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SEM Analysis of Ablated Carbon Felt–Carbon Composite Samples

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Abstract: Ablated carbon felt–carbon composite samples were analysed using scanning electron microscopy (SEM). Results indicate processing-induced microstructure change in preoxidized and intermediate strength PAN carbon fibres. Structural features were analysed accordingly. © 1998 Published by Elsevier Science Limited and Techna S.r.l.

1 INTRODUCTION

Carbon fibres are a novel kind of reinforcing material, presently widely used in composite materials. The structure and defects in carbon fibres directly influence composite performance. In recent years, X-ray diffraction analysis and high resolution transmission electron microscopy (TEM) have been utilised in investigation of carbon fibre microstructure, and significant achievements have been made. While the former mentioned analysis technique usually returns statistically average results, the latter requires excessive experimental work because its results are limited to localized regions. With the improvement in scanning electron microscopy (SEM) technique and its resolution, SEM analysis of 3-dimensional structure in carbon fibres improved the care and reliability of evaluation of composites performance based on microstructure.[1]

Ion etching or ablation can unveil the intrinsic structure and defects in carbon fibres and their composites, and SEM analysis can reveal those features.

This paper presents results of systematic observation on ablated samples of carbon felt–carbon composite materials. Emphasis was put on microstructure in pre-oxidised filaments after composite processing, distribution of defects and impurities; structural characteristics of deposited carbon and impregnated carbon. In addition, the ablation process of carbon felt–carbon composite materials was also analysed.

2 SAMPLES AND EXPERIMENTALS

The carbon felt–carbon composite materials were fabricated using preoxidised PAN carbon fibre felt billets. The billets were processed by CVD, several cycles of pitch impregnation, several cycles of high temperature graphitisation, and were finally finished by a relatively longer period of high temperature graphitisation.

Preoxidized PAN fibres possess initial carbon structures, but a substantial amount of organic carbon is also present.

During processing, the fibres experience carbonisation and graphitisation, showing significant microstructural change, and many intrinsic defects occur.

The CVD carbon and impregnated carbon are also prone to structural change during high temperature graphitisation.

The ablation of carbon felt–carbon composite materials is equivalent to a high dosage ion etching, which unveils detailed structures. The ablated samples should be preserved appropriately and should be subject to no damage or contamination.

The ablated samples were coated with a layer of gold about 100 Å in thickness on a 1B–3 ion

sputter system and were observed on H-700H TEM utilising its 7010 A scanning accessories.

3 RESULTS AND ANALYSIS

3.1 Fibre structure and defects

X-ray linear structure analysis and high resolution electronic microspectroscopy show: PAN carbon fibres are composed of large amounts of fibrils, and surrounded by poorly crystallized carbon or amorphous carbon, and, in between, there are holes and cavities. The fibrils are polycrystalline, with the thickness between 250 Å and 1000 Å.

Ablation of carbon felt–carbon composite materials clearly unveils the fibrils whose diameter is around 250 Å. The diameter of the fibrils is basically uniform, and intrinsic defects are rarely found. The arrow pointed region in Fig. 1 is the skin of the fibre which is relatively thin. It can be see that the fibrils are randomly distributed, and the arrangement of the skin region, the intermediate region and the core region are identical. The fibres appear slightly graphitised fibres.

EM observation revealed a large amount of defects in the majority of those fibres, and those defects mainly include long holes, cavities and aggregated impurities.

