



Characterization of the microstructure of mechanically-activated olivine using X-ray diffraction pattern analysis



Jiajie Li*, Michael Hitch

Norman B. Keevil Institute of Mining Engineering, University of British Columbia, 517-6350 Stores Road, Vancouver, B.C. V6T 1Z4, Canada

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ABSTRACT

During mechanical activation, disorder in the crystal structure of olivine has been recognized as one of the most important factors for enhancing the reaction rate of downstream carbonation processes. This paper investigates the detailed microstructure of olivine after mechanical activation in a SPEX 8000 mill using the X-ray diffraction technique. In order to successfully extract information from the microstructure using X-ray diffraction patterns, various models were applied and compared, including the Scherrer method, Williamson–Hall method, Multiple Whole Profile fitting method, and Rietveld method. Results show that with 120 min of milling, the media particle size initially decreased dramatically, and then increased slightly, the BET surface area and amorphization degree increased, the lattice was initially compressed and then expanded, the crystallite sizes decreased, and strains increased. The strain value for 120-min mechanically-activated olivine, calculated by the Scherrer method, Williamson–Hall method, Rietveld method, and MWP method, was 2.15×10^{-3} , 1.64×10^{-3} , 0.73×10^{-3} , and 0.28×10^{-3} , respectively. The Rietveld method was found to be the most accurate method and therefore most suitable method for characterizing the microstructure of olivine and mine waste materials containing olivine.

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1. Introduction

Mechanical activation by high intensity milling has been commonly used as an effective pretreatment method in various areas, such as mining, metallurgy and materials engineering. Mechanical activation is one of the three broad catalogues for grinding, the other two being coarse grinding and fine grinding (Boldyrev et al., 1996). In mineral processing, the aim of coarse and fine grinding is liberate the ore for downstream separation. Furthermore, fine grinding also used after mineral separation in order to give concentrates the right particle size distribution or a sufficiently high surface area. Whereas mechanical activation aims to enhance a reaction (Baláž, 2008). A level of mechanical activation can be achieved as the material is milled to the brittle-ductile transition range (Boldyrev et al., 1996). Beyond the integration of particles and the formation of new surfaces, mechanical activation alters the properties of a material (Tkáčová, 1989). Additional changes such as structural distortions are crucial in determining the reactivity of material. These distortions result in the storage of excess enthalpy, which lowers the activation energy of subsequent reactions (Boldyrev et al., 1996). The main advantages of mechanical activation include: (a) its simplicity as a process, which

can be completed over a single grinding stage; (b) its attractiveness as a more environmentally friendly process, as it does not use solvents, gas, etc.; (c) its ability to obtain metastable state products or nano-particles, which are hard (if not impossible) to achieve using traditional methods (Boldyrev, 2006). The only drawback to mechanical activation is its intensive energy consumption. However, when integrated as part of the total milling process, the operational energy consumption can be reduced to a practical level (Haug, 2010).

Olivine is an industrial mineral composed of an independent SiO_4 tetrahedral linked by divalent ions in 6-fold coordination (such as Mg^{2+} , Fe^{2+}), with a theoretical formula of $(\text{Mg}, \text{Fe})_2\text{SiO}_4$ (Klein et al., 1993). Traditionally, olivine is used for refractory material, slag conditioner, blasting sand and foundry sand. When the reactivity of olivine's surface is enhanced, it has many new applications, such as a neutralizer for sulphuric acid, a heavy metal adsorbent, a catalyst for decomposing toxic organic chemicals, a magnesium source for soil improvement in agriculture, and an additive in water treatment (Kleiv and Thornhill, 2006). Most recently, olivine, especially its magnesium end-member forsterite, is recognized as one of the most promising feedstock in CO_2 sequestration by mineral carbonation.

In recent years, mechanical activation on olivine has been a popular object of study, and specifically the importance of crystal structure changes for downstream reactions (Fabian et al., 2010;

* Corresponding author.

E-mail address: jiajie.li@alumni.ubc.ca (J. Li).

