Rev.Adv.Mater.Sci. 18(2008) 326-328

NANOCOMPOSITES INTERMETALLICS / OXIDES, PRODUCED BY MA SHS

N.Z. Lyakhov[1], P.A. Vityaz[2], T.F Grigoryeva[1], T.L. Talako[2], A.P. Barinova[1], I.A. Vorsina[1], A.I. Letzko[2] and S.V. Cherepanova[3]

[1]Institute of Solid State Chemistry and Mechanochemistry of Siberian Branch of Siberian Branch of Russian Academy of Sciences, Russia
[2]Powder Metallurgy Institute of National Academy of Science, Belarus Republic
[3]Boreskov Institute of Catalysis of Siberian Branch of Russian Academy of Sciences, Russia

Received: March 29, 2008

Abstract. MA SHS was used for preparation of oxide / intermetallics nanocomposites. Mechanochemical reduction of Fe_2O_3 was investigated in conditions of high dilution by iron and aluminium. It shows that excess of Al content significantly alters the process. Mechanical activation of mixture 60.9 wt.% Fe + 26.6 wt.% Al + 12.5 wt.% Fe_2O_3 leads to formation of nanocomposite Al_2O_3/Fe/Al, and following SHS creates nanocomposites Al_2O_3/FeAl.

1. INTRODUCTION

The combination of mechanical activation (MA) and self-propagating high-temperature synthesis (SHS) allows to extend the possibilities and to reduce disadvantages for both methods**.** The different nanosized intermetallic compounds (FeAl, $MoSi_2$, NiAl, NiTi, $NbAl_3$) were obtained by MA SHS [1-7].

The purpose of this work is to use MA SHS for preparation of oxide/intermetallic nanocomposites.

If a mixture of metals that can form intermetallic compounds in the course of SHS, one of the components being able to reduce the initial oxide, is used as a diluent, mechanochemical activation can result in metallic composites with uniformly distributed oxides. Thus, in the course of mechanical activation of the Me'_xO_y + Me'' + Me' mixture, the chemical reaction Me'_xO_y + Me" $\rightarrow$ Me''_zO_x + Me' yields Me'/Me"/Me''_zO_k composites and subsequent SHS from these materials will give $Me'_nMe''_m$ /Me''_zO_k composites.

2. EXPERIMENTAL

AGO-2 planetary ball mill with water cooling was used (jar volume 250 cm^3; ball diameter 5 mm; ball charge 200 g; sample weight 10 g; rotation speed of grinding jars around the common axis ~1000 rpm). Activation was carried out in argon atmosphere.

The activated mixture was compacted in a mould 17 mm in diameter and 25 mm in height. The synthesis was carried out in argon atmosphere. The sample was ignited by an electrically heated tungsten coil. After the combustion process, the sinter was powdered.

XRD-patterns were obtained with URD-63 diffractometer. Microstructure was studied by scanning electron microscopy (a Camscan scanning electron microscope with a Link Analytical AN10000 energy dispersive X-ray analyzer, Link ZAF4-FLS software was used for quantitative analysis), and