The impurity aggregated region forms a sphere after ablation, which is about 2000 Å in diameter, and caves occur wherever such spheres are formed. In Fig. 2, the arrows A and B pointed spheres are located inside the fibres, whereas arrow C pointed sphere is in the deposited carbon. Figure 2(b) shows the ablation morphology of three fibres, there are caves on the surfaces of fibres A and B,

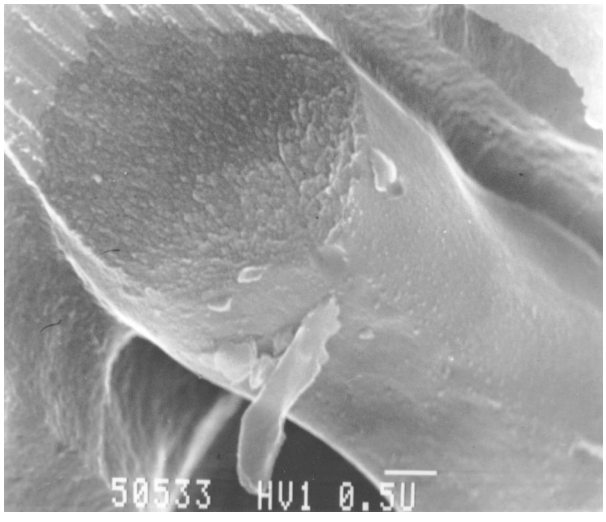


Fig. 1. Fibrils revealed by ablation.

fibre C is relatively small in diameter and ablated evenly.

There are a variety of holes inside the fibres, among them the elongated holes are the principal part whose longer axis is aligned along the fibre axis. There are also cylindrical holes. A minority of fibres have excessive holes inside, and there are particles in the holes. Figure 3 shows the morpholgy of various holes. In Fig. 3(a), the holes in fibre C are very large, and the holes in fibre D differ in size, with the average diameter being 0.5 μm, the holes in fibre E are also very large, and arrow A points to the propagation of the interfacial gap during ablation, whereas arrow B points to a well-bounded interface between the deposited carbon and the fibre. The arrow pointed area in Fig. 3(b) shows the existence of contaminant on the fibre surface which is present at a relatively large area around the interface.

It follows from the above observation that there exists a large amount of elongated holes inside the fibres after processing and the holes are aligned along the fibre. These holes are caused by high temperature processing.

SEM observation of ablated 3D carbon–carbon surfaces has revealed the existence of caves on the fibre surface and the presence of spherical particles in the caves. These particles contain elements Na, C, O, Si, K and Mg.[2] There are similar impurities in carbon felt–carbon composites, which reduce the oxidisation resistance of the impurity aggrevated regions, causing caves in ablation.

The ablation performance of the fibres show that the fibres have a low degree of graphitisation: as pointed out in Ref. 4, PAN-based felts decompose at 840°C, whose d_{002} is 3.53 Å, L_c is 20 Å. At 2000°C, d_{002} is 3.52 Å, L_c is 23 Å, whereas at 3000°C, d_{002} is 3.40 Å, L_c is 68 Å. This suggests that the preoxidised PAN fibres in the carbon felts have a low degree of graphitisation after high temperature processing, and the microcrystals are very small in size.

3.2 Structural features of deposited carbon and impregnated carbon

Ablation reveals the structural features of deposited carbon and impregnated carbon to a satisfactory extent. The deposited carbon compactly surrounds the fibres. Its thickness is dependent on deposition speed and deposition time.

Figure 4 illustrates a series of morphologies of deposited carbon. Figure 4(a) shows the bonding between deposited carbon and the fibres, indicating denudation propagation along the interface. Figure 4(b) is the locally magnified picture of Fig. 4(a), it

