INFLUENCE OF HOT PRESSING ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF CVI-INFILTRATED C/C-COMPOSITES

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Introduction

The isothermal isobaric chemical vapor infiltration (I-CVI) process is widely used to manufacture C/C-Composites for high performance applications. The main drawback of the I-CVI-process is the low deposition rate resulting from the use of a low gas pressure, which enhances the deposition into the center of the porous substrate. Therefore, infiltration cycles of many days or many weeks are required in order to obtain sufficient dense C/C-Composites. To overcome this drawback thermal/mechanical treatments like hot pressing (HP) can be used to post densify CVI-infiltrated samples with lower initial densities. During the HP process a simultaneous graphitization of the Pyrocarbon matrix can be achieved. In this work investigations were carried out in order to study the influence of hot pressing treatments on the microstructure and mechanical properties of CVI-infiltrated carbon fiber felts.

Experimental

Uniaxial hot pressing (HP) experiments were performed on carbon fiber felts with a fiber volume content of 12% randomly oriented chopped fibers infiltrated by an I-CVI process. Figure 1 shows the principle of the hot pressing process. The sample is enclosed in a graphite mold and undergoes the mechanical pressure of the graphite punches. The experiments were carried out at the temperatures 2000 and 2150°C. The applied loads were set to 10 and 15 kN which corresponds to axial pressures of about 19 and 29 MPa, respectively. The process duration was varied between 2 and 20 h.

The bulk density and the open porosity of the samples before and after hot pressing treatments were determined using the buoyancy method. X-ray diffraction measurements were carried out before and after hot pressing treatments using a Siemens D500 diffractometer. The apparent interlayer spacing \(d_{002}\) and the apparent layer stack height \(L_c\) were calculated from the (002) diffraction peak using the Bragg and the modified Scherrer equation, respectively [1-2].

Three-point bending tests were carried out on samples before and after hot pressing in order to characterize the influence of HP process on the mechanical properties of the CVI-infiltrated carbon fiber felts. Rectangular bars of 8 x 3.5 x 0.5 mm³ were cut using a diamond-tip saw. The tests were carried out on a universal testing machine (UTS, Germany) using a load cell of the type KAP-S/100N/0,05 (A.S.T., Germany). The specimens were placed on rollers, 3 mm in diameter. A span of 7 mm was used, giving a span-to-depth ratio of 14. The tests were carried out with a constant cross head speed of 10 mm/min. At least twenty specimens were tested for each composite. The load and deflection values were recorded as a function of time. The nominal bending stress (\(\sigma\)) and the nominal outer fiber strain (\(\varepsilon\)) were calculated according to [3]. The ratio of the secant modulus, i.e. the slope of the line from the origin to the stress at failure in the stress-strain curve, to the origin modulus, i.e. the slope of the linear part of the stress-strain curve, is used here to compare the quasi-ductile fracture behavior of the samples before and after hot pressing treatments (Fig. 2).

The interpretation of the bending strength results was made with the help of the widely used Weibull distribution [4]. This choice is confirmed by the fact that the experimental strength data fit very well with a straight regression line when plotted in a Weibull diagram. The slope of the straight line gives the shape parameter (m) of the distribution and the strength at a failure probability of 63.21% gives its scale parameter (\(\sigma_0\)).

After the bending tests the fracture surfaces of the samples were examined by means of the scanning electron microscope (SEM) LEO 440C.

