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Effects of mechanical activation on the structural changes and microstructural characteristics of the components of ferruginous quartzite beneficiation tailings

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ABSTRACT

The effects of mechanical activation in a planetary mill on the structural changes and microstructural characteristics of the components of ferruginous quartzite beneficiation tailings generated by wet magnetic separation process were studied using X-ray and laser diffraction methods. The results revealed the relationship between variations in the mean particle size of activated powders and the milling time. The crystallite size, microstrain, lattice parameters and unit cell volumes were determined for different milling times in powder samples of quartz, hematite, dolomite, and magnetite from the beneficiation tailings. The main trends in the variation of the crystallite size of quartz, hematite, dolomite, and magnetite as a function mean particle size of powder samples were revealed. Changes in the particle shape as a function of the activation time was also investigated.

1. Introduction

At present, more than one-half of commercial iron ore production in Russia comes from the Kursk Magnetic Anomaly (KMA) basin. Similarly, this region accounts for more than one-half of the tailings from ore beneficiation. Estimates indicate that the amount of iron ore tailings generated and stockpiled on site (together with the existing tailings) within the KMA using the common beneficiation methods may reach 21–24 billion tons over the next fifty years. Tailings from beneficiation process containing up to 80–95% free silica pose potential risk of silicosis. Long-term storage of tailings is usually the biggest environmental concern. Effective utilization of massive tailings resources can require modification of the structure and properties of waste materials.

Mechanical activation or activation grinding is one of the most effective means to cause changes in the structural characteristics and properties of the material during higher-energy milling under the action of mechanical forces compared to the traditional ball millings. Such grinding results in the structural-chemical transformations of minerals, formation of lattice distortion and dislocations and point defects.

The waste materials generated during beneficiation of ferruginous quartzite using the wet magnetic separation process are complex multicomponent mixtures the microstructural changes of which during comminution have yet to be studied.

The goal of this study is to investigate structural and microstructural changes of waste materials induced by mechanical activation, which can be used to develop a novel composite material consisting of dispersed fine particle with substantially enhanced properties. The trends in the change of their microstructure as a function the specific surface area can provide the basis for developing quick test methods for assessment and ranking waste materials in terms of their potential for effective backfill mix design. Void filling using mill tailings especially in metal mining is one of the best techniques [1].

2. Materials and methods

The chemical analysis by X-ray fluorescence on the ARL Optim'X spectrometer revealed that the waste material generated during beneficiation of ferruginous quartzite using the wet magnetic separation process contains (in wt.%) about 59. 82% SiO_2, 20.36% Fe_2O_3, 6.67% CaO, 2.20% MgO, 7.63% CO_2, 1.09% P_2O_5, 0.91% Al_2O_3, 0.438% K_2O, 0.34% Na_2O, 0.274% TiO_2, 0.147% MnO, 0.0705% SO_3, 0.0355% WO_3, 0.0302% SrO, 0.0089% CuO, 0.0026% ZrO_2, and 0.0021%

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Table 1
Summary of particle size distribution data for powder samples.

Characteristics	Starting size	Milling time 1 h, in ethanol	Milling time 2 h, in ethanol	Milling time 6 h, in ethanol	Milling time 6 h in ethanol +6 h in air	Milling time 12 h, in ethanol
D10[a] (10% of particles) (μm)	11.336	1.020	1.003	0.856	0.727	0.471
D50 (50% of particles) (μm)	59.024	6.035	3.983	2.601	2.071	1.187
D90 (90% of particles) (μm)	141.340	25.770	11.143	7.454	7.252	2.017
Mean particle size, (Mean volume diameter), d[4.3] (μm)	69.16	9.65	5.23	3.46	3.10	1.22
Fraction	Content of fine-grained fractions in the bulk sample (%)					
0–1 μm	1.61	12.27	14.85	17.29	22.76	47.90
0–3 μm	4.30	36.24	40.15	49.98	66.82	99.91
0–5 μm	5.63	45.38	53.14	72.28	74.21	100.00
0–10 μm	9.17	65.66	85.54	97.38	97.36	100.00

Note:
[a] D10 is the particle size at which 10% of the particles are finer; D50 is the grain size at which 50% of the particles are finer; D90 is the particle size at which 90% of the particles are finer.

Y_2O_3. The XRD spectra were recorded using an Ultima IV Rigaku diffractometer (Cu K_α, $\lambda = 0.154059$ nm, in the range of $2\theta = 10$–$110°$ at the step size of $\Delta(2\theta) = 0.02°$ and scanning speed of 2.5 s) and contained the diffraction lines from quartz SiO_2 (ICDD PDF-2# 00-046-1045), hematite Fe_2O_3 (ICDD PDF-2# 01-086-0550), magnetite (ICDD PDF-2# 01-086-1346) Fe_3O_4, and dolomite $CaMg(CO_3)_2$ (ICDD PDF-2# 01-073-2361).

