

Laboratory technology of nanopowder SiC – ceramics for SOFC applications

Introduction

For a long time, obtaining products of complex shape from SiC was complicated by extremely difficult conditions of micro-powder sintering. It was necessary to use hot pressing methods (pressure up to 1000 MPa) at operating temperatures of about 2100°C, followed by finishing with a diamond tool. Low technology and high cost of production of SiC products limited the applicability of this material in these sectors of the national economy. A unique opportunity to sinter high-density silicon carbide ceramics within the framework of ceramic technology was appeared due to reducing the particle size to the nanoscale range.

Obtaining experimental samples of silicon carbide ceramics became possible under laboratory conditions.

Research aims

1. Developing of the unique laboratory technology for production of SiC ceramic s from nanopowder without using of hot pressuring technology.
2. Investigation of the microstructure and elemental composition of the obtained samples.

Methodology

The starting material is a commercial (Saint-Gobain Company) nanoscale powder of silicon carbide α -SiC (Fig.1) with additives of up to 2% by weight of boron nitride (B_4N_3) as dopant (flux). The samples will be made in the form of prismatic bars with a size of 4 x 4 x 30mm. The sample producing technique involves the sequential processing of uniaxial pressure molding ($P = 40\text{MPa}$), high hydrostatic pressure pressing ($HDD = 400\text{MPa}$) and sintering in an induction furnace in argon atmosphere for 1-2h. Values of temperatures of agglomeration for various samples from a series: 1900°C - 2200°C.

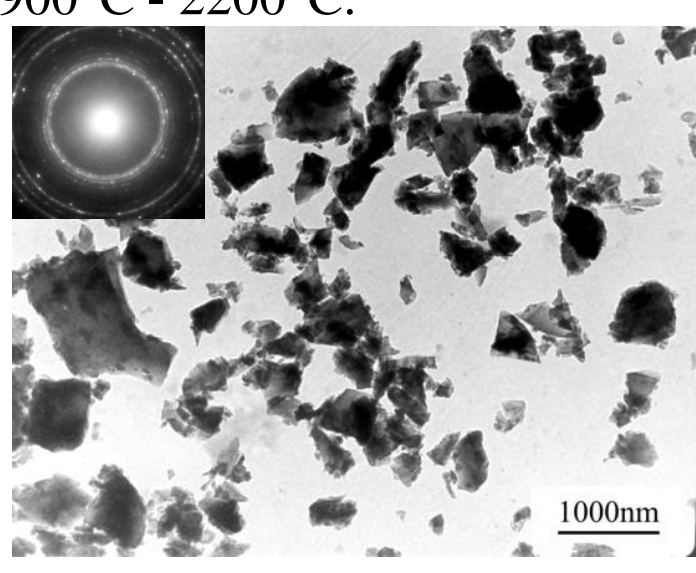
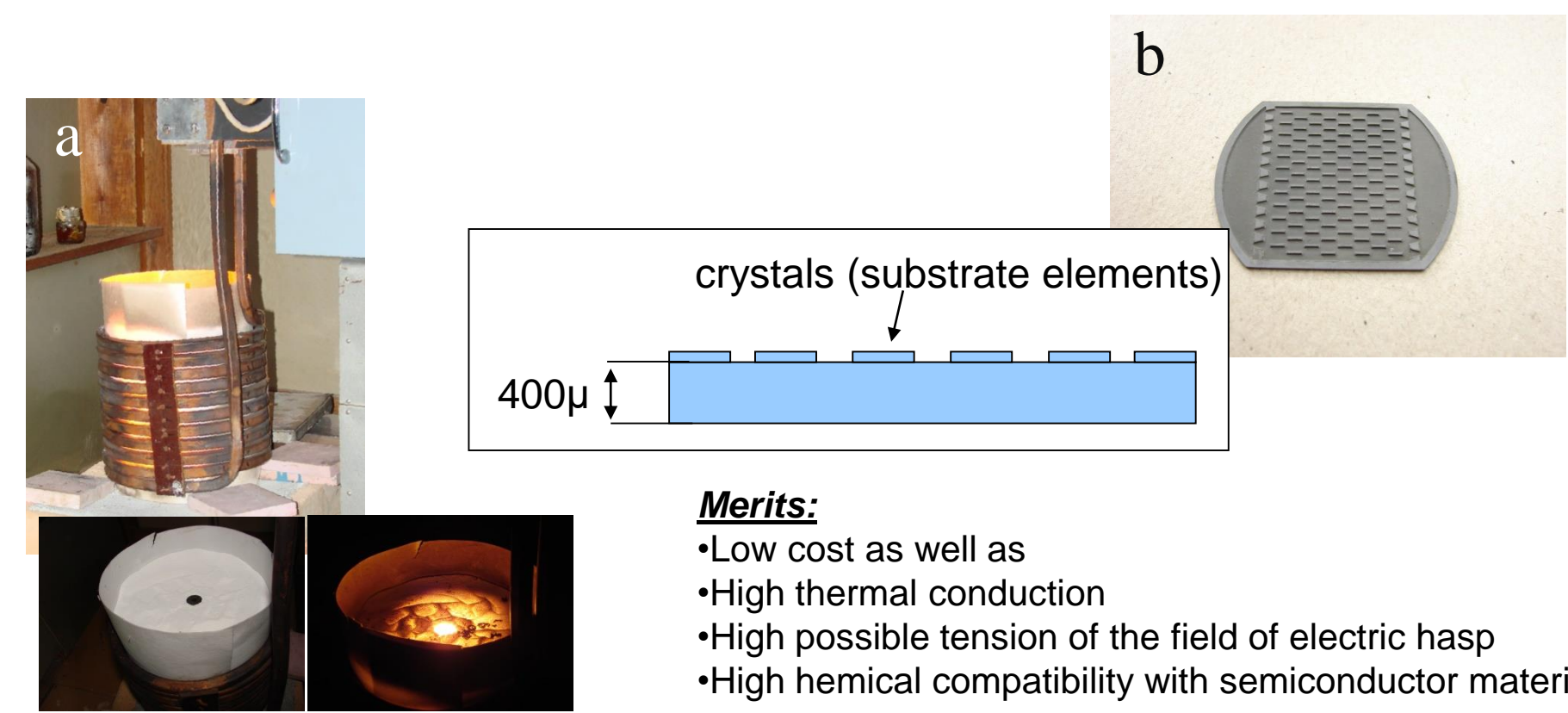


