Mesoporous silicon carbide via nanocasting of Ludox® xerogel

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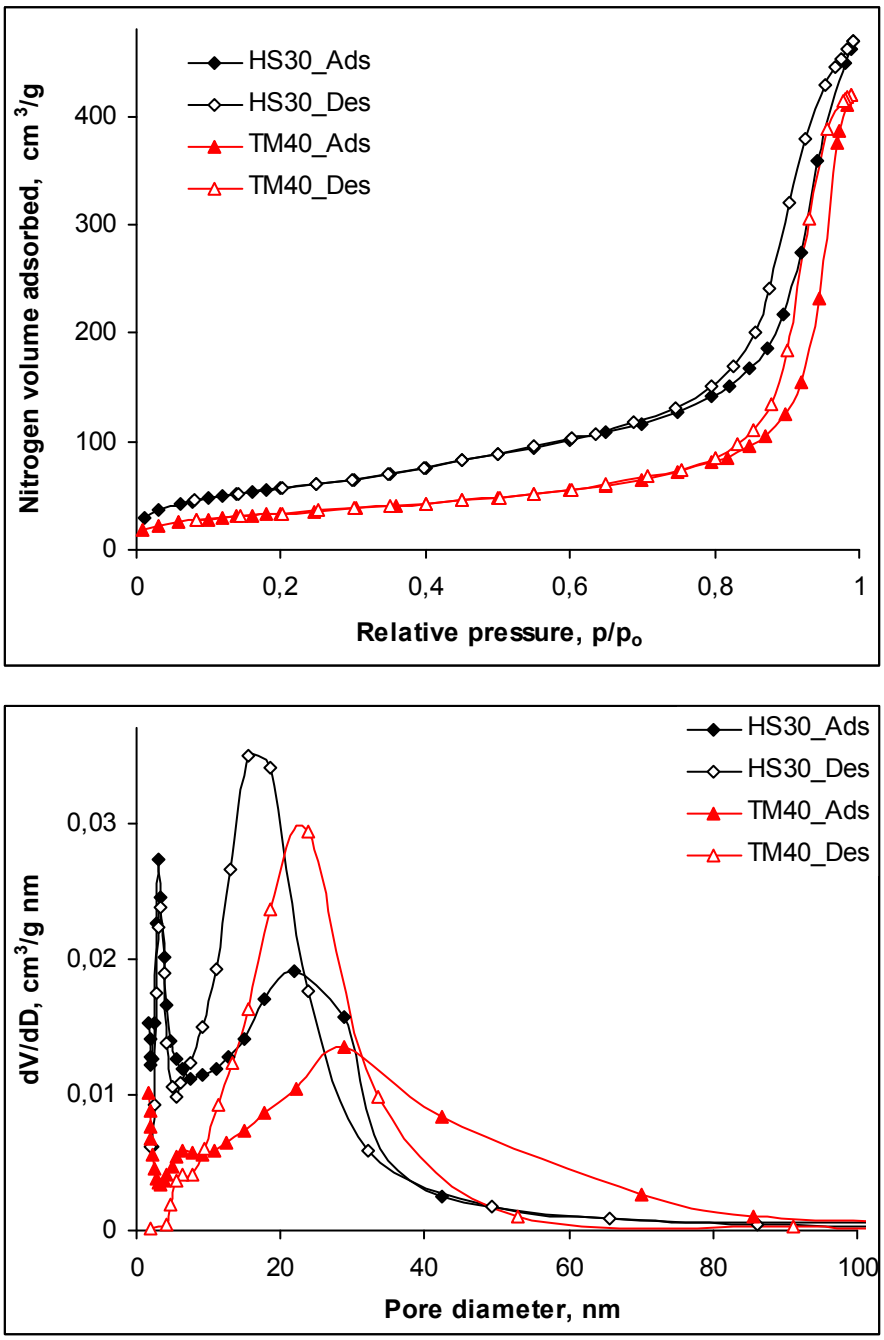

Fig. S1. Nitrogen physisorption isotherms (a) and BJH pore size distributions (b) of Ludox silica xerogels.