The quantitative analysis of XRD patterns and phase identification were performed using the PDXL RIGAKU software. The diffraction data were smoothed by the Savitsky-Golay method [2] and the background line was subtracted using the Sonneveld-Visser method [3]. The diffractions were described by a Gaussian function and the Rachinger correction was applied to separate a diffraction peak doublet into the $K_{\alpha1}$ and $K_{\alpha2}$ components [4]. The mean crystallite (Coherently Scattering Domains (CSD)) size (D) for quartz, hematite, magnetite, and dolomite was determined from the β line broadening analysis. The instrumental angular resolution function of the diffractometer $\mathrm{FWHM_R} = (utg^2\theta + vtg\theta + w)^{1/2}$, where $u = 0.0093$, $v = -0.0090$ and $w = 0.0078$ was determined in a special diffraction experiment using a standard sample of the lanthanum hexaboride LaB_6 (PRF-12). The X-ray diffraction peak broadening $\beta = (\mathrm{FWHM}^2_{exp} - \mathrm{FWHM}^2_R)^{1/2}$ was determined by comparing the full width at half-maximum of an experimental diffraction reflection FWHM^2_{exp} with the instrumental angular resolution function of the diffractometer FWHM^2_R [5]. The mean crystallite size D and the contribution from the microstrain ε were determined using the Williamson-Hull plot [6] based on an approximation that the functions of size and strain contributions are described by the Gaussian functions [7]:

$$\beta^2 \cdot \cos^2\theta = (4\varepsilon \sin\theta)^2 + \left(\frac{\lambda}{D}\right)^2 \quad (1)$$

where θ is the Bragg angle, β is the broadening of the diffraction line, ε is the microstrain, and λ is the X-ray wavelength.

The plot of $\left(\frac{\beta\cdot\cos\theta}{\lambda}\right)^2$ vs. $\left(\frac{4\sin\theta}{\lambda}\right)^2$ is a straight line with the tangent of the inclination angle proportional to ε^2 and the y-intercept inversely proportional to D^2.

The tailings samples were ground to powder in a Pulverisette 5 laboratory planetary ball mill at 400 r/min with automatic time reverse rotation every 30 min. For each run, maximum powder batches of 210 g were ground using one 250 ml stainless steel jar with balls of 5–20 mm diameter and 790 g total weight. The powders were milled in air and 60 ml ethanol for intervals of 1–12 h and dried after milling.

The particle size distribution and elongation ratio were determined with an Analysette 22 NanoTec Laser Diffraction Analyzer using the wet dispersion and ultrasonication of the sample powder [8]. The Analysette 22 NanoTec is the instruments which produces a particle size distribution and particle shape analysis (elongation ratio) in a single measurement.

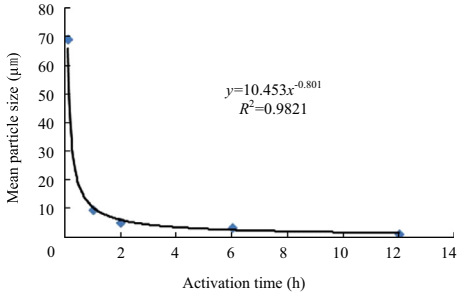

Fig. 1. Variation of the mean particle size as a function of the activation time.

Fraunhofer theory was applied for interpretation of diffraction pattern for determining particle size. The elongation was defined as the ratio of the radii of the ellipsoid.

3. Results and discussion

3.1. Particle size and shape

The tailings samples with an initial mean particle size of 69.16 μm were milled in ethanol at intervals of 1, 2, and 6 h to sequentially reduce the mean particle sizes to 9.65, 5.23 and 3.46 μm, respectively, then the powders were milled for 6 h in ethanol and 6 h in air to particle size of 3.1 μm; and finally the samples were milled in ethanol for 12 h to a mean particle size of 1.22 μm. The data on the particle size distribution in fine-grained fractions are summarized in Table 1. Changes in the mean volume diameter of the particles as a function of the milling time are shown in Fig. 1.

The experimental data indicate that after 2 h of milling over 85% of the material was milled to the particle size below 10 μm. The degree of crystallinity of fine mineral particles below 10 μm is becoming increasingly important because fine milling is capable of producing novel crystalline materials with crystallite sizes at the nanometer scale in which tailored properties can be used to improve hydrometallurgical processes, reduce annealing temperature, accelerate the reactivity of the binders, allow for the formation of metastable phases and particles with high surface energies [9]. Although a substantial difference was found in the particle size after 1, 2, 6, and 12 h of milling, intensive grinding

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