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# Synthesis of Microforsterite using Derived-Amorphous-Silica of Silica Sands

Upik Nurbaiti<sup>1,2,a)</sup>, Triwikantoro<sup>1</sup>, Mochamad Zainuri<sup>1</sup> and Suminar Pratapa<sup>1, b)</sup>

<sup>1</sup> *Department of Physics, Faculty of Mathematics and Natural Sciences  
Institut Teknologi Sepuluh Nopember*

*Jl. Arief Rahman Hakim, Surabaya 60111, Indonesia*

<sup>2</sup> *Department of Physics, Faculty of Mathematics and Natural Sciences  
Semarang State University*

*Jl. Raya Sekaran GunungPati, Semarang 50221, Indonesia*

<sup>a)</sup>upik\_nurbaiti@mail.unnes.ac.id

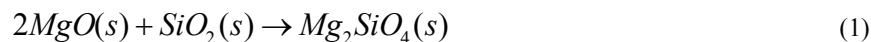
<sup>b)</sup>suminar\_pratapa@physics.its.ac.id

**Abstract.** Synthesis of microforsterite ( $Mg_2SiO_4$ ) has been successfully done by a simple method benefiting of the local silica sands from Tanah Laut, Indonesia. The starting material was amorphous silica powder which was processed using coprecipitation method from the sands. The silica powder was obtained from a series of stages of the purification process of the sands, namely magnetic separation, grinding and soaking with HCl. The microforsterite synthesis followed the reaction of stoichiometric mole ratio mixing of 1:2 of the amorphous silica and MgO powders with 3 wt% addition of PVA as a catalyst. The mixture was calcined at temperatures between 1150-1400°C with 4 hours holding time. XRD data showed that calcination at a temperature of 1150°C for 4 hours was optimum where the weight fraction of forsterite can reach as much as 93 wt% with MgO as the secondary phase and without  $MgSiO_3$ . SEM photograph of the microforsterite showed tapered morphology with a relatively homogeneous distribution.

**Keywords:** amorphous silica, local silica sands and microforsterite

## INTRODUCTION

Naturally, forsterite ( $Mg_2SiO_4$ ) formed solely to maintain the stability of magnesite in the Earth's mantle through a chemical reaction equilibrium between enstatite ( $MgSiO_3$ ) and magnesite ( $MgCO_3$ ) [1]. Therefore, the availability of natural forsterite is not abundant as compared with its applications in various fields. This fact leads to the requirement of synthetic forsterite as one product of MgO-SiO<sub>2</sub> system. The main reaction of the formation of forsterite is [2]



Forsterite has a melting point at about 1890°C, and hence is used as a refractory in high temperature applications [3]. Many synthesis methods of forsterite have been reported [4]–[12], but usually required thermal treatment up to more than 1200°C to obtain pure forsterite. The methods to fabricate forsterite can be classified into solid reactions [4]–[7] and wet reactions [8], [9], [13]. It is, however, very difficult to avoid the secondary phase formation such as the polymorphs of  $MgSiO_3$  and  $SiO_2$  or/and MgO. This paper describes a facile method to synthesis pure forsterite powder in a micrometric scale by making use of natural silica sands with relatively high purity products.

## EXPERIMENTAL

The natural silica sands from Tanah Laut were first purified. There was gradually steps for purification of the sands: cleaning & sieving, magnetic separation, milling, another magnetic separation and soaking with hydrochloric acid prior to a hydrothermal treatment. The previous steps would produce purified silica powder which were ready for coprecipitation. The cleaned silica powders were dissolved in a 7M NaOH and then were stirred in a hydrothermal process to obtain  $\text{Na}_2\text{SiO}_3$  solutions. A 2M hydrochloric acid was then added by titration to form silicic acid ( $\text{Si}(\text{OH})_4$ ) in a form of gel. The gels were then dried at  $100^\circ\text{C}$  to obtain amorphous  $\text{SiO}_2$  powders.