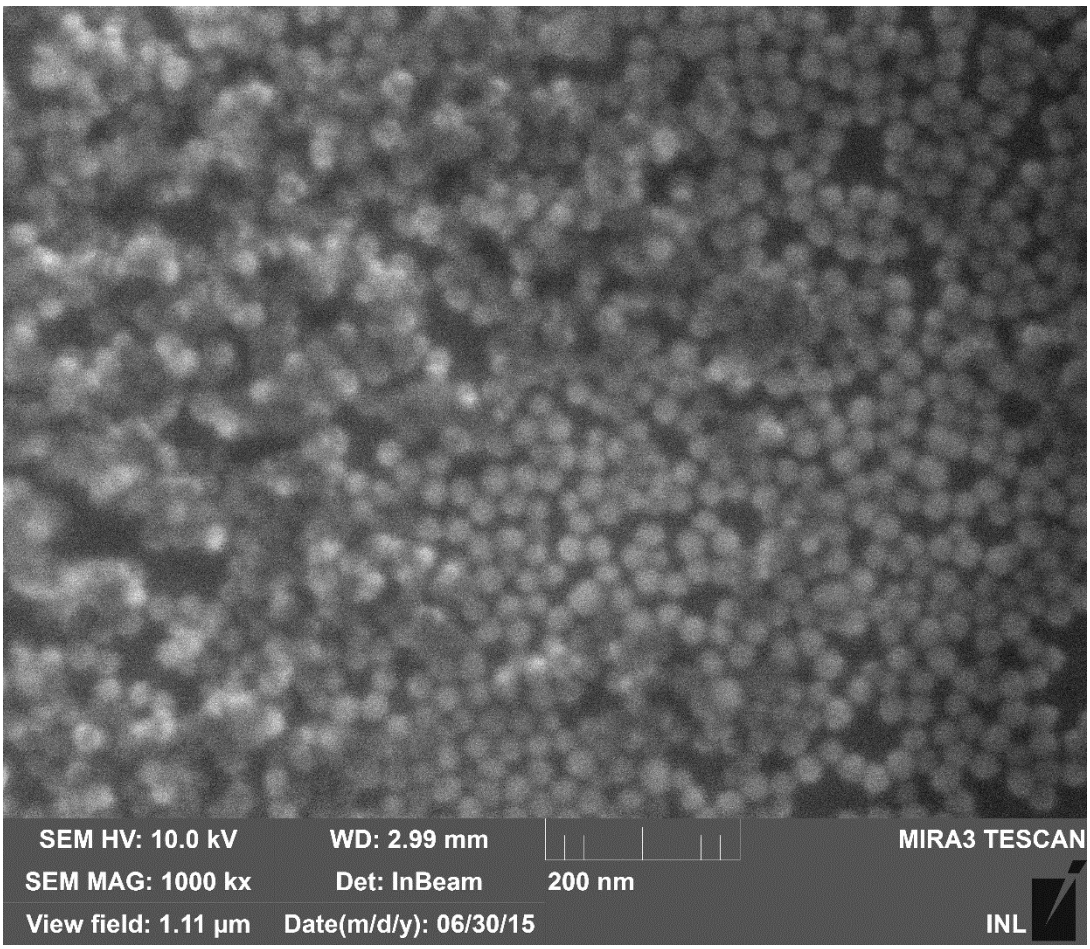

Fig. S2. SEM image of Ludox TM40 xerogel.

