A Mechanistic Study on the Structure Formation of NiCo2O4 Nanofibers Decorated with In Situ Formed Graphene-Like Structures

Nickel cobaltite (NCO) nanofibers were synthesized using poly(styrene-co-acrylonitrile) (SAN) as the polymeric binder through sol–gel assisted electrospinning. Defect-free precursor nanofiber mats were pyrolyzed at 773 K at three different pyrolysis soaking times $t = 2$, $4$, and $6$ h. The SAN present in the precursor nanofibers caused morphological changes in the NCO nanofibers during their thermochemical degradation. Consequently, fractal aggregates of NCO nanoparticles were formed along the length of the nanofibers. X-ray photoelectron spectroscopy (XPS) revealed both $+2$ and $+3$ oxidation states for Ni and Co, with spinel crystal defects due to oxygen rich atmosphere. XPS, high-resolution transmission microscopy, and optical analysis showed graphene-like structures embedded within the NCO nanofibers. With increase in pyrolysis soaking time, the morphology of the NCO particles markedly changed from spherical to rod-like. We propose a mechanism for the morphological change of NCO nanoparticles on the basis of crystallite splitting accompanied by particle splitting and reordering.

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