The Influence of Carbon on the Microstructure of Sintered Alumina

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Introduction

Alumina is one of the most used ceramic materials, and as such understanding its sintering and densification processes is important. It is known that the sintering behavior is strongly affected by dopants, such as MgO, which promotes sintering and limits grain growth.

The present study focuses on the influence of carbon on the sintering of alumina. High hardness [1] and improved wear resistance [2] was observed for alumina doped with carbon.

Carbon segregation is difficult to analyze by conventional electron microscopy. However, carbon segregation at grain boundaries in alumina was analyzed by atom probe tomography (APT). Using APT segregation of C to a grain boundary was shown (for alumina doped with 0.012wt.% C). The C concentration was <1 monolayer for the observed boundary (no amorphous film or grain boundary phase) [3].

Methods

Commercial ready-to-press (RTP) alumina specimens were sintered to full density (98%) at 1600°C for 2 hr in air and in a graphite furnace using flowing He. The green bodies were prepared by uniaxial pressing (27 MPa) and CIP at 200 MPa. The pressed green bodies were placed in a carbon crucible on top of pure alumina powder and were covered by pure alumina powder. In addition, a specimen was fired at 850°C for 2 hr *in air* and then sintered in the graphite furnace in flowing He.

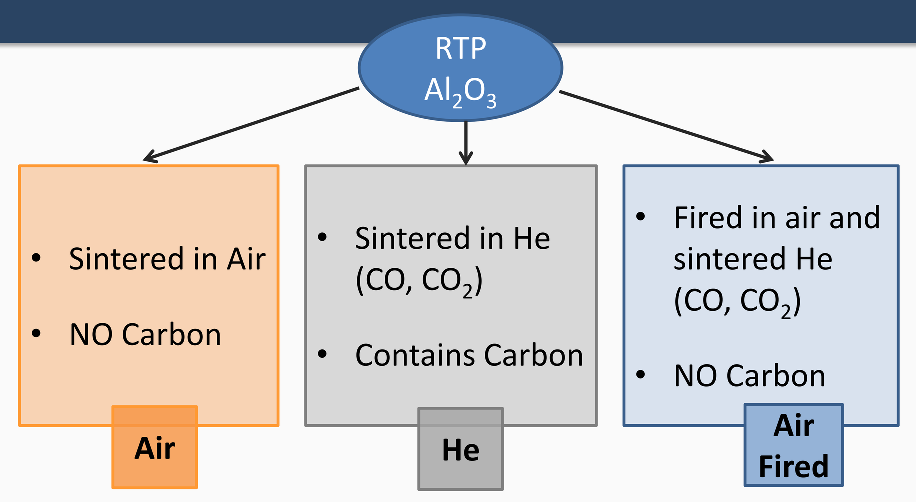

Fig. 1: Schematic of the sample sintering process.

The mechanical properties were evaluated. Three-point-bending was used to measure the flexural strength. The wear resistance of the specimens was evaluated by diamond-cutting experiments, where the time to cut through a specific cross sectional area was measured.

Results & Discussion

Powder and Specimens

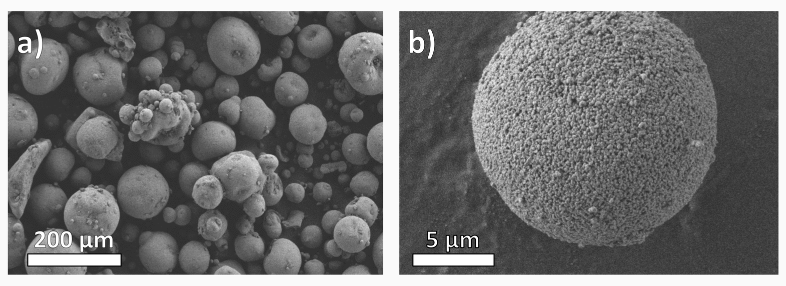

Fig. 2: SEM micrographs of (a) the RTP Al_2O_3 granules and (b) one granule composed of crystallites with a diameter of ~150 nm.

