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Investigation of Chemical Vapor Infiltrated Carbon Fiber Felts by Combined Scanning Force Techniques

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Abstract

Carbon fiber felts are an ideal system for studying chemical vapor infiltration with carbon. To understand the influence of different parameters of the infiltration process on the microscopic properties of the infiltrated felts it is necessary to investigate not only the local surface structure, but also local mechanical properties like elasticity, adhesion and friction. Here we report on a study of infiltrated carbon fiber felts applying a combination of different scanning force techniques including lateral force microscopy (LFM), force modulation microscopy (FMM) and adhesion force imaging in the pulsed force mode (PFM). The results are compared with polarized light microscopy and scanning confocal optical microscopy data. It is shown that LFM, FFM and PFM allow to distinguish not only carbon fiber and matrix but also different carbon microstructures within the matrix due to their different mechanical and adhesive properties. As a result, three different microstructures of carbon could be distinguished by scanning force techniques within the matrix: low, medium and highly textured pyrolytic carbon. Furthermore carbon fiber felts were infiltrated at two different gas pressures to study the influence of the total gas pressure during infiltration on the pyrocarbon microstructure.

Keywords: A. carbon/carbon composites; B. chemical vapor infiltration; C. atomic force microscopy, optical microscopy; D. frictional properties

1. INTRODUCTION

Carbon fiber felts represent an ideal three-dimensional structure for studying chemical vapor deposition and infiltration of carbon. Corresponding studies are a major source of present knowledge about various microstructures of pyrolytic carbon: isotropic and low, medium or highly textured [1].

The present paper is concerned with the study of microstructures of pyrolytic carbon, deposited in a carbon fiber felt, using various techniques of atomic force microscopy (AFM). With conventional contact mode AFM, surfaces can be investigated from the micrometer down to the atomic scale. Besides the calibrated measurement of the real three-dimensional topography direct local probing of mechanical properties and therefore the visualization of material contrast at surfaces are possible [2-4], but corresponding studies with different scanning force techniques on chemical vapor infiltrated carbon fiber felts have not yet been reported.

In the following first AFM studies of infiltrated carbon fiber felts are presented in combination with lateral force microscopy (LFM), force modulation microscopy (FMM) and AFM in the pulsed force mode (PFM).

2. EXPERIMENTAL

A carbon fiber felt was infiltrated with pyrolytic carbon as

described in [5]. After cutting slices from the felt the thickness was reduced by grinding and polishing. In the final stage the sample was further treated by double-side dimpling with 3 µm and 0.25 µm diamond paste; afterwards the surface was cleaned by Ar-ion beam milling.
The optical micrographs of the samples were taken with a polarized light microscope (Leica DM LM). All AFM measurements were performed with a homebuilt AFM, equipped with commercial control electronics (Park Scientific Instruments) in air and at ambient conditions.

3. RESULTS AND DISCUSSIONS

Fig. 1 shows three micrographs of the same surface area within a cross section of the carbon infiltrated carbon fiber felt. The circular and elliptic structures seen in the images represent the cross sections of carbon fibers running almost perpendicular to the plane of imaging, whereas the oblong rod in the center of the images can be assigned to a longitudinal cut of a single fiber lying almost parallel to the sample surface. Observation of the cross section with polarized light (Fig. 1(a)) shows several layers of pyrolytic carbon which were successively deposited during the chemical vapor infiltration process. These carbon layers surrounding the carbon fibers significantly differ in their optical activity. In the polarized light microscopy image, the carbon fibers show low or no optical activity. The first layer of low textured (LT) pyrolytic carbon around the carbon fiber seems to be comparable to the fiber concerning the optical contrast. The second layer exhibits small radial extinction crosses which indicate a medium textured (MT) carbon. The third layer exhibits a high density of irregular extinction crosses typical for highly textured (HT) pyrolytic carbon. Both the crystalline order and material density increase in the order LT, MT, HT carbon [6].
Fig. 1(b) shows a topographic AFM image of the sample. In this case it is easy to distinguish between carbon fiber and MT carbon but there is only a weak contrast between MT and HT carbon. Some of the cross-sectional areas of the carbon fibers are below the average surface height of the carbon matrix (dark circular areas in Fig. 1(b)), while other fibers are lying higher. In addition, pyrolytic carbon layers of the same type of microstructure exhibit a similar topographic height. This formation of plateaus is the result of the grinding and dimpling process during sample preparation. As the amount of material removed by the polishing procedure depends on the local properties of the sample, dark areas in the image of Fig. 1(b) should indicate softer, less wear-resistant carbon structures (higher rate of material removal) while the higher or brighter parts should represent the more wear-resistant ones (lower rate of material removal). This conclusion is confirmed by results of Reznik et al. [7] showing that the fracture energy can be converted into deformation in the case of MT carbon whereas HT carbon exhibits high crack densities.

