POSTER

CATALYTIC GRAPHITIZATION OF GLASSY CARBON MATRIX IN BORON-DOPED CARBON/CARBON COMPOSITES

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INTRODUCTION

Carbon/carbon composites, due to their superior properties at high temperature,have attracted considerable attention as ideal materials for aerospace, including rocket nozzles, nose tips of spaceshuttle . Nevertheless,in the manufacture of c/c, a few inherent problems are existing: such as long period, great energy consumption and high heat treatment temperature. Heat treatment temperature is concerned about not only energy consumption and cost, but also the residual stress and properties of materials. As a result, in this article, by addition of boron and its compounds, the reducing of graphitization temperature in c/c is especially researched,in the name of mechanisms, it's called catalytic graphitization[1,2].

The studying of reducing graphitization temperature can simplify equipments, save on energy source,lower residual stress, shorten graphitization time , cut down c/c's production cost. So it's important in use and practice.

EXPERIMENTAL

In this experiment, furfural acetone was used as binder, grain of furfural acetone resin carbon,boron catalyst and chopped PAN carbon fiber as additives, through molding, carbonation,impregnation,graphitization, the boron contained c/c composites were made. The extent of graphitization, G parameter, was tested by X-ray diffraction(XRD) . the formula is $G=(0.344-d_{002})/(0.344-0.354)$;where, 0.344nm is the interlayer spacing of amorphous carbon,0.3354nm is the interlayer spacing of ideal graphite crystal, d_{002} is the interlayer spacing measured by XRD. By studying the influence to G of graphitization temperature(1800,2100, 2500℃), catalyst varieties(B,B_4C,B_2O_3) , catalyst's amount(0,1.5,3,6wt%),the optimum process of catalytic graphitization was acquired. The state of boron in c/c was considered by XRD and x-ray photoelectron spectroscopy(XPS), the special catalytic graphitization mechanisms by boron were obtained.

RESULTS AND DISCUSSION

Table-1 is the extent of graphitization in this experiment.For the c/c without catalyst, the G is 71% at graphitization temperature of 2500℃, while the c/c with catalyst, the maximum G is 85% at 2100 ℃ . The graphitization temperature drops 400℃, but the G increases by 14%.Hence, the catalytic effects is obvious, it makes the non-graphitizing carbon---glassy carbon, graphitizing perfectly and rapidly.

In view of table-1, the optimum catalytic graphitization process can be obtained. According to the G, graphitization temperature is 2100 or 2000 ℃ ; catalyst variety is B_4C firstly, B secondly; as for catalyst's amount,it's clear from table- 1 that G increases at first,then G drops, as the amount increases; so we can determine that the good amount is about 3wt%. Consequently, the optimum catalytic graphitization process is: temperature--2100 or 2000℃, catalyst variety--B_4C, amount--about 3wt%.

The catalytic action for glassy carbon by boron was examined by XRD and XPS.In XRD,the diffraction degree is from 18° to 64° , the results only showed the three characteristic peaks of graphite, but no boron peaks. So there may be two situation:one is that boron evaporates completely, the other is that boron forms solid solution in c/c. When studyed by XPS,the boron's peak appeared, so we can conclude that boron in c/c, furthermore,boron is solid solution in c/c. It's reported that there were two kinds of solid solution: substitutional and interstitial solid solution,the former can lead to the decrease in interlayer spacing, while the latter increase[3] . It was also reported that the substitutional solid solution's amount is fixed at certain temperature[4]. When the amount of boron exceeded the fixed value, boron transferred from substitutional to interstitial place. This can explain the change of G when the catalyst's amount is increasing.

General speaking, glassy carbon is not graphitizing even when heated to 3000℃. But the G in table-1 indicates the glassy carbon is graphitized perfectly, this entirely due to the introduction of boron. Boron has the following features: strongly attracting electron and replacing carbon in the lattice of graphite. So boron can cut down bond between carbon atom by attracting electron; at the same time,boron takes the place of carbon atom to form the layer plane of graphite.Because of above reasons, glassy carbon is able to graphitize, as well, the processing of graphitization can be carried out perfectly and rapidly.By means of theory analysis and experiment demonstration,we can make the following catalytic graphitization mechanisms by boron[1] : (1) the strongly attracting electron can lead to the breaking of bond between carbon atom, which promotes the carbon atom's reconstruct in graphitization processing; (2) boron forms substitutional solid solution in c/c, replaces carbon atom in turbostratic structure,removes the defects in layer plane and interlayer, producing marked graphite crystal.(3)boron combines with carbon to form carbides, then carbides decomposes, forming a graphite crystal.

CONCLUSION

1) Catalytic graphitization, in reducing graphitization temperature of c/c, is practicable and remarkable.
2) In such catalytic graphitization process as that graphitization temperature is 2100℃; catalyst is B_4C;catalyst's amount is about 3.0wt%, the extent of graphitization is as high as 85%.
3) Boron promotes glassy carbon's graphitization by way of mechanisms of breaking bond by attracting electron and removal of defects by replacing carbon atom in graphite lattice and so forth.

REFERENCE

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Table-1 the extent of graphitization in boron- doped c/c

	0%	1.5%(B)	3.0%(B)	6.0%(B)	3%(B_4C)	3%(B_2O_3)
1800℃	/	/	54%	/	52%	57%
2100℃	/	/	82%	/	85%	76%
2500℃	71%	77%	84%	74%	80%	76%