

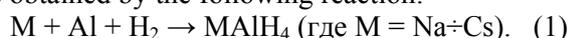
MECHANO-CHEMICAL METHOD OF ALKALINE EARTH METAL ALUMINUM HYDRIDE AND LANTHANIDES BOROHYDRIDES OBTAINING

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Metal aluminum hydrides and f-elements borohydrides are valuable specimens in chemistry and technology. In particular, there is a great demand for them, with the purpose of power-consuming materials creation for hydrogen power engineering purposes.

In [1-2] works, the alkali element aluminum hydrides in the presence of solvent and solutions are obtained by the following reaction:



Besides, many metals borohydrides are obtained in solvated medium. [3, 4].

In the present work, with the use of mechano-chemical processes the alkaline earth metal aluminum hydrides (AEM) and lanthanide borohydrides are obtained without solvated chemical agents.

Ball mill with the reactor pot volume 300 ml [5] was operated for realization of interaction.

Mechano-chemical approach to synthesis $M(AlH_4)_2$ (where $M = Ca, Sr, Ba$) allow to obtain AEM aluminum hydrides by the following reactions:



Metals aluminum hydrides synthesis was carried out in argon atmosphere. AEM hydrides are obtained by direct interaction of AEM with hydrogen, AlH_3 - is obtained by chlorine-benzyl method [8].

AEM aluminum hydrides after the synthesis were extracted as tetrahydrofuran (THF) and diglym (DG).

The product was evolved from clean solution by the help of solution evaporation.

Physical and chemical properties of AEM aluminum hydrides were studied by RFA and IR – spectrums were registered in spectrograph IR–20. The properties of obtained $M(AlH_4)_2$ are compatible with literature data.

In the present work the interaction of sodium borohydride by lanthanide chlorides of cerium subgroup by force of mechano–chemical activation and without solvent participation was also studied.

The equipment presented in work [5] was used during the work. Lanthanides chlorides solid phase reactions with $NaBH_4$ is studied, the possibility of lanthanum, cerium, praseodymium and neodymium lanthanides borohydrides obtaining is presented.

Reactor loading, mechanical processing of reaction mixture in ball mills and sample

preparation for investigation were carried out in inert atmosphere. The technical $NaBH_4$ was recrystallized from diglym. Mechanical processing of $NaBH_4$ - $LiCl_3$ mixture was carried out during 10-30 minutes. Mole ratio $LiCl_3 : NaBH_4 = 1 : (3,75 \div 4,0)$.

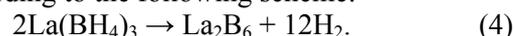
It turned out, that the reaction of $LnCl_3$ with $NaBH_4$ (where $n = La, Ce, Pr, Nd$) at sufficiently intensive mixing, in excess of $NaBH_4$ 20÷30% behaviors at 10-30 minutes and produce quantitative output of lanthanides borohydrides.

In typical test 15 gr. of $LnCl_3$ and 8, 5÷9, 3 gr. of $NaBH_4$ were taken. It is obvious from the table that the most optimal process time is 15 minutes, where the product output is 56%.

The reaction product was dissolved in benzol after the synthesis and product was evolved from clean solution by the help of solution evaporation. Product analyses are presented in the table. Thus, individual lanthanum, cerium, neodymium and praseodymium borohydrides are obtained.

Physico-chemical properties of obtained lanthanides borohydrides were investigated by roentgen-phase analysis. IR-spectrums were registered in spectrophotometer UR-20.

$La(BH_4)_3$ thermovolumogram is differ by its considerable thermal sustainability and decomposed by one stage at 265°C. Apparently, the decomposition of $La(BH_4)_3$ is behaviors according to the following scheme:



Thus, the synthesis way of light lanthanide borohydrides (La, Ce, Pr, Nd) by mechano-chemical methods with non-solvated product obtaining is worked out.

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References

1. Ashby E.C., Brendel G.J., Redman H.E. Direct synthesis of complex metal hydrides. Inorg. Chem. 1963; 2(3): 499-504.
2. Dimova T.N., Eliseeva N.G., Bakum S.I. et. al. – Doklady AN SSSR, 1974; 215(6): 1369.
3. Mirsaidov U.M. Borogidridi metallor. – Dushanbe: Donish, 2006, 139 p.
4. Mirsaidov U.M., Dimova T.N. Borogidridi peregodnikh metallov. – Dushanbe: Donish, 1984, 107 p.
5. Avakumov E.G. Mekhanicheskiye metodi aktivatsii khimicheskikh protsesov. – Novosibirsk: Nauka. Sibirskoye otdeleniye, 1986, 204 p.