UDC 546.65’74

NONSTOICHIOMETRIC OXIDE FORMATION IN SYSTEM Nd-Ni-O.


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The non-stoichiometric compositions \( \text{Nd}_{2-x}\text{NiO}_{4.3x/2+\delta} \) were obtained using coprecipitation method from nitrates solutions by \( \text{K}_2\text{CO}_3 \) followed by calcination of the obtained blend at 900°C (10 hours). It was found that full coprecipitation of ions begins at mole ratio of coprecipitated ions to precipitator \( n=1.75 \) in range \( \text{pH}=10-10.2 \).

The thermogravimetric investigations established that coprecipitated carbonates decompose in the four stages.

The first – (25 – 392°C), removing of crystallisation and adsorbed water evaporation. The second - (392 – 520°C), \( \text{OH}^- \) - groupes decomposition. The third (622 - 720 °C), carbonates groupes decomposition. At 850°C - neodymium nickelate phases formation.

According to X-ray phase analysis \( \text{Nd}_2\text{NiO}_4.392(5) \) consists of orthorhombic neodymium nickelate and a small of \( \text{Nd}_2\text{O}_3 \) impurity (specific reflex at \( d=0.3026 \) nm).

\( \text{Nd}_2\text{NiO}_4.392(5) \) unit cell parameters - \( a=0.545(0) \) nm, \( b=0.536(8) \) nm, \( c=0.123(3) \) nm, space group Bmab.

Samples \( \text{Nd}_{1.9}\text{NiO}_{4.113(5)} \) and \( \text{Nd}_{1.8}\text{NiO}_{4.006(5)} \) consist of non-stoichiometric phase \( \text{Nd}_{2-x}\text{NiO}_{4+\delta} \) with neodymium ions deficient in sublattice.

The orthorhombic unit cell parameters for \( \text{Nd}_{1.9}\text{NiO}_{4.113(5)} \) - \( a=0.541(9) \) nm, \( b=0.535(4) \) nm, \( c=0.123(2) \) nm, \( \text{Nd}_{1.8}\text{NiO}_{4.006(5)} \) - \( a=0.541(5) \) nm, \( b=0.535(4) \) nm, \( c=0.123(2) \) nm.

The stabilization of non-stoichiometric neodymium nickelate phases likely is due to ion vacancies formation.
Nd_{2-x}NiO_{4-3x/2+δ} samples with 0.2<\textit{x}\leq0.5 are polyphase and contain Nd_2NiO_4, NiO and Nd_2O_3. It was not observed Nd_3Ni_2O_7 phase formation in our experimental conditions.