Occurrence of fibers in partially formed Ca-K geopolymers

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Geopolymer matrix filled with SiC grains in for polishing purposes

- Usually used material for grinding and polishing are based on ceramic matrixes and these materials are fired in ceramic kilns on temperature up to 1280 °C.

- Polishing tools (for ceramic, stone, etc.) are also made with dolomite matrixes filled by different grain sizes of corundum or SiC.

The silicon carbide

- Very hard and thermally resistant material (SiC) is produced in electric arc furnace when layers of pure silica sand are sandwiched by egg-coke. All circa 15 m long and 1,8 m high sarcophagi is coated by layer of clayed shell.

- Electric arc creates extremely high temperature (3000 °C) and when all content is cooled the SiC chains (necklace like formations) appear between non reacted sand and coke (efficiency is about 10-12 % only).

- The high temperature effects on silica sand and secondary and non desirable product of SiC fabrication is amorphous silicon.

- The SiC grains should be then washed in alkalis and neutralized in acids **to remove all amorphous silicon content**.

Potassium activated geopolymers

- Materials:
- blast furnace slag, dominantly containing gehlenite $Ca_2Al(AlSi)O_7$ complemented by merwinite $Ca_3 Mg (SiO_4)_2$), bassanite ($CaSO_4$. 0.5 H_2O), syngenite ($K_2Ca (SO_4)_2.H_2O$) and wolastonite ($CaSiO_3$).
- Waste clay, washed from sandstone deposit (snow-white sand for glass production), containing 50 wt. % of small particles of quartz (50-200 μm).
- Shistous clay (upper layers of the old coal mining area)
- For better sliding on polished material the biomass ash was used instead of plastics used in traditional polishing tools (max. 10 wt. %).

