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# CARBON-CARBON COMPOSITE MATERIALS (CCCM). INFILTRATION AND CARBONIZATION UNDER HIGH PRESSURE IN THE PRODUCTION OF CCCM. METHOD AND EQUIPMENT

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**Abstract**

*A method and equipment for the realization of infiltration and carbonization processes in the production of carbon-carbon composite materials (CCCM) have been suggested. The method is based on the use of a high pressure apparatus (HPA) of a "cylinder-piston" type and hydraulic press of the appropriate capacity. A CCCM blank with a rated amount of a pitch are inside a sealed thin-walled metal container which is placed in the HPA .The dry river sand is used as a pressure transmission medium.*

**Key words:** *carbon-carbon composite materials, high pressure apparatus, pitch, coke, carbon fiber*

## 1. INTRODUCTION

The development in the 1960s of high-strength and high-modulus carbon fibers made possible the creation of carbon-carbon composite materials (CCCM) whose strength properties and fracture toughness at high temperatures are much higher than those of other known structural materials. The production of the CCCM is a long-term multi-stage process [1]. The process involves the weaving of a multidirectional carbon fiber preform, the densification of the preform by its infiltration with liquid molten high-molecular organic substances and subsequent high-temperature treatments resulting in the formation of a carbon matrix. Once the product is infiltrated, it is subjected to carbonization by heating at a preset rate up to temperatures of 650-1100 $^{o}$C, which results in the formation of a carbon coke matrix. Then the obtained material is graphitized by heating up to temperatures of 2600-2800 $^{o}$C and holding at this temperature for long time, which leads to the transformation of the matrix structure from a coke to graphitic one. After the "infiltration-carbonization-graphitization" cycle is complete, the carbon-carbon composite material has a high open porosity and, correspondingly, low density of the order of 1.0 -1.2 g/cm$^3$. Performance characteristics of the CCCM to a greater extent depend on the density of the material. For example, it has been proven in practice that the density value of about 1.93 g/cm3 is required and sufficient for securing the appropriate erosion and oxidizing resistance of the working surfaces of a nozzle in jet engines for virtually all kinds of solid fuel. Therefore, to attain high density, the infiltration and carbonization are carried out under high pressures up to 100 MPa, which permits the infiltration of an organic binder into the smallest pores and affects chemical reactions of the decomposition of the binder so that the carbon coke yield is increased. In so doing, the infiltration and carbonization steps under high pressure, followed by graphitization, are repeated five-six times. Organic binders are usually pitches derived from coal tar or petroleum. In most known cases, an isostatic carbonization is carried out in an autoclave [2]. It is necessary to have very expensive large volume autoclaves to produce large CCCM blanks (about 500 mm in diameter and height) for making, for example, some parts of nozzles of advanced missile systems. An alternative, rather cheap method is proposed for the realization of the impregnation and carbonization under high pressure.

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## 2. METHOD AND EQUIPMENT

A "piston-cylinder" high pressure apparatus (HPA) is used to carry out infiltration and carbonization. Fig. 1 shows a cross-sectional diagram of the HPA.

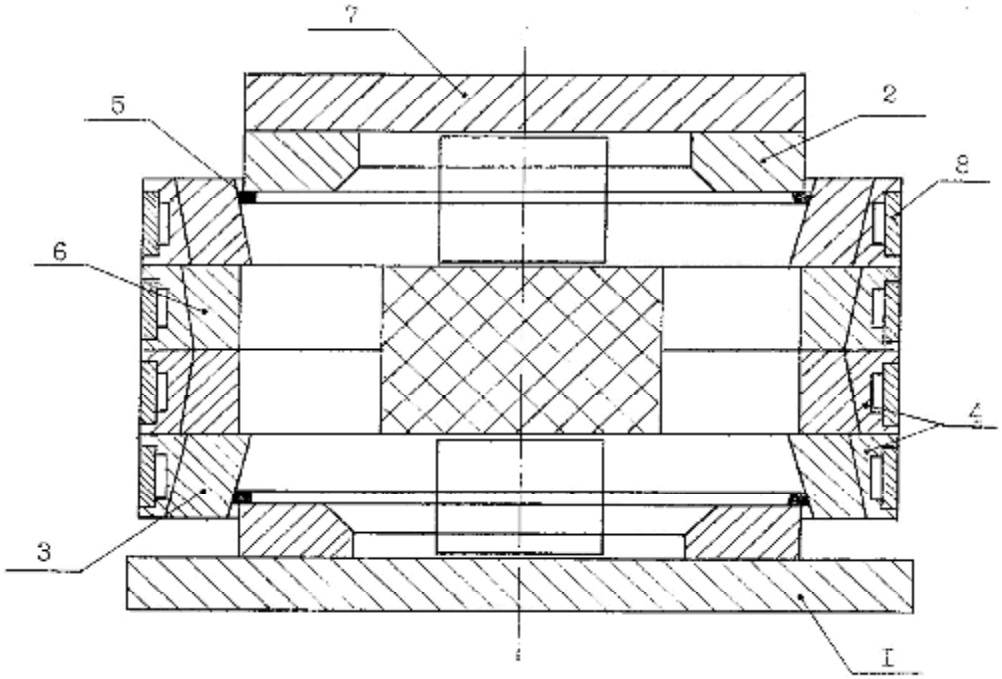



Fig.1. Cross-sectional diagram of the high pressure apparatus
(1) lower support plate, (2) piston, (3, 4, 6, 8) cylinder parts, (5) anti-extrusion ring,
(7) upper support plate



