



Mechanochemical reduction of natural pyrite by aluminum and magnesium



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ABSTRACT

Mechanochemical reaction of pyrite with aluminum and magnesium were carried out for pyrite reduction using a planetary ball mill in an inert atmosphere. Thermodynamic studies indicated that the mechanochemical reactions are mechanically-induced self-propagating reaction (MSR). The XRD patterns of pyrite and magnesium mechanochemical reaction proved the reaction could not be complete after 280 min milling under the test conditions. On the other hand, the mechanochemical reaction between aluminum and pyrite completed after 120 min under similar milling conditions. The results confirm that in the primary MSR reactions troilite (FeS) phase was formed as an evidence for non-stoichiometric mechanochemical reaction. Thus, the mechanochemical reaction primary test of pyrite and aluminum was corrected with addition of more aluminum. The XRD results verified the troilite could be completely eliminated from the final product and the iron and aluminum phases contained main constituents of the obtained powder. The FE-SEM images indicated that the particles are about 100 nm to less than 1 μm in size. Finally, from SEM/EDS and XRF results, the zero-valent iron (ZVI) particles were surrounded by aluminum sulfide phase comparing.

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1. Introduction

In mechanochemistry, mechanical energy was applied for reactivity enhancement, performance of alloying and synthesis processes. The processes have widely used in various fields such as hydrometallurgy and preparation of advanced and nano structured materials [1]. There are different methods for preparation of nano structured materials and among them, mechanochemical synthesis of nano materials are often faced intrinsic problems due to its top-down nature. A part of the problem arises from agglomeration phenomenon when ultrafine particles with high surface energy aggregate and stick to each other, hindering formation of nano particles. Also, due the exerted impact during intensive milling, the solid composite phases produced during mechanochemical reactions are not well separable when needed. Although many efforts were conducted to solve the problems by using additives and selection of proper mechanochemical reactions, the agglomeration and separation problems are essential and create general challenges in mechanochemical preparation of nano structured

materials [1–3]. The complete reaction between reactants during mechanochemical reaction is first criterion to find a solution for the problems, especially the separation problem. The mechanically-induced self-propagating reaction (MSR) is a mechanochemical reactions type with high potential capability to proceed completion. In the MSR reactions, the ratio of standard reaction enthalpy to product heat capacity ($\Delta H/\Delta C_p$) is higher than about 2000 K [4,5]. The displacement type of MSR reactions are known as a powerful tool for reduction of sulfide compounds during metal-sulfide mechanochemical reaction. The reactive metals such as Al and Mg are supposed to reduce the less reactive metals in sulfide compounds [4]. Nevertheless, over the last years, many investigations were developed for mechanochemical reactions wherein advanced nano structured materials were prepared [2–8].

Preparation of nano zero-valent Iron (NZVI) is classified in nano scale environmental technology due to appreciable capability of NZVI for removing of various contaminants such as chlorinated organic solvent, organochlorine pesticides, organic dyes, various inorganic compounds and metal ions such as Pb (II), As(III), Cu(II) [9]. Various chemical methods have been used for preparation of NZVI generally based on reduction of ferric or ferrous iron from aqueous solutions by using related reducing agents such as sodium

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borohydride [10]. Due to the chemical procedure performed in aqueous media, the NZVI surface were oxidized converting to iron oxide nano powders [9–11]. Although in most cases, it has been tried to eliminate oxygen during preparation operation and maintenance condition, the NZVI powders have oxidized. Therefore, the mechanochemical synthesis methods having tougher controlled atmosphere (reaction media) can be employed to suppress oxidation process during NZVI synthesis. Regarding to existed solid and natural sources of iron, iron sulfides such as pyrite are suggested as one of the best nominates for preparation of NZVI by using mechanochemical methods.

It should be noted that natural pyrite (FeS_2) with two main constituents of iron and sulfur is the most common gangue mineral associated with valuable minerals in ore deposits and it can be processed easily by conventional flotation methods. The pyrite concentrate is a refractory concentrate due to difficulty in its treatment [12–15]. The gangue pyrite disposal is unavoidable in mineral processing plants raising environmental concerns. Previous investigations indicated that pyrite can be used as an adsorbent [15–17], precursor for superconductor preparation, high-energy density batteries [18] and photocatalyst [19], opening new opportunities for the gangue mineral of pyrite to be applied. The applications of pyrite have advantages e.g. it is cost-efficient, abundant and easier to handle due to its simple chemical composition.