The microforsterite powders were synthesized as follow: firstly, starting materials were prepared to set  $\text{SiO}_2$ :MgO molar ratio 1:2 according to the theoretical value of pure forsterite prior to mixing. Next, 3 wt% PVA was added into the mixture. Then, all ingredients were mixed by a mortar for 30 seconds. The powders were calcined at temperatures between 1150 and  $1400^\circ\text{C}$  for 4 hours holding time. XRD (radiation  $\text{CuK}\alpha$ ) measurements were conducted for phase identification and phase composition. Finally, SEM imaging was used to reveal the microstructure of the forsterite powders.

## RESULT AND DISCUSSION

The contents of Si in the samples after purification of the natural silica sand are shown in Table 1. The purification process has increased the Si content from 94% in the sand sample (TLT) to 98% in the powder sample after coprecipitation (ATL). The coprecipitation powder has an amorphous structure, as shown in Figure 1.

This amorphous silica powder was then mixed with the periclase powder. The powder mixtures were calcined at several temperatures to produce powders where the phase characteristics were identified by x-ray diffraction technique (see Figure 2). It appears that forsterite (AMCSD No 0000328) is the dominant phase in all of the calcined powders. This synthesis method, however, also produced secondary phases, namely periclase (AMCSD No 0000501) at all temperatures and cristobalite (AMCSD No 0017659) at a temperature of  $1400^\circ\text{C}$ . Forsterite was formed following Eq.(1) [2]. The presence of secondary phases were also detected in the previous syntheses with a solid reaction as performed by Rani *et al* [3] and Wahsh *et al* [14]. Our results are altered from those reported previously in that (1) we did not detect polymorphs of  $\text{MgSiO}_3$ , either in the form of enstatite, clinoenstatite or protoenstatite [3] and (2) the powder milling and mixing time as well as the calcination temperature were more efficient in producing forsterite. The latter can be explained by the following. The peak intensity ratio between periclase (111) at  $2\theta \approx 43^\circ$  and forsterite (211) at  $2\theta \approx 37^\circ$  for our  $1150^\circ\text{C}$  sample is lower than that for Wahsh [14], ie  $\frac{1}{4}$  and  $\frac{1}{3}$  respectively.

For all samples, the diffraction peak width is relatively narrow, ca  $<0.2^\circ 2\theta$ , which indicates that forsterite crystal size is relatively large and not nanometric [15]. Furthermore, the observation using SEM showed that the dominating grain size is about 3-7 microns – see Figure 3. The SEM image also reveals the presence of numerous grains which indicate that the powder contains crystallites, as claimed from the XRD data. Therefore, here, we have successfully synthesised microforsterite powders.

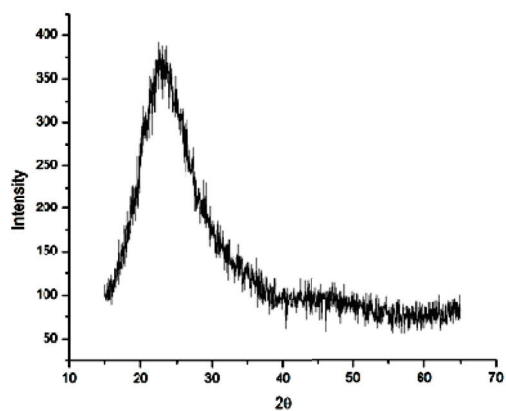
The Rietica XRD quantitative analysis results for all powders are presented in Table 2. The  $1300^\circ\text{C}$  sample exhibit the highest microforsterite relative weight fraction, ie  $93.5 \pm 9$  wt%. The value for the  $1150^\circ\text{C}$  sample is lower, but it agree well with that to the  $1300^\circ\text{C}$  sample within one standart deviation. By considering these results as well as the energy required for the formation of mikroforsterit, preferably calcination at  $1150^\circ\text{C}$  than at  $1300^\circ\text{C}$ . Thus, the synthesis mikroforsteri with the starting material of amorphous silica powder and periclase powder by this solid reaction quite efficiently performed at temperatures of  $1150^\circ\text{C}$  during 4 hours.