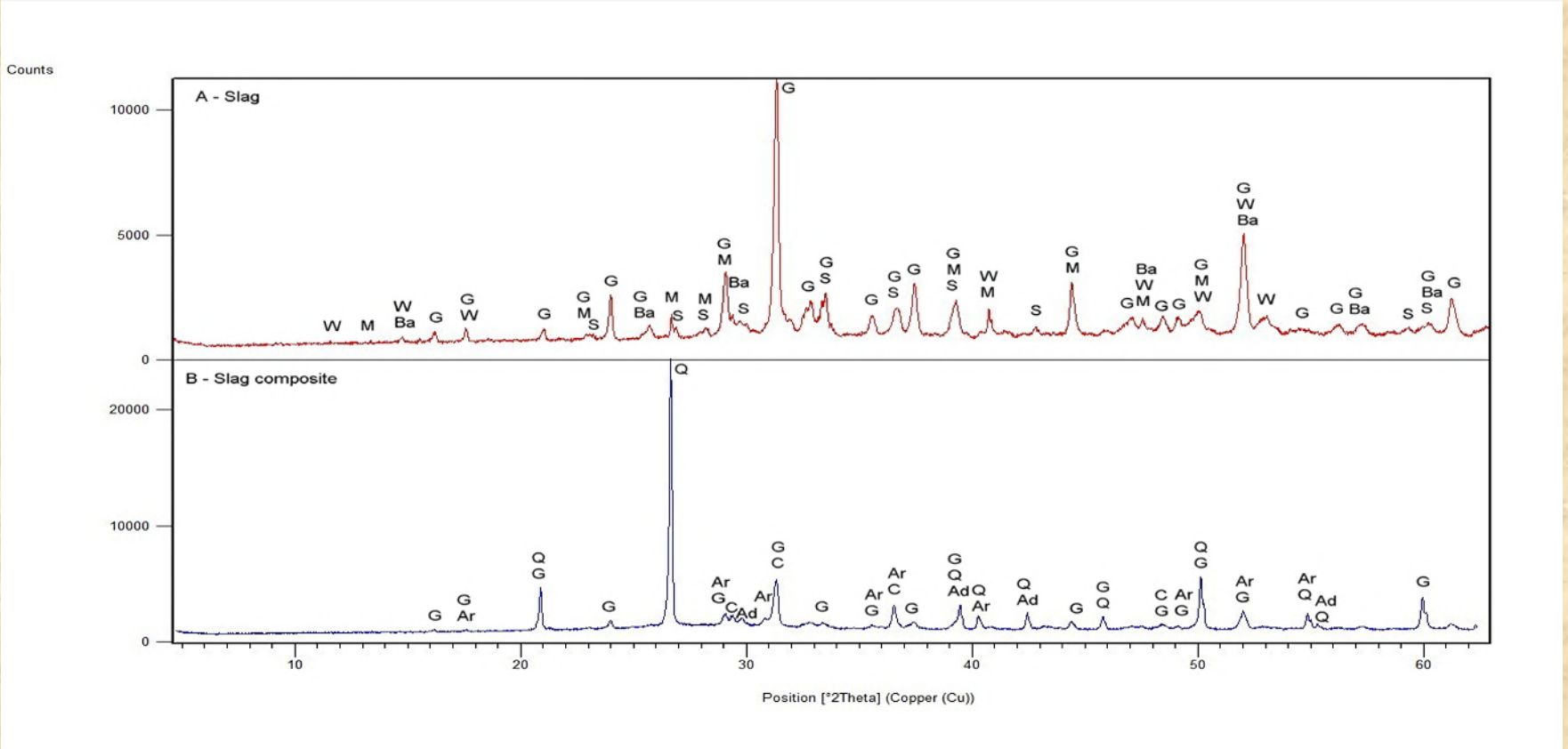
Chemical composition

Oxide/ used materials	SiO_2	Al_2O_3	CaO	Fe_2O_3	MgO	K_2O	Na_2O	LOI
Blast slag	22.38	8.01	37.44	2.31	3.51	1.27	<0.11	14.7
Shistous clay	54.69	28.63	0.59	7.07	0.62	3.72	<0.11	1.60
Waste clay*	67.86	24.69	0.03	0.35	0.0	0.28	<0.11	6.08

*/ Clayed material contains about 50 wt. % of fine quartz sand.

Biomass straw ash	45.35	2.93	13.95	1.60	3.58	21.47	<0.11	1.74

XRD comparative patterns



•The upper line corresponds to the blast furnace slag and the lower one depicts the combined matrix of slag and waste clay from sand washing. (W-wolastonite, G-gehlenite, Ba-bassanite, Ar-aragonite, C-calcite)
•Lower matrix pattern contains amorphous phases.

Circumstances

- The use of untreated SiC was a coincidence and misunderstanding between our laboratory and supplier of the materials.

- The broken part of the prepared sample was left in laboratory without any specific attention and before thrown away was observed with a laboratory microscope looking first of all for the distribution of SiC grains in matrix.