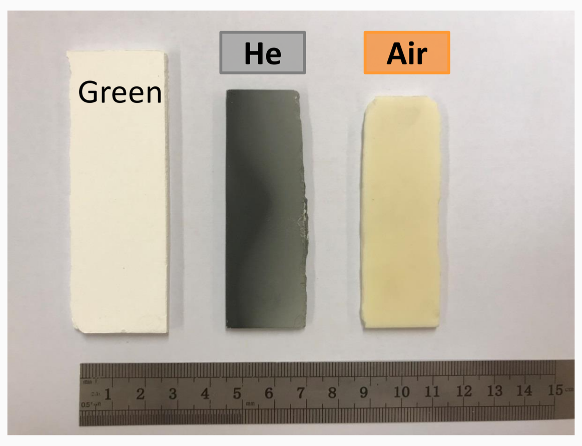

Fig. 3: Specimen before sintering (green), sintered in He, and sintered in air. The specimens sintered in He (graphite furnace) developed a black color, which indicates a carbon content originating from the organic compounds in the RTP powders (~3wt.% C). In comparison, alumina specimens sintered in air, where the carbon decomposes during sintering at elevated temperatures, were white. The relative density of the specimens according to the Archimedes method is 97-98%.

Microstructure

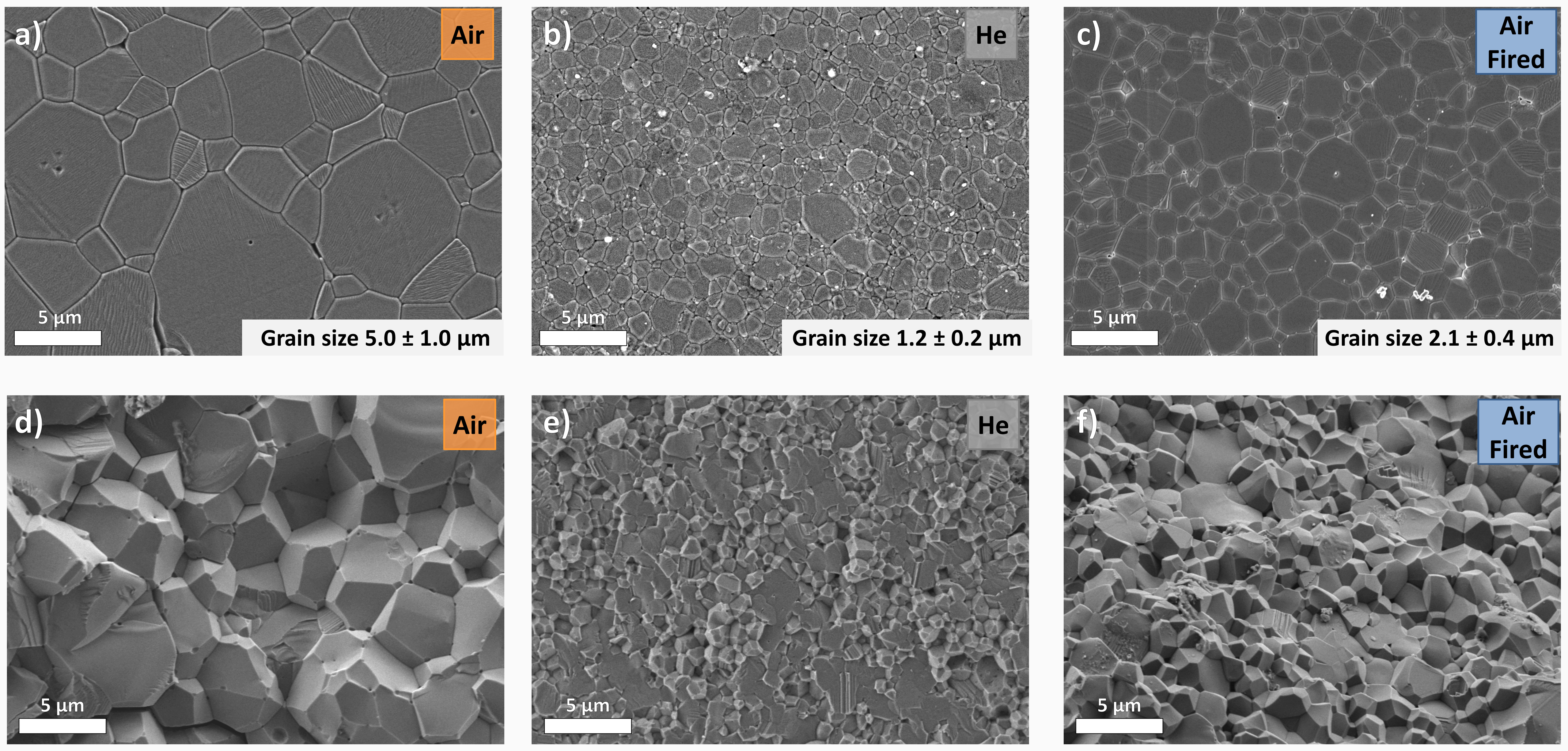

Fig. 4: : Secondary electron SEM micrographs of (a, b, c) polished and thermally etched and (d, e, f) fracture surfaces of RTP alumina sintered at 1600°C for 2 hr in (a, d) air and (b, e) in the graphite furnace in flowing He. The