Results and Discussion

Figure 3 shows the influence of the hot pressing conditions as well as the initial bulk properties of the composites on the densification effect after the hot pressing treatments. It points out that the densification effect after HP treatments depends strongly on the initial bulk properties of the composites. Indeed, with decreasing initial bulk density and increasing initial open porosity of the composites the densification effect becomes more pronounced (Fig. 3). It demonstrates also that with increasing pressure, temperature or duration both the bulk density and the open porosity rates of all investigated composites decrease after
hot pressing (Fig. 3) showing an intensification of the densification effect. Figure 4 shows two SEM micrographs of polished cross sections of a CVI-infiltrated carbon fiber felt before and after hot pressing treatments. It visualizes clearly the densification occurring during the HP process. In fact, after hot pressing treatments both the number and the size of pores decreased dramatically. Figure 5 shows the influence of hot pressing treatments on the crystallinity of the samples. The apparent layer stack height $L_c$ increased while the interlayer spacing $d_{002}$ decreased with increasing HP temperature or duration. This proves the advance of the graphitization of the samples during the hot pressing process. In order to study the influence of the microstructural changes after hot pressing treatments on the mechanical properties of the CVI-infiltrated carbon fiber felts, more than twenty bars were cut from the composite before and after the HP process (20h/2150°C/29MPa) and were tested in three-point bending mode. The typical nominal stress-nominal strain curves are shown in figure 6. It shows a distinct increase of both, flexural strength and quasi-ductile fracture behavior of the composite after hot pressing treatments. Figure 7 shows the Weibull distributions of the flexural strengths of the investigated samples. It confirms statistically the results of Fig. 6. In fact, the scale parameter of the distribution (flexural strength with a failure probability of 63.2%) increases more than 21% after hot pressing treatments from about 55 MPa up to 67 MPa. This increase in the flexural strength must be the result of the densification effect after HP treatments mentioned previously (Fig. 3).

The $E_{\text{secant}}/E_{\text{origin}}$ ratio, used here to describe the quasi-ductile fracture behavior of the composites (Fig. 2), drops after hot pressing treatments from 86.0% down to 70.6% showing a pronounced elongation of the quasi-plastic strain part in the nominal stress-nominal strain curves. This was clearly confirmed with SEM investigations showing a distinct pullout increase after hot pressing treatments (Fig. 8). In fact, the length of the pulled out part of the fibers is distinctly higher after the hot pressing process than before it (Fig. 8, lower magnification).

The quasi-ductile fracture behavior of CVI-infiltrated carbon fiber felts is strongly governed by the crack deflection at the interfaces of the numerous sublayers within high textured (RL, rough laminar) pyrocarbon layer [5, 6]. After hot pressing treatments the sublayer architecture of the matrix becomes more pronounced resulting in lower interlayer spacing $d_{002}$ and higher layer stack height $L_c$ (Fig. 5). This means that the crack must overcome more sublayer interfaces, perfectly oriented parallel to the fiber axis, during its propagation. This requires more fracture energy and leads to a higher quasi-ductile fracture behavior of the samples. On the other hand, it was shown in a previous work that low textured (SL, smooth laminar) pyrocarbon graphitizes hardly in comparison with RL pyrocarbon. As a consequence of this effect, a misfit between SL and RL pyrocarbon layers must occur after the hot pressing process resulting obviously in a higher crack deflection at the interface between the two layers. This enhances the quasi-ductile fracture behavior of the samples and leads to the more pronounced pullout effect observed in the SEM micrographs (Fig. 8).

**Conclusions**

During hot pressing treatments a simultaneous densification and graphitization of C/C-Composites occurs. The densification effect leads to a distinct increase of the flexural strength of the samples. The graphitization of the matrix enhances distinctly the quasi-ductile fracture behavior of these materials.

**References**


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Figure 1. Principle of the hot pressing (HP) process

Figure 2. Schematic stress-strain curve showing the definition of the $E_{\text{secant}}/E_{\text{original}}$-ratio

Figure 3. Influence of hot pressing conditions and initial bulk properties of the composites on densification effect after hot pressing treatments

Figure 4. SEM micrograph of polished cross section showing the densification effect after hot pressing
Figure 5. Influence of hot pressing (HP) conditions on the crystallinity of the samples

Figure 6. Typical stress-strain curves of the investigated felt before and after hot pressing

Figure 7. Flexural strength distributions of the investigated felt before and after hot pressing
Figure 8. SEM micrographs of the fracture surfaces of the C/C-felt after three-point bending tests before and after hot pressing treatments.