Proportions of matrix with unwashed SiC

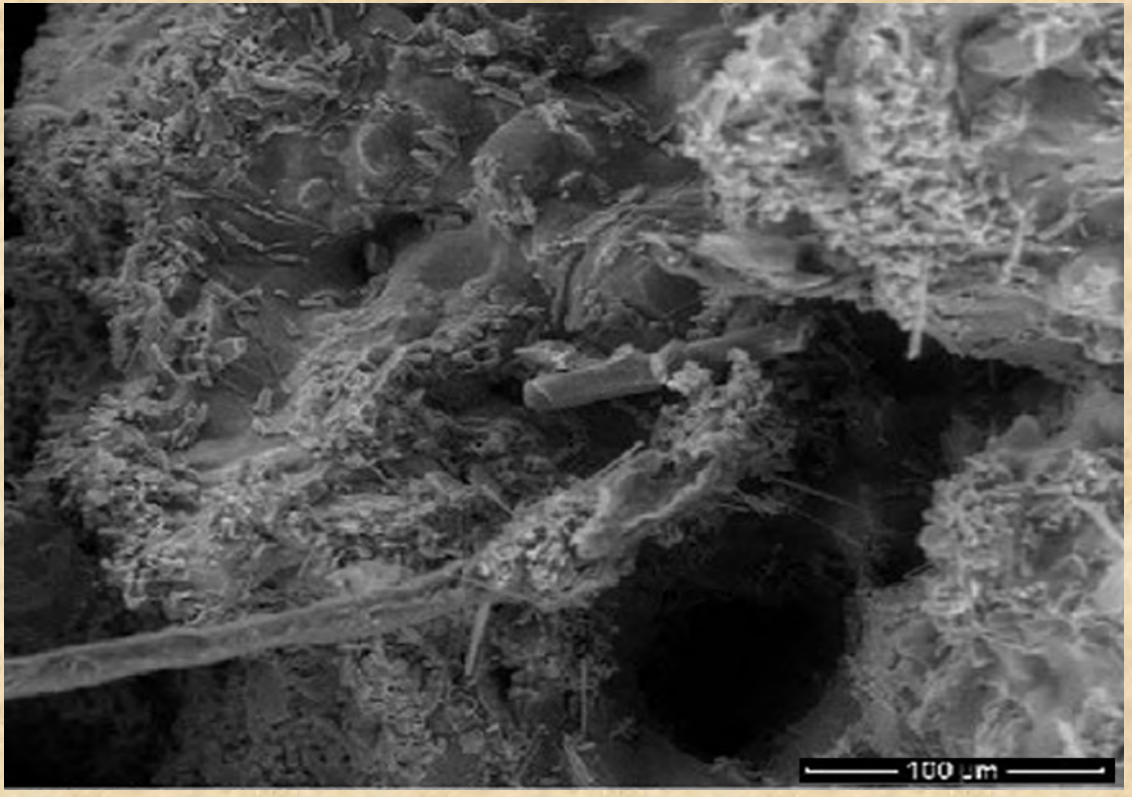
- The above mentioned matrix combining 3 wt. parts of slag and 2 wt. parts of particular clay washed from sand deposit (clay : quartz in proportion 1:1), was mixed with 3 wt. parts of untreated SiC grains. Alternatively was used the combination of 3 wt. parts of slag and 2 wt. parts of shistous clay with the same amount of SiC grains.

- The reaction of amorphous silicon in aqueous alkali conditions (activated by KOH) produces hydrogen and the final stage is a highly porous geopolymer solid.

