SUMMARY OF STAGE 2

At this project stage, researches were widened on obtaining the two ceramic powders targeted in the project: silicon carbide and silicon nitride.

INCDMNR-IMNR Partner continued studies on the characteristics of Dubova serpentinite and on the technological processes of the use of this cheap raw material and few other applications to obtain mesoporous silica as silicon source for advanced ceramic powders: silicon carbide and silicon nitride.

It was aimed at carrying out experimental work to produce mesoporous silica from serpentinite through the application and demonstration of technological options proposed in the previous stage of the project, selecting alternative technologies that enable the production of mesoporous silica with the most favorable characteristics which, in terms of its chemical composition, is considered to meet the requirements necessary to obtain high quality SiC and Si_3N_4 for the applications covered in this research project.

Following undertaken research it was developed the technology for mesoporous silica production and for residual MgO recovery and its use in the composition of abrasive ceramic materials.

Analyzing the results obtained in this current phase of the project it can be assumed that to produce a mesoporous silica from serpentinite which exhibits the compositional features necessary for obtaining high quality SiC and Si_3N_4 for applications covered in this research project developed, the technology must include the following stages: 1) physical-mechanical treatment of serpentinite (grinding + sorting), to reduce the size of grain solid material particle to less than 63μm; 2) acid dissolution, carried out in concentrated mineral acid medium, followed by separation of the phases (solid, liquid) by filtering, washing the separated silica residue in multiple steps with the hot water- operation performed to remove the residual acid solution- followed by the magnetic fraction separation (for example, magnetite) from the silica residue; 3) drying of the silica residue to remove moisture excess from the material.

To recover residual MgO, the concentrated acid solution which was separated after serpentinite dissolution in acid medium was processed as follows: 1) it undergoes deferrization operations, 2) liquid and solid phases are separated by filtration 3) separation of MgO from solution which is clarified by precipitation, followed by separation of the precipitate obtained by filtration, 4) washing with water in order purify the precipitate, 5) drying and calcination in the electric furnace at a temperature depending on the envisaged application.

INCDCP-ICECHIM Project Coordinator has studied the process of obtaining new silica- PAN composites based on mesoporous silica produced by IMNR from serpentinite. There were used different concentrations of monomer to silica, applying host-guest polymerization technique in ultrasound field. There were varied different parameters of the hybrid synthesis process: initiator concentration, time of impregnation with sonication and time of polymerization with sonication, respectively, the remaining parameters being kept constant. Organic and inorganic components were favorably combined to obtain composites having improved mechanical and thermal properties.

FTIR results of the new composites, synthesized by varying different synthesis parameters, showed all the characteristic bands of the inorganic host compound and of the vinyl polymer guest. The thermal stability of the polymer constrained in the inorganic structure has been improved compared to polyacrylonitrile (PAN). Thermal Gravimetric Analysis also provided important information on the carbonization process (1^{st} and 2^{nd} stage of the process of polymer degradation) which can then be used to adjust the parameters of the ceramic powders obtaining process. Thermo dynamo mechanical analysis has revealed the formation of polymer layers on the surface of the host material, and an improvement in elasticity of up to 180 °C compared to neat silica. All tests and measurements showed the reinforcing effect of the silica to the polymer.

This study provides information on thermal generation of silica-carbon nanocomposites. It was shown that in silica matrix various concentrations of carbon can be incorporated, but more important is that carbonic phase can be controlled by the amount of initial incorporated polymer.

An intimate silica - carbon mixture may be obtained by carbocatenar polymer oxidation and graphitization in silica pores after thermal treatment in nitrogen atmosphere.

It was proposed the polymer nanocomposites production technology and polymer carbonization technology, which is common for the obtaining technologies of the both ceramic powders studied in the project: SiC and Si_3N_4.

SC Caloris Group SA Partner has conducted an experimental study aimed at producing SiC and Si_3N_4 ceramic powders. In this study, the following features were observed:

The quantity and morphology of the obtained product after nitridation-carbothermal reduction depend on many synthesis factors and on the involved constituents. Porous materials precursors for obtaining composites along with the impurities contained in their structure play an important role on Si3N4 formation and on α / β Si3N4 phase ratio;

Silica sorts that were the basis for subsequent obtaining of different composite, time and temperature of the thermal treatment were varied, finally yielding Si3N4 ceramic powder in percentage of over 90%.

By varying both the maximum treatment temperature and samples maintaining time at constant temperature, it was determined the optimum temperature of the carbo thermal nitridation process.

Silicon carbide was obtained through pellet making technique.

Technology of SiC and Si3N4 nanocomposites obtaining includes the technology for obtaining silica-carbon nanocomposites via polymer nanocomposites plus heat treatment step at high temperatures.