

NOVEL PROCESSING OF NICKEL CONTAINING CORDIERITE GLASS-CERAMICS FOR MICROPOROUS GAS SEPARATION MEMBRANES

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The production of microporous membranes using the well-established glass-ceramic process combines the advantages of both amorphous silica and zeolites. In this process, traditional glass forming methods are employed, and the pore structure is controlled by the crystalline phases. The process involves the exposure of a glass-ceramic to a reducing atmosphere to produce a microporous membrane with evenly dispersed nickel metal colloids. Stoichiometric Ni-doped cordierite glasses having the composition $(2-x)\text{MgO} \cdot x\text{NiO} \cdot 2\text{Al}_2\text{O}_3 \cdot 5\text{SiO}_2$ are synthesized, where $0 \leq x \leq 1$. The glasses crystallize to form varying amounts of spinel, cordierite and β -quartz phases depending on the heat treatment temperature and time. Subsequent exposure of the glass-ceramic to a reducing atmosphere results in a hydrogen reduction reaction with nickel. Divalent nickel (Ni^{2+}) is reduced to the metallic state (Ni^0), while the Al-O-Si framework remains intact. Selective reduction of transition metal ions from crystalline aluminosilicate phases presents a novel route to produce nanoporous materials.

Spinel and spinelloids are investigated for this method because of the large range of transition metal ions that can be incorporated into the structure. The spinel solid solution series $\text{Mg}_{1-x}\text{Ni}_x\text{Al}_2\text{O}_4$, where $0 \leq x \leq 1$ is studied for this method using *in-situ* high temperature diffraction with full atmosphere control. Rietveld analyses for phase quantification and cell refinements are presented. The $\text{Ni}_x\text{Mg}_{1-x}\text{Al}_2\text{O}_4$ spinel framework remains intact following the complete reduction of nickel for compositions where $x \leq 0.50$. For $\text{Ni}_x\text{Mg}_{1-x}\text{Al}_2\text{O}_4$ spinel containing $x \geq 0.75$, decomposition of the spinel framework is marked by the formation of $\text{NiAl}_{32}\text{O}_{49}$. The weight percent of nickel formed during the reduction reaction directly relates to the formation of nickel vacancies in $\text{Ni}_x\text{Mg}_{1-x}\text{Al}_2\text{O}_4$ spinel.