Ultra-high-temperature cell for toroid-type high-pressure apparatus. Melting of B_4C and SiC under pressure

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Boron carbide (B_4C) and silicon carbide (SiC) are superabrasives with outstanding mechanical properties, low density and high chemical resistance that have a wide rang of industrial applications. They are also used as refractory materials due to their high melting temperatures and superior thermal stability. At ambient pressure the melting temperatures of B_4C and SiC may be evaluated as 2720±50 K and 3100±40 K, respectively. However, in the literature there is no data on their melting at high pressures. In the present study the pressure dependencies of melting temperatures of boron carbide and silicon carbide were investigated for the first time at pressures up to 8 GPa..

The experiments were conducted in a specially designed high-temperature (up to 3400 K) cell (Fig. 1a) of toroid-type high-pressure apparatus at LSPM–CNRS. The cell was pressure calibrated at room temperature against phase transitions in Bi (2.55 and 7.7 GPa), PbSe (4.2 GPa), and PbTe (5.2 GPa). The temperature calibration under pressure was made using well-established reference points: melting of Si, NaCl, CsCl, Pt, Rh, Mo, Al_2O_3 and Ni–Mn–C ternary eutectic. In the case of Pt, Rh, Mo and Al_2O_3, the melting points were found in series of quenching experiments from the change of shape and microstructure of the reference material pressed into a cylinder of graphite-like boron nitride, while in the other cases the melting temperature was fixed in situ from the electrical resistance jump in the cell (the reference material was in a direct contact with a heater). In the pressure range under study the dependencies of temperature in the center of the cell on the electric power input are almost linear (Fig. 1b). The line slopes decrease from 2.0 K/W at 2.55 GPa to 1.5 K/W at 7.7 GPa due to an increase of heat losses with increasing pressure. The error of the temperature evaluation in the 2400-2800 K range is ±60 K, and according to the finite element method calculations of temperature fields in the framework of the stationary thermal conductivity problem, the temperature gradients in the sample do not exceed ~15 K/mm in the radial and ~10 K/mm in axial directions.

The melting of both carbides in the 2.5–7.7 GPa pressure range was studied by quenching. The isothermal holding time at a desired pressure was 60–90 s, the cooling rate at the initial stage after switching-off the power was ~300 K/s. No signs of chemical interaction between carbides and boron nitride capsule were observed over the whole pressure – temperature range under study.

Both melting curves have negative slope i.e. –13±6 K/GPa for B_4C and –44±4 K/GPa for SiC that points to a higher density of carbide melts as compared with the solid phases in the pressure range under study.

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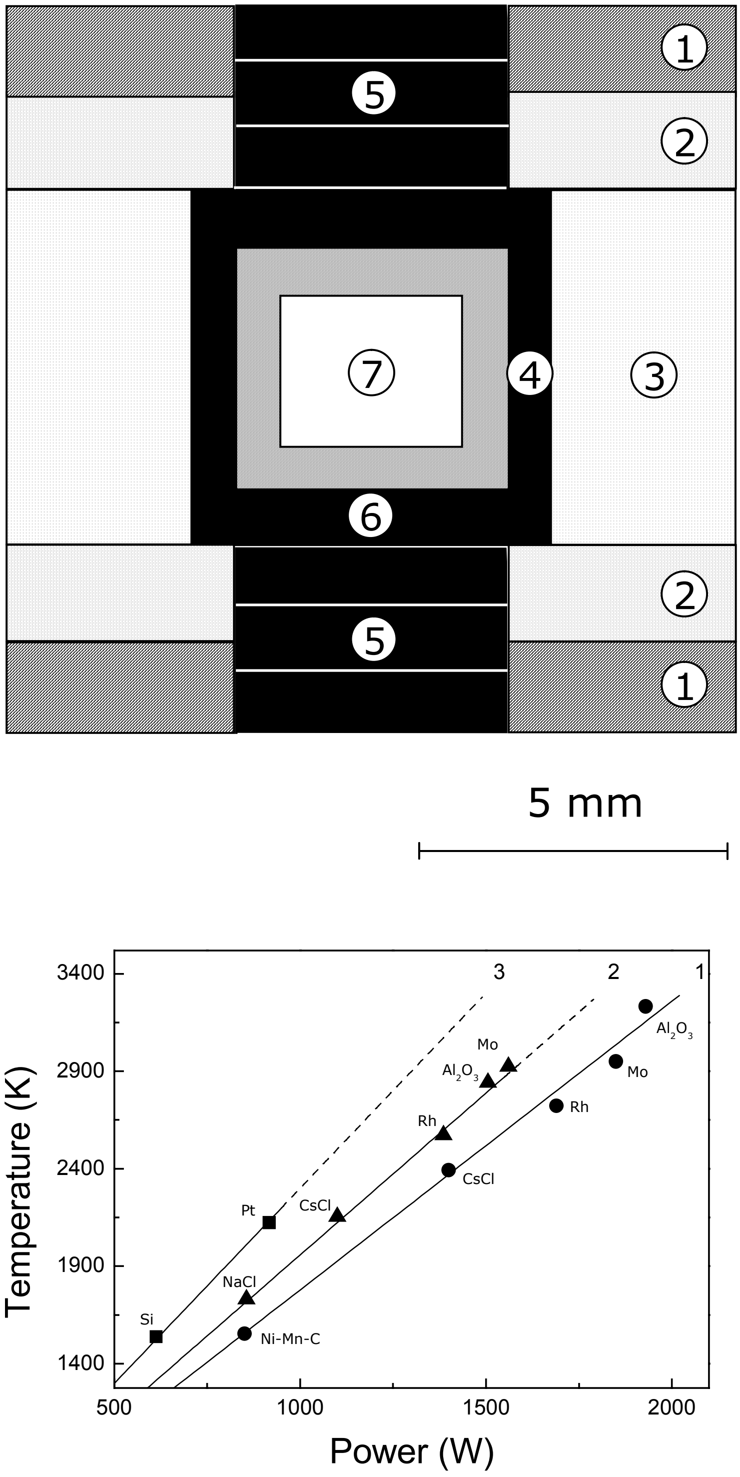

Figure 1.

(*a*) High-temperature cell of the toroid-type high-pressure apparatus: (1) pyrophyllite ring; (2) ring of pressed fianite (-100 µm); (3) sleeve of pressed fianite (100–200 µm); (4) cylindrical graphite heater; (5) discs of pressed Ceylon graphite; (6) boron nitride capsule; (7) sample.

(*b*) Temperature in the center of high-pressure cell *vs* electric power at 7.7 (1), 5.2 (2) and 2.55 GPa (3).

References :

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