Sensing Properties of Nanocrystalline Silicon Carbide in Wet Condition

A. Gaiardo1, 2, B. Fabbri1, M. Valt1, G. Pepponi2, C. Malagù1, A. Giberti3, P. Bellutti2, V. Guidi1
1 Department of Physics and Earth Science, University of Ferrara, Via Saragat 1/c, 44122 Ferrara, Italy
2 MNF - Micro Nano Facility, Bruno Kessler Foundation, Via Sommarive 18, 38123 Trento, Italy
3 MIST E-R s.c.r.l., Via P. Gobetti 101, 40129 Bologna, Italy
Corresponding author’s e-mail address: andrea.gaiardo@unife.it

Abstract
Silicon carbide is a well-known material with high thermal and chemical stability. In this work, we present an investigation on the chemoresistive properties of nanostructured Silicon Carbide (SiC). A commercially available nanopowder of silicon carbide was first purified and then its morphology, structure and thermal stability was characterized. Afterwards, the powder was mixed with suitable organic vehicles and screen-printed onto alumina substrates. SiC thick films were tested as chemoresistive gas sensors in thermo-activation mode. In this way, they were exposed to 13 gases belonging from different chemical classes. The sensing characterization showed that this semiconductor is an extremely selective functional material for the detection of sulphur dioxide (SO2) at temperature higher than 600°C, useful for harsh environments. Also, the sensing properties of SiC sensors were strongly improved by the presence of humidity. The sensing mechanism was also investigated and a possible interpretation has been proposed.

Key words: Chemoresistive gas sensors, nanostructured silicon carbide, sulphur dioxide, high selectivity, wet condition.

Introduction
Silicon carbide is a long-time known material, massively produced starting from 1890. Ceramics obtained by sintering SiC grains are very robust, then they find employment in car brakes, bullet-proof vests and in general in high endurance applications [1]. SiC thermal strength is also extraordinary, it holds stability even above 1000°C, making it possible high-temperature applications (SiC melting point is above 2500°C at 35 atm). Electronic applications of SiC are light-emitting diodes and high-temperature and/or high-voltage devices, as JFETs and MOSFETs rated at 1200 V [2]. Encouraged by the diverse fields in which SiC can be applied and the possibility to use it in form of a nanosized powder, we decided to investigate its possible chemoresistive properties for gas-sensing applications. There are no studies about chemoresistive properties of SiC nanoparticles at the best of our knowledge, apart from our previous study [3].

Experimental
First, commercial nanostructured SiC was purified by means of subsequent cycles of powder, then it was characterized from the morphological, thermal and structural point of view. These characterizations highlighted the high chemical purity and the nanometric size of the SiC powder (Fig. 1). Furthermore, the structural characterization showed the presence of one predominant crystalline phase in the SiC sample. Afterwards, the material was screen-printed onto alumina substrates and tested as chemoresistive gas sensor. The electrical characterization of the SiC sensors was carried out in a test chamber by means of the flow-through technique. SiC thick films were exposed to 13 different gases, and the responses of the sensors were investigated at operating temperatures ranging from 250 °C to 800°C. The measurements have been performed both in dry and wet conditions. The most interesting result was that SiC sensors proved to be insensitive to almost all gases analysed, while showed a significant response to sulphur dioxide in the concentration range of some ppm, in dry and in wet conditions (Fig. 2).
Fig. 1. SEM Image and EDX analysis of SiC nanopowder.

Fig. 2. The sensing selectivity of SiC films to SO\textsubscript{2}, in presence of humidity.

The presence of the humidity increased the stability and the response of the SiC films vs. SO\textsubscript{2} (Fig. 3).

Fig. 3. Comparison of SiC gas sensing responses, vs. SO\textsubscript{2}, in dry air, RH=33% and RH=60%.

The calibration curve of SiC thick film vs. SO\textsubscript{2} concentrations highlighted a nonlinear correlation, similar to the trend of common metal oxides. The detection limit was about lower than 1 ppm of SO\textsubscript{2} (Fig. 4). Further investigations highlighted that the interesting sensing property of SiC films was due to the formation of a SiO\textsubscript{2}/SiC core shell, starting from a temperature of 600°C (Fig. 5).

Fig. 4. Sensitivity of the SiC thick film vs. SO\textsubscript{2} concentrations.

Indeed, the formation of this core shell increased the reactivity of SiC films.

Fig. 5. XPS analysis of SiC nanopowder before the heat treatment (red line) and after 2 hours at 650°C (green line) and at 850°C (black line).

Finally, a gas sensing mechanism has been proposed. With this aim, exhaust gases of the reaction between SO\textsubscript{2} and SiC films at 650°C were analysed by means of PTR-MS technique, to investigate the possible chemical reaction. Applications of such a sensor could span many fields, since sulphur dioxide plays an important role in air pollution, industrial processes and winemaking.

References

