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SINTER OPTIMIZATION FOR A SILICON NITRIDE

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2
Specimen treatment

2.1
Delivery of specimens

The material has been delivered as 28 bending bars with the size of 45x4x3 mm³. One of the bars was not completely manufactured in height but all bars have been received without visible damage.

2.4 Finish treatment

To avoid the influence of surface flaws on the strength data of the material and to achieve plan-parallelism, the surface was ground on all sides to a final shape of 2x3x36 mm³.

2.5 Microstructural investigations

Fracture surfaces have been analized with the scanning electron microscope (SEM) S260 (Leica). Microstructural examinations were carried out on the field emission SEM NVision40 (Zeiss). For the determination of crystalline phases via x-ray diffraction (XRD), the diffractometer D8 (Bruker AXS) with Cu-K-alpha radiation was used. Density was measured with Archimedes method in water.

2.6 Strength measurement

Strength was measured with 4-point-bending method. The span of the outer bearing was 32 mm, the span of the inner bearing 16 mm. The test velocity was 0,5 mm/min (relative movement between inner and outer bearing).

3 Results

3.1 Density and shrinkage

The mass loss from green stage to the sintered stadium was 28,65 +- 0,13 %. The inner density is 3,235 +- 0,025 g/cm³ and the open porosity is 0,13 +- 0,14 %. All values have been calculated from the measurement of 8 specimens.
The linear shrinkage was following:

- Length: 19,51 +- 0,10 %
- Width: 20,21 +- 0,47 %
- Height: 21,98 +- 0,43 %

3.2 Strength

To ensure reproducibility, strength has been measured on specimens fabricated in two sintering cycles. The name of the specimens in this report is combined from the sintering cycle and the specimen number, i.e. C1_7 is specimen 7 from cycle 1 (Table 2).

Table 2: Strength values from 4-point-bending test

Specimen nr. (C1)	Strength (C1) [MPa]	Specimen nr. (C2)	Strength (C2) [MPa]
1	865	1	722
2	858	2	887
3	799	3	859
4	849	4	734
5	608	5	928
6	897	6	808
7	990	7	879
8	541	8	1069
9	959	9	705
10	801	10	936
11	996	11	512