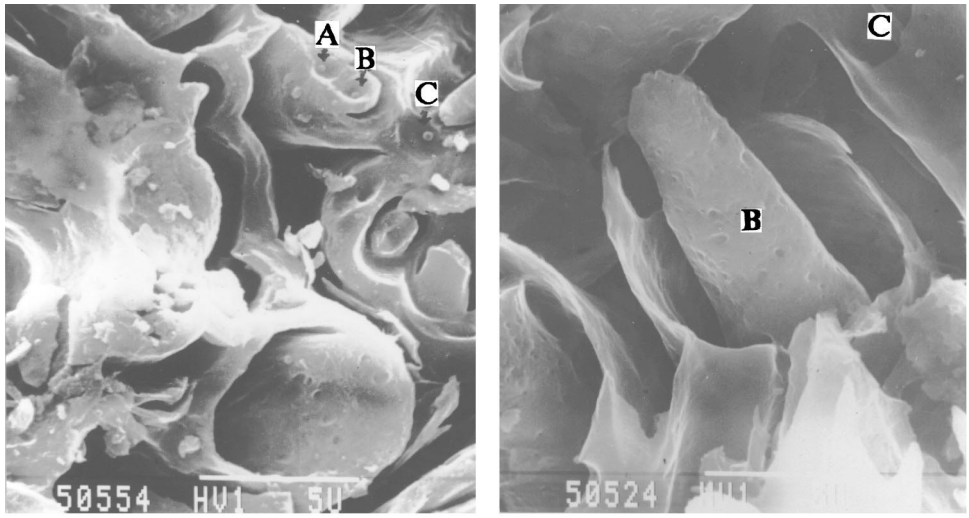


Fig. 2. Impurities aggregated regions after ablation.

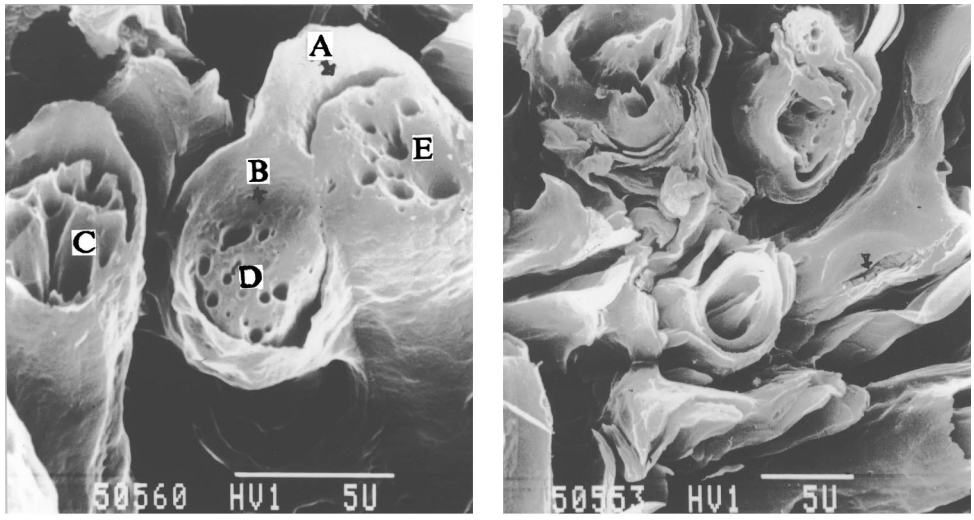


Fig. 3. Defects, interfacial gaps and contaminants.

