HIP-TREATMENT OF CARBON/CARBON COMPOSITE

Toshinori HOSHINO,Masashi TAKADA and Shigehiko YAMADA
Department of Materials,
Teikyo University of Science & Technology
Uenohara,Yamanashi 409-01 JAPAN

Abstract

HIP(Hot Isostatic Press)-treatment of normally hot-pressed C/C composite upto 600℃ under 20MPa in accordance with Okura et al's process, being well known as a very rapid preparing method of the composite.The operating temperaure and pressure covering 1000-2000℃ and 10-200MPa,respectively.Changes in bulk density,electrical resistivity,as well as flexural strength and modulus,were determined as functions of temperature and pressure.In parallel,XRD was inverstigated to elucidate the changes.Additionally,the same starting specimens were graphitized at 2700℃ under normal pressure to compare the graphitizability with that of HIP-ed ones above mentioned.

1.Introduction

Development of C/C composite in industrical fields has been accelerated by the success in rapid processing originated by Okura et al[1].However,repeated impregnation followed by carbonization, taking as much time as the regular process,is needed for its densification to improve the mechanical properties.To solve the problem,HIP-treatment was carried out,first on the specimens carbonized above 1100℃ under normal pressure to avoid the danger due to its volatile materials.In this article,just formed ones prior to the carbonization are treated,so that the effect of HIP can be more exhibited for the purpose.

2.Experimcnt

1D(uni-directional)- and 2D(two directional)-specimens to be HIPed,were prepared in Across Co.,Ltd.,by above menthioned process,employing resin coated high strength carbon fibers,coke powder and bulk meso-phase powder,having a size of 5(1D) and 6mm(2D) in diameter and 40-45mm in length.Hot pressing tempreature was 600℃ and pressure 20MPa.HIP apparatus was 'Dr.HIP' prepared by Kobe Steel Co.,Ltd.The range of operating temperature by HIP was 1000-2000℃ and pressure 10-200MPa.Under normal pressure, HIP is hard to treat in reality,and therefore 10MPa was used as the most close to the normal pressure for comparison.

Density was determined by size and weight,and electrical resistivity was obtained by holding the specimen between copper meshes,using a digital mulutimeter,TR6845.Bending strength and modulus were determined by a three point method.

Graphitization under normal pressure for comparison was carried out at 2700℃ in an industrial furnace by the courtesy of Tokai Carbon Co.,Ltd.

Parameters about XRD are the same as defind in JSPS-117, and Flanklin's p-value is calculated by

$$1-p^2=(3.440-c_0/2)/0.086$$

.3.Result and discussion

3.1 Chage in strength and modulus

According to the result as shown in Figs.1 and 2,the increase of bending strength was observed only in the case of 2000 ℃ treatment of 1D specimens,whereas modulus was improved only by 1000 and 1500 ℃ treatment of 1D. As a whole,the HIP effect was not so remarkable.

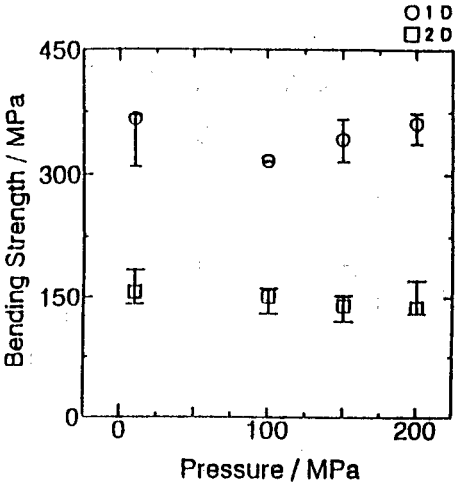

Fig.1 Bending Strength as a function of pressure of 1D and 2D composites, treated at 2000℃ for 1h

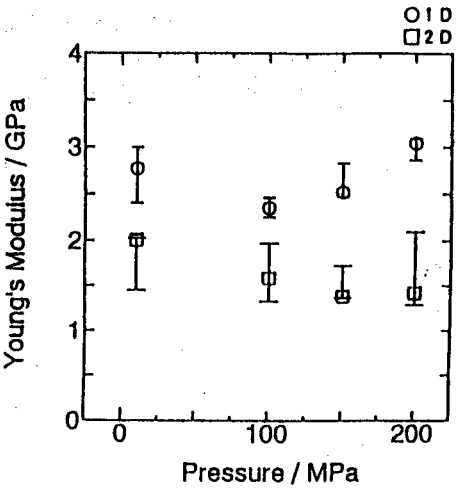

Fig.2 Young's Modulus as a function of Pressure of 1D and 2D composites, treated at 2000℃ for 1h

3.2 Change in density and electrical resistivity

As shown in Fig.3,a slight increase of the relative density in the case of 2000 ℃,was observed,while the increase in the cases of 1000 and 1500 ℃ was less.In comparison with such a simple increase,resistivity change was not so simple as illustrated in Fig.4 where HTT is 2000 ℃, similarly to the result on 1000 and 1500 ℃ treatment.The reason why the decrease under a low pressure (10MPa) in all cases,was considerably more than the others,under 100,150 and 200MPa,is not sure,almost no effect of HIP on the resistivity change was also found,suggesting that there might be almost no development of graphite crystallite.