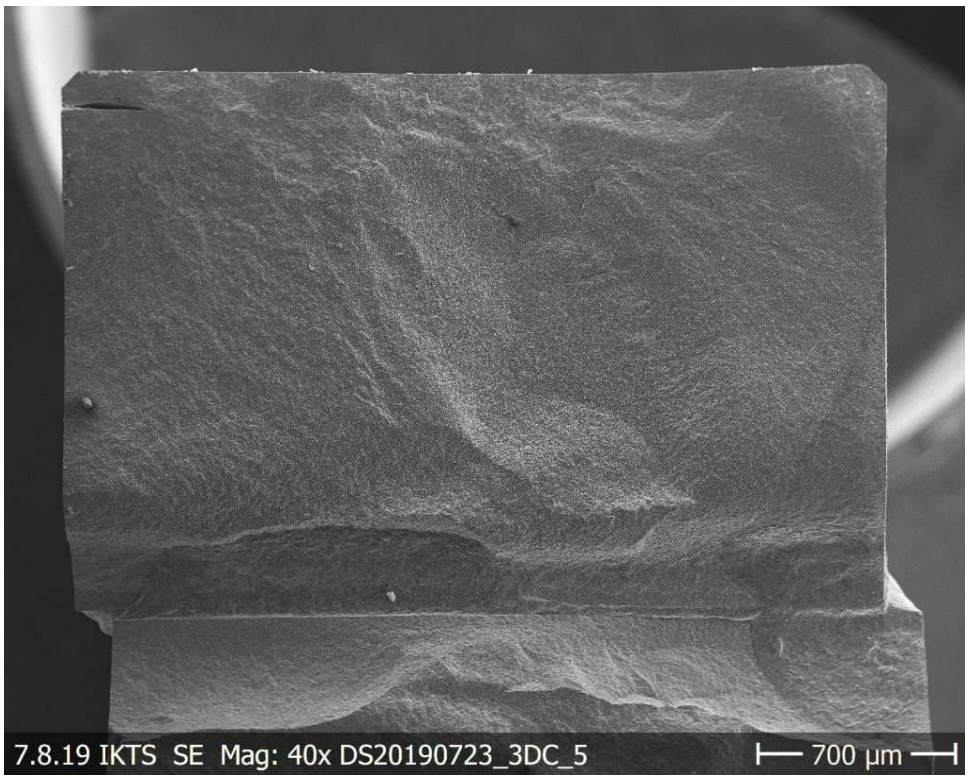

Figure 3: Fracture surface of specimen 5 of cycle 1 (C1_5); flaw on the top left

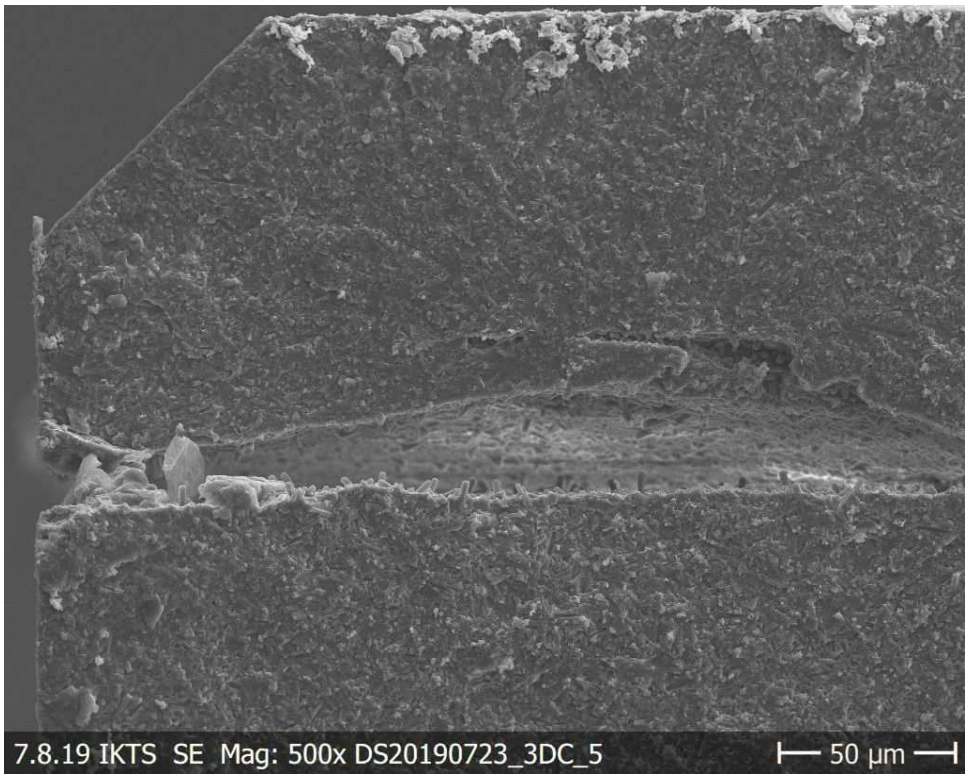

Figure 4: Higher magnification of the flaw in Figure 3

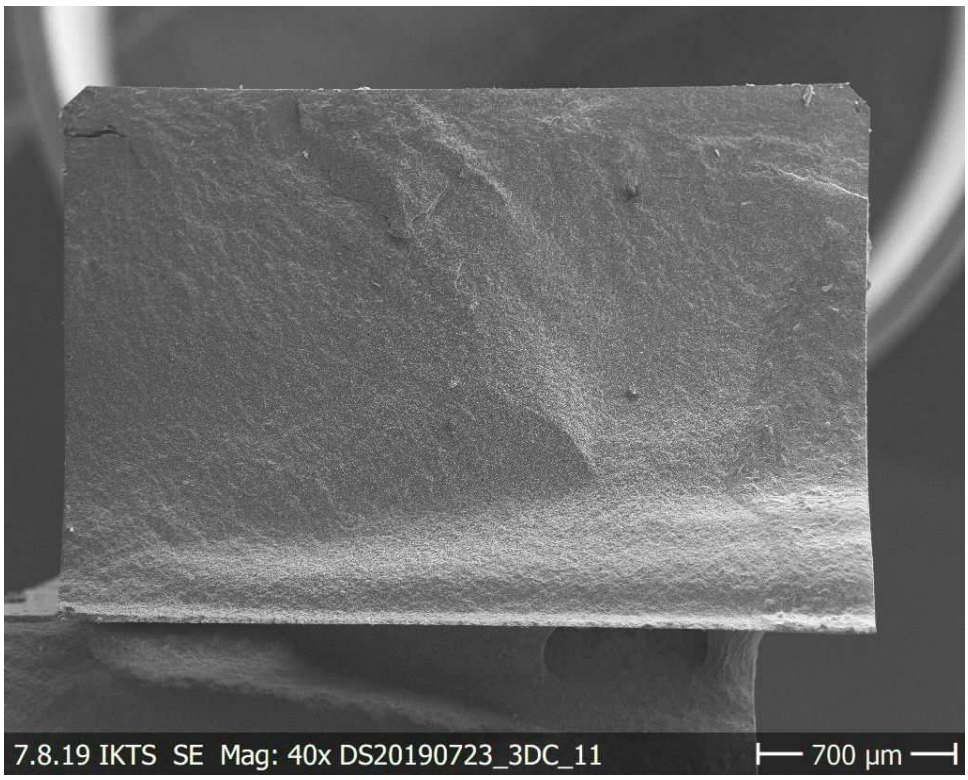