Fig.1 TEM snapshot of powder ready for sintering.

Nanopowders were sintered in laboratory induction furnaces of the VCI-25 type with a volume of 2 litter in the temperature range of 2000-2200°C. The phase composition (XRD), density (gravimetry), microstructure and morphology of the structural elements (SEM, JSM640LV), elemental composition (Rutherford Backscattering Spectrometry RBS). of the samples was studied depending on sintering temperature.

Results

The unique laboratory technology for production of SiC ceramic from nanopowder without using of hot pressuring technology was developed.



Fig/2. Work cell of high-temperature furnace (a) and the sample plate, the SOFS of the heat exchanger (b).

The unique samples with fine features, which is suitable for use as the SOFS heat exchangers was obtained (Fig.2).

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Microstructure

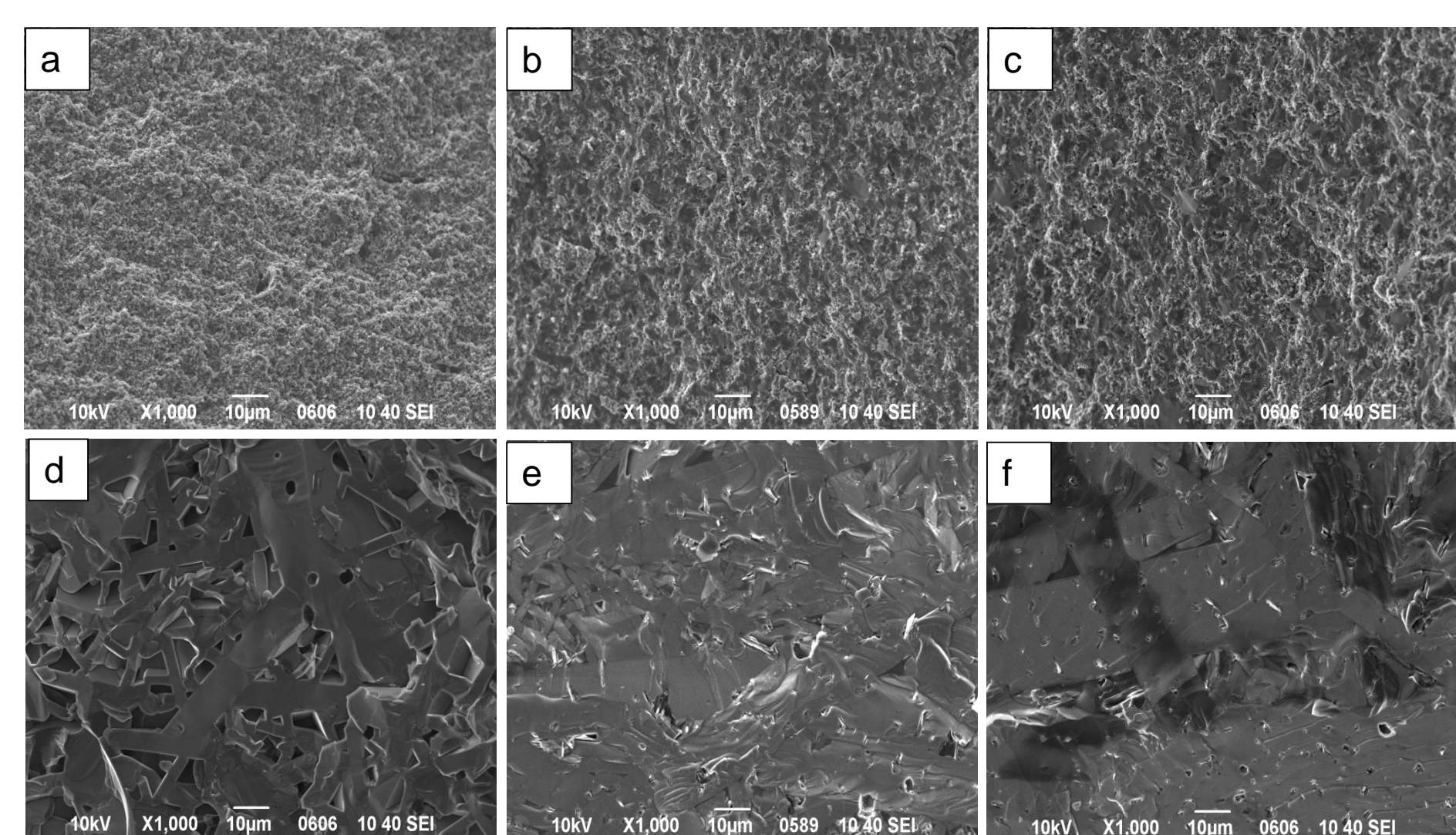


Fig. 3. The topology of fractures of SiC samples obtained at different sintering temperatures: 2000°C (a), 2050°C (b), 2080°C (c), 2100°C (d), 2150°C (e), 2200°C (f), 1h.

