NEW METHOD FOR PREPARATION NANOCOMPOSITE BASED ON PYRYTE

Urakaev F. Kh. *, Shevchenko V.S.
*(United Institute of Geology, Geophysics and Mineralogy, Siberian Branch of RAS, Novosibirsk, Russia; *Novosibirsk State University, Novosibirsk, Russia)*
urakaev@uiggm.nsc.ru

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A new method for performing mechanochemical reactions with the participation of the material of milling tools is proposed. The method is based on using abrasive additives, e.g. fused quartz. The essence of the method is the abradant-reaction wear of the milling tools of a mill. The method has been tested for the preparation of nanocomposite based on pyrite during mechanical activation of a mixture of amorphous quartz and sulfur in AGO-2 planetary ball mill with steel furniture.

In the recent years, we evidence a rapid increase in the number of investigations in mechanochemistry [1-3]. As a rule, the role of milling tools in mechanochemical processes is limited only to ascertaining the possibility of the contamination of the products by the material of milling tools [1,4]. On the other hand accumulation of sulfur at the enterprises of the petroleum and gas complex puts a problem of it recycling. Because of this, the goal of the present investigation was to study the treatment of a mixture of sulfur with quartz as an abrasive in a mill with steel furniture.

Sulfides are synthesized from the elements by heating. However, the degree of homogeneity of the final product are far from being perfect; this depends on many factors, in particular on the size and surface state of initial reagents. Mechanochemical reactor eliminates these shortcomings. Iron sulfides are at present used in the many specific area [5].

The results of modeling of mechanochemical reactions involving sulfur show that the most important process for the synthesis of sulfides, for example zinc sulfhide, is plastic yielding of sulphur which leads to its amorphization - polymerization with the transition to vitreous state [3]. The characteristics of this process are determined by the thickness of layers lined with softer sulphur on the surface of zinc particles. However, no experimental confirmation of this process has been obtained. Because of this, in order to confirm the theory, it was necessary to choose such a system for mechanical activation that would contain, along with sulfur, an inert, desirably amorphous component in order to simplify the interpretation of the results of X-ray powder analysis. Usual glass and fused quartz, which are fully amorphous materials, are proper for this role. The use of these additives can help proving amorphization of sulfur. This has also been the subject of the present investigation.

Sulfur crystals (see PDF 83-2285) in amount of 0.4 to 2 g, was added to the weighed portions (3 g) of crushed object-plate glass and quartz tube. The samples were preliminarily ground and homogenized in Fritsch Pulverisette mill equipped with agate furniture. Experiments on mechanical activation Experiments on mechanical activation (MA) of thus prepared samples and sulfur crystals alone (2 g) were performed in the steel two-drum ball planetary mill AGO-2. The volume of each drum was ~140 cm^3, the number of balls was 400, ball diameter 0.4 cm. The relative velocity of collisions of milling tools was ~11 m/s [6].

The results of X-ray of sulfur alone (for MA time up to 3 h) show that sulfur does not undergo any structural rearrangements (see PDF 83-2285): all the reflections are conserved without any noticeable broadening or changes in intensities, except for the absence of the halo which is due to the presence of particles of glass or amorphous quartz. The situation changes (Fig. 1) when sulfur is treated in the presence of inert glass particles: the structural changes that occur with sulfur in accordance with the results of modeling [3]. Crucial changes occur during the MA of samples based on quartz. The formation of pyrite (FeS_2, see PDF 71-2219) is shown in Fig. 2. In this case, amorphization of sulfur [3] and it chemical reaction in combustion regime [2] with iron nanoparticles formed as a result of abrasive wear of steel milling tools by quartz occur concurrently. It is most likely connected by that hardness of quartz particles is much higher than that of steel or glass, and the melting point of quartz is much higher than that of glass.

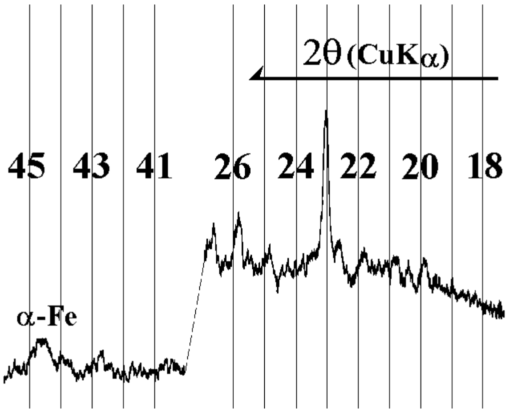

Fig. 1. The X-ray data for the system glass (3 g) – sulphur (0.4 g) for MA time 2 h.

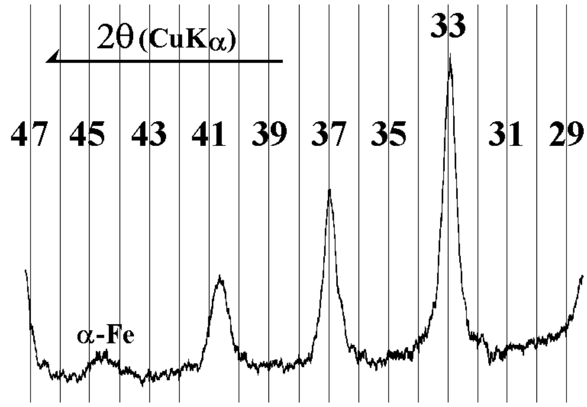

Fig. 2. The X-ray data for the system fused quartz (3 g) – sulfur (1.6 g) for MA time 2 h.

Using the X-ray data, we applied the procedure [1,5] to calculate crystal blocks and distortions in the structure of the resulting pyrite. The fine lattice parameters were calculated from half-width of the profile of the diffraction peaks (220) and (440), accepted for pyrite. In order to determine the instrumental broadening, we used the profile of crystal pyrite lines. The obtained size of blocks in pyrite was ~24 nm (for iron particles, ~10 nm), distortion value was ~1%. Similar results were obtained also in [5], but for more than 110 h of mechanical treatment of Fe + 2S powder mixture.

Thus, nanocomposite based on pyrite and amorphous quartz is obtained during the mechanical activation of a mixture of quartz and sulfur in AGO-2 planetary mill with steel furniture (it is possible to use scrap of other metals as a milling tools). The time necessary for the process is 1-2 orders of magnitude shorter than that required for traditional mechanical alloying of iron and sulfur powders [5]. The method can also find broad application both for obtaining chalcogenide glasses and processing of geological and technogeneous materials, e.g. complex sulfide concentrate [7].

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