FRACTURE BEHAVIOR OF COARSE GRAIN GRAPHITES INVESTIGATED BY ACOUSTIC-EMISSION AND CT-ANALYSIS

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Introduction

Fracture mechanics investigations on graphites with grain sizes larger than 10 mm are difficult to evaluate due to problems with crack length measurement. Methods like compliance, optical or direct current potential drop method which are successfully used in the field of material science are not applicable due to the macrostructure of these graphites and required sample sizes. A combination between acoustic-emission and computer tomography (CT) analysis is used in this paper to characterize the sample microstructure under loading and crack propagation.

Experimental

Material

For this paper three different trial materials with a coarse grain recipe are used:

1. Trial recipe 1, max. grain size around 10 mm
2. Trial recipe 2, max. grain size around 15 mm
3. Trial recipe 3, max. grain size up to 20 mm

Besides coke and pitch an anti puffing agent is added to the mix of all three materials. This addition forms small precipitations while the production process.

The material properties include an apparent density of about 1.70 g/cm^3, dynamic Young's moduli of around 9 GPa and flexural bending strengths of 9.5 to 10 MPa (4-point bending). The average sound velocity of all three trial recipes is around 1200 m/s.
For the fracture experiments the sample geometry is a single-edge notched beam (SENB) where W = 70 mm, B = 70 mm and L = 490 mm (Fig. 1). With respect to the grain size (max. grain up to 20 mm) the notch length a_0 is only 7 mm which corresponds to an a_0/B ratio of 0.1 . The notch is set with a band-saw with a 2 mm width.

Acoustic emission measurement

Acoustic emissions are recorded using a 6-channel PCI-2 system (Physical Acoustics) and a pre-amplification of 40 dB with a threshold of 35 dB. To filter background noise and ambient noise a band pass between 20 kHz – 1 Mhz is used.
In total six WD wide-band sensors (Physical Acoustics) are used to detect the acoustic emissions. Vacuum cups which are continuously evacuated during the experiment are used to fix the sensors on the sample surface. The recorded acoustic emissions hits are localized by the software PolarAE and visualized by the software "DensityVille" [1,2].

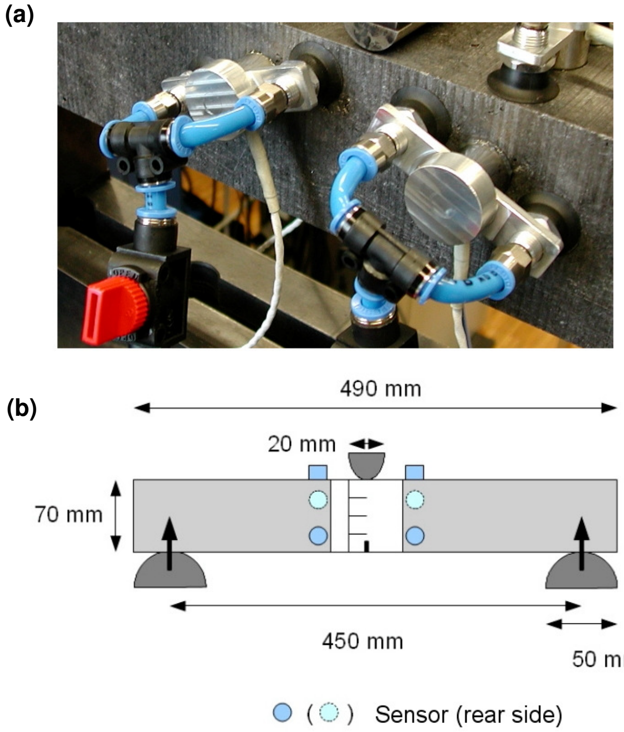

Fig. 1 Vacuum punch to fix the sensors at the sample surface (a); schematic view of the experimental setup and the used sample size (b)

3-point bending measurement

Figure 1b shows a schematic view of the experimental setup.
The cross head speed of the testing machine is 0.002 mm/s with a constant feed. The optical measurement of the crack propagation is done by video capturing. The designated crack area is coated with a white paint to increase the contrast between the crack and the sample surface.

Results and Discussion

Microstructure

CT-analysis clearly documents the differences between the three materials (Fig. 2). It is a very valuable tool to describe and to optimize the micro-/macrostructure of the graphites.
The coke grains with their porous and needle-shaped structure are clearly visible. By moving through the sample their three-dimensional orientation in the graphite block can additionally be investigated which is a big advantage when compared to classical micrographs. For each recipe the bigger coke grains are very well embedded in the fine-grained matrix – neither cavities nor gaps can be located.
In contrast, the distribution of the precipitations, seen as white spots in the CT-pictures is not very homogenous. Furthermore small micro-cracks are visible, especially in the trial 1 material (Fig. 2a) distributed across the graphite block.