Picture of damaged edge

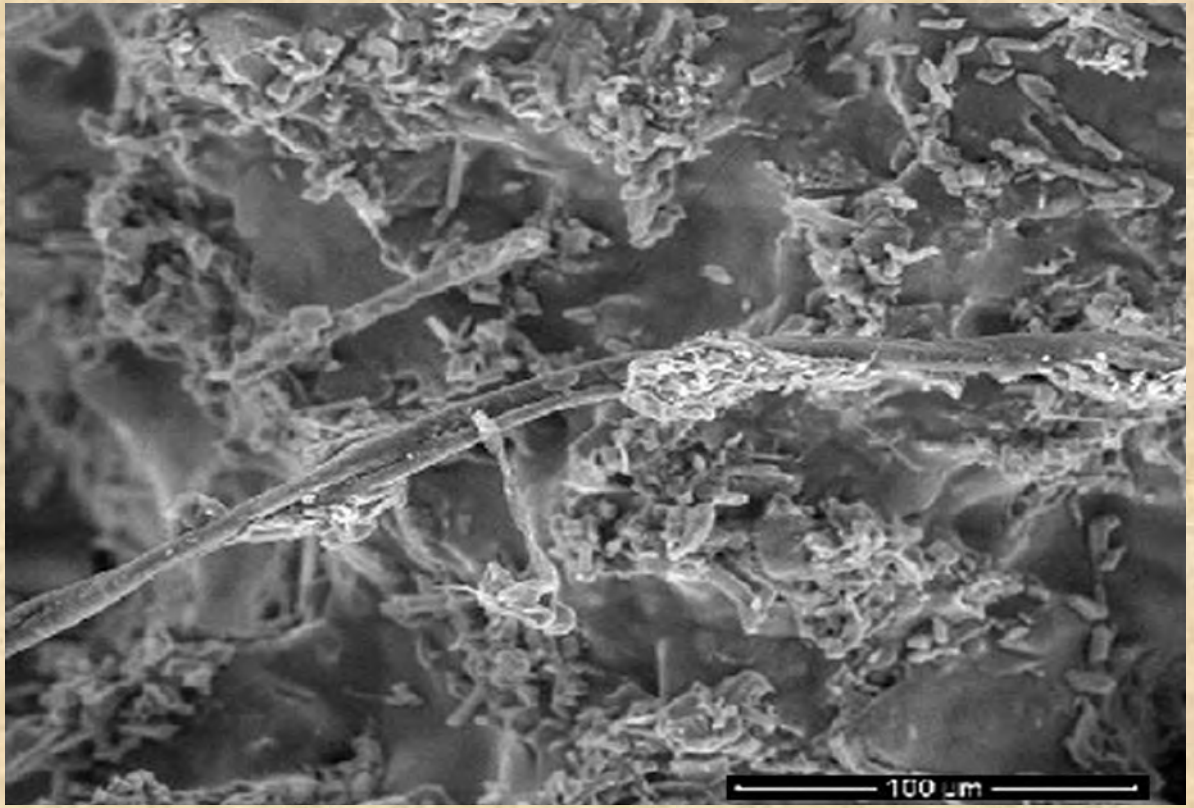
Pores, SiC grains and strange fiber-like formation
(magnitude 60 times)

Scanning electron microscope (SEM)



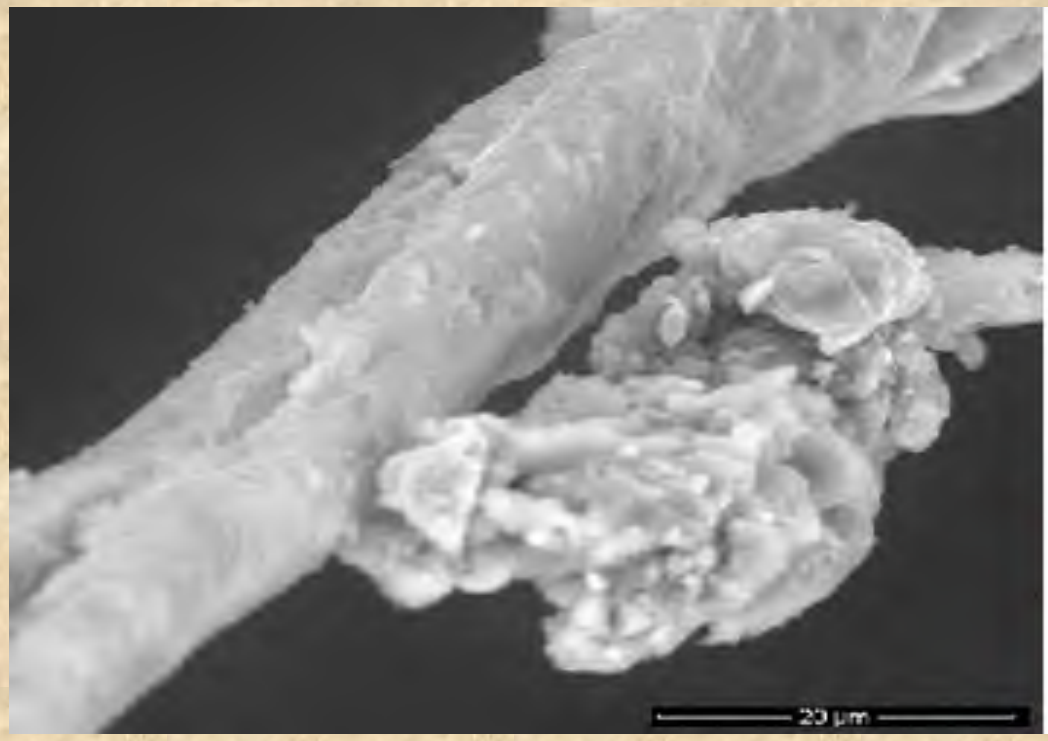
The SEM picture shows one of the nascent point of one long and one broken fiber, some small and thinner fibers are also detected (right part of the picture).

