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Formation of Microvoids during Tensile Deformation of Composite Elastomers

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1 Introduction

Carbon materials possess various advantageous properties such as high thermal and electrical conductivity, high thermal and chemical resistance, high tensile modulus and strength, and high adsorption capacity with porous carbons. These properties make them an important class of material in various application fields. The brittleness, however, is their drawback in some application fields. It is desired to develop a material which possesses both the properties of carbon materials and deformability.

There are two approaches to obtain such a material: one being the control of the structure of the carbon material for enhancing deformability, and the other being dispersing carbon-based fillers to a deformable material. The feasibility of the former approach is demonstrated in the experimental results of the transverse compression tests on carbon fibers which showed that carbon fibers can be deformed reversibly up to a displacement of about 10% of the fiber diameter. [1] It has also been reported that the in situ small-angle X-ray scattering (SAXS) during axial compression tests on carbon fiber composites showed that the longitudinal length of the microvoids in carbon fibers decreased by about 4–10 % by compression, indicating that a local region of carbon fibers can be deformed to a strain larger than the macroscopic compression fracture strain of the fiber. [2] The use of the second approach, on the other hand, is advantageous to obtain much larger deformability although this can impart the properties of carbon materials to the material only to a limited extent.

In this study, the second approach was used, and carbon nanotubes (CNT) and graphene nanoplatelets (GNP) were dispersed in a fluorine elastomer. The structure changes of these composite elastomers during stretching was analyzed with the synchrotron radiation SAXS. The fluorine elastomers have excellent thermal and oil resistance while their tensile strength is rather low and their high chargeability causes dust deposit. These disadvantageous properties will be improved by the dispersion of the carbon-based fillers.

2 Experiment

A copolymer of tetrafluoroethylene with perfluorovinyl ether (FFKM, Daikin Industries, Ltd.) was used for the matrix elastomer. The CNT with a length of 10–20 μm and a diameter of 150 nm (VGCF-H, Showa Denko K. K.) and GNP with a width of 15 μm and a thickness of 6–8 nm (Strem Chemicals, Inc.) were used for the fillers.

CNT and GNP were dispersed in a solvent of FFKM, sonicated, and added with FFKM swollen with the solvent. The mixture was mixed with a planetary centrifugal mixer. The solvent was evaporated by heating the mixture at 80 °C for 3 hrs. The mixture was pressed into films with a thickness of 500 μm using a hot press. During the hot pressing, Si_3N_4 and urea were placed around the film and the pressure was kept for 20 min at 140 °C to form crosslinking by triazine rings.

The SAXS measurements were performed during the stretching of the composite elastomers using the facilities at PF, BL-6A. The X-ray wavelength was 0.15 nm, the sample-to-detector length was 1.5 m and Pilatus 3 was used for the detector.

3 Results and Discussion

The tensile modulus and strength of FFKM were effectively increased by dispersing fillers for both CNT and GNP. These values increased with increasing filler contents up to 10 phr (10 mass % relative to the mass of FFKM) and GNP showed higher reinforcing effect than CNT except for 10 phr GNP dispersion.

Fig. 1 shows the changes in the SAXS patterns of the composite elastomers by stretching. With increasing elongation, the scattering patterns are gradually elongated in the horizontal direction, i.e. perpendicularly to the stretching direction. There are two possibilities for these changes. One is that the fillers are oriented in parallel to

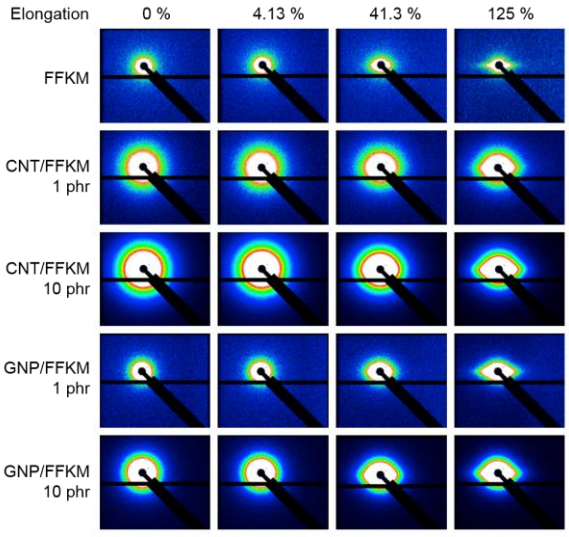

Fig. 1: SAXS patterns of FFKM dispersed with CNT and GnP by 0, 1 and 10 phr measured at elongation of 0, 4.13, 41.3 and 125 %. Stretching direction was vertical.

the stretching direction. The other is that microvoids elongated in parallel to the stretching direction were formed. Since the scattering intensity along the horizontal direction is increased while that along the vertical direction does not, the changes in the scattering patterns can be attributed to the formation of microvoids. The amount of microvoids formed during stretching was larger for the GNP dispersion compared with the CNT dispersion. This is considered to be the reason for the lower tensile strength of the composite elastomers dispersed with 10 phr GNP as compared with the composite elastomers dispersed with the same amount of CNT. Further study is continued.

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