Corresponding author: T. F. Grigoryeva, e-mail: grig@solid.nsc.ru

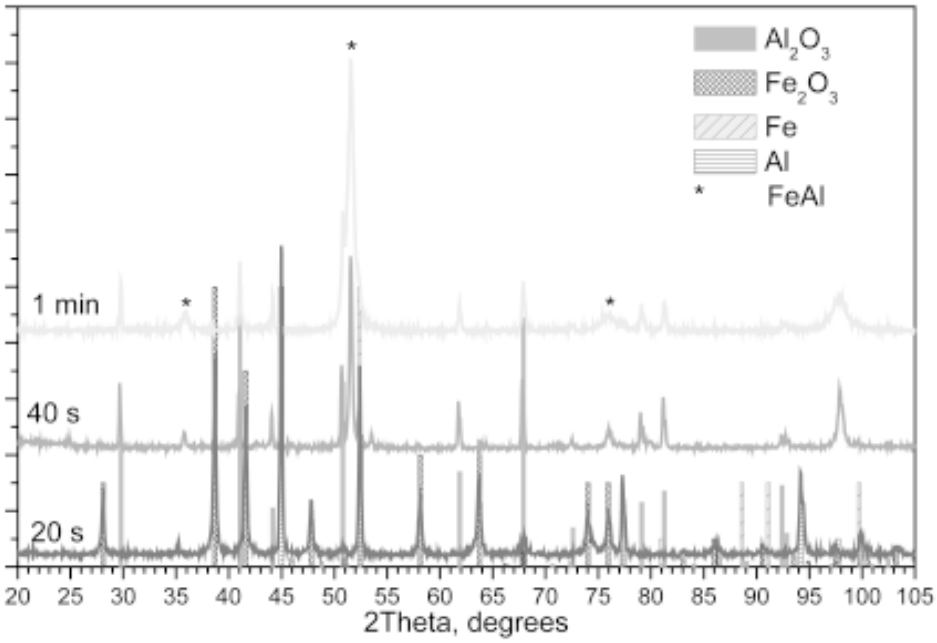

Fig. 1. X-ray patterns of activated mixtures Fe_2O_3 + 4 Al.

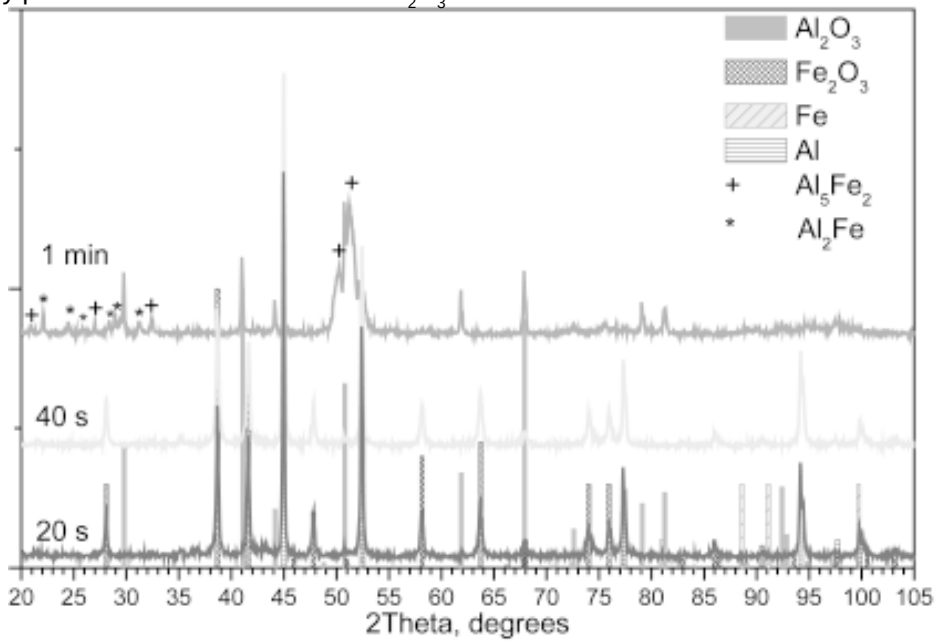

Fig. 2. X-ray patterns of activated mixtures Fe_2O_3 + 6 Al.

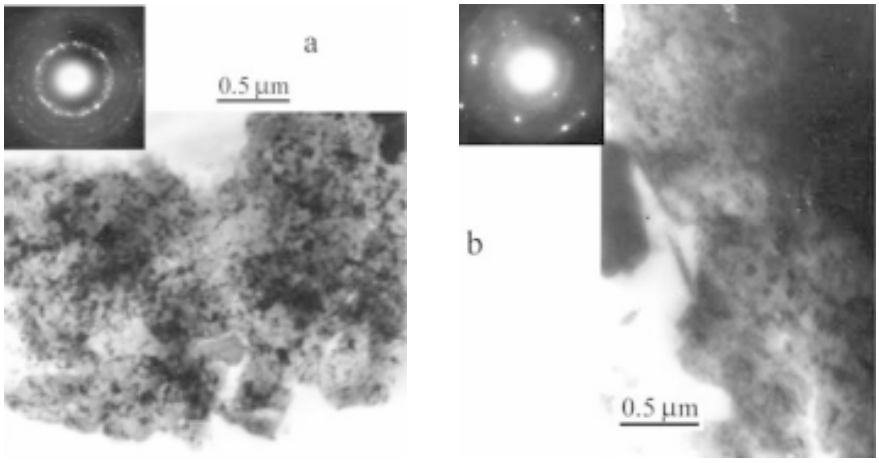

Fig. 3. High-resolution microphotographs of a Fe_2O_3 + Fe + Al sample after (a) mechanical activation and (b) SHS.

transmission electron microscopy (a JEM 1000x microscope).

