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Effect of wet and dry milling on properties of sintered hydroxyapatite

Siripan NILPAIRACH

Matullergy and meterial science research institute Chulalongkorn University

Abstract

The commercial hydroxyapatite was milled by wet and dry method using porcelain ball mill. The effect of wet and dry milling on the sintering behavior of hydroxyapatite was investigated. The result of this study show that based on the particle size and sintered behavior, wet milling appears do be more effective to improve sintering charecterization of hydroxyapatite powder.

Introduction

The milling of material is widely used in comminution process to reduce the average particle size of material , to modify the particle size distribution , to disperse agglomerate and aggregate ,to modify the shape of particle , to increase the content of colloids and to mixing or blending of two or more material or mix phase.

The ball millis are generally known as method by which numerous ceramic powder including hydroxyapatite can be commimute easily and economically. They are typically used to produced -200 and -325 mesh material wide size distribution and deagglomerate and mix slurries and powder.

It was purepose of the presest work to study the effect of wet and dry milling on milling behavior of hydroxyapatite. The powder charecterization and mechanical properities of sintered hydroxyapatite was studies.

Experiment procedure

The commercial hydroxyapatite (Merck Co., Darmstadt, Germany) was used as a starting material. The particle size distribution, of as-received hydroxyapatite powder was determined by sedimentography using a Malvern Particle Size Analyser. The powder shape was examined SEM

There are two processes for milling : wet and dry. About 80g of hydroxyapatite powder were milled in porcelain ball mill using alumina balls as milling media. In the wet process methanol was added to hydroxyapatite. The powder was dried and crushed in a mortar, after milling. Then the particle size distribution was measured as a function of the milling time. After 250 minute milling, the hydroxyapatite powder were pressed into stainless steel mould 10 ton force. The compact powder specimen was sintered at 1250°C for 4 hour with heating rate of 20 °C/min. The density of sintered compacts were measured using Archimedes principle and the percentage densification calcutated assuming the theoretical density of 3.156 g/cm^3.

The average grain size was determined using the linear intercept method from SEM micrographs. The grain boundary was revealed by etching the sample with a 10% phosphoric acid solution for 1 minute at room temperature. The hardness of each sample was tested using a Shimadzu Microhardness Tester machine with a 1 ton load. The fracture toughness was calculated by an indentation method according to the following equation formulated by Laugier (1)

$$k_{IC} = k^{P} \frac{(C-a)^{-\frac{1}{2}}}{a}\left[\frac{E}{H}\right]^{-\frac{2}{3}}\left[\frac{P}{C}\right]^{-\frac{3}{2}}$$

where ;

k = Calibration Constant

E = Young's modulus

P = Load, Kg

c = Crack Length, um

H = Hardness

2a = Identdiagonal

Result

Figure 1 shows the particle of starting hydroxyapatite powder (HA_p) consists of small granula and its size rang from 0.22-122 μm. Particle size of hydroxyapatite obtained from wet milling was found to be smaller than one from dry milling process when comparing at different time i.e. 15, 40, 90, 120 and 250 minute (Fig 2). The physical properties of sintered compact got from the wet and dry processes are shown in table 1. The fracture toughness and density of both sintered compact are closely, but the microhardness from the wet process shown higher than dry one. Under the dry and wet process, the average grain size is 3.12 and 2.96 um, respectively as shown in (Fig 3).

These value are reasonably and corresponds with microhardness. The hardness is always dependent on grain size. A decrease in grain size is accompanied by an increase in mircohardness.

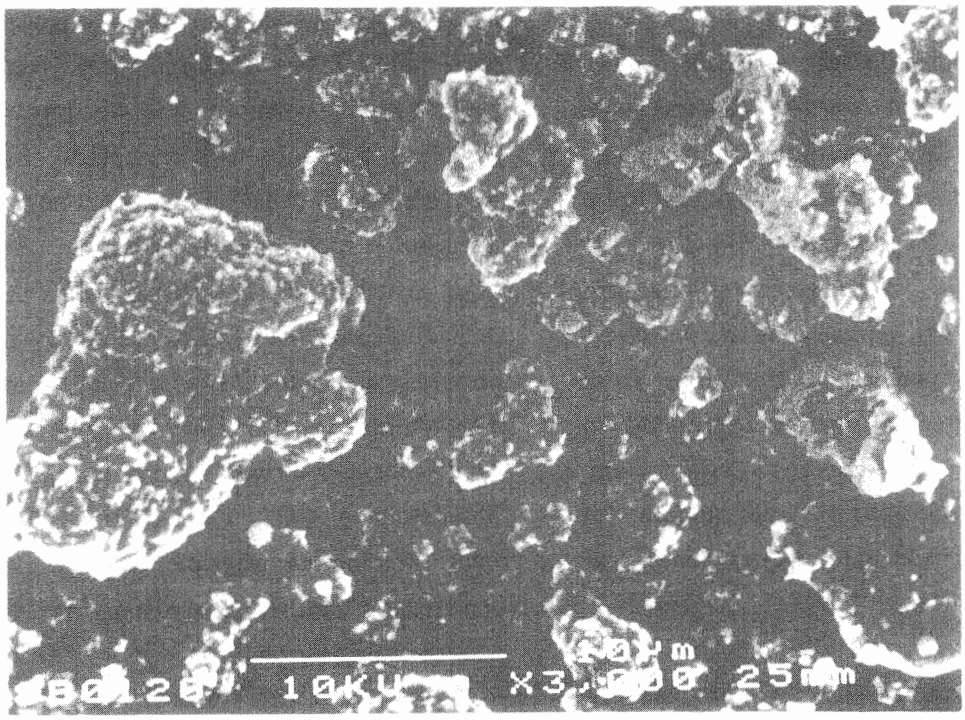


Fig.1 Scanning electron mircograph before processing of hydroxyapatite powder.