Figure 5: Fracture surface of specimen 8 of cycle 1 (C1_5); flaw on the top left; please note: the specimen number in the figure is not correct!

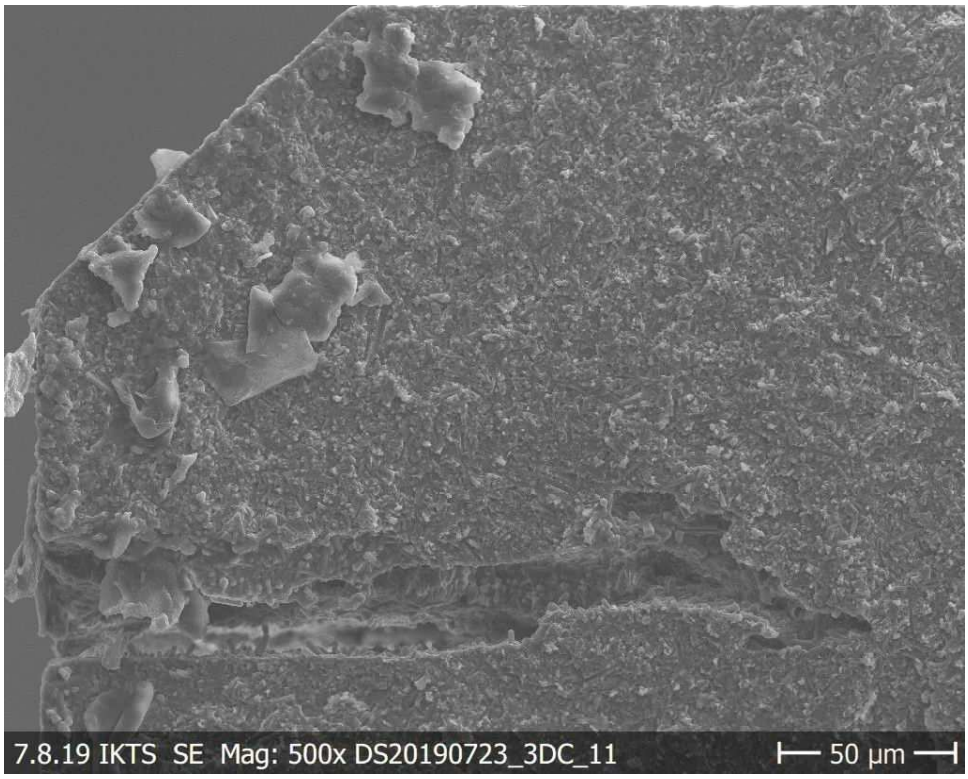

Figure 6: Higher magnification of the flaw in Figure 5; please note: the specimen number is the figure in not correct!

