PREPARATION AND RESEARCH OF TaC COATINGS ON THE SURFACE OF MESOPHASE PITCHCARBON FIBERS

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Introduction

Carbon fiber has been widely used as reinforcement materials of composites due to its excellent properties [1-2]. However, oxidation resistance of carbon fiber was poor. The significant weight losses of carbon fiber in the air occurred above 400°C, and resulting in lower fiber strength, decrease the mechanical properties of composites, and even led to material failure [3]. The research results showed, that the carbon fiber surface coated with SiC, TaC, HfC and ZrC coating can effectively improve the its oxidation resistance. Among those materials, TaC was a broad application prospects coating material due to its melting point up to 3880°C, high hardness, chemical stability, corrosion resistance, thermal shock and high temperature oxidation resistance. Coating process of TaC mainly include CVD, PVD, Sol-gel, cathode sputtering, and liquid precursor method, etc. [4-6]. In this paper, the sol - gel method for prepare TaC coating on mesophase pitch carbon fiber was researched.

Experimental

Tantalum pentachloride was dissolved in ethanol to prepare alcohol based sol, and added certain amount of DMAC to the sol, then aging 12 hours. Mesopahse pitch carbon fiber was coated by the Tantalum pentachloride sol, and dried in room temperature for 10 hours into dry gel. After high temperature treatment, the dry gel coating transferred into TaC coating. In order to improve the interface bonding strength between carbon fiber and TaC coating, the carbon fibers should be surface treated by electrochemical oxidation method. The tantalum sol-gel was analyzed by FT-IR and TaC coating on carbon fibers was analyzed by SEM, EDS, XRD, etc. The TG curve of coated carbon fiber and uncoated carbon fibers were compared.

Results and Discussion

In the tantalum sol preparation process, at first, $TaCl_5$ reaction with ethanol as below reaction equation to form alcohol based tantalum [7]. After adding dimethylacetamide, alcohol based tantalum and dimethylacetamide undergo sequestration transesterification reaction, resulting in increased crosslinking degree and viscosity, and formation of a complex chain or network structure.

$$TaCl5 + C_2H_5OH \rightarrow Ta\text{-}OC_2H_5 + HCl + 2\ Cl_2$$

Fig.1 was infrared spectrum of Ta sol (a), and gel (b). Fig. 2 was the XRD of coating treated at different temperature. The coating was Ta_2O_5 at 1000°C and transfer to TaC at 1400°C. When the treatment temperature increased to 1600°C, TaC the diffraction peaks become very sharp and almost no deviation compared with the standard spectra, indicating that the TaC crystalline tends to complete and high ordered. Figure 3 was 1600°C heat treatment coated fiber. It showed clearly that the surface of carbon fiber was coated by a gray layer. Combination of EDS and XRD results, that the layer was TaC coating. Fig. 4 showed that the coating layer consist two parts, one was TaC particles, the other one was pyrolysis carbon which connected TaC particles. There were also some micro holes which caused the gel shrinking during carbonization process. Fig. 5 was the backscatter SEM of coating fiber after heat treatment at 1600°C. It was showed that the carbon fiber cross section was uniform covered by TaC coating. The thickness of the layer was about 150nm. The two lines in fig. 6 were TGA curve of coated carbon fibers (b) and uncoated carbon fibers (a). As can been seen from the curve, uncoated carbon fibers (a) was easy oxidized. When temperature raised to 400°C, the fibers start weight loss, and weight lost obviously at 700°C till 840°C. While the coated fiber (b) were more stable, the weigh loss start at 650°C and up to 850°C the weight loss become obviously.

