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X-ray diffraction and Mössbauer characterization of Raney Fe-Ni catalysts

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Raney type catalysts were prepared by means of a two step procedure: (1) Mechanical alloying of the metals and, (2) alkaline aluminum leaching. Mechanical alloying is a novel alternative related to the synthesis of skeletal Ni catalysts. X-ray diffraction, and Mössbauer spectroscopies were performed for catalysts characterization. Binary Al-Ni and ternary Al-Ni-Fe alloys were produced by mechanical alloying from pure metallic powders; in particular, the intermetallic β-(AlNi) phase was formed with a fine microstructure as a non-equilibrium phase; then, aluminum was selectively removed. After aluminum leaching the β-(AlNi) phase was transformed into the more stable nickel fcc structure.

Introduction

Raney-type catalysts were first introduced in 1924. Since then, these catalysts, both in powdered and granular form, have been widely applied in many commercial processes, including hydrogenation of unsaturated organic compounds.[1,2] Conventional Raney-Ni catalysts (with fcc structure) are produced by removing the aluminum from binary Ni-Al alloys by leaching with an aqueous alkaline solution.[3] In contrast to conventional methods, mechanical alloying (MA) is a process used to produce alloys at room temperature having a fine microstructure; furthermore, it can be applied to alloy incompatible materials.[4]

IVANOV et al.[5] reported that after leaching Al atoms from β-AlNi structures of mechanically alloyed Ni and Co aluminides, the catalysts remained on the metastable bcc structure leading to a very active Ni and Co Raney-type catalyst. The bcc structure converts into the more stable nickel fcc structure at about 463 K. In this work, MA was applied to obtain Al-Ni and Al-Ni-Fe alloys from elemental metallic powder blends.

Experimental

Catalysts preparation

Binary Al-Ni and ternary Al-Ni-Fe were prepared from powder mixtures, with nominal atomic compositions $Al_{65}Ni_{35}$, $Al_{65}Ni_{30}Fe_5$, and $Al_{75}Ni_{15}Fe_{10}$ (in atomic percent). The mixtures were milled in an attritor ball mill (Union Process, model 1-S), equipped with a water-cooled jacket and a 3.8 l stainless steel vessel container. Stainless steel balls (3.2 mm diameter) were used as milling media.

Due to the pyrophoric properties of the alloyed material, a slow surface oxidation is taking place "passivating" the alloy. The alloyed samples were then treated for 2 hours with a 20 wt% KOH aqueous solution, held at its boiling point (digestion), in order to leach away selectively the aluminum atoms.

Characterization techniques

X-Ray diffraction (XRD) experiments were carried out in a Shimadzu XD-3, using Cu Kα radiation.

Mössbauer spectra were collected at room temperature using an Austin Science Associates Model S-600 Mössbauer Spectrometer. The spectrometer was operated in constant acceleration mode with a 10 mCi (370 MBq) Co/rhodium source. Isomer shift values are related to metallic iron.

Results and discussion

X-ray diffraction (XRD)

Both mechanically alloyed samples $Al_{65}Ni_{35}$ and $Al_{65}Ni_{30}Fe_5$ produced similar diffraction patterns corresponding to the bcc structure of NiAl alloy (β2), as shown in Fig. 1. The formation of the β2 structure is probably due to the introduction of defects by the MA process, because their compositions were out of the range in which this phase is present at equilibrium conditions.

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