Fiber shape



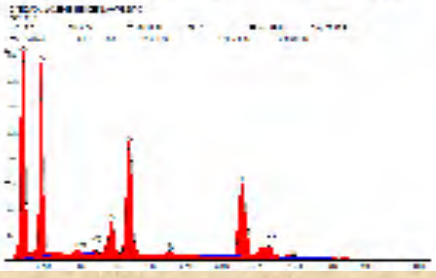
Across the picture we could observe twisted fiber with small particle glued on it.

Detailed view on glued particle



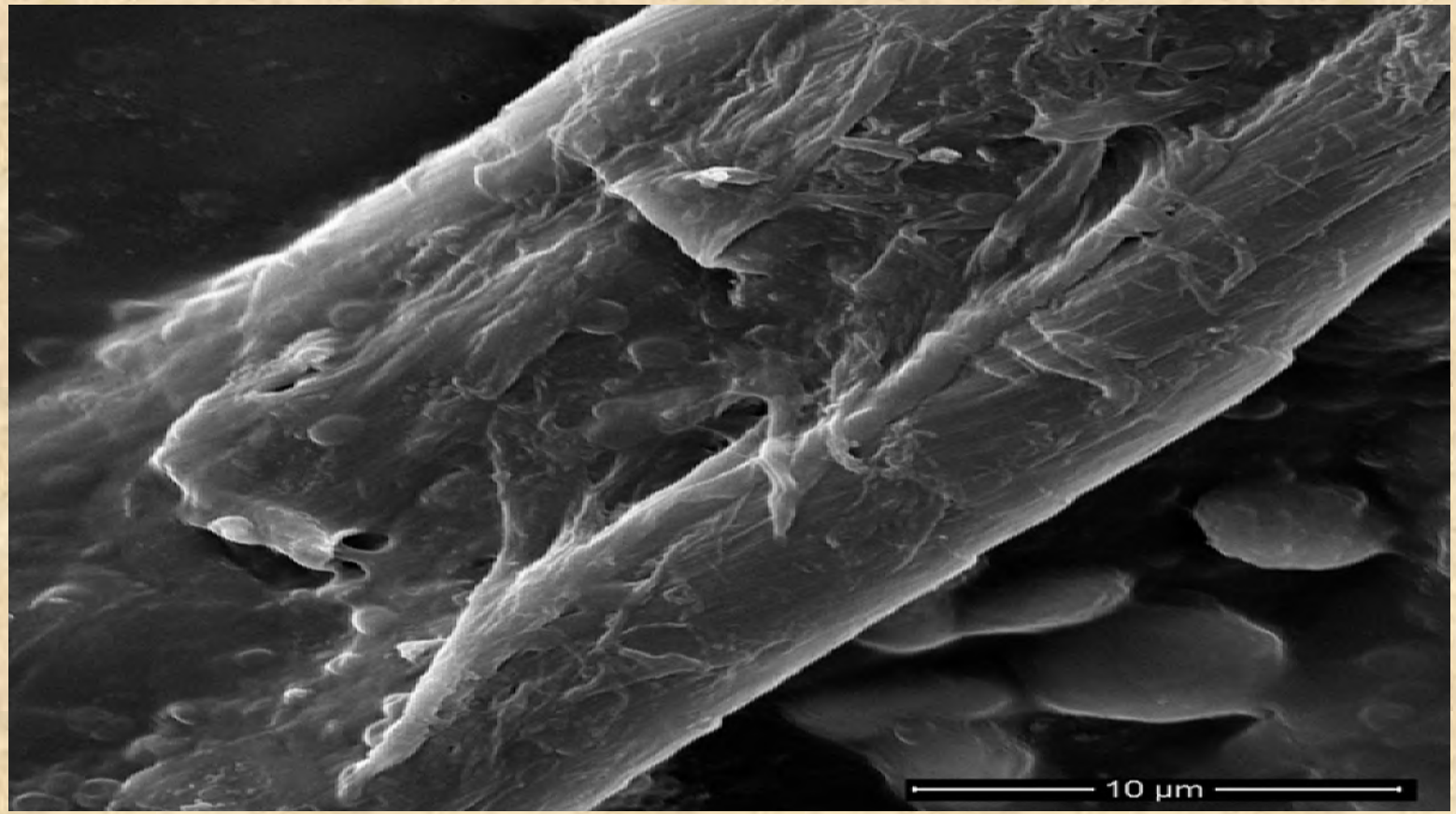
