



## Structural Analysis and Morphological Study of Al<sub>2</sub>O<sub>3</sub> nanofluids in Microchannel Heat Sink

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### ABSTRACT

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Microchannel heat sink is a small device with the ability of creating an innovative cooling technology to remove large amount of heat from small areas. Recently, nanotechnology has gained interest to explore the microchannel cooling benefits of water containing small concentration of nanoparticles as working fluid. It has been found that nanoparticles dispersed with water have increased the heat transfer coefficient in the microchannel. There were number of studies on improving heat transfer in the microchannel using Al<sub>2</sub>O<sub>3</sub> nanofluids. However, there is less study on the effect of the nanoparticles structure after it is used as working fluid in the microchannel heat sink. Therefore, this present study investigates the structures of Al<sub>2</sub>O<sub>3</sub> nanofluids after 10 hours application of Al<sub>2</sub>O<sub>3</sub> nanofluids. It was confirmed by XRD method that the heating process in the microchannel has not changed the structure of Al<sub>2</sub>O<sub>3</sub> nanofluids. According to the XRD, patterns show that the diffraction peaks are sharper after an increase in the temperature. Besides that, the morphological study found that the heating process within the microchannel has increased the grain size of Al<sub>2</sub>O<sub>3</sub> nanofluids. This investigation concluded that the structures of Al<sub>2</sub>O<sub>3</sub> nanofluids have not changed after 10 hours in the heating side whereas an increase in the grain size occurred due to the agglomeration of the particles.

#### Keywords:

Microchannel heat sink, nanofluids, heat transfer, structural analysis, morphology

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## 1. Introduction

Microchannel heat sink is a device with very fine channel the width of a normal human hair and is widely used in electronic cooling. As the size of the channel reduces to micron, the heat transfer coefficient can increased thousand times from the original value [1]. It is combines the characteristics of very high surface area to volume ratio, large convective heat transfer coefficient, small mass and volume with small coolant inventory [2]. The heat sink usually made from a high thermal conductivity material such as copper, silicon or aluminum. The dimensions ranging is from 10 to 1000 micron and serve as flow passages for cooling fluid [3].

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In a microchannel heat sink, the microchannel is stacked together in order to increase the total contact surface area for heat transfer enhancement and reduce pressure drop. As coolant, fluid flow through the microchannel, their large surface enables them to take large amounts of energy per unit time per unit area while maintaining a considerably low device temperature.

Recently, nanofluids gain interest to explore the microchannel cooling benefits of water containing small concentration of nanoparticles as working fluid. The idea of using metallic nanoparticles was proposed by previous study to increase the thermal conductivity of fluids. The nanofluids remarkably increased the forced convective heat transfer performance of the base fluid. There have found that nanofluids flow through small channel under constant heat is efficient only when the heat transfer irreversibility is dominant [4]. The increasing of heat transfer coefficient is not only affected by thermal conductivity but it is also affected by increasing of Reynolds number and the concentration of nanoparticles [5]. The presence of nanofluids instead of traditional fluids (water) provided higher heat transfer with increasing Reynolds number as well as particle volume concentration [6]. It was reported that the nanofluids have high thermal conductivity and have better performance for heat transfer in order to decrease of using energy [7]. It is not only be a better medium for heat transfer but also can be ideal for applications in microchannel where high heat loads encountered. That combination of nanofluids and microchannel will provide both highly conducting fluids and large heat transfer area. It can reduce chances of erosion. According to the previous finding, the heat transfers are achieved at inlet region by increasing the concentrations of nanoparticles [8]. The enhancement of the nanoparticles concentration appears more prevalent in the entrance region than the downstream fully developed.

However, there is lack information of effect of the nanofluids after apply as working fluids in the microchannel heat sink. Thus, the fine grades of nanoparticles increase the heat transfer rate but it has poor stability after being used for a long time [9]. The effect of particles is an obvious concern with solid particle suspensions that are intended for cooling applications. The nanoparticle suspensions are more stable compare to larger particles suspensions. Therefore, x-ray diffraction method (XRD) is useful to investigate about arrangement of atoms within crystalline materials and particle size with the crystallographic phase [10]. Through the X-ray diffraction method (XRD), information provided that the characterization of crystalline materials represented crystal structure, phases, preferred crystal orientation and other structural parameters [11]. High-resolution surface images of the superlattices obtained by Field Emission Scanning Electron Microscope (FESEM) showed excellent ordered arrangements of nanoparticles with either close-packed or non-close-packed structures[12]. Therefore, the structural analysis and morphology of  $\text{Al}_2\text{O}_3$ nanofluids in the microchannel is important in order to see the capability of the nanofluids after it is used as working fluids in the microchannel heat sink. In addition, it may further help in the investigation of the agglomeration occurrence in the  $\text{Al}_2\text{O}_3$ nanofluids after being used in the microchannel heat sink.