Nomenclature

A	the degree of amorphization	m	the median of the log normal crystallite size distribution calculated by the MWP method
A_L	the cosine Fourier coefficients of the peak profile	n	the order of reflection ($n = 1, 2, 3 \dots$)
A_L^S	A_L relates to the crystal size	R	the residual value of the figure-of-merit function in Rietveld method
a, b, c	the lattice parameters	R_{WP}	the weighted profile residue in line profile fitting method
B	a parameter in the Williamson–Hall plots, where $B = \frac{\beta_f \cos \theta}{\lambda}$	R_e^*	the effective outer cut-off radius of dislocation
B_0	the background of the diffraction peak for a non-activated mineral and	S_G	granulometric surface area
B_X	the background of the diffraction peak for the mechanically activated mineral	S_{BET}	the BET surface area
b	the Burgers vector of dislocation	t_M	the time of mechanical activation
\bar{C}	the average dislocation contrast factor	V	the volume of a lattice
D	the crystallite size	w_i	the coefficient in R , which is $1/y_i(obs)$
D_V	the volume weighted crystallite size	W_N	the specific milling energy input
d	the lattice spacing of an unstrained sample	$y_i(calc)$	the calculated intensity at i th step
Δd	the change in d at a strained status	$y_i(obs)$	the measured intensity at i th step
d_{10}	10% passing size	β	the integral breadth of a XRD peak
d_{50}	50% passing size	β_C	the Cauchy components of β
d_{90}	90% passing size	β_G	the Gaussian components of β
d_{BET}	the BET size	β_{SC}	the Cauchy component of size-integral breadth
d^*	a parameter in the Williamson–Hall plots, where $d^* = \frac{2 \sin \theta}{\lambda}$	β_{SG}	the Gaussian component of size-integral breadth
FWHM	the full-width at half of the maximum height of a XRD peak	β_{DC}	the Cauchy component of strain-integral breadth
g	the diffraction vector	β_{DG}	the Gaussian component of strain-integral breadth
h, k, l	Miller indices of diffraction planes	β_S	the total-size-integral breadth
I_B	the intensity of background	β_f	the integral width of the sample
I_{max}	the maximum height of a XRD peak	β_D	the total-strain-integral breadth
I_0	the integral intensity of a diffraction peak for a non-activated mineral	ε	the strain, which is defined as $\varepsilon = \Delta d/d$
I_X	the integral intensity of a diffraction peak for the mechanically activated mineral	$\langle \varepsilon_T^2 \rangle$	the mean square strain
K	a unit cell geometry-dependent constant in Scherrer equation	$\langle \varepsilon_T^2 \rangle^{1/2}$	the root mean square strain (RMSS)
L	the Fourier length	σ	the variance of the log normal crystallite size distribution calculated by the MWP method
L_0	the edge length diameter of a crystallite	θ	the diffraction angle, and 2θ is the peak position of XRD profile
MWP	multiple whole profile fitting method	λ	the wave length
M^*	the arrangement of dislocations	ρ	the average dislocation density
		ρ'	the density of olivine

Haug, 2010; Kleiv and Thornhill, 2006; Turianicová and Baláz, 2008). Kleiv and Thornhill (2006) found that structural disordering of the olivine surface is largely responsible for the disproportionate increase in the reactivity of acid leaching. Similarly, Summers et al. (2004) found that dry grinding that caused structural damage improved reactivity by changing the diffusion rate in direct aqueous mineral carbonation. Atashin et al. (2015) suggested that the most desirable microstructure for CO₂ sequestration is about 55 kJ/g milling energy input.

To quantify the structure changes X-ray diffraction patterns were widely applied in many studies. The crystal structure of olivine has been presented using several parameters from peak geometry, such as full width at half maximum (FWHM) (Kleiv and Thornhill, 2006; Summers et al., 2004), peak intensity (Rigopoulos et al., 2015), and integral area of peak (Baláz et al., 2008; Haug, 2010). Haug (2010) has found that the crystallinity calculation based on peak area of the XRD pattern demonstrated the best correlation with energy consumption. The main reason for this is that the crystallinity calculation contains all the factors influencing the shape of the XRD pattern, such as crystallite size, grain size of particles and amorphization materials. However, it is better to analyze the individual structural characteristics in order to have a better understanding of how mechanical energy

activates olivine. At present, little work has been done on accurate characterization of structural distortions caused by the mechanical activation of olivine, such as changes in crystallite size, micro-strain, lattice parameter, and phase transformation.

The aim of the present study is the quantitative and qualitative characterization of microstructural changes in olivine in response to dry mechanical activation, based on X-ray diffraction pattern. Various models for microstructure characterization are discussed, including the Scherrer method, Williamson–Hall method, Multiple Whole Profile fitting and Rietveld method.

2. Material and experimental

The olivine foundry sand (olivine) used in this study was purchased from Ward's Canada Limited, and was from the Twin Sisters ultramafic complex in northwest Washington State, USA. The chemical composition of olivine was analyzed using X-ray fluorescence (XRF) at Acme Analytical Laboratories Ltd. The major oxides of olivine powder were 50.94% MgO, 40.7% SiO₂, 8.39% Fe₂O₃, 0.68% Cr₂O₃, 0.15% Al₂O₃, 0.09% CaO, 0.02% TiO₂, and 0.12% MnO, and loss on ignition (LOI) was 0. X-ray powder diffraction analysis (XRPD) detected approximately 97.9% pure olivine, 0.2% lizardite, 1.5% chromite, and 0.4% quartz in the foundry sand. Normalization of

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