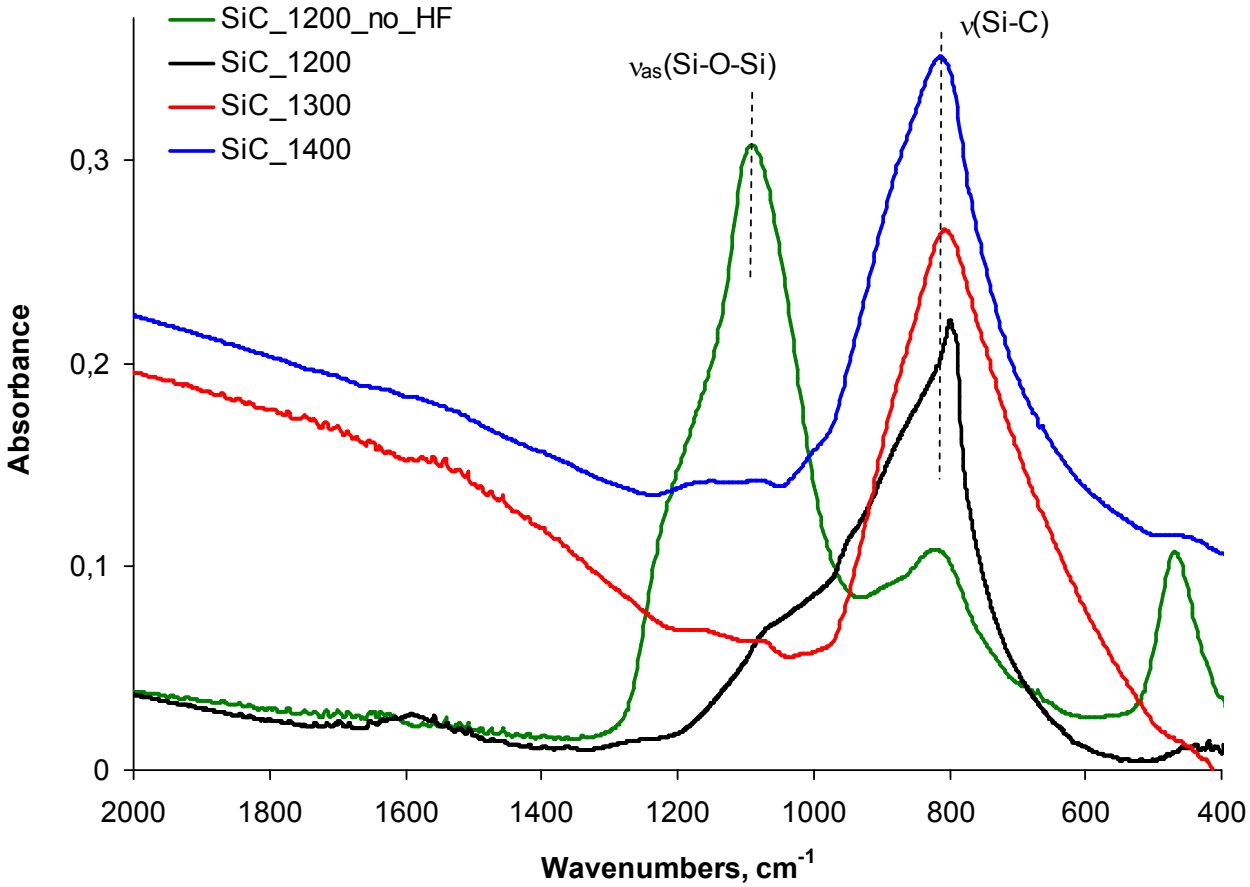

Fig. S3. Absorbance FTIR spectra for the series of SiC-HS-0.6 samples, prepared at different temperatures, before (SiC_1200_no_HF) and after SiO_2 template leaching.

An intense ν(Si-C) band at 805 cm^{-1} in the spectra of leached samples is typical for mesoporous SiC; the completeness of the SiO_2 template removal is confirmed by disappearance of ν(Si-O-Si) band at 1100 cm^{-1} after HF treatment.