## 2. Methodology

### 2.1 Experimental Setup

The concept used in this investigation was modified from a previous study by Parida [13]. The experiment starts by using water as working fluid. The heat transfer was measured according to fluid flow range. After that, the experiment of heat transfer used 1.0 wt. %  $\text{Al}_2\text{O}_3$  nanofluids and 2.5 wt. %  $\text{Al}_2\text{O}_3$  nanofluids as working fluids. The working fluid is taken out from the tank and circulated through water pump. Upon exiting the pump, the flow is controlled before entering the test section. The flow rate was adjusted within the range of 1.5 LPM to 3.0 LPM in order to maintain the flow in laminar form. The working fluid then flows through the microchannel with constant heat produced in the

specimen test (325 W). The temperature was read using K-type thermocouple. After leaving the specimen test, the fluid then returned to the liquid tank and then circulated to the heat sink for several times. The experiment was running continuously. After the flow rate stabilized, the cartridge heater was switched on to provide heat to the specimen test. The temperature of copper block was then increased rapidly. The experimental work was carried out for 40 – 50 minutes for each flow rate.

## 2.2 Preparation of $Al_2O_3$ Nanofluids

$Al_2O_3$  nanofluids used in this experiment are made by Sigma Aldrich and ready in 20.0 wt. % volume of concentration were dispersed in water. The dilution process was carried out to reduce the concentration of  $Al_2O_3$  nanofluids to 1.0 wt. % and 2.5 wt. % concentration. This process requires using volumetric flask due to its accuracy. The volume of 20.0 wt. %  $Al_2O_3$  nanofluids was calculated first before added and mixed with water. The calculation is explained in Equation 1.

$$m_1v_1 = m_2v_2 \quad (1)$$

where  $m$  is mass concentration and  $v$  is volume for concentration. After the dilution process, the color of 1.0 wt. % and 2.5 wt. % are considerably paler than the 20.0 wt. % concentration. In order to maintain the performance of  $Al_2O_3$  nanofluids during the experiment, the  $Al_2O_3$  nanofluids were stored in glass container and placed in cool and dry area. The glass container needed to be shaken for a while before used to ensure that the particles not agglomerated.

## 2.3 Structure Analysis and Morphology of $Al_2O_3$ Nanofluids

The characterization was carried out to analyze the nanoparticles based on the volume concentration. It is to analyze the effect of nanoparticles towards microchannel heat sink. The process was done by using X-ray diffraction and FESEM. The XRD analysis was carried out based on reference code of 98-008-5137 by a component named Aluminum Oxide- $\alpha$ . This technique was generated by a cathode-ray tube filtered to produce monochromatic radiation, collimated to concentrate, and directed toward sample. The interaction of the incident rays within the sample produces constructive interference and a diffracted ray. The conditions required for constructive interference are determined by using Bragg's Law as shown in the equation as follow;

$$n\lambda = 2d \sin \theta \quad (2)$$

Where  $d$  is the separation between atomic planes (the d-spacing),  $\theta$  is the half of diffraction angle,  $n$  is an integer and  $\lambda$  is the X-ray wavelength. Average crystallite size ( $D_N$ ) of the synthesized fluid, was estimated through XRD analysis by calculation of the full-width at half maximum FWHM value by using Scherrer approximation. It is assumed that the small crystallite size to be the cause of line broadening as in the following equation;

$$D_N = \frac{k\lambda}{\beta \cos \theta} \quad (3)$$

where  $D_N$  is the crystallite mean size,  $k$  is a shape function,  $\lambda$  is the wavelength of the radiation,  $\beta$  is the full width at half maximum (FWHM) in radians in the  $2\theta$  scale and  $\theta$  is the Bragg angle. The field

emission scanning electron microscope FESEM is used to visualize very small morphology details on the surface fractioned objects. The electrons was liberated from a field emission source and accelerated in high electrical field gradient. In order to scan the image easier, gold coating process is necessary for this sample. The gold coating process were carried out in FISON SEM Coating System. The process was running for 10 minutes to coat the sample. The samples are then scanned in FESEM to visualize the particles as seen in Figure 1. The image seen from figure below was used to analyze the morphology of  $\text{Al}_2\text{O}_3$  nanofluids before and after apply to the microchannel heat sink.