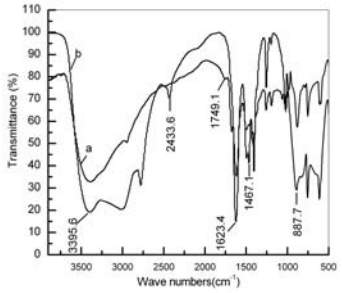

Fig. 1 FT-IR spectrum of precursor sol-gel of Ta

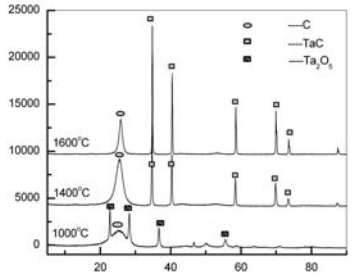

Fig.2 XRD patterns of the coating fibers after heat treatment at different temperature

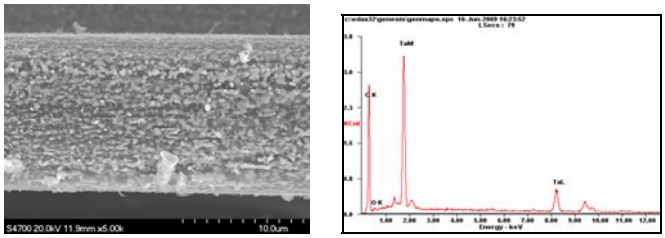

Fig. 3 SEM photos and EDS of coated fiber after heat treatment at 1600°C

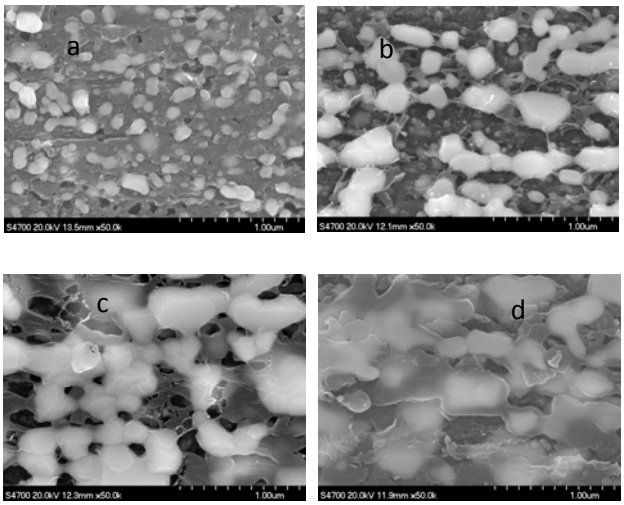

Fig. 4 SEM of coating fiber after heat treatment at 1600°C

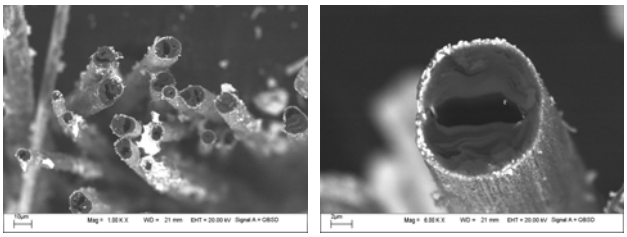

Fig. 5 Backscatter SEM of coating fiber after heat treatment at 1600°C

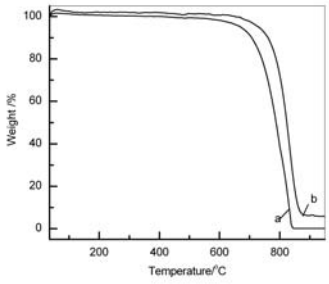

Fig.6 TGA of un-coated carbon fibers and TaC coated fibers

Conclusions

TaC coating on surface of mesophase pitch-based fibers was prepared by sol-gel method. The coating was compact and no obvious holes, the thickness was about 150nm. The coating is a continuous layer structure and consists of TaC grains and pyrolytic carbon. TaC coating can effectively block the invasion of oxygen, and improves the oxidation resistance of carbon fiber. Compared with uncoated carbon fibers, the initial oxidation temperature of coated increased 250°C, and weight loss obviously temperature up to 800°C.

References

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