Using Scanning Electron Microscopy, we study how the nickel-graphite composites (Fig.1) obtained from nickel-amorphous carbon powder mixtures containing 50 vol. % Ni by graphitization-accompanied Spark Plasma Sintering (SPS) respond microstructurally to phase separation. The in situ graphitization implies intimate contact between nickel and graphite; the latter forming by a dissolution-precipitation mechanism. The treatment of the composites — removal of nickel by dissolution in acid and graphite by annealing in air — was aimed at creating porous structures. The burnout of graphite during annealing of the composites in air proceeded parallel to the formation of NiO. The removal of graphite, however, did not lead to the formation of structures with uniform porosity distribution. The morphological outcome of the oxidation treatment appears to be dependent on the relative density of the Ni-C compacts as well as on the presence of Ni(C) solid solutions. During oxidation, the presence of carbon in the solid solution results in the evolution of CO thus preventing the formation of a continuous NiO film. A longer annealing of the compacts in air led to the formation of NiO structures shown in Fig.2. The NiO-based compact that formed did contain porosity; however, that porosity was inherited from the nickel-graphite composite that was not fully densified; in addition, the hollow NiO structures contained closed porosity. The NiO network formed as a result of nickel oxidation simultaneous with oxidation of carbon did not structurally repeat the pre-existing nickel network. A conclusion was drawn that in the microstructural evolution accompanying the burnout of carbon from the nickel-graphite composites, sintering of NiO, rather than porosity creation, dominates. By dissolving nickel from the SPS-ed specimens in acid, porous pellets were obtained that perfectly retained their shape and consisted of graphite platelets with diameters ranging from 0.3 to 2 μm and a thickness of 0.2 μm. The porous graphite formed from mechanically milled mixture was of uniform structure. In the networks formed from the non-milled nickel-amorphous carbon mixtures, pores 4-5 μm in diameter were present (Fig.3). The platelets of porous graphite obtained from the compacts consolidated in the solid state were smaller than those of the graphite crystallized in contact with molten nickel.
Fig. 1: Microstructure of the SPS-ed nickel-graphite composite, SPS-temperature 1000°C.

Fig. 2: NiO structures evolved during oxidation of the SPS-ed nickel-graphite compact during annealing in air.

Fig. 3: Porous graphite obtained by phase separation in the nickel-graphite SPS-ed composite.