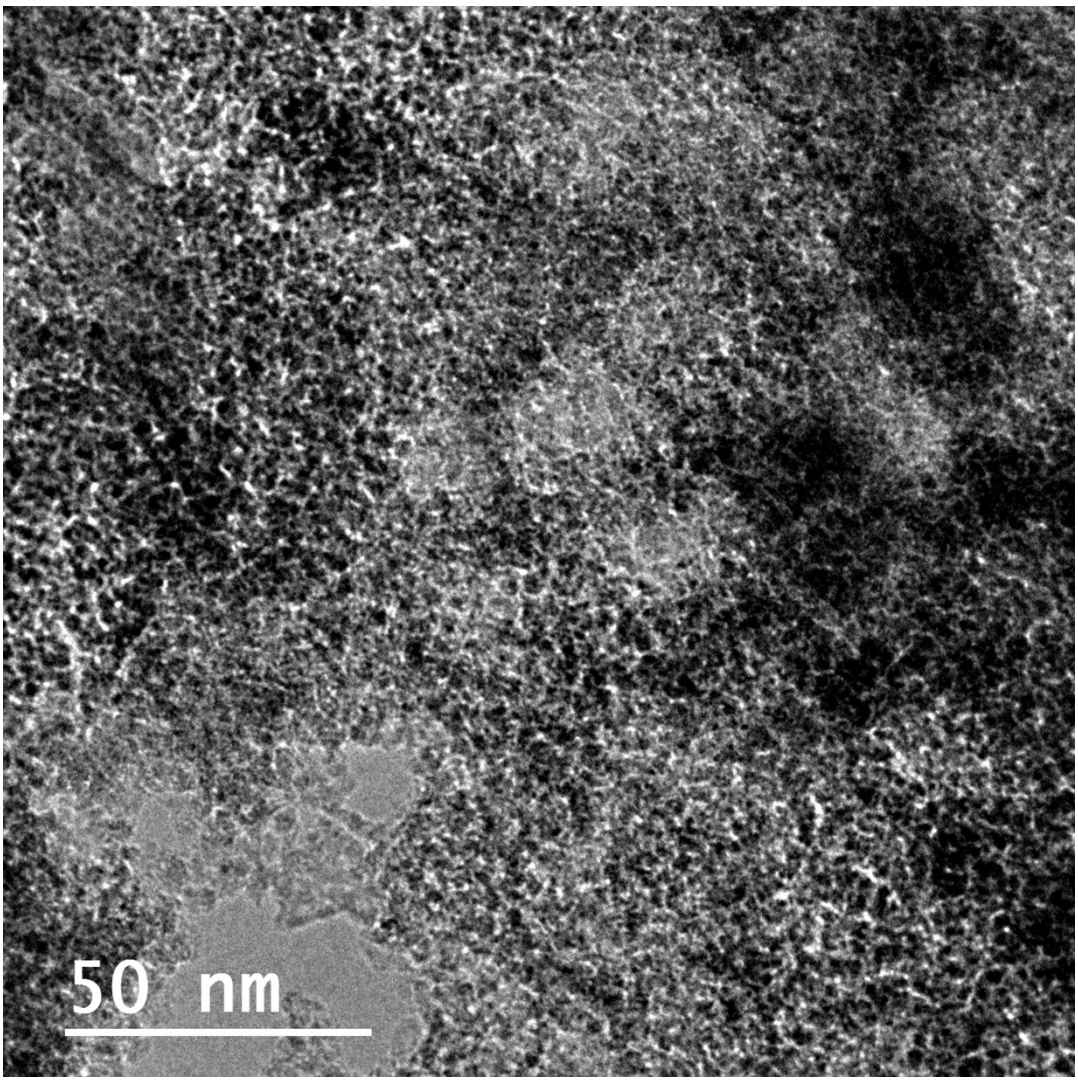

Fig. S4. TEM image of SiC-TM-1200-0.7 sample. SiC pore walls are composed of 2-10 nm crystallites.

Table S1. Parameters of powder XRD (022) peak for SiC-HS-0.6 series and por-SiC after thermal oxidation and HF wash treatments.

Sample	τ_1, nm	τ_2, nm	S_1, %
SiC-HS-1200-0.6	25.9	1.97	10.6
SiC-HS-1300-0.6	20.3	2.03	13.1
SiC-HS-1400-0.6	27.3	2.28	14.9
SiC-1200-O2_HF	23.0	1.98	13.8
SiC-1300-O2_HF	23.9	2.03	20.6
SiC-1400-O2_HF	23.1	2.19	20.9

τ_1 is the crystallite size of narrow Lorentzian peak component estimated by Scherrer equation;
τ_2 is the crystallite size of wide Lorentzian peak component estimated by Scherrer equation;
S_1 is the area of narrow Lorentzian component related to overall peak area.

The size of large crystallites is independent on the pyrolysis temperature within the experimental error. The size of small crystallites demonstrates a slight growth with the PT increase. Fraction of the large crystallites which is determined by S_1 parameter rises significantly with the PT as well after the oxidation/washing cycle. This fact demonstrates oxidation instability of small crystallites.