specimen in (c, f) was fired in air at 850°C for 2 hr and sintered in the graphite furnace in flowing He at 1600°C for 2 hr. The grain size was analyzed from SEM micrographs of the polished and thermal etched specimens, according to the linear intercept method. Between 600 and 1000 grains per specimen were counted for the analysis. The thermal etching was performed at 1250°C for 2 hr. Sintering with carbon in He resulted in specimens with a finer microstructure.

Mechanical Properties

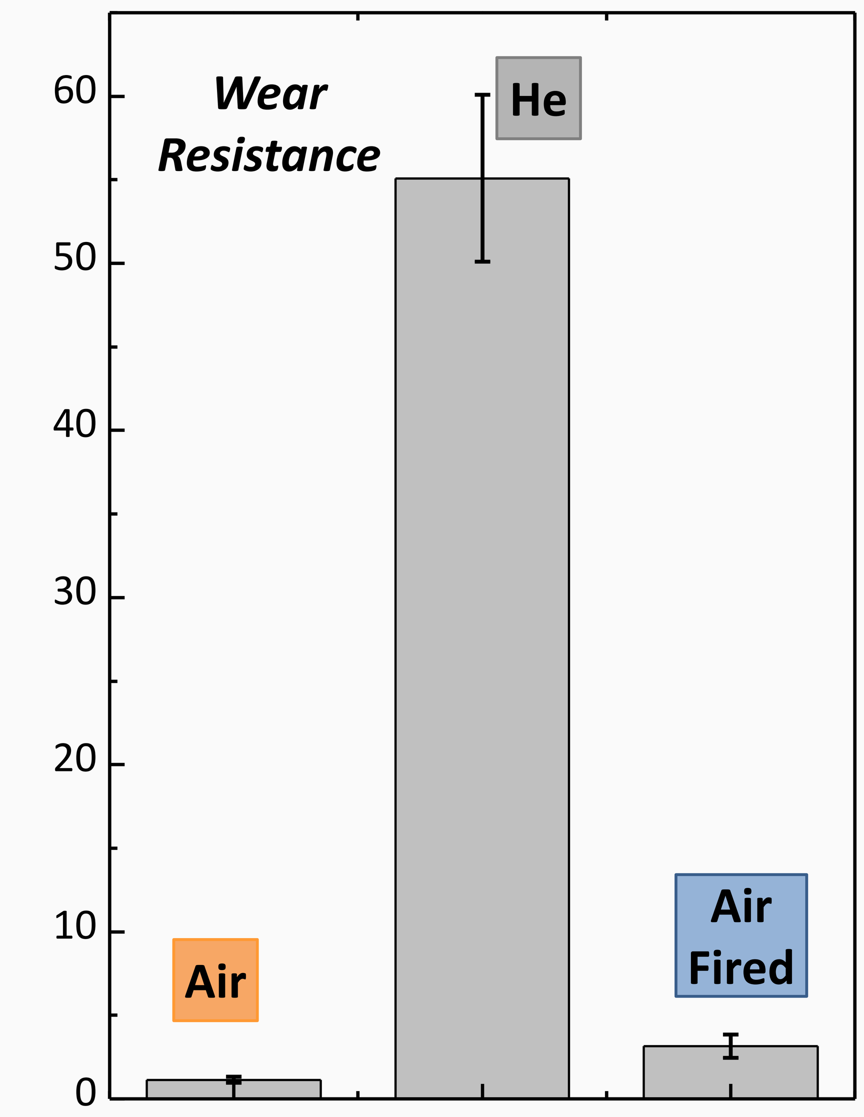

Fig. 5: In order to evaluate the wear resistance of the specimens, diamond-cutting experiments using a diamond wafer blade were conducted. In these experiments, specimens of similar cross sectional area were cut with a diamond wafer blade and the normalized time was evaluated.