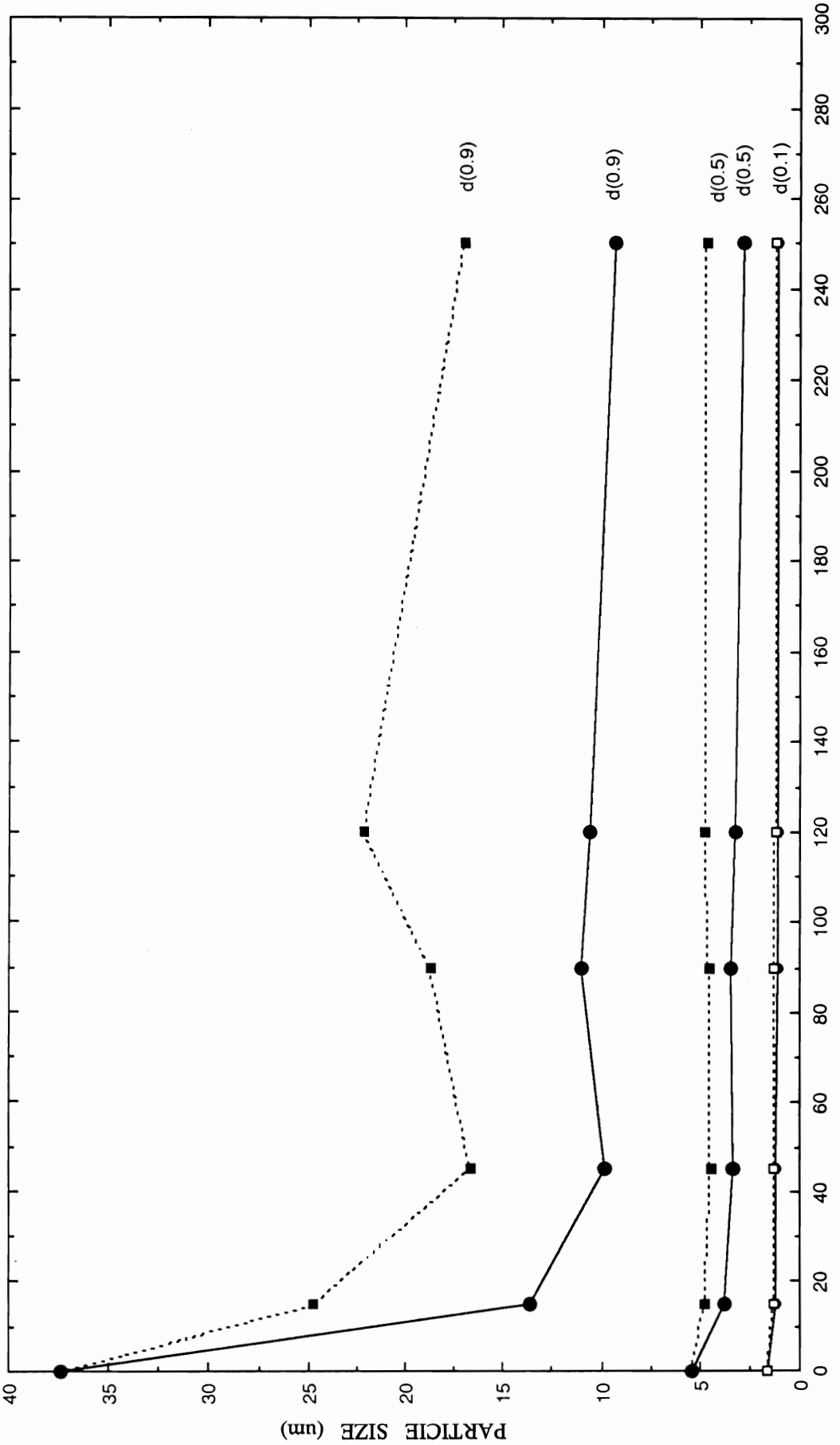
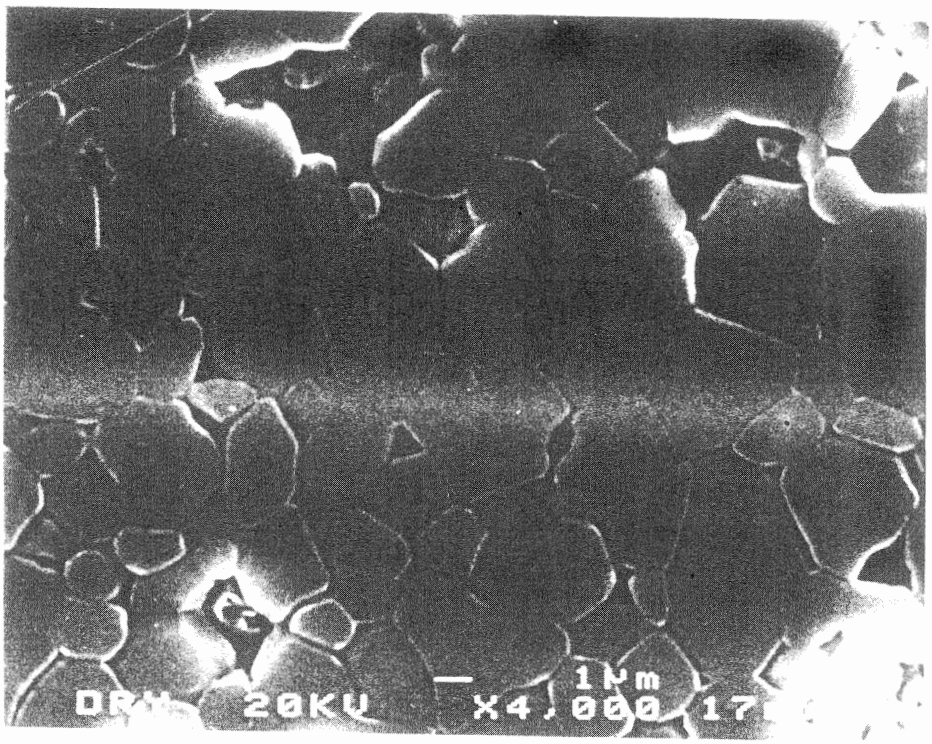
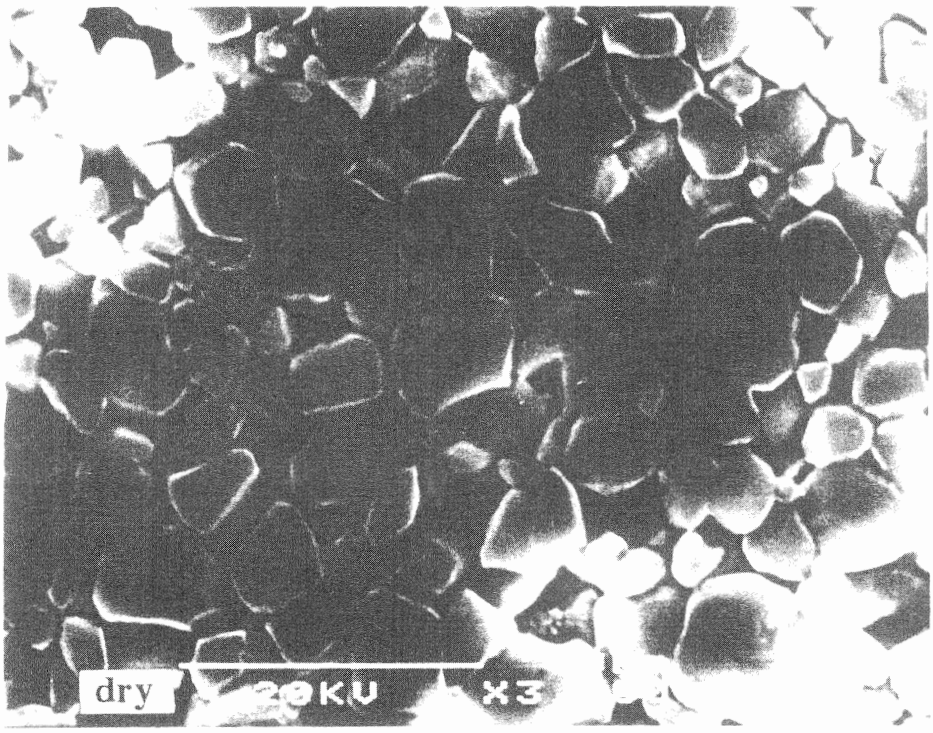


Fig.2 Comparison of particle size of hydroxyapatite at d0.9, d0.5 and d0.1 as a function of milling time
(a) Wet milled hydroxyapatite (–) a (b) Dry milled hydroxyapatite (----)



(a)



(b)

Fig. 3 Scanning electron micrographs of polished surfaces etched by 10% H_3PO_4 solution for a) wet mill sintered b) dry mill sintered speciments

Table 1. The properties of sintered hydroxyapatite

Property	Process	
	Wet milling	Dry milling
Hardness (Hv, 1000g)	458.16 ± 94.67	293 ± 85.59
K_{IC} (Mpa $m^{1/2}$)	0.100 ± 0.04	0.156 ± 0.02
Density (Mg . m^{-3})	3.06	3.02
Average grain size (μm)	2.96 ± 0.47	3.12 ± 0.61

Conclusion

Based on the particle size result and sintered behavior, wet milling appears to be more effective method to improve the sintering characterisation of the Merck hydroxyapatite.

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