Fig.2. Overall view of the high pressure apparatus

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A cylinder of the HPA is about 1000 mm in inner diameter. The cylinder houses a sealed thin-walled metal container with a pitch-infiltrated CCCM blank and rated amount of a pitch. The remaining free space inside the container is filled with dried river sand that is used as a density-transmitting medium. To make the assembling and dismantling of the HPA convenient, two pistons (lover and upper) and a cylinder consisting of four composed rings are employed. A 0.1 mm thick band of stainless steel, having a sufficiently high specific resistance, is used as an electric heater. The band encloses the container with the CCCM blank; in doing so, it is separated from the container surface by a layer of an electric insulating material. The heater is powered through the pistons and current leads from a welding transformer used as a power supply. This kind of heater allows one to produce and maintain temperatures up to 800-900$^0$C throughout the entire carbonization process. Heating is controlled by four thermocouples. A series of the experiments has been carried out to determine a relation between the temperatures of the heater and CCCM blank. For this purpose, twenty one thermocouples were placed into the CCCM blank at different radii and heights. The arrangement of thermocouples is shown in Fig. 3.

Some results of the above experiments are presented in Figs. 4 and 5.

A high pressure infiltration-carbonization regime is shown in Fig. 7.

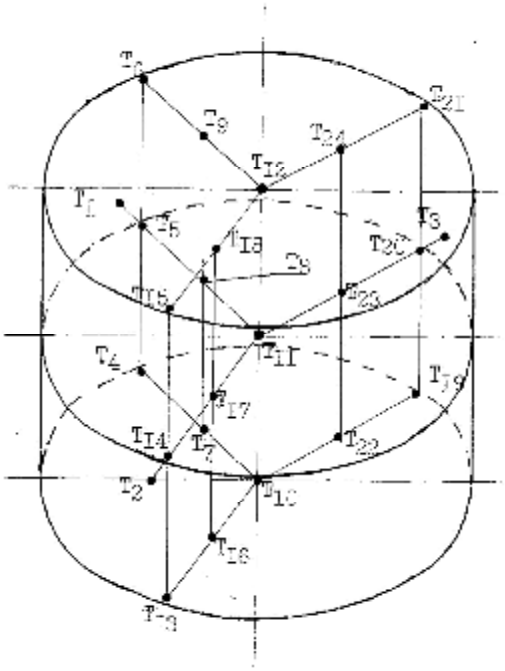


Fig. 3

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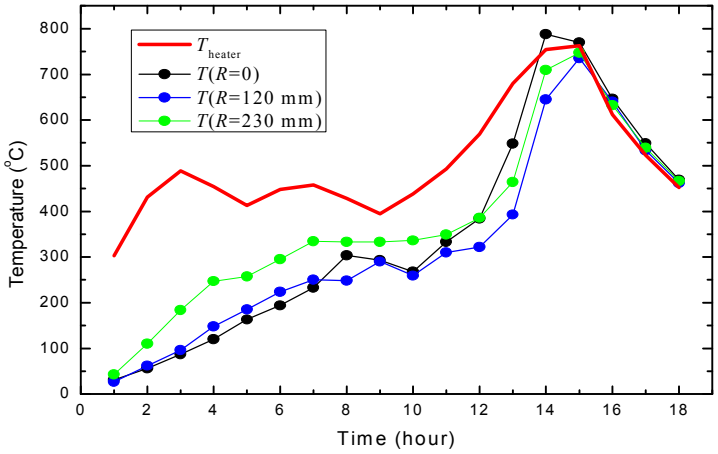



Fig. 4. Mean temperatures at different radii of the CCCM blank.

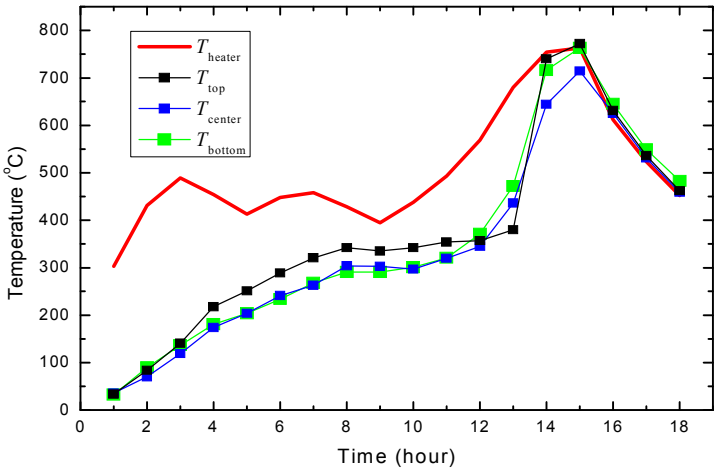



Fig. 5. Mean temperatures at different heights of the CCCM blank.