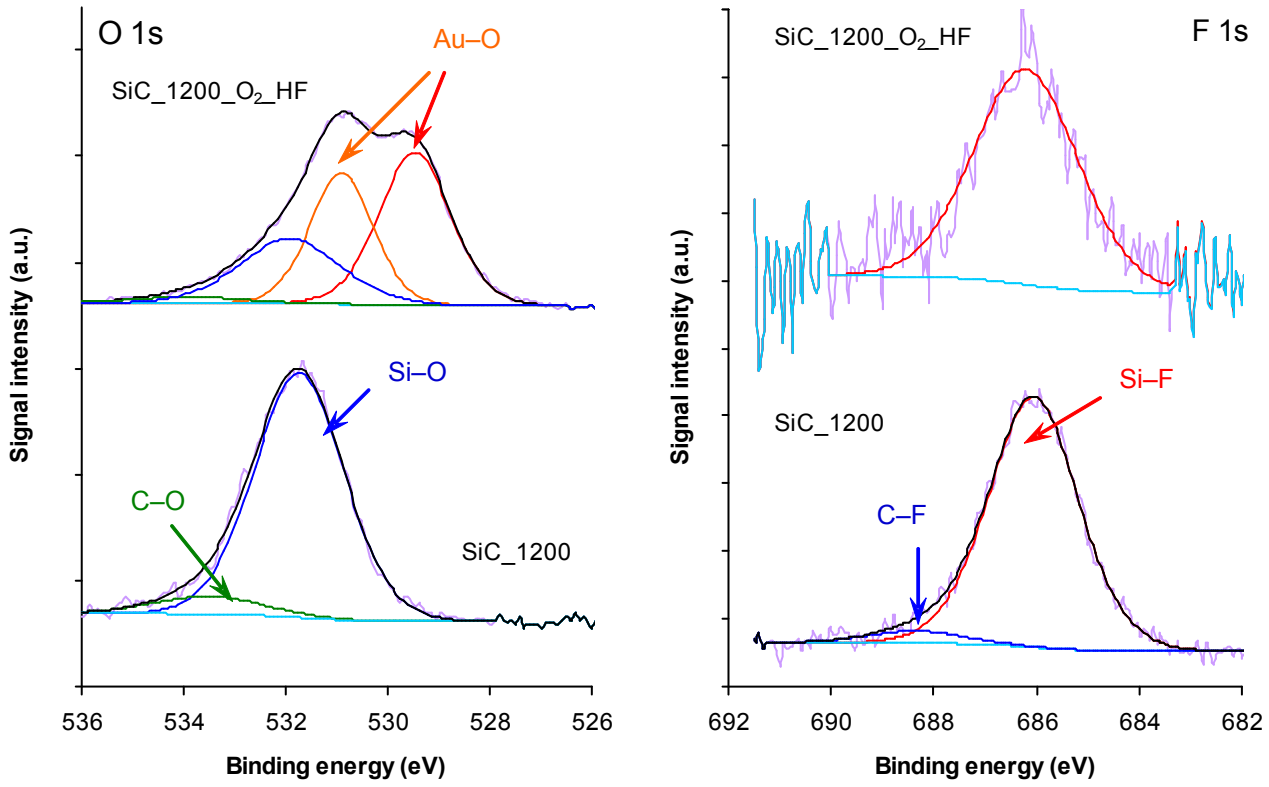

Fig. S5. XPS spectra (O 1s and F 1s) of SiC-HS-1200-0.6 and SiC-1200-O_2_HF samples.

The lines are presented mainly by Si–O (531.7 eV) and Si–F (686.0 eV) components. The components of O 1s line at 530.7 and 529.3 eV arise most probably due to the oxygen, chemisorbed on gold XPS support

Table S2. Chemical composition of SiC-HS-1200-0.6 and SiC-1200-O_2_HF samples derived from XPS (standard RSF values were used).

	Si	C	O	F
SiC-HS-1200-0.6	29.3	45.8	17.6	7.3
SiC-1200-O_2_HF	43.6	49.1	6.1	1.1

Table S3. Textural characteristics of por-SiC obtained from 22 nm silica xerogel template, PCS: SiO_2=0.7 by weight with addition of $Ni(acac)_2$. The samples are indexed as SiC_T_X_Ni, where T is the pyrolysis temperature, X is the initial Ni : PCS ratio (% wt.).

Sample	S_{BET}, m^2 g^{-1}	$V_{pore,}$ cm^3 g^{-1}	D_{ads}, nm	D_{des}, nm	D_{BET}, nm	h, nm	τ, nm
SiC_1200	438	0.97	22	13	9	2.8	4,6
SiC_1200_1.5Ni	396	0.96	25	18	10	3.1	7,0
SiC_1200_2.5Ni	313	0.97	31	22	12	4.0	9,4
SiC_1200_3.5Ni	288	0.98	41	24	14	4.3	10,7
SiC_1200_4.5Ni	270	0.92	51	30	14	4.6	15,3
SiC_1400	351	0.72	22	13	8	3.6	6,6
SiC_1400_1.5Ni	245	0.76	32	24	12	5.1	11,8
SiC_1400_2.5Ni	198	0.75	43	30	15	6.3	13,1
SiC_1400_3.5Ni	180	0.66	58	35	15	6.9	14,8
SiC_1400_4.5Ni	155	0.66	60	35	17	8.0	15,8
SiC_1300	426	0.89	22	11	8	2.9	-
SiC_1300_1.5Ni	338	0.93	22	15	11	3.7	-
SiC_1300_2.5Ni	300	0.91	29	20	12	4.2	-

S_{BET} is the surface area; V_s is the pore volume at $p/p_0 = 0.98$; D_{ads} and D_{des} are the maxima of pore size distributions from adsorption and desorption branches of the isotherm, respectively; D_{BET} is the average pore size, calculated from the cylindrical pore model by formula $4V_s/S_{BET}$, $h = 4/(\rho_{SiC} \cdot S_{BET})$ is an average pore wall thickness in estimation of pore walls as the SiC cylinders, τ is the crystallite size, derived from Scherrer equation.