3.3 Microstructure

The microstructure of the material is very homogeneous and shows no pore clusters, big pores or former pores filled with intergranular glassy phases (Figure 7 to Figure 10). Only some sub-micron pores can be seen near the surface but these are typical for silicon nitride materials and have no influence on the materials´ strength. The shape of the silicon nitride grains is typically hexagonal (beta-Si_3N_4) and shows a usual aspect ratio (Figure 9 and Figure 10).

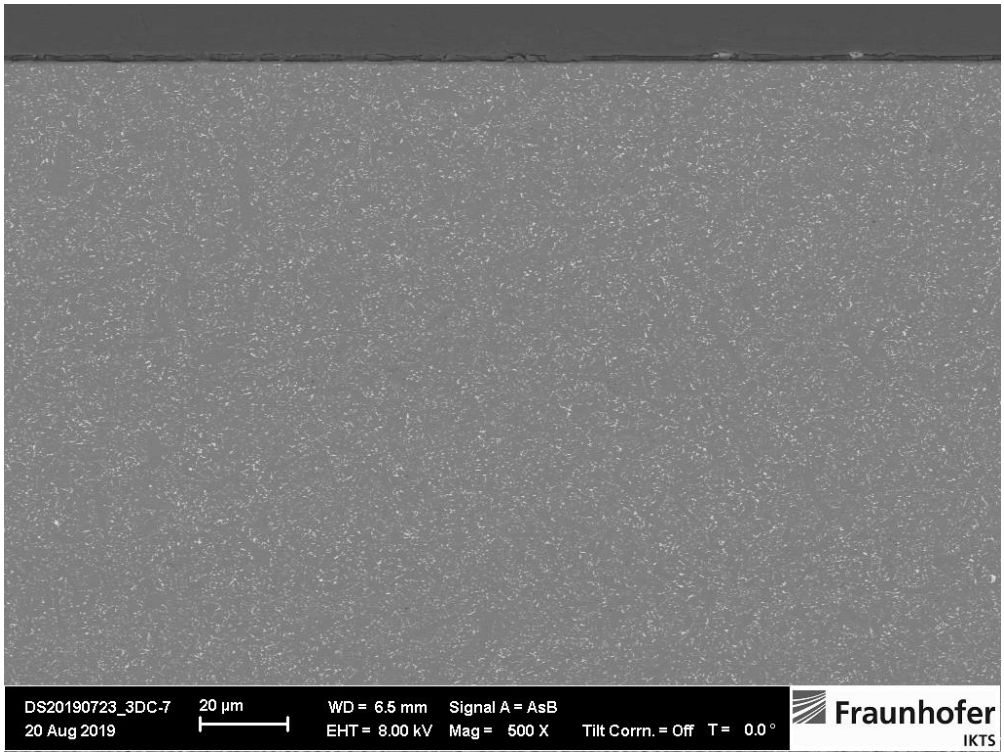

Figure 7: Microstructure of C1_7

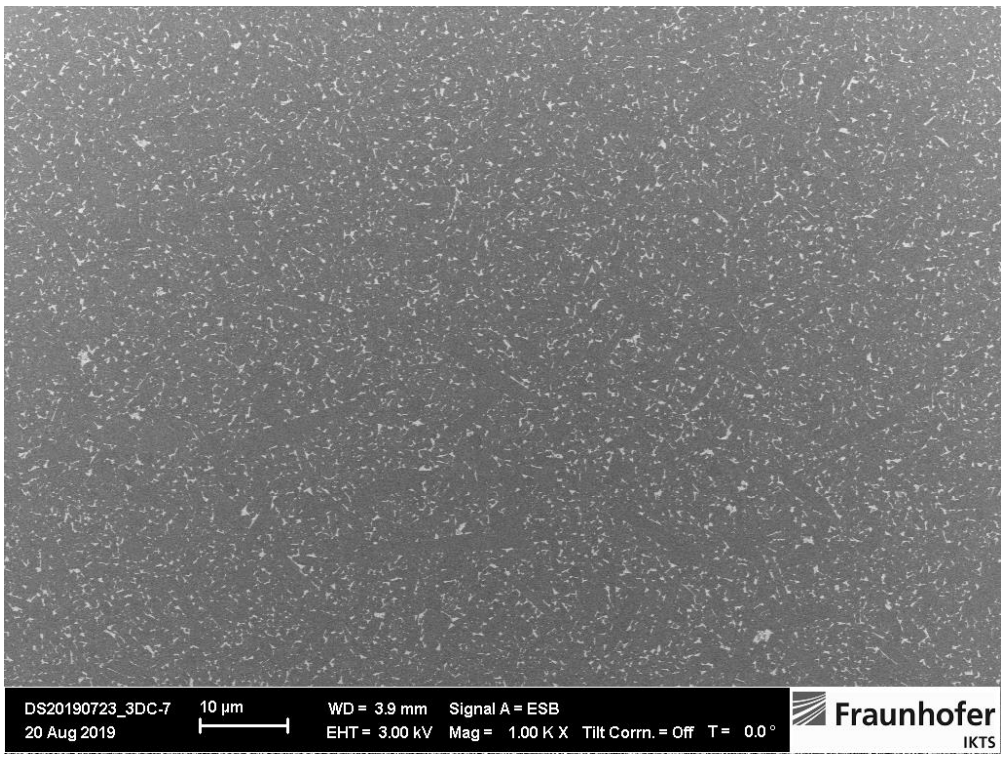

Figure 8: Microstructure of C1_7

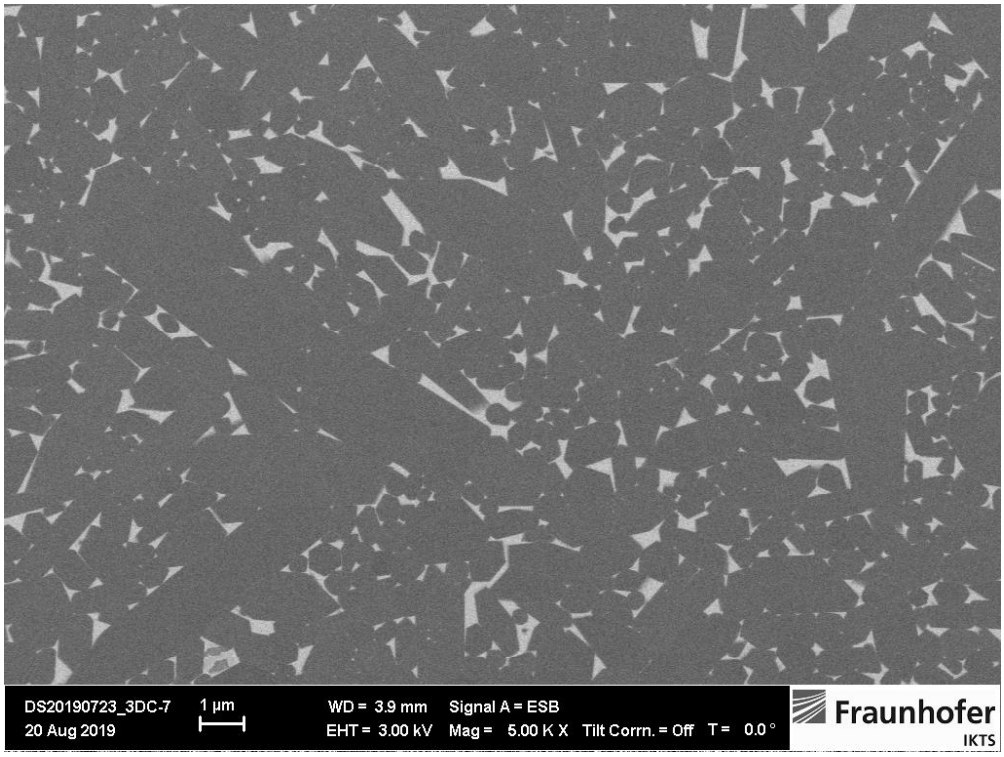

Figure 9: Microstructure of C1_7