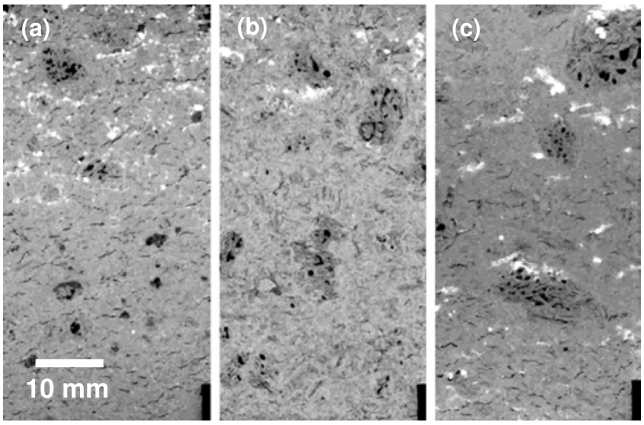

Fig. 2 Microstructure of the three different coarse grain graphite recipes; trial recipe 1 (a); trial recipe 2 (b); trial recipe 3 (c)

Acoustic emission combined with CT-analysis

The combination of acoustic emission density plots and CT-analysis yields a very good insight on the fracture process and the loaded microstructure. Figure 3 shows three slices through the fracture surface profile of the trial material 3 with the corresponding acoustic emission density. The CT-analysis is overlaid by the acoustic emission densities at the same distance referred to the sample surface.

In general the pictures show a very broad distribution of the acoustic emissions around the crack path. Sliding and partially expanding micro-cracks during the loading process combined with the coarse microstructure are responsible for this behavior. This can been seen for all three material types. The width of this process zone can reach ± 15 mm around the crack path. The knowledge of the process zone dimensions could further be used to optimize the modeling of fracture processes of a coarse grain material.

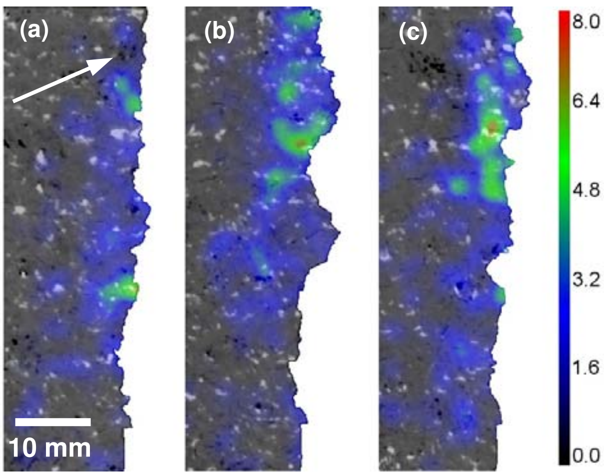

Fig. 3 Combination of acoustic emission density plots with the CT-analysis; distance of the slice from the sample surface: 25 mm (a); 40 mm (b); 50 mm (c)

Precipitations are obviously sources of high acoustic emission densities (green to red color). These parts emit acoustic signals even if the distance between crack path and precipitation exceeds 20 mm (Fig 3b). Furthermore a network of acoustic emissions between the precipitation can be observed (Fig 3c). These results show that the major part of the external loading energy dissipates at these precipitations. A better distribution of these particles combined with a smaller size could lead to a better material performance under mechanical loads due to reduction of stress peaks.

In contrast, the large coke grains itself show almost no acoustic emissions even if they are embedded directly beside the crack path (Fig 3a arrow). At the edge of these bigger grains (>10 mm) a slightly increased acoustic emission density can be found which represents a good adhesion of coke grains to graphite matrix.

These observations are made independently for all three trial materials with a similar amount of acoustic emissions and acoustic emission energies.

Crack length measurement

Acoustic emission measurements are also used to determine the crack propagation length [3, 4]. This method is also applicable for the tested coarse grain graphites.

Knowing the initial crack length a_0 and the final crack length a_{total} which corresponds to a complete crack through the sample, the crack propagation can be estimated by following equation:

$$a(t) \propto \sum_{i=0}^{N} E_i(t)$$

where E_i represents the acoustic emission energy. The crack length in turn can be used to calculate fracture properties like K_{1c} [4].

Conclusions

The method of combined acoustic emission measurement and CT-analysis is successfully used for coarse grain graphites to describe the events happening during a fracture process. This combination connects the microstructure of the sample directly to the acoustic emissions and leads therefore to a better understanding of the fracture process.

Additionally the crack propagation length can be determined which is hardly accessible with commonly used techniques like compliance or potential drop measurement.

References

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