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François Cheviré, Arthur Pallu, Franck Tessier

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François Cheviré, Arthur Pallu, Franck Tessier. Characterization of rare-earth aluminum oxynitrides synthesized by carboreduction and nitridation. ISNT 2009 - 6th International Conference on Nitride and Related Materials, Mar 2009, Karlsruhe, Germany. hal-01261259

HAL Id: hal-01261259

<https://hal.science/hal-01261259>

Submitted on 25 Jan 2016

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Characterization of Rare-Earth Aluminum Oxynitrides Synthesized by Carboreduction and Nitridation



François Cheviré, Arthur Pallu, Franck Tessier

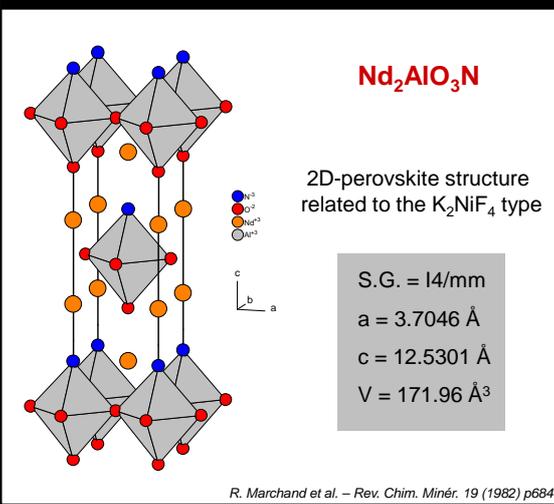


UMR CNRS 6226 "Sciences Chimiques de Rennes" – Equipe Verres et Céramiques
Groupe Matériaux Nitrures, 35042 – 35042 Rennes Cedex – France

Background

Over the recent years, a great interest has been shown for multinary oxynitrides containing main group elements especially in the fields of phosphors or visible-light driven photocatalysts. However if a tremendous work has been done on nitridosilicates and nitridoaluminosilicates based materials, very little is known about multinary (oxy)nitride systems based on aluminum (R_2AlO_3N , $Al_{11}O_{12}N$), gallium (La_2GaN_3) or phosphorous (Zn_2PO_3N). A large part of our research is then devoted to the preparation of new materials among those systems by exploring less-common synthesis routes than the usual solid state reaction or the ammonia flow-based nitridation. As a first step of the present work, we have successfully reinvestigated the preparation of the R_2AlO_3N oxynitrides ($R = Nd$ and Sm) using the carboreduction and nitridation process (CRN).

Structure...



Synthesis routes...

Previous synthesis : mixture of Nd_2O_3 or Sm_2O_3 and AlN sealed under N_2 in nickel tubing and fired at 1350°C with repeated heating.

Long reaction process (~ 6 x 24h)
Very difficult to obtain pure samples → Al_2O_3 and R_2O_3 minor phases

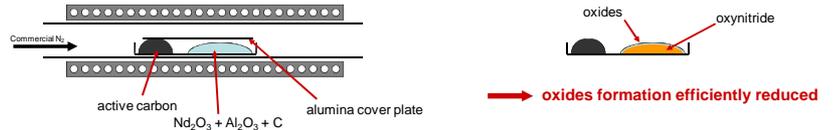
Carboreduction & nitridation (CRN):



Regular apparatus

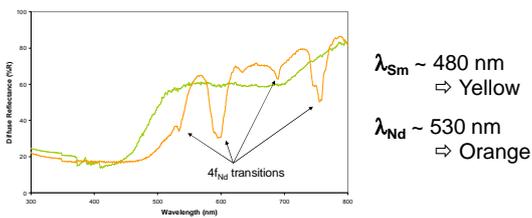


"Oxygen trap" apparatus



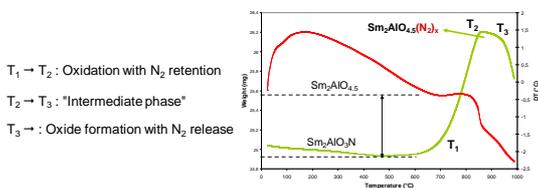
Optical properties...

Nd_2AlO_3N → orange powder
 Sm_2AlO_3N → greenish yellow powder



Thermal analyses...

Oxidation under air starts ~ 600°C

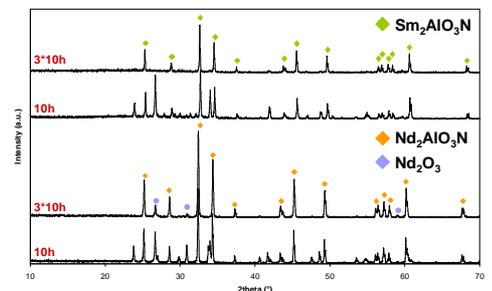


X-Ray Diffraction...

⇨ Sm_2AlO_3N can be obtained as a nearly pure sample after only 3 heating at 1250°C

⇨ In the same conditions, a Nd_2O_3 impurity phase still be present along with Nd_2AlO_3N

little oxidation remains



Conclusion...

▶ Carboreduction & Nitridation (CRN) is a convenient way to prepare aluminum based oxynitrides with interesting optical properties and good thermal stability under air.

▶ Apparatus will be upgraded with a nitrogen gas purification system (moisture and oxygen traps, hot titanium wire) to avoid reoxidation of the samples.

▶ Other systems are under investigation such as $A_{2-x}R_xAlO_{4-x}N_x$ solid solution with A = Alkaline earth and R = Rare earth.

Contact informations:

francois.chevire@univ-rennes1.fr +33 (0) 2 23 23 62 34
http://www.verceram.univ-rennes1.fr Fax +33 (0) 2 23 23 56 83