AFM investigations of fracture surfaces of filler-reinforced elastomers

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The starting point of the motivation for the presented work was to find a correlation between the morphology of the fracture surfaces and fracture mechanics parameters of filler-reinforced elastomeric materials [1] to get a deepened understanding of the process of crack propagation in such materials. The processes leading to a macroscopic material separation in form of crack propagation have not yet been clarified for elastomers. One of the main problems is the lacking of extensive plastic deformation compared to thermoplastics or metals for example. As a result, the fracture surfaces of (highly) filler-reinforced elastomers are macroscopically often less structured (**Figure 1 a**) and so, the interpretation of the crack process is more difficult. However, it is to assume that a connection between the morphology of the fracture surface and the crack propagation characteristics exists also for elastomers. It is well known that fracture surfaces of carbon-black reinforced styrene–butadiene rubber vulcanizates (SBR) become macroscopically increasingly smooth with increasing filler content. However, own pre-examinations by using the atomic force microscopy (AFM) have shown that on the micro-scale a different trend can be observed (**Figure 1 b**). Furthermore, these pre-examinations showed clearly that it is necessary to perform systematic AFM investigations regarding the definition of the optimal test parameters such as type of cantilever, scan size, amplitude and scan frequency. These results should be shown in this contribution.

The AFM examinations concerning the influence of the experimental parameters were performed with a fracture surface of an SBR vulcanizate with 50 phr (parts per hundred parts of rubber) by using the AFM Q-ScopeTM 250 of the company Quesant Instruments Corporation. A measuring head of 40 µm (x and y direction) and maximum 4 µm in z direction was applied and the tests were performed at room temperature in the intermittent mode (wavemode). For the quantitative characterization of the fracture surface roughness, the arithmetic and square mean roughness values R_a and R_z according to DIN EN ISO 4287 were selected. Three different cantilever types were used. Details are shown in Table 1.

Table 1. Details of the used cantilever types

Type	Resonance Frequency (kHz)	Length (µm)	Thickness (µm)	Tip Angle (°)
NSC 14 AL/Bs	160	125	2	30
NSC 16 Q-WM 190	170	230	7	20
HF Q-WM 300	300	125	4	10

It was found that the cantilever type and the scan frequency especially have a strong influence on the mean roughness value R_a. Some selected results illustrating this fact are shown in **Figure 2**. From these results, it was derived that the cantilever NSC 16 is the

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optimal choice for the quantification of the variation of the further experimental parameters. Furthermore, with NCS 16 the highest reproducibility of the test results was obtained. It is not surprising that the lowest scan frequency led to the most detailed mapping of the fracture surface. The variation of the amplitude of the cantilever oscillation was found to be of relatively low importance in this case. Furthermore, concerning a complete scan and an optimum test time, the scan size must be well adjusted, also in connection with the other test parameters, especially the cantilever type (see **Figure 2**).

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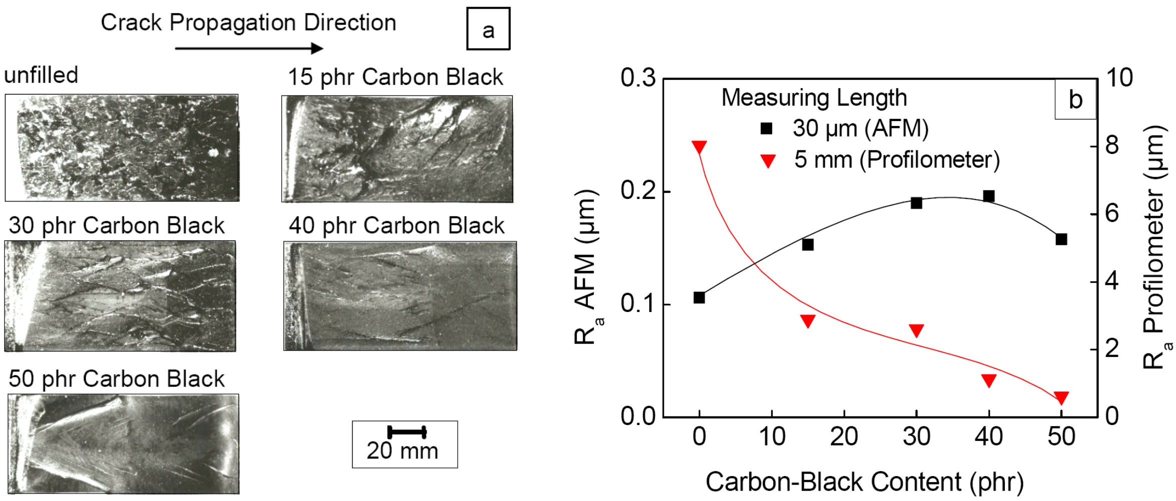

Figure 1. Light microscopic images of fracture surfaces of SBR vulcanizates showing decreasing macroscopic roughness (a) and mean roughness values *R*a from AFM and preinvestigations and tests with a profilometer depending on the content of carbon black

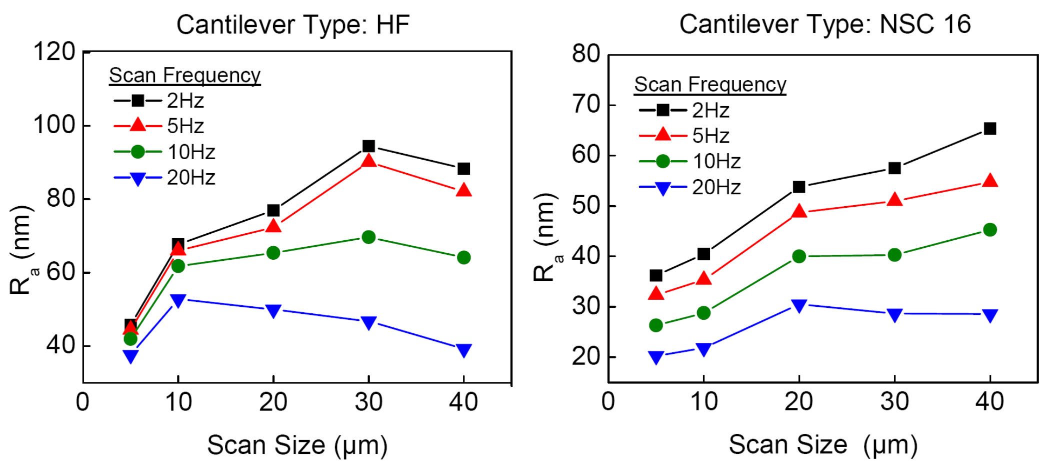

Figure 2. Dependence of the mean roughness value *R*a on the scan size and the scan frequency for two different cantilever types; material was an SBR vulcanizate reinforced with 50 phr carbon black

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