EDS Quantitative Results

Element	Wt%
CK	26.44
OK	49.03
NaK	0.44
MgK	0.25
AlK	2.59
SiK	8.91
SK	0.45
KK	10.40
CaK	1.50



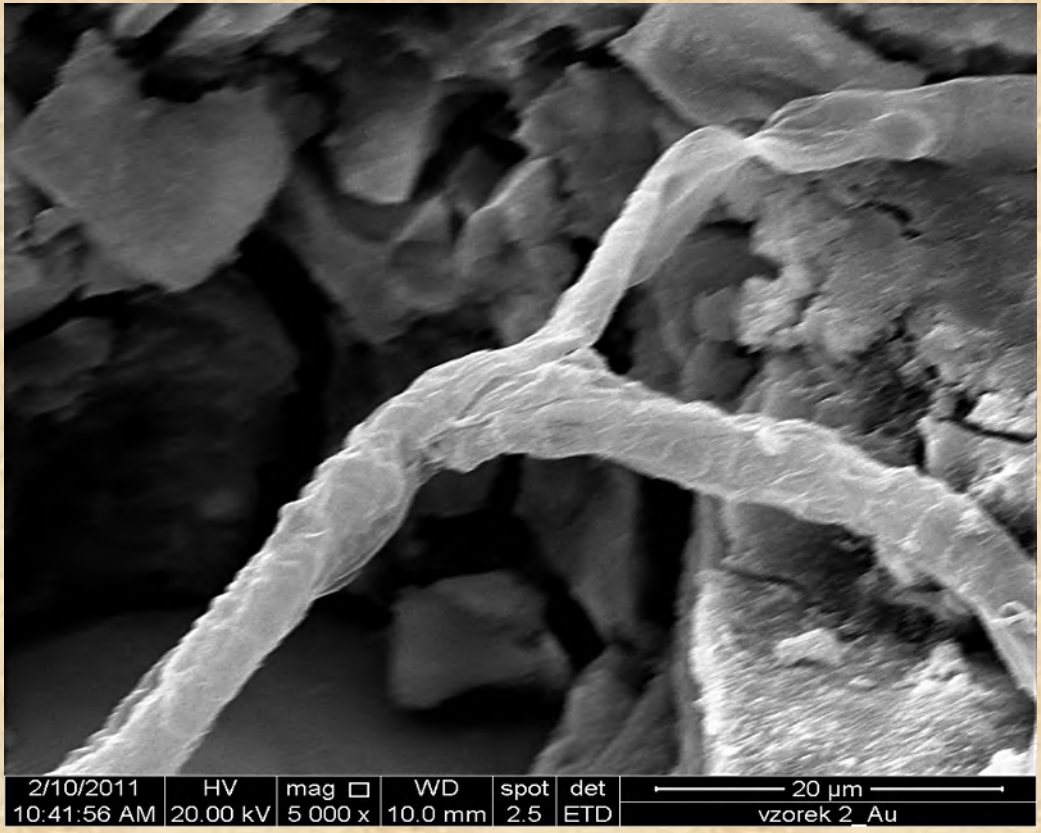
The shape and structure of twisted fiber is shown better

