Plasma treatment of Silicon Carbide: preliminary experimental results

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Introduction

Silicon carbide (SiC) is one of the best materials for advanced applications in several industrial sectors thanks to its outstanding properties, such as high hardness and strength, excellent corrosion/oxidation resistance, good high-temperature strength, and high thermal conductivity; it is then an optimal choice for many high added values applications, for instance diesel particulate filters, measurement instruments, ceramic membranes, heating elements, and catalytic support products – among many others. In addition, the new frontiers of additive manufacturing (AM) potentially promise to boost the application of ceramics, and SiC among the others, in many industrial fields. Basically, silicon carbide powders can be produced by pyrolysis of silane compounds, direct carbonisation of Si metal, and carbothermal reduction of SiO_2. Additionally, the production of SiC particles of nanometric dimensions is particularly important because it allows the development of technical applications with improved mechanical properties. For this reason, thermal plasma processing technique has gained more and more attention for the preparation of submicronic and nanosized SiC powders, starting from different types of precursors, such as SiO_2 /CH_4, Si /CH_4, coarse SiC, or using plasma source like transfer arc, DC or RF thermal plasma. In this frame, ENEA developed a prototypal plant for the production of powders for AM. The system based on DC thermal plasma technology was own designed and installed at ENEA Portici Research Centre. In this poster we will present the experimental work carried out on coarser angular silicon carbide powder with the aim of exploring and identifying the best process parameters to produce submicronic powders. The powders were analyzed thoroughly with respect to the morphology, the crystalline phase and the further processability in view of tailored industrial applications.

Experimental

Commercial SiC powders supplied by Kyocera, type StarCeram S Raw UF15 (d_{50}=0.75 micron), were used for experimental test. The raw powders were processed by a DC thermal plasma plant installed at ENEA Portici Research Center composed by a jacketed-cylindrical stainless steel reactor of 13 cm inner diameter, and 185 cm length cooled with circulating cold water. The reactor is equipped with a collection tank, where the produced powders are collected. The powders were fed directly by an injector (d=2.0mm) using argon (Ar) as carrier into plasma flame at the top of the reactor and their mass flow was changed by a GTV PF 2/2 feeder.

The plasma jet was carried out by a nozzle (d=6mm) using argon (Ar) as main gas to light the plasma and helium (He) as secondary gas to improve the flame conditions.

The identification of the crystalline phases was performed by Malvern Panalytical XRD X'Pert while the morphological analysis was with LEO 1530 SEM.

Table 1 shows the best experimental conditions for SiC powder tests.

Test code	Power (kW)	Ar (slm)	He (slm)	Ar carrier (slm)	Feeding rate (g/min)
PL5	20	40	10	5	1
PL6	20	40	10	7	3
PL7	20	50	10	5	3
PL8	15	40	10	3	3
PL9	14	40	10	3	3
PL26	11	40	10	2,5	5

Tab 1 : experimental conditions for SiC powders

Results and Discussion

During the process, the high processing temperature partially vaporized all reactants, resulting in the formation of nanoparticles which deposited on the reactor wall together with the biggest particles, according to the power of the plasma torch. All tests showed that the formation of nanosized SiC already occurred at 14 kW although starting material residue was still present, as showed in Fig.1.

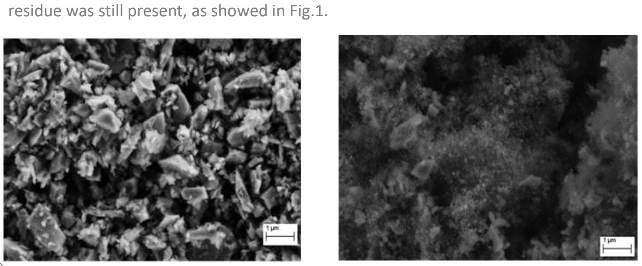

Fig.1 – SEM images on powders before (left) and after (right) plasma treatment (PL9)

The power affected the particle size distribution and the reactivity of the powders, so at the highest power, the test produced nanoparticles below 200 nm with no starting material, as showed in Fig.2.

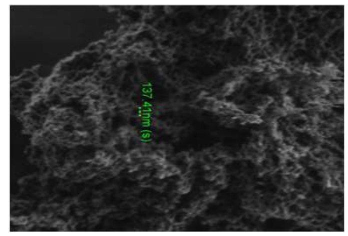

Fig.2 –SEM images on powders after plasma treatment (PL5)

The main phase of all treated powders was the alpha SiC, similarly to the starting material, as showed in Fig.3. The presence of weak free silicon peaks (2θ= 28.48, 47.3, 56.08, 69.12) could be due to the dissociation of the SiC at the highest power during the plasma treatment. The formation of silica (SiO_2) is also detected (shoulder about 2θ=22) due to post oxidation into the reaction chamber.

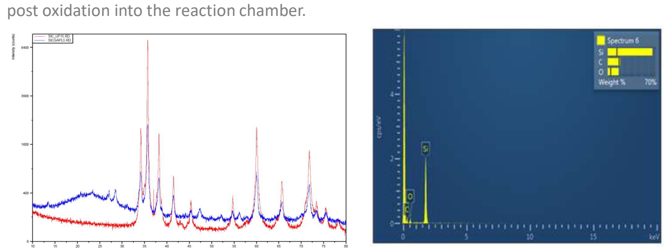

Fig.3–XRD on powder before (red line) and after plasma treatment PL5 (blue line); EdX PL5

The further test were performed changing the process conditions until obtaining a powder with low Si and without SiO_2 as showed in Fig.4.

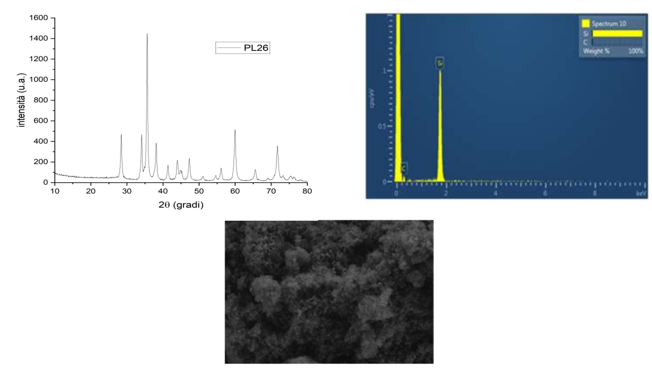

Fig.4 – XRD PL26 (left), EdX PL26 (right) and SEM PL26 (under)

PL26 powder shows the absence of Si and SiO_2 peaks in the XRD, followed by the absence of oxygen in the EdX .

Conclusions

The experimental tests conducted on SiC powder, using a DC thermal plasma plant installed at ENEA Portici Research Center, have shown that this technology can be considered as one of the most promising methods to produce nanosized SiC for industrial applications. All samples showed the formation of nanoparticles but the higher the power the more SiC dissociation. The best results were obtained under lower power torch and resolving the oxidation problem. The analyzes of the morphology and the crystalline phase were performed to explore the final industrial application but further tests are definitely necessary.

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