3. RESULTS AND DISCUSSION

Mechanochemical activation of mixture 60.9 wt.% Fe + 26.6 wt.% Al + 12.5 wt.% Fe_2O_3 was investigated. According to equation of chemical reaction (Fe_2O_3 + 2 Al = Al_2O_3 + 2 Fe; ΔH = - 840 kJ/mol) the 4.2 g of Al is required for reduction of 12.5 g of Fe_2O_3. The mechanochemical reaction of reduction takes place in a condition of high dilution by iron and aluminium. The influence of Fe, Al excess content on the process and phase composition of product was studied.

At equimolecular ratio, the reaction between Fe_2O_3 and Al may would finish during 30-40 s of mechanical activation. At the double Al excess the appearance of α-Al_2O_3 was detected after 40 s by IR study. X-ray data of this reaction product indicates on formation of intermetallic and a-Al_2O_3 (Fig. 1). At triple Al excess, Fe and mixture of intermetallic compounds were revealed after 1 minute of MA (Fig. 2). Further increasing of Al content extends the induction period considerably.

In conclusion, an excess of Al content significantly alters the process.

Formation of lower iron oxide (FeO) after 5 min of mechanical activation was established for the mixture α-Fe_2O_3 + Fe by IR study.

The X-ray powder diffraction pattern of the 12.5 wt.% Fe_2O_3 + 60.9 wt.% Fe + 26.6 wt.% Al mixture after 2 min of activation resulted two phases: Fe and Al. No oxide phases, including the initial iron oxide, were revealed by X-ray diffraction. The absence of reflections due to the initial phase of ferric oxide implies that it was reduced by aluminum, and the lack of reflections due to alumina is associated either with the formation of an X-ray amorphous modification or with the fact that fine alumina particles are coated and spatially separated by plastic metals.

Transmission electron microscopy showed that all components of the mechanochemically synthesized composite are nanosized (Fig. 3a). These data allowed us to assume that, in the course of mechanical activation, ferric oxide is reduced with aluminum to give highly dispersed alumina and the Fe/Al/Al_2O_3 composite is formed. This composite has a layered structure. Thus, mechanochemical stage for mixture Fe + Al + Fe_2O_3 results in the reduction of Fe_2O_3 and creation of contact surface between Al and Fe.

The mechanochemically prepared nanocomposite was used as the initial material in the SHS process, leading to the formation of the FeAl intermetallic compound. The SHS product inherits the morphology of the initial composite, although the initial material contained a mixture of the metals, while the product has their chemical compound. The retention of morphology can be evidence of a highly uniform distribution of the initial metals, Fe and Al, in the mechanically obtained nanocomposite. Transmission electron microscopy showed that the use of nanocomposites as the initial material ensures a high dispersion of the resulting SHS products (Fig. 3b).

The similar results were produced for initial mixtures Fe + Al + Cr_2O_3 and Ni + Al + NiO.

ACKNOWLEDGEMENTS

The investigation has been performed within the integration project of Siberian Branch of RAS No 98 “Mechanocomposites are precursors for creation of materials with new properties”.

REFERENCES

[1] T.Grigorieva, M.Korchagin and N.Lyakhov // *KONA* **20** (2002) 144.

[2] V.Itin, T.Monasevich and A.Bratchikov // *Fizika goreniya i vzryva* **5** (1997) 48, In Russian.

[3] V.Gauthier, C.Josse, F.Bernard, E.Gaffet and J.P. Larpin // *Mater. Sci. Eng.* **A265** (1999) 117.

[4] F.Charlot, E.Gaffet, B.Zeghmati, F.Bernard and J.C.Niepce // *Mater. Sci. Eng.* **A262** (1999) 279.

[5] C.Gras, F.Charlot, E.Gaffet, F.Bernard and J.C. Niepce // *Acta Mater.* **47** (1999) 2113.

[6] E.Levashov, V.Kurbatkina and K.Kolesnichenko // *Izvestiya vusov. Tsvetnaya metallurgiya* **6** (2000) 61, In Russian

[7] V.Gautier, F.Bernard, E.Gaffet, D.Vrel, M.Gailhanou and J.C. Larpin // *Intermetallics* **10** (2002) 377.