Magnitude of 1000 times



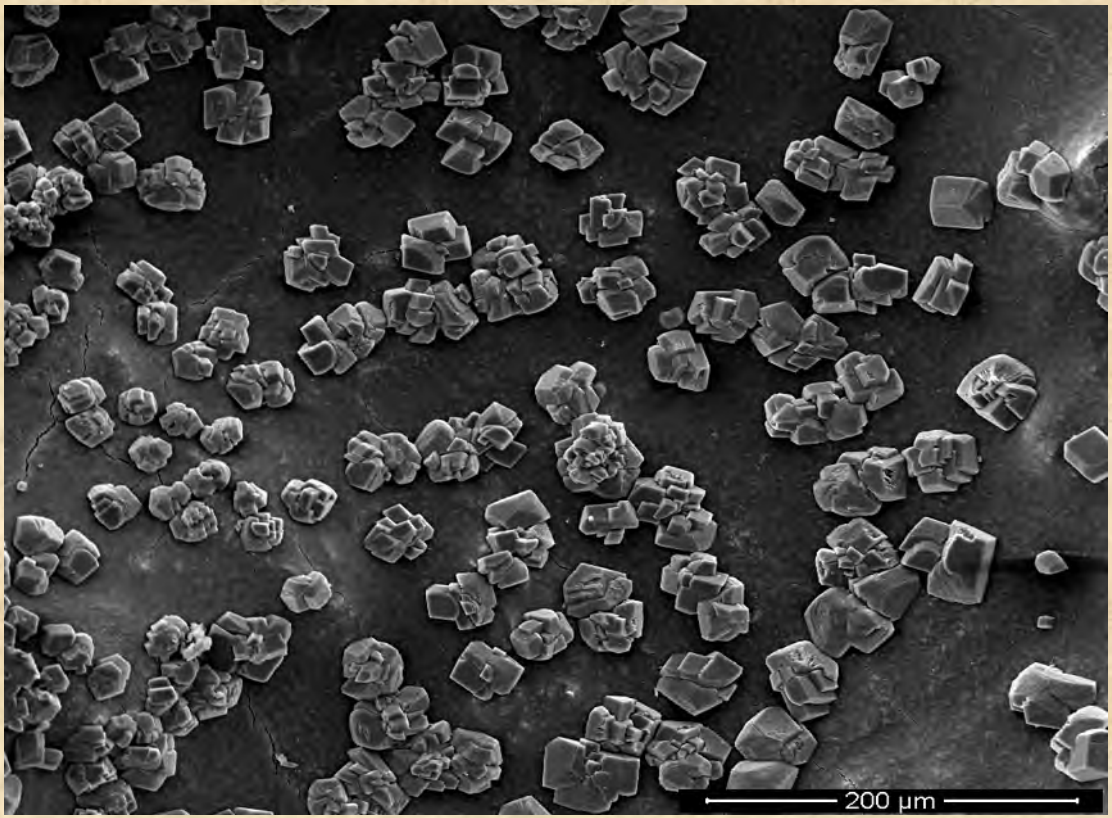
The borders are rounded and stronger than central part of the fiber and probably these differences produce twisting effect of the fibers.

