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Synthesis of graphite-metal composite materials by salt thermolysis

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Using electron microscopy, Auger spectroscopy, and X-ray diffractometry, the characteristics of thermoexfoliated graphite-cobalt (TEG-Co) nanocomposite materials (NCM) have been investigated. The pre-treatment of TEG by different substances (H_2O_2, H_2SO_4, HNO_3) has been shown to favor the effective impregnation of TEG by cobalt salt solutions and formation of nanoscaled Co particles (60 to 70 nm) at a relatively high metal content in the NCM (up to 30 mass.%). It was found that the type of salt used to prepare TEG-Co NCM defines the phase composition and morphology of the samples at each stage of metal nanoparticle formation on TEG surface. The preliminary thermal shocking of TEG-Co acetate or TEG-Co nitrate powders provides formation of smaller metal particles on TEG surface after reduction in hydrogen flow than the direct reduction of TEG-Co salt powders.

Методами электронной микроскопии, Оже-спектроскопии и рентгеновской дифракции исследованы характеристики приготовленных нанокомпозитных материалов (НКМ) терморасширенный графит-кобальт (ТРГ-Co). Показано, что предварительная обработка терморасширенного графита различными веществами (H_2O_2, H_2SO_4, HNO_3) благоприятствует эффективной пропитке терморасширенного графита солями кобальта и формированию наноразмерных частиц Co (60–70 нм) при высоком содержании металла в НКМ ТРГ-Co (до 30 % мас.). Обнаружено, что тип соли одного и того же металла, используемой для приготовления НКМ ТРГ-Co, определяет фазовый состав и морфологию образцов на каждой стадии формирования наночастиц металла на поверхности ТРГ. Предварительный термоудар порошков ТРГ-ацетат Co и ТРГ-нитрат Co приводит к образованию более мелких частиц металла на поверхности ТРГ после восстановления в потоке водорода, чем прямое восстановление порошков ТРГ-соль Co.

In development of graphite-metal nanocomposite materials (NCM), the type of graphite support and its structural and phase state are of great importance in providing metallic component homogeneous distribution in the NCM. The method of graphite impregnation by metal salt solutions followed by salt thermolysis and reduction to pure metal in hydrogen flow is widely used in production of graphite-metal NCM [1]. In our opinion, the thermoexfoliated graphite (TEG) is the most promising support to prepare graphite-metal NCM. First, TEG is characterized by high specific surface and porous structure that improves essentially its impregnation by metal salt solutions [2]. Second, the graphite oxidation results in formation of oxygen-containing groups on its surface, which will act as exchange centers for metal cation fixing in graphite-metal (or graphite-metal oxide) composite material [3–5]. The graphite oxidation results also in an essential imperfection of graphite surface, formation of projections, cavities, roughness, acting also as for metal ion fixing centers on graphite surface from water solution. Finally, graphite possesses chemical inertness with respect to metals and their compounds that provides high stability of composite. The aim