Table 1: Flexural strength, Weibull modulus, grain size, and density of RTP alumina sintered in air and in flowing He at 1600°C for 2 hr.

	Specimen	Sintering atmosphere	Flexural strength [MPa]	Weibull	Number of specimens	Grain size [µm]	Rel. Density [%]
Air	Al_2O_3	Air	438 ± 28	15.2	12	5.0 ± 1.0	98
He	C doped Al_2O_3	Flowing He	447 ± 32	13.4	10	1.2 ± 0.2	97

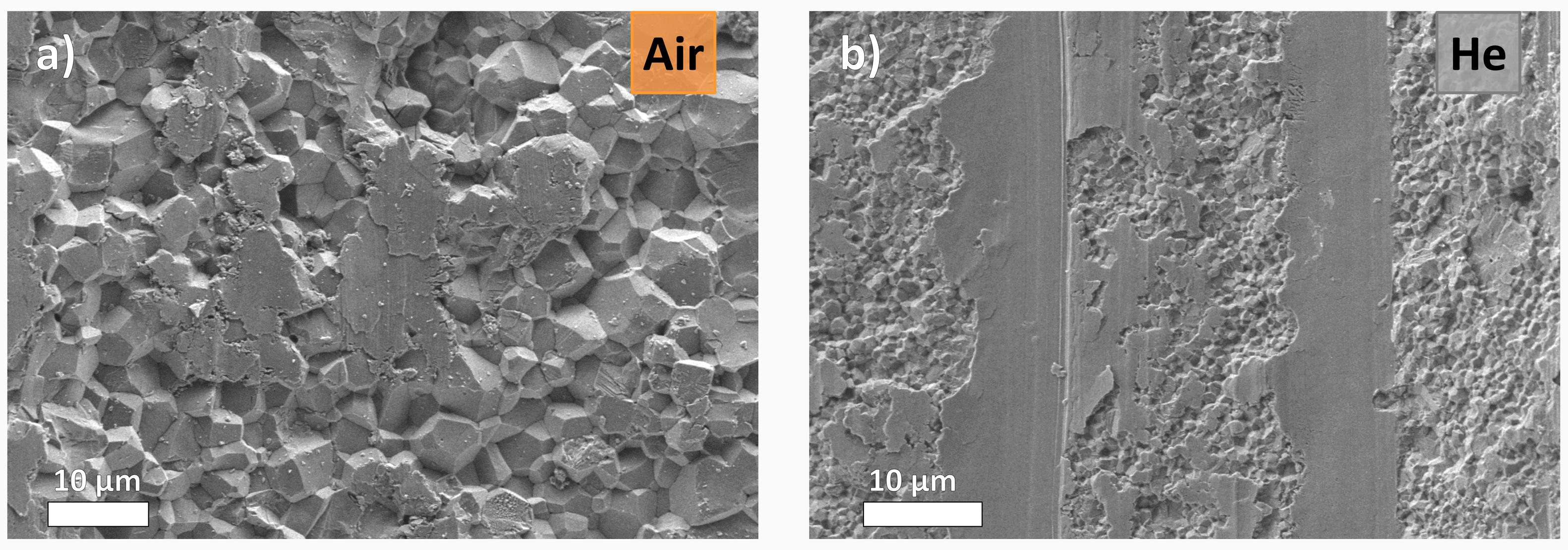

Fig. 6: SEM micrographs of the surface sliced with a diamond saw. (a) alumina and (b) alumina doped with carbon. Pull out is clearly visible.