The fiber branching



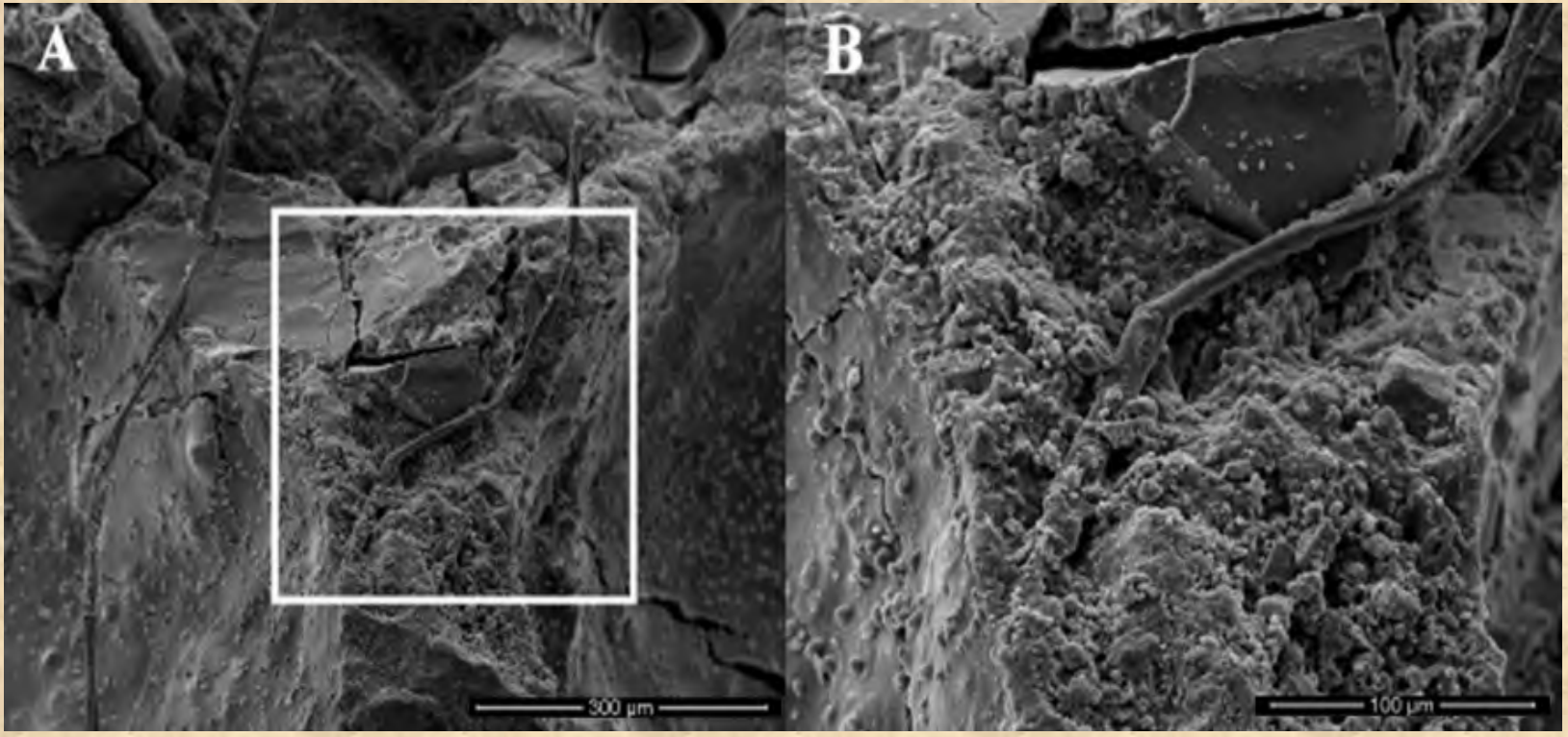
The fiber is crossing the pore - visible highly heterogeneous structure of the surroundings.

The gehlenite crystals



Crystals of gehlenite identified also by XRD analysis (bottom of convex part of a big pore)

Nascent point of fiber



The other nascent point of fiber growing from the pore edge
(see detail B of the central part A when also a long fiber cross the picture)

Conclusion and appeal

- Occurrence of fibers-like formations, their composition in highly porous matters and their influence on solidified bodies could be important.

- The partially formed geopolymers surrounded in calcareous porous matter could answer the questions of long term stabile materials (e.g. Pre-Portland mortars combining calcareous materials with thermally treated clays or with ceramic shards and quartz sand).

- For the confirmation of fibers formation in other type of geopolymer composites we ask you to repeat and confirm results of this experiment.

Thanks for your attention