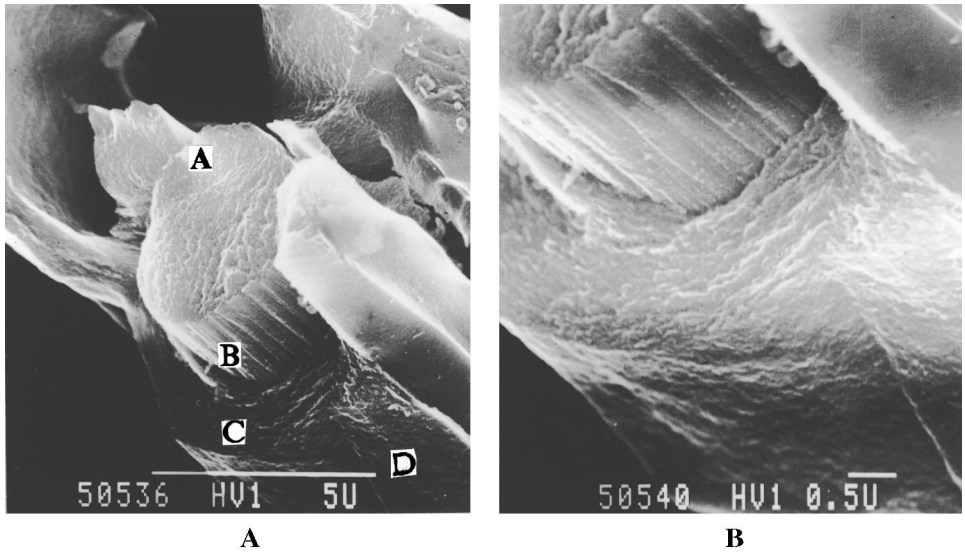


Fig. 4. Morphologies of deposited carbon.

falls into four parts: A—the fibre cross-section with fibrils and microholes clearly present; B—fibre surface with a small amount of carbon particles bonded to it; C—deposited carbon with layered structure and microholes inside; D—surface of deposited carbon. The deposited carbon particles range 300–600 Å in diameter.

It can be seen that the deposited carbon is particle shaped, with microholes inside. It is relatively compact and is not easily denuded.

Ablation reveals the layered structure of impregnated carbon. The layers are not in parallel alignment, and there are gaps between them, as shown in Fig. 5.

The roles of deposited carbon in carbon felt–carbon composite materials include: protecting the fibres, the fibres ablated slowly where the fibres and the deposited carbon is well-bonded; improving the ablation performance of the composite material.

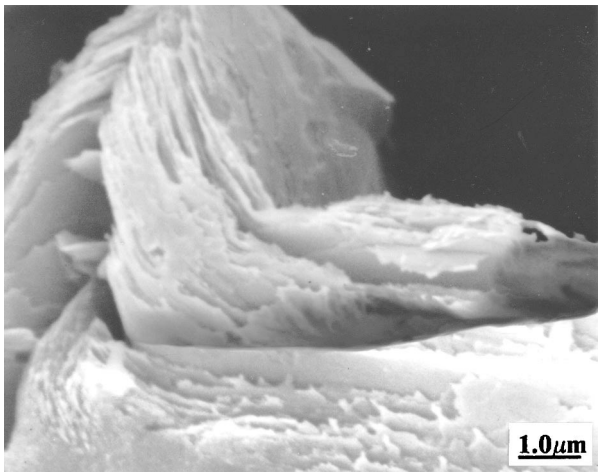


Fig. 5. Layered structure of impregnated carbon.

3.3 Ablation process

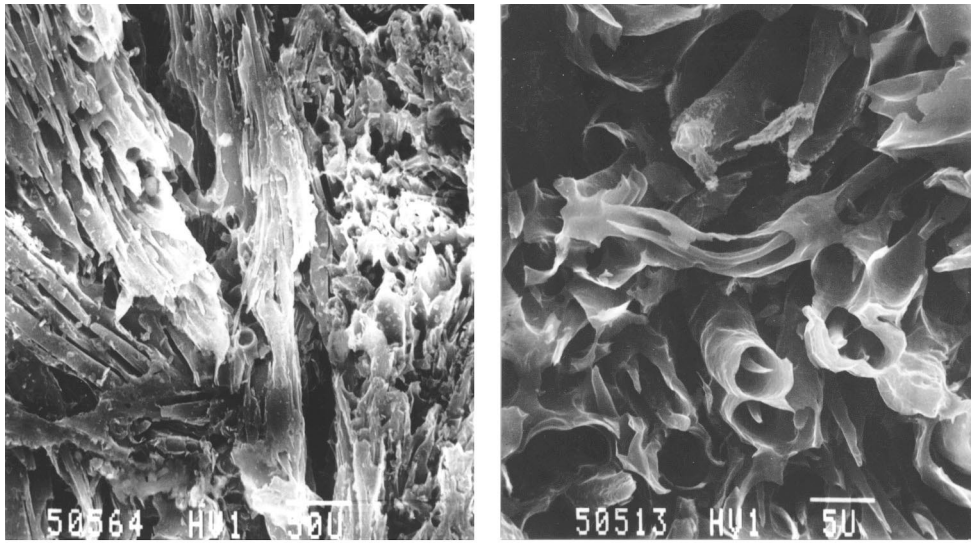
SEM observation results are analysed to trace the ablation process of carbon felt–carbon composite materials. And the assumed process is presented below.