Elemental composition

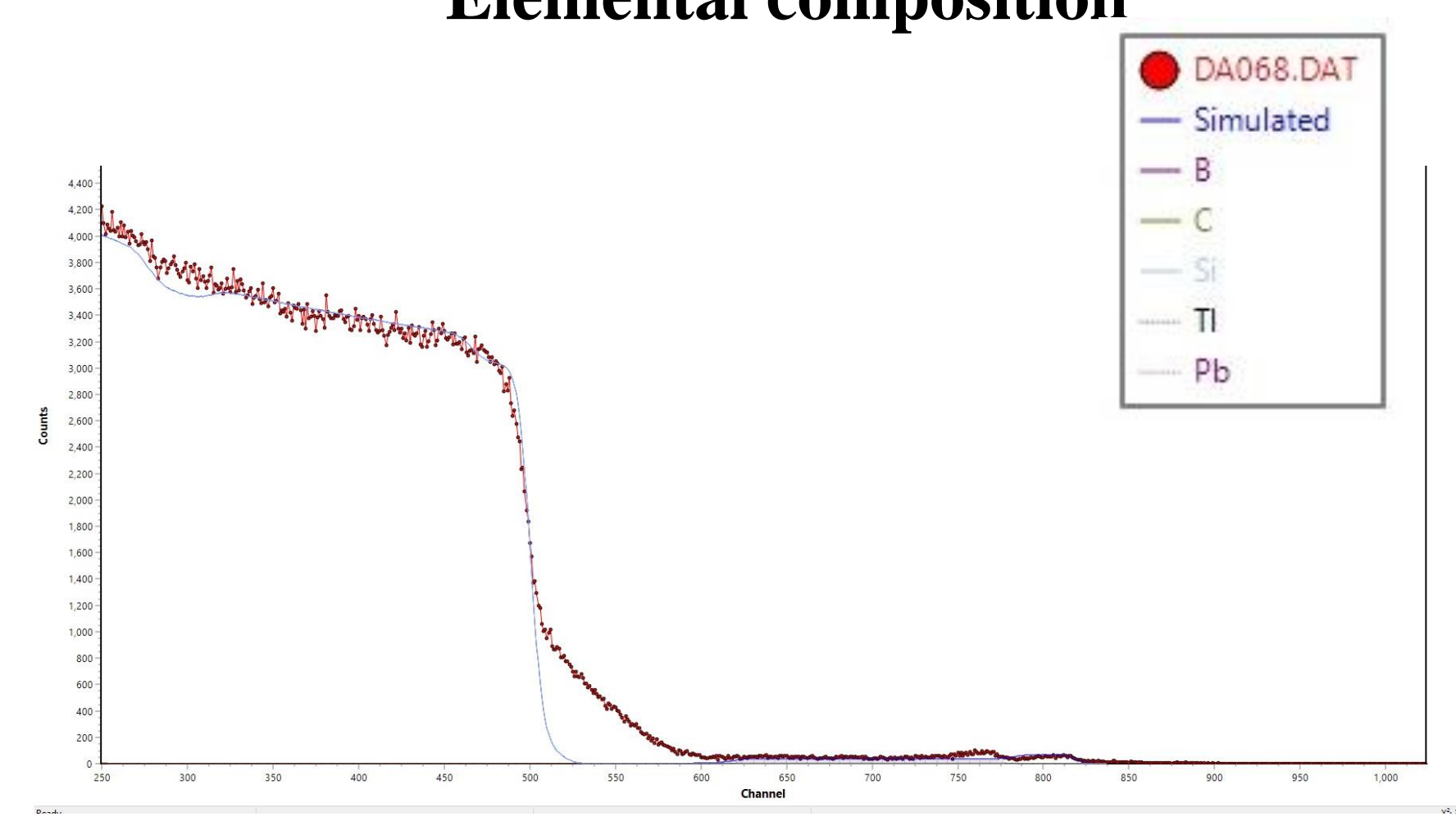


Fig. 4. Measured by RBS method and calculated by Simnra 7.02 concentration profiles of nanosized SiC ceramics// Energy 2000 KeV, incident geometry angle – 60 deg, energy per channel – 1,9845keV/ch)

Conclusions

1. A new laboratory facility for sintering SiC nanopowders using ceramic technology has been developed.
1. The new technology for sintering of SiC ceramic with a density of at least 98% of the theoretical [1] without using hot pressing has been developed.
2. The test samples of complex shapes were synthesized and studied.
3. Insignificant (about 0.01% at.) impurities of boron and heavy elements (Pb, Ti) were detected.

[1] O. S. Doroshkevych, A. V. Shylo, O. V. Saprukina, I. A. Danilenko, T. E. Konstantinova, L. A. Ahkzov Impedance Spectroscopy of Concentrated Zirconia Nanopowder Dispersed Systems Experimental Technique // World Journal of Condensed Matter Physics. - 2012. - №2. - C.1-9. doi:10.4236/wjcmp.2012.21001