To apply an axial thrust on the pistons, the HPA is placed in a hydraulic press. It is necessary to have a hydraulic press of 5-7 thousand ton press force to create pressures required for the HPA of the above sizes. A general view of the setup consisting of a HPA and hydraulic press is shown in Fig. 6.

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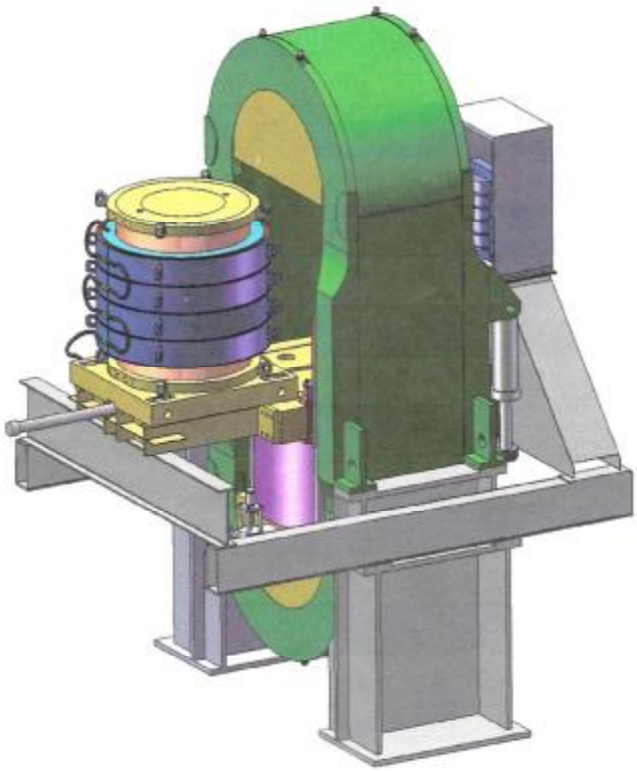


Fig. 6

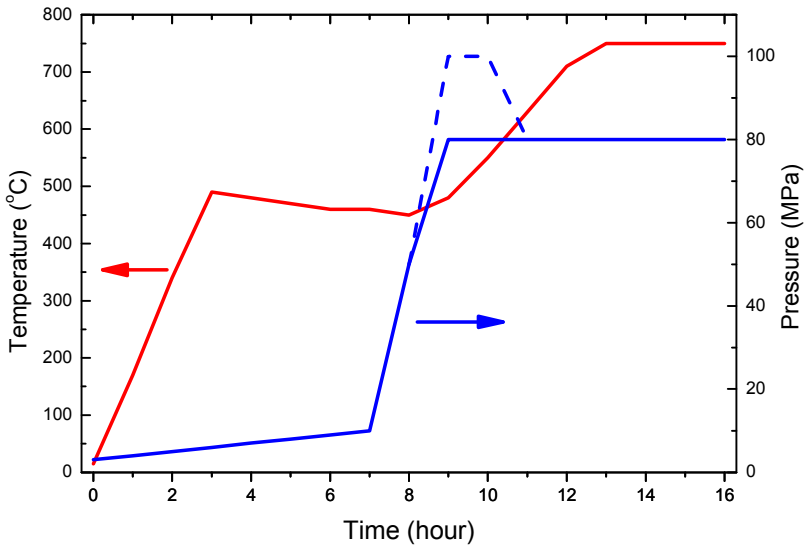



Fig. 7

The initial axial pressure applied to the HPA pistons is comparatively low so that a reliable electrical contact between the heater and current leads can be ensured. Then, at this low pressure, the heating is applied to melt the pitch. When the pitch melting stage is complete, the infiltration of the CCCM blank with a liquid pitch begins: the pressure is increased and transmitted through the sand and container walls to the melted pitch. This is followed by a gradual build-up of temperature and pressure until the values required for carbonization are achieved. The achieved P,T-values are maintained throughout the carbonization process. The proposed method and equipment for carrying out infiltration and carbonization under high pressures are successfully employed for the production of the CCCM in a number of Russian enterprises. In particular, friction discs fabricated from the CCCM are used by Russia's leading aircraft companies in aircraft braking systems [3].

## 3.CONCLUSION

An alternative method and equipment for the realization of infiltration and carbonization processes in the production of carbon-carbon composite materials have been developed and put into commercial production.

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