## CONCLUSIONS

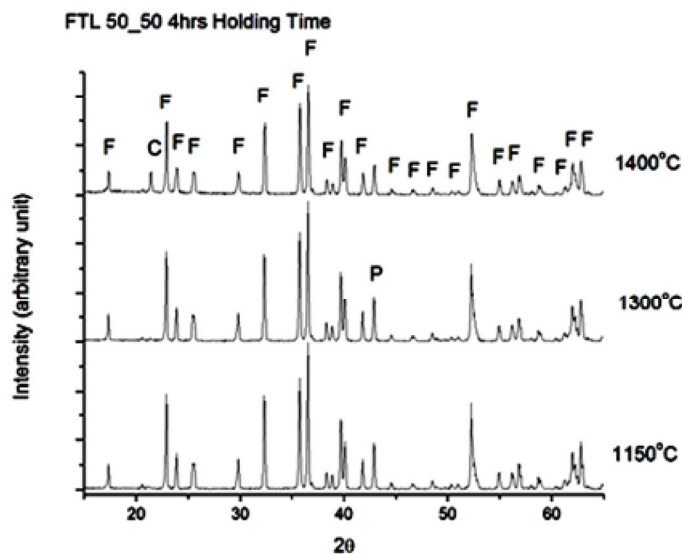
Microforsterite powders have been successfully synthesised through a solid-state reaction involving amorphous silica powder from a silica sand from Tanah Laut. The results showed that fair purity of microforsterite can be obtained by the method ( $> 92$  wt%). The optimum temperature in the formation of microforsterite is a sample calcined at  $1150^\circ\text{C}$  for 4 hours.

**TABLE 1.** XRF weight percentage of Si for the silica sands from Tanah Laut after purification steps. The standard deviations of the average values for the last significant figure are given in parantheses.

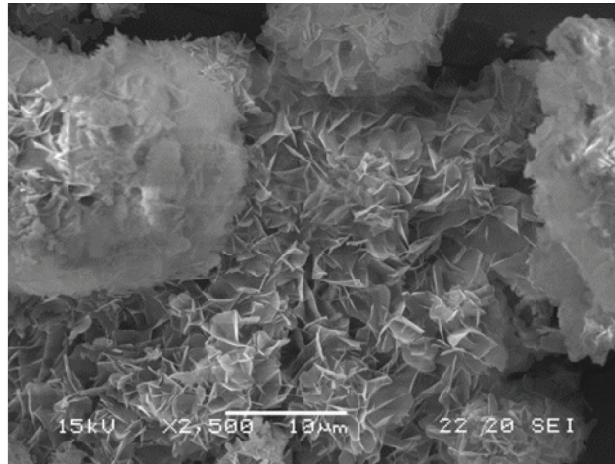
Steps	Numenclature	Quantity Si (wt%) Tanah Laut
As-received sand	TLT	94.7(2)
Washed sand	TLA	95.6(2)
Milled sand	TLG	97.7(1)
Coprecipitated powder	ATL	98.0(2)



**FIGURE 1.** XRD pattern (CuK $\alpha$  radiation) for the amorphous silica from Tanah Laut.



**FIGURE 2.** XRD patterns (CuK $\alpha$  radiation) for the microforsterite powders after calcination at various temperatures for 4h. C=Cristobalite, F=Forsterite, P=Periclase and FTL=forsterite from Tanah Laut silica sand.



**FIGURE 3.** A Secondary Electron SEM image of microforsterite powder calcined at 1150°C for 4h.

**TABLE 2.** Rietveld relative weight fractions in the forsterite powders and their standard deviations, where the least significant digits are given in parantheses.

Sample	Quantity(wt%)		
	Forsterite(F)	Periclas(P)	Cristobalite(C)
FTL_50 1150_4	92.6(9)	7.4(5)	-
FTL_50 1300_4	93.5(9)	6.5(4)	-
FTL_50 1400_4	90.1(9)	6.2(9)	3.7(9)

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