SI: Supporting Information

SI-1: Electron tomography study of the selective localization of Fe₃O₄ nanoparticles

inserted in the porosity of SiC through an impregnation process using ethanol as solvent.



Fig. SI 1: (A) Bright field TEM image from the tilt series used to reconstruct the volume of the chosen fragment of the Fe_2O_3/SiC sample. (B), (C) Typical slice and closer view, extracted from the reconstruction, showing the preferential location of the particles inside the R-pores.

SI-2: Analysis of the reconstructions obtained by electron tomography at the SiC surface

A detailed analysis of the SiC reconstructions obtained by electron tomography in BF mode suggests the existence of a difference in chemical composition between the R-pore and C-pore surfaces. Indeed, a relatively thick fringe-like contrast can be observed at the surface of the chosen fragment of SiC (Fig. SI 2). Its origin is certainly not the residual Fresnel diffraction contrast due to the acquisition of the initial images in bright-field mode, because this fringe is present only at the surface defined by the C-pores and not at the surface of the R-pores as can see from the figureSI4. This difference in contrast between the C-pores and the R-pores surfaces can be related to a difference in oxide layer thickness. Its presence especially at the C-pore surface suggests a difference in oxidation rate between the surfaces proposed by the two types of pores, in agreement with the previous hypothesis.



Fig. SI 2: (A) One of the classical TEM images used to compute the 3D reconstruction of the Pd/SiC system (the Pd nanoparticles were deposited by the incipient impregnation method using the ethanol as solvent). (B) Example of slice extracted from the reconstruction suggesting the presence of a new phase with a different chemical composition at the external surface (corresponding to the C-pores).

SI 3: 2D chemical composition analysis of a SiC grain: illustration of the signal extraction procedure for: a) oxygen from a STEM-EELS spectrum; b) a typical C elemental map from the EFTEM tilt series.



Fig. SI 3a: Extraction of the O elemental signal for the SiC grain presented in A), which was analyzed in STEM-EELS along the green line drawn on the STEM-HAADF image. The EELS spectrum is shown in Fig. B and the extracted O signal in Fig. C.



Fig. SI 3b: (A) Typical image recorded in the Zero Loss mode on the SiC grain selected for the analytical tomographic analysis. (B) EELS spectrum recorded on the whole object, illustrating the C K-edge (at 284eV) and O K-edge (at 532eV), as well as the characteristics of the energy windows used to acquire the tilt series of filtered images for the carbon. (C) Corresponding filtered images recorded at the K-edge of the carbon at a given tilt angle. (D) Elemental projection of the carbon obtained from the three filtered images, after the background extraction using the three-window method. The scale bar is 100 nm.

SI 4: TEM analysis of the filling of a C-pore by the SiO₂ after an oxidation process at

high temperature



Fig. SI 4. TEM micrograph showing a C-pore, localized between two SiC islands, plugging by the formed SiO_2 phase after air oxidation at 1000°C.

The 2D-TEM micrograph of the oxidized SiC(ca. 10 wt.% of SiO₂)is presented in Figure SI4. The TEM micrograph clearly shows the presence of the SiO₂ phase within the C-pore channel bridging the two SiC particles. Statistical TEM analysis indicates that a large part of the SiO₂ phase was grown up from the C-pore surface. Such phase formation was expected to be responsible for the pore plugging and for a significant specific surface area loss of the SiC material.