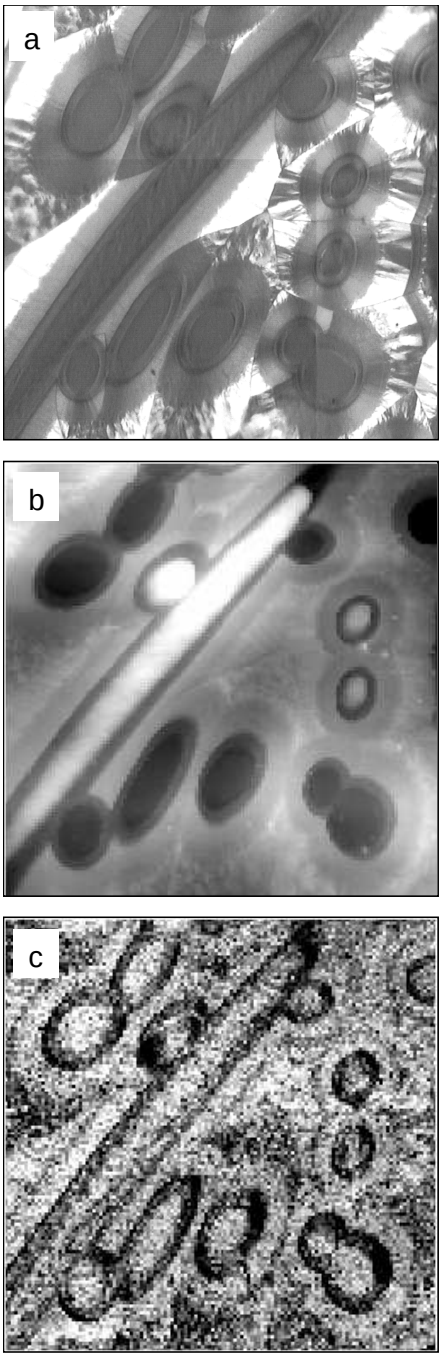

Fig. 1. Micrographs of a cross-sectional area of a carbon infiltrated carbon felt taken by (a) polarized light microscopy (PLM), (b) atomic force microscopy (AFM, topography) and (c) pulsed force microscopy (PFM).

Pulsed Force Microscopy

Fig. 1(c) shows an adhesion force image of the same surface area as shown in Fig. 1(a)-(b). Adhesion imaging was performed in the pulsed force mode of the AFM [8]. The bright regions represent a high adhesion force between AFM tip and sample surface. In contrast to the topographic image of Fig. 1(b), the micrograph in Fig. 1(c) shows no differences between the cross-sectional areas of the higher and lower carbon fiber cross sections, because the adhesive force between tip and sample surface does not depend on topographic height. The adhesion force between tip and sample has been determined to ~1.5 µN for the region of the fiber, ~0.5 µN for the LT, ~1.0 µN for the MT and ~1.5 µN for the HT regions at a rel. humidity of 40 %.

4. CONCLUSION

The studies of cross-sections of chemical vapor infiltrated carbon fiber felts show that different AFM techniques, light and polarized light microscopy complement one another when investigating material properties. While the optical methods can be used to distinguish different carbon microstructures due to their varying optical reflectivity, atomic force microscopy can be applied to investigate not only surface topography but also local mechanical and adhesive properties of the different carbon microstructures. Thus, it is possible to obtain material contrast, allowing to distinguish between different carbon microstructures such as isotropic, low, medium and highly textured carbon.

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