s10.m2.p1 **Decomposition of the (Gd, Ho,Tb, Er)Ni_5 Induced by High-Energy Ball Milling.** A. Tonejc[1], M. Stubicar,[1] Ž. Blažina[2], *[1]Faculty of Science, Department of Physics, Bijenicka 32, 10002 Zagreb, Croatia, [2]Ruder Boškovic Institute, Bijenicka 54, 10001 Zagreb, Croatia.*
Keywords: rare earth-intermetallic compounds, high-energy ball millingm structural changes.

The previous X-ray diffraction studies[1,2] of milled $GdFe_2$, $Sm_2Fe_{17}C_x$ and $Sm_2Fe_{14}C$ compounds under purified argon atmosphere showed the segregation of metallic iron from the compounds and the appearance of an amorphous phase. However, the milling of YFe_2 compound showed no segregation of iron but a direct transformation to the amorphous phase[3].

In the present work we have milled the $GdNi_5$, $HoNi_5$, $TbNi_5$ and $ErNi_5$ intermetallic compounds, prepared previously by arc melting of the components under argon atmosphere[4]. Milling was carried out in a planetary ball mill (Fritsch P7) in air, using the balls and the vial made of agate.

X-ray diffraction results were obtained with a Philips PW 1820 powder diffractometer employing Cu$K\alpha$ radiation. At the initial time, the diffraction lines agree with those of $GdNi_5$, $HoNi_5$, $TbNi_5$ or $ErNi_5$. With milling time the intensity of compounds diffraction lines decreases and the shape of the lines broadens as the consequence of the crystallite size decrease. The lines of crystalline nickel become also visible, and at the same time a very broad maximum around $2\theta = 30^0$ appears, indicating the formation of an amorphous phase.

After about 3 h of milling only a mixture of an amorphous phase and nanocrystalline nickel could be observed on the X-ray diffraction patterns. Beyond 3 h milling no change in amorphous phase/crystalline nickel was observed up to the moment the nickel began to oxidize (more than 30 h of milling).

We can conclude that severe milling of the $GdNi_5$, $HoNi_5$, $TbNi_5$ and $ErNi_5$ intermetallic compounds induces their decomposition into the crystalline nickel and into an amorphous (Gd, Ho,Tb, Er)Ni phase. In our experiments we used considerably different milling conditions in comparison with those reported for $GdFe_2$, $Sm_2Fe_{17}C_x$ and $Sm_2Fe_{14}C$ compounds[1,2], the milling of which shows also the decomposition effect. This indicates that experimental procedures should not be the cause for the effect observed, thus further study is needed to explain the decomposition behavior.

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s10.m2.p2 **In situ X-ray Diffraction of Co/Al_2O_3 Catalyst during the Disproportionation of Carbon Monoxide.** P. Pinheiro, M.-C. Schouler, P. Gadelle, *LTPCM, ENSEEG, BP 75, 38402 St Martin d'Hères*, E. Dooryhée, *ESRF BP220, 6 rue J. Horowitz, BP220, 38043 Grenoble Cedex*.
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Carbon filaments or nanotubes can grow from the decomposition of hydrocarbons or from disproportionation of CO over the VIII group metals [1,2]. The aim of the present work is to study the filamentary growth of carbon (2 CO $\rightarrow$ CO_2 + C) and to characterize the concomitant chemical transformation (carbide, metal, oxide) of the Co particles deposited in a refractory alumina powder. The poisoning of the Co particles and the de-activation is related with the formation of carbide phases, which decompose at room temperature and which should be characterized in situ at high temperature during the disproportionation process. The experiment uses the high-T reaction chamber at the ESRF powder diffraction beamline (BM16). The diffraction data are collected while the sample is being treated: a) before and during the reduction of the supported oxide in hydrogen at 600C; b) during and after the catalytic reaction under a CO or CO/H_2 flow between 420 and 600C. The gaseous reaction products are examined by on-line gas chromatography. Depending on the absence or the presence of hydrogen, TEM images of the deposits show that CO disproportionation produces nanotubes with co-axial cylindrical graphene layers or filaments made of stacked graphene cone segments[3]. The d spacing of the carbon 002 diffraction line is slightly different depending on the conical or cylindrical morphologies of the carbon layers. A major difficulty in the Rietveld refinement of the patterns is to disentangle the Co and C phase diffraction signals from the alumina diagram and from the scattering arising from the capillary container.

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