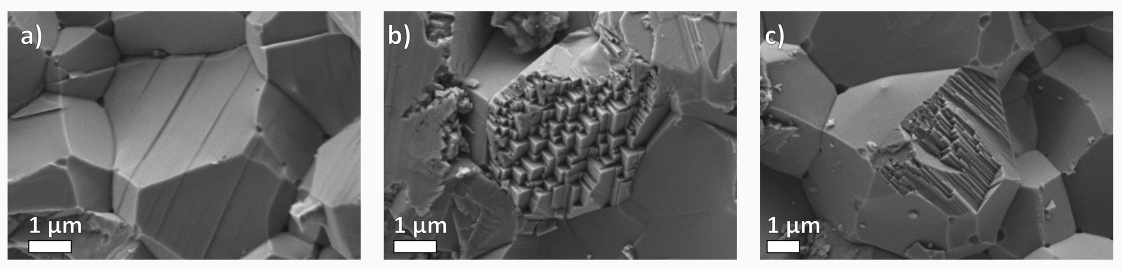

Fig. 7: Higher magnification micrographs of the surface in figure 6 (a).

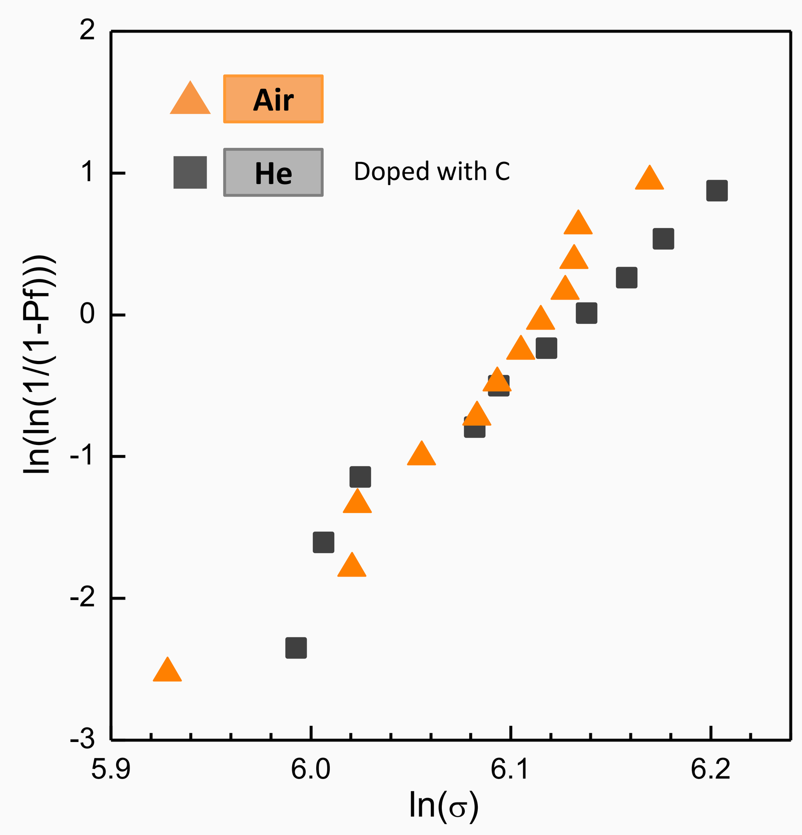

Fig. 8: Flexural strength measurements of specimens sintered in air and in flowing He (carbon furnace). The mean strength and Weibull modulus are listed in Table 1.

Summary and Conclusions

- The **presence of carbon retards grain growth**, most probably by **solute drag**.
- The time to cut (diamond wafering) specimens **containing carbon** was **more than 40 times longer** compared to the specimens sintered in air.
- The combination of flowing He and carbon content has a **positive effect** on the **microstructure** and **wear resistance**.
- No difference in mechanical strength was measured for alumina doped with carbon and alumina without carbon.

References

[1] Liu, X., Fan, Y.-C., Li, J.-L., Wang, L.-J. & Jiang, W. Preparation and Mechanical Properties of Graphene Nanosheet Reinforced Alumina Composites. *Adv. Eng. Mater.* **17**, 28–35 (2015)

[2] Yahya, N. A. & Todd, R. I. Influence of C doping on the fracture mode and abrasive wear of Al2O3. *J. Eur. Ceram. Soc.* **32**, 4003–4007 (2012).

[3] Marquis, E. A., Yahya, N. A., Larson, D. J., Miller, M. K. & Todd, R. I. Probing the improbable: imaging C atoms in alumina. *Mater. Today* **13**, 34–36 (2010).