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Supplementary Information

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High modulus polyimide particle-reinforcement of epoxy composites

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S1 – Comment on Nanoindentation Testing and Determination of the Indentation Modulus

Due to the low thickness of the PPPI particles it is very challenging to measure hardness and modulus by nanoindentation of single separated particles with a thickness of 0.2 µm. It is known that the indentation depth should be not higher than one tenth of the thickness. Consequently, a flat-wise testing will not work properly with the used devise, especially if one considers that the thickness will be thinner by polishing. Furthermore, the resulting roughness after polishing requires a minimum depth of indentation, which is in contrast to the “one-tenth” rule. On the other hand, an edge-wise testing is not possible because it has to be remembered that the indentation size for the used Berkovich tip is seven times the indentation depth, thus a sufficient distance from the edge of the particle has to be considered. Also, a direction-dependent behaviour of the highly crystalline particles cannot be excluded.

Taking into account all these things, nanoindentation of PPPI particles (or agglomerates) was done in a special way, by testing particles mixed in a polyimide (PI) matrix. This has two advantages for testing: On the one hand, large, dense agglomerates are present, which allows the testing with larger indentation depths. On the other hand, the matrix itself has an indentation modulus of 6 GPa, which is relatively close to the modulus of PPPI particles. This guarantees that even in the case when the elastic field around the indentation reaches the matrix to some extent, the influence on the result will be comparatively small. Furthermore, if one looks at the morphology of the epoxy composites with 10 and

15 Vol.% PPPI particles, a high number of small agglomerates of at least two or three particles can be observed, meaning that the matrix is in fact reinforced by small agglomerates of PPPI particles.

In a first step, indentation testing in the above-mentioned dense agglomerates was conducted at a very low load of 50 µN (10 s loading, 2 s holding, 3 s unloading), yielding an indentation modulus of 9.1 GPa. Due to the relatively high roughness resulting from the brittleness of the particles, the agglomerate nature, as well as possible orientation effects, the shapes of the resulting load-indentation curves were rather heterogeneous (Figure S1, left). In this case, the requirement that the indentation depth has to significantly exceed the roughness cannot be fulfilled.

Due to this fact, the indentation load was increased to 500 µN (Figure S1, right), leading to a modulus of 8.7 GPa. Due to the significantly more robust appearance of the indentation curves, this value was consequently assumed as the resulting modulus value of the particles (in addition to still being within 5 % of the value obtained at lower loads).

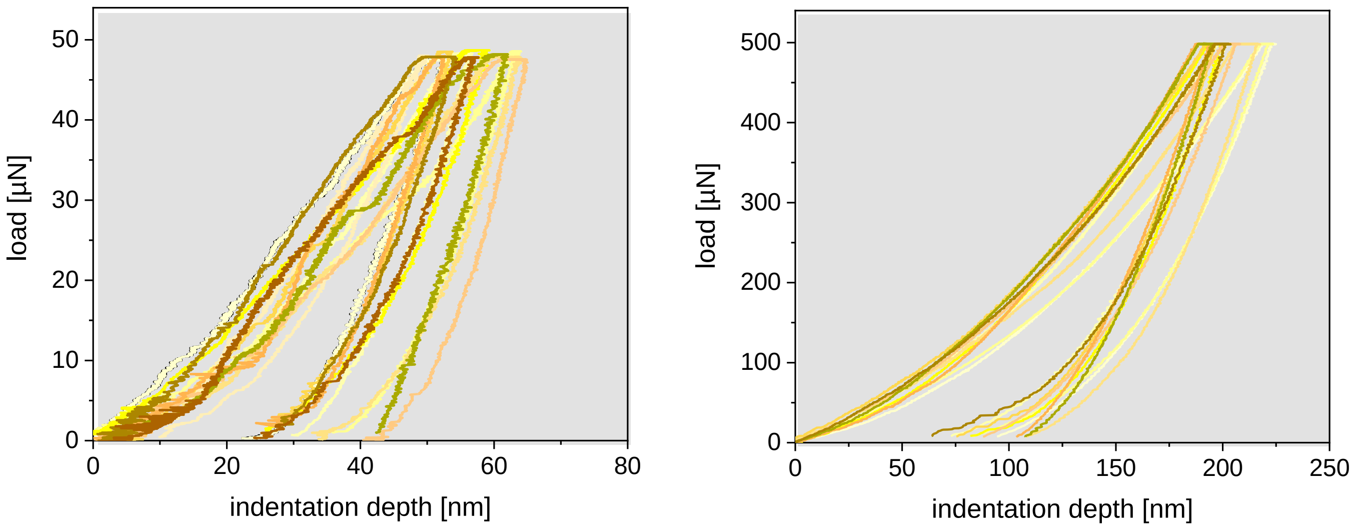

Figure S1: Load-depth curves from indentation testing of PPPI particle agglomerates at different maximum loads (left: 50 µN; right: 500 µN).