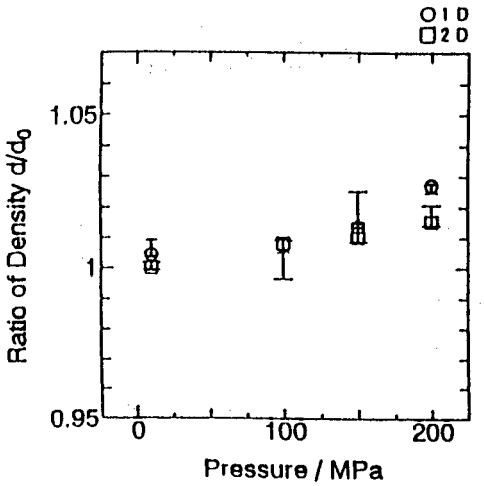

Fig.3 Ratio of Density as a function of Pressure of 1D and 2D composites, treated at 2000℃ for 1h

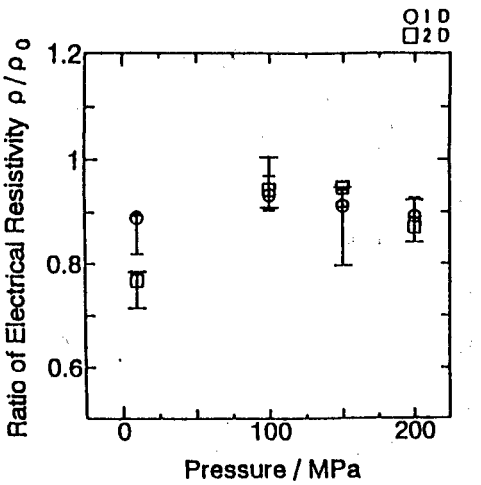

Fig.4 Ratio of Electrical Resistivity as a function of Pressure of 1D and 2D composites, treated at 2000℃ for 1h

3.3 Comparison of graphitization

Changes in c_0(002) and Franklin's p-value of 1D and 2D specimuns are illustrated in Fig.5 and 6.,where the treatment-1 means prior to HIP,-2 means HIP whose condition is as described in the figures,and -3 is graphitized specimens at the same time(2700 ℃).There is such a clear diffrence between 1D and 2D in every change,based upon the bigger effect of fibers in 2D than that in 1D;however,the graphitized values are almost all the same.Similar tendency is observed in Lc change shown in Fig.7.As shown in Figs.8 and 9,the changes in 2D-specimens are very small also in these cases,differing from the casesin 1D-specimens.