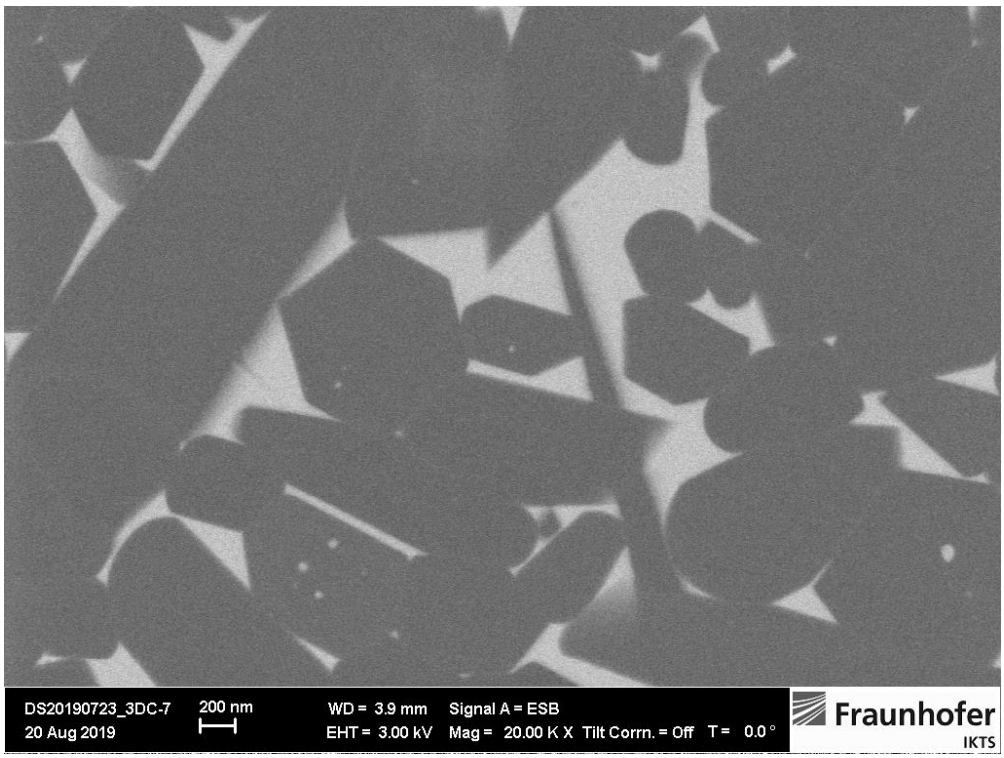

Figure 10: Microstructure of C1_7

The XRD measurement shows, that the main constituent is beta-Si_3N_4. Some small peaks indicate the presence of SiAlON-phases and of Y_2SiO_5N, which is a metastable phase that crystallizes instead of YAG (Figure 11).

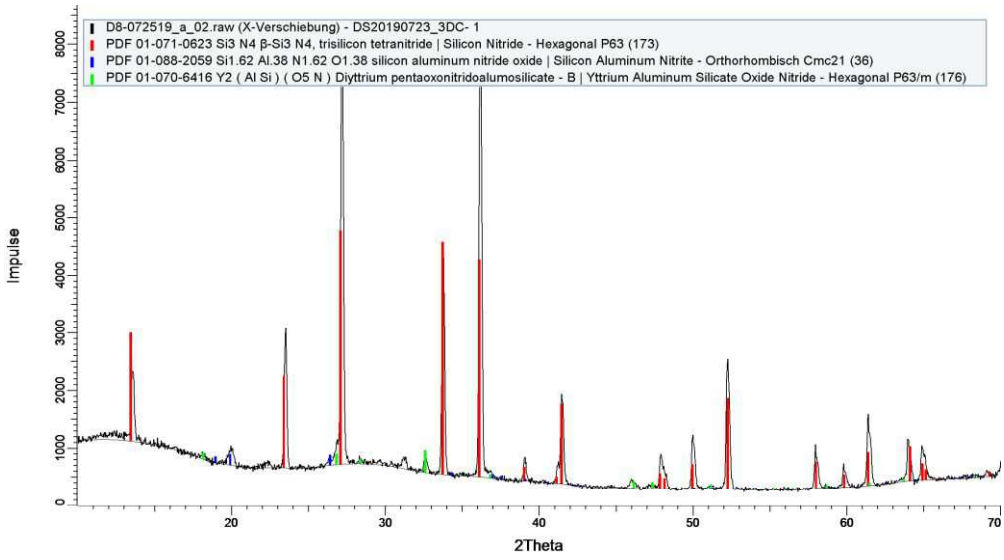

Figure 11: XRD-plot of C1_1 after sintering