiC or TiO<sub>2</sub> powder added C/Cs with the increase of HTT was brought by the development of graphite crystallite in the matrix.

In the case of inorganic powder added C/Cs, the progress of graphitization of C/C matrix offered much more significant effect on the shear strength than the disorder of graphitic structural unit.

On the other hand, there is a discrepancy between on the shear strength of without powder addition in figure 1 and 2. It will depend on the curing process, but it is now under inspection.

### CONCLUSIONS

The important factor to obtain C/C with high shear strength is as follows;

1. Suppress the progress of matrix graphitization,
2. Disturb the orientation of graphitic structural unit in the matrix.

It was also found that factor 1 was predominate over factor 2.

### REFERENCES

- [1] E.Fitzer, K.-H.Huchtner and R.Weiss, "Chemical interactions between the carbon fiber surface and epoxy resins," *Carbon*, **18**, 389-393 (1980).
- [2] Y.Hishiyama, M.Inagaki, S.Kimura and S.Yamada, "Graphitization of carbon fiber/grassy carbon composites," *Carbon*, **12**, 249-258 (1974).
- [3]. E.Yasuda and Y.Tanabe, "Matrix modification by graphite powder additives in carbon fiber/carbon composite with thermosetting resin precursor as a matrix," *carbon*, **26**, 225-227 (1988).
- [4] J.Ariyoshi, T.Akatsu, Y.Tanabe and E.Yasuda, The 21th Annual Meeting of the Carbon Society of Japan, 152-153 (1994).

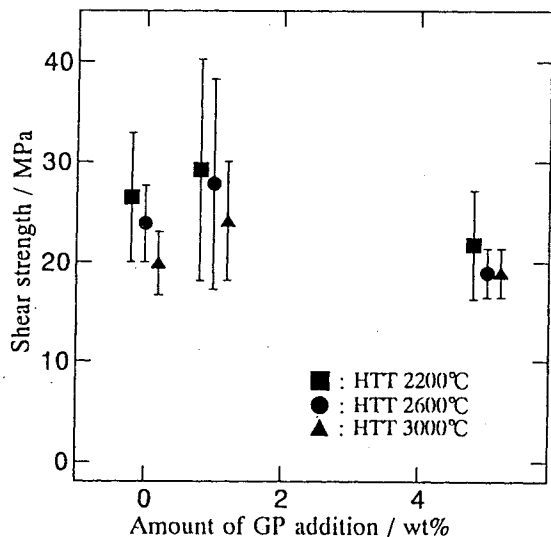


Fig.1 Shear strength of C/C as a function of the amount of GP addition

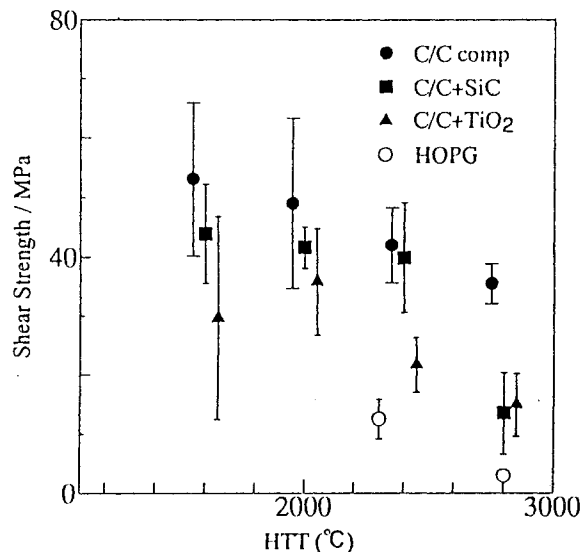


Fig2 Shear Strength of C/C with and without additives as a function of HTT

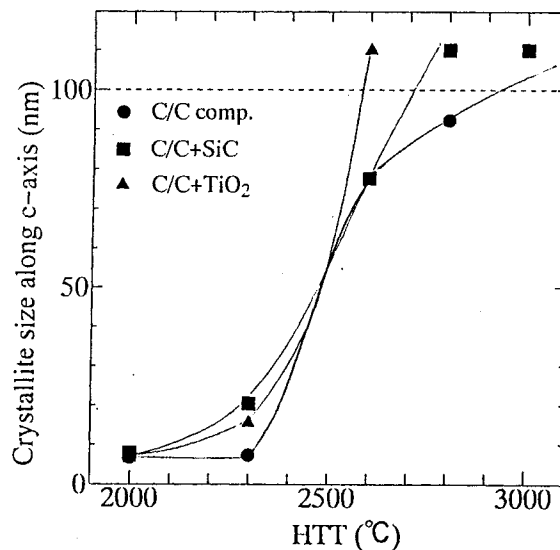


Fig3 Change in crystallite size(Lc) of C/C composite with and without additives as a function of HTT