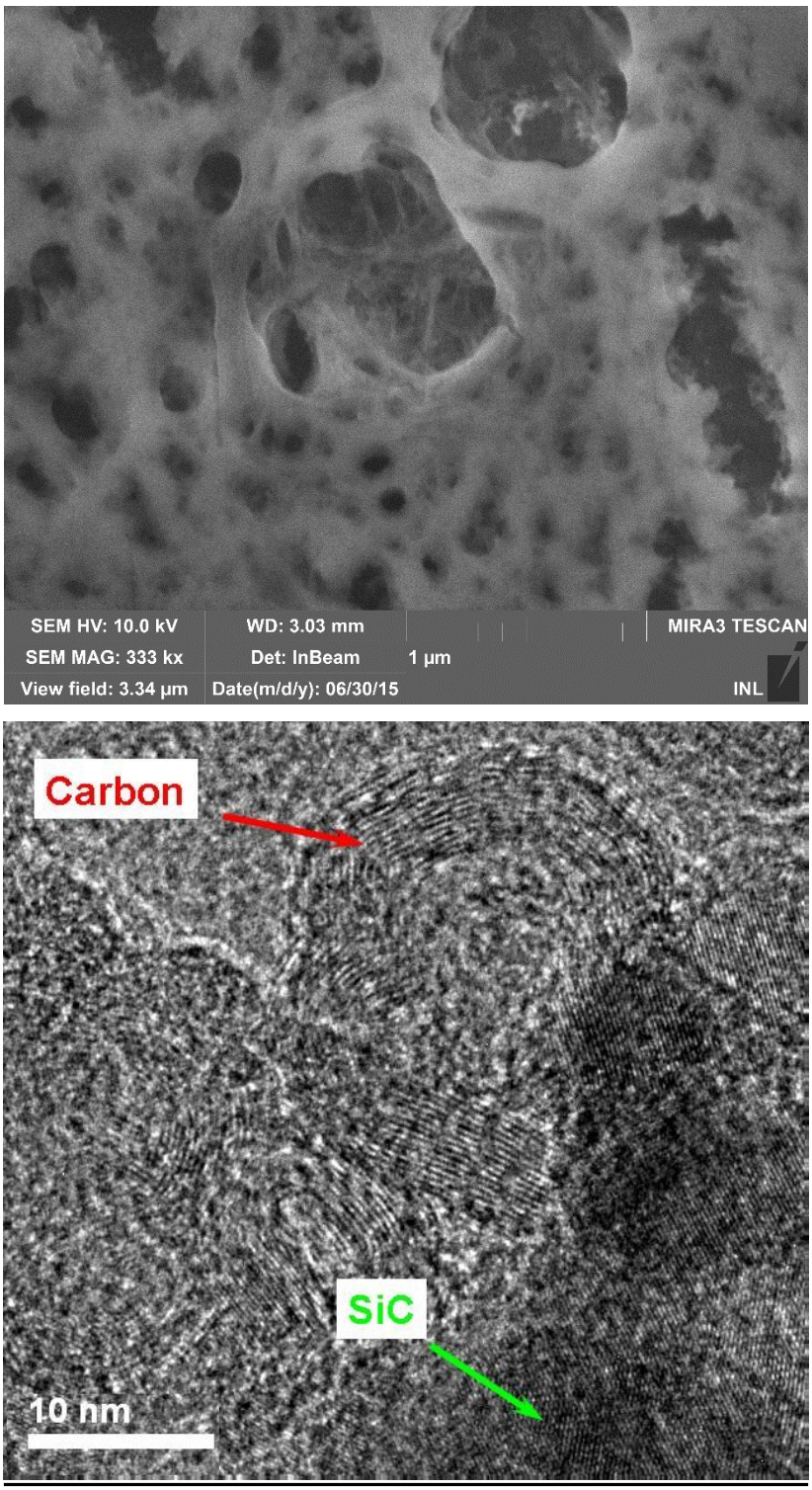

Fig. S6. SEM and HR-TEM images of SiC_1200_4.5Ni sample (see Table S2).