Regarding to the facts that the application of pyrite as an initial source for preparation of NZVI is not reported, the present study aimed to investigate mechanochemical reduction of pyrite by reactive metals of aluminum and magnesium metal powders as first step for preparation of NZVI from natural pyrite by using mechanochemical methods. The progress of mentioned MSR reactions are evaluated using X-ray diffraction analysis and an attempt is exerted to complete at least one of them.

2. Experimental

2.1. Materials

A high-purity pyrite concentrate was obtained using conventional beneficiation methods. XRD analysis of the pyrite concentrate disclosed that the pyrite reflections solely corresponded with JCPDS No. 1-1295 (Fig. 1).

The aluminum (Merck 1.01056.0250) and magnesium (Merck

art 5815) metal powders were used for mechanochemical reaction of pyrite as will be described in the following section.

2.2. Mechanochemical reactions

Mechanical reactions of pyrite was conducted in a planetary ball mill (Pulverisette 6, FRITSCH, Germany) in a 250 cm^3 vial with a rotation speed of 400 rpm under argon atmosphere to prevent unwanted reactions. 10 stainless steel balls having diameters of 20 mm were used for milling by the ball to powder weight ratio of 40:1. After per 20 min milling, 20 min rest time was considered as operational instruction. The milling conditions during mechanochemical reaction of the pyrite with magnesium and aluminum were similar. For mechanochemical reaction of pyrite, the natural pyrite and the mentioned powders were co-milled in accordance with the stoichiometry ratios presented in the following reactions and just in final step, the minor changes were considered for improving mechanochemical reaction of pyrite and aluminum. The thermodynamic parameters of the reactions are obtained by HSC 6.0 software (Table 1).



2.3. Characterization

The X-ray diffraction patterns of natural pyrite and products were collected with Bruker Axs D8 advance instrument (Germany), applying $\text{Cu K}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$) at 50 KV and 250 mA in the two theta ranges of $25\text{--}70^\circ$. The patterns were recorded using a step size of 0.02 and a counting time of 3 s per step. The chemical composition of final product was analyzed using X-ray fluorescence

Table 1

Thermodynamic parameters of the reactions of pyrite with aluminum and magnesium metal powders.

	ΔH (kJ)	ΔS (J/K)	ΔG (kJ)	$\Delta H/\Delta C_p$ (K)
MSR of pyrite with Al	-782.5	43.67	-795.52	2749.83
MSR of pyrite with Mg	-519.9	9.5	-519.9	4484.21

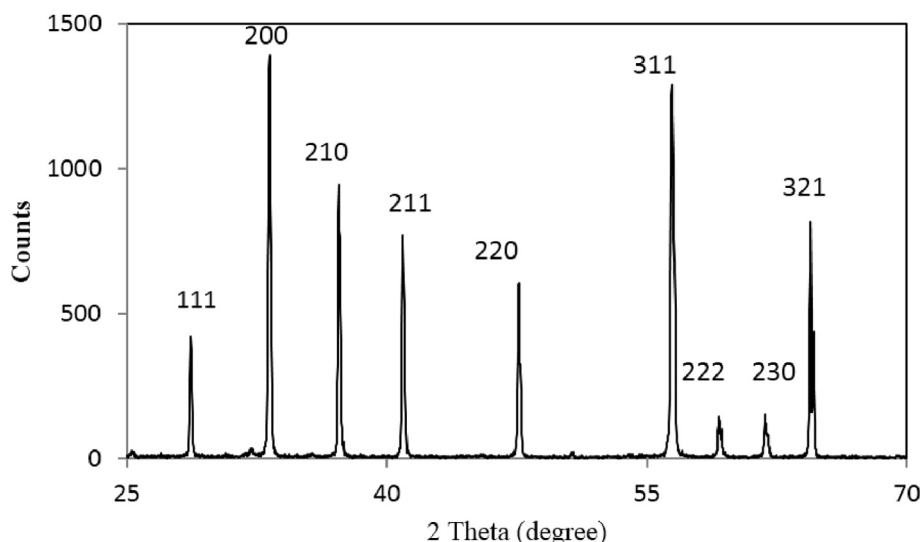


Fig. 1. XRD pattern of the initial natural pyrite.

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