Impregnated carbon denudes first, because it is not well-bonded to deposited carbon. And there are interface gaps between the two kinds of carbon, with many intrinsic defects inside the impregnated carbon itself, thus denudation occurs easily. In the same time, the carbon fibres are severely ablated because of the poor ablation resistance of carbon fibres. And the existence of interfacial gaps which are prone to early ablation. Ablation of the fibre starts from the fibre side surface until it ablates completely.

The ablation of deposited carbon comes second, and partial denudation occurs. The deposited carbon sublimes at high temperatures, showing good ablation resistance.

Figure 6 is a typical set of ablation morphologies of carbon felt–carbon composite materials. Figure 6(a) and (b) shows the prior ablation of impregnated carbon, with deposited carbon and carbon fibres remaining, and ablation of the carbon fibres starts from the side surface. The ablation characteristics of impregnated carbon are shown in Fig. 7. And the ablation process of deposited carbon is shown in Fig. 8.

It follows from the above results that: the intrinsic structure of carbon felt–carbon composites is directly related to their ablation resistance; the interface has significant influence on composite performance; and, defects and impurities in the material deserve attention.



A B

Fig. 6. Ablated surfaces of carbon felt–carbon composite materials.

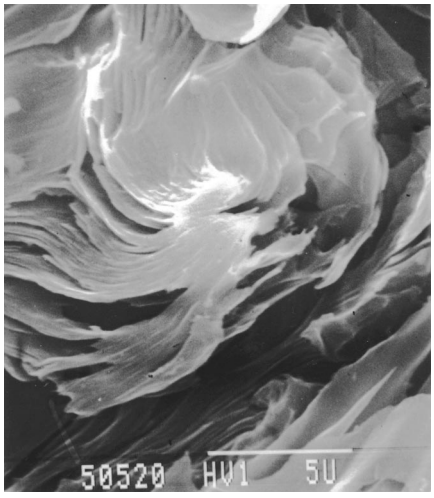


Fig. 7. Ablation of impregnated carbon.

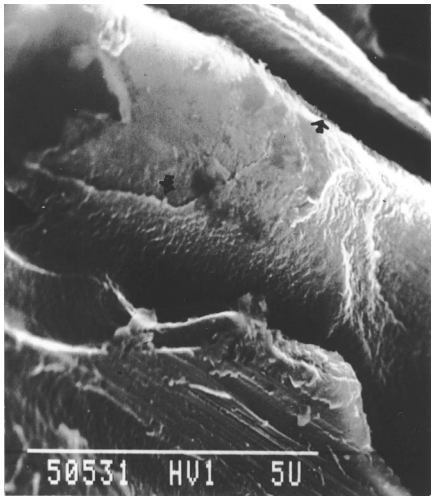


Fig. 8. Ablation of deposited carbon.

4 CONCLUSION

1. There are many defects and impurities in carbon felt–carbon composite materials after processing, which adversely affects composite ablation performance.
2. Defects in carbon felt–carbon composite materials mainly include interfacial gaps and inter-layer cracks in impregnated carbon, the former causes prior ablation of the interface, whereas the latter causes denudation.
3. Deposited carbon is relatively ablation resistant and denudation does not easily occur. But its bonding with the fibres is an important factor to take into account.

ACKNOWLEDGEMENT

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