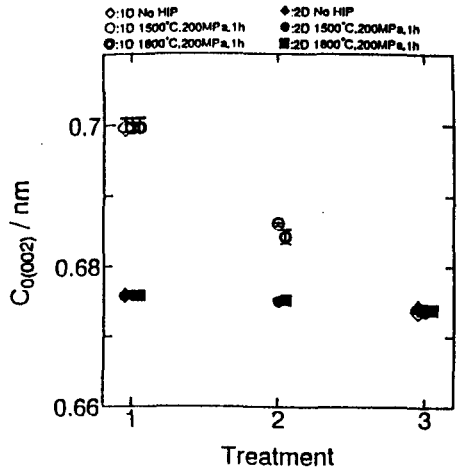

Fig.5 Change in $C_{0(002)}$

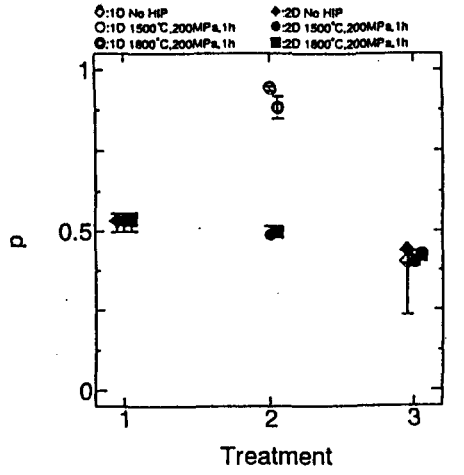

Fig.6 Change in Franklin's p-value

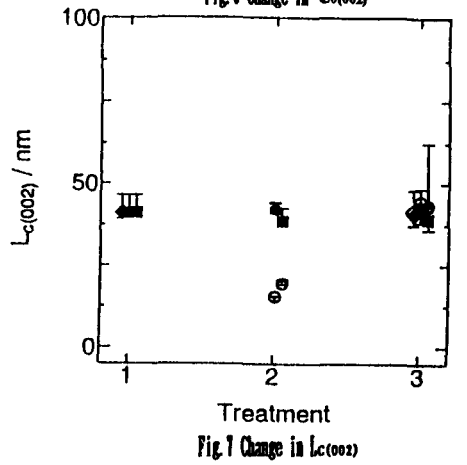

Fig.7 Change in $L_{c(002)}$

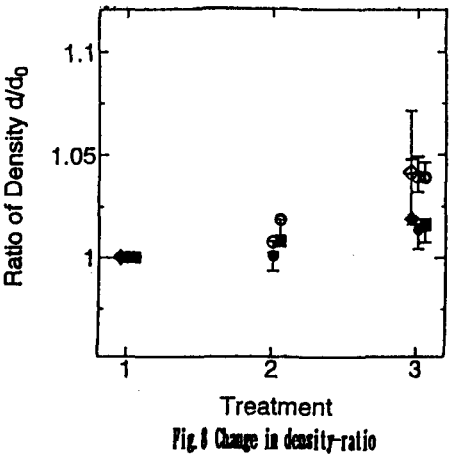

Fig.8 Change in density-ratio

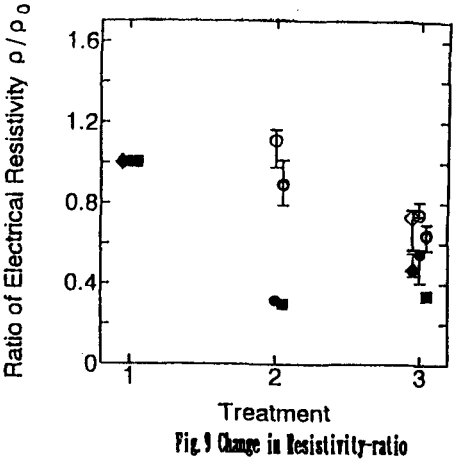

Fig.9 Change in Resistivity-ratio

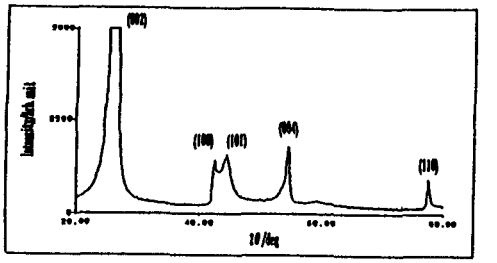

Fig.10 Example XRD profiles of graphitized 1D

Fig.10 shows an example XRD profiles of graphitized 1D,covering a wide angle-range.All 3 sorts of 1D,-1,-2 and -3 treated,showed the same profiles of (002),(100),(101),(004) and (110),having the same as intensity,as anticipated in Fig.5-7.The profiles of 2D specimens of treatment-1 showed (100),(101),(004) and (110),while those of 1D prior to HIP showed only (002) and (004) and the 1D-specimen after HIP (before graphitization) showed no (101)-profile,differing from corresponding 2D-ones.Although the intensity of all 5 profiles of 2D-specimens prior to graphitization were slightly smaller than those after graphitization,every intensity and p-value of graphitized 3 sorts of 1D and also 2D specimens are approximately the same each other.It may suggest that the final graphitized crystallite structure of all sorts is similar,being independent of HIP route,as well as of texture. It seems that HIP-treatment did not play any role for graphitization.

In order to investigate the effect of HIP,any other property such as tribological one,should be determined,which is now being carried on.

4.Conclusion

The effect of HIP treatment of a C/C composite,being not completely carbonized,on the strength and modulus was investigated as functions of operating temperature and pressure.As a result,almost no improvment was found,except a slight densification.Concerning the effect on the effect was also hardly found out.Any other effect such as triboligical one should be considered.

Acknowledgment

The authors are indebted to Tokai Carbon Co.,Ltd,for the graphitization process.

Reference

1) Tongshik CHANG,Takano NAKAGAWA and Akimitsu OKURA,Report of the Institute of Industrial Science, The University of Tokyo, Vol.35 No.8(1991)

Received September 25, 1997
Accepted January 23, 1998