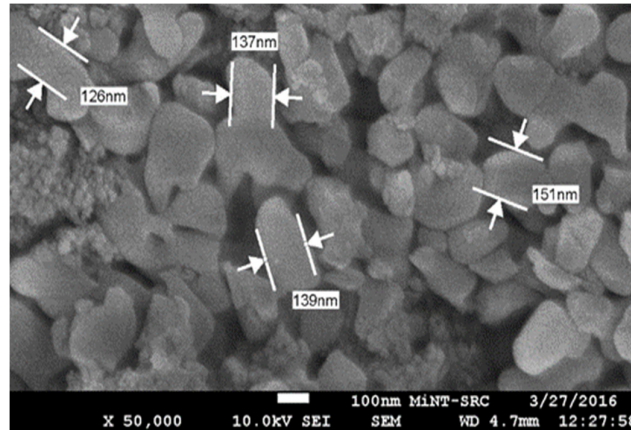


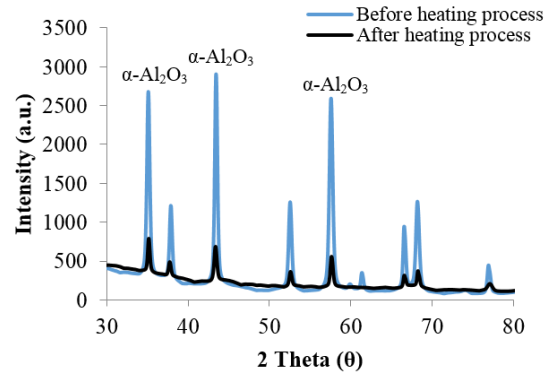
Fig. 1. Image of  $\text{Al}_2\text{O}_3$  nanoparticles

### 3. Results and Discussion

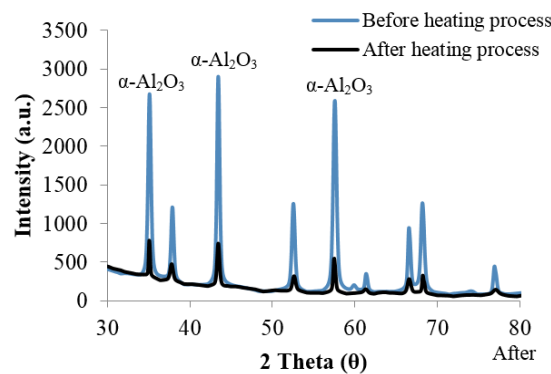
#### 3.1 Structural Analysis of $\text{Al}_2\text{O}_3$ Nanofluids after Being used in the Microchannel Heat Sink

The structural analysis were carried out for both 1.0 wt. % and 2.5 wt. %  $\text{Al}_2\text{O}_3$  nanofluids. The XRD patterns for 1.0 wt. %  $\text{Al}_2\text{O}_3$  nanofluids showed diffraction peaks at its highest intensity at position  $35.1^\circ$ ,  $43.4^\circ$  and  $57.5^\circ$  as seen in Figure 2. The data obtained matched with the database of ICSD collection code of 85137. Both samples have diffraction peaks at same position. It is shown that the structure of 1.0 wt. %  $\text{Al}_2\text{O}_3$  nanoparticles has not changed after it's used in the microchannel and its contact with constant heat input of 325 W. However, it has been observed that after being used for the duration of 10 hours in the microchannel, the FWHM showed signs of an increase from the original. The result indicates the crystallite size increasing from its original size. Therefore, it can be concluded that 1.0 wt. %  $\text{Al}_2\text{O}_3$  nanoparticles increase its crystallite size after it is used in the microchannel.

The concentration of  $\text{Al}_2\text{O}_3$  nanofluids increased to 2.5 wt. % and the heat transfer performance in the microchannel also increases due to the quantity of nanoparticles being increased in order to absorb more heat. It has been observed that the quantity of  $\text{Al}_2\text{O}_3$  nanoparticles improved the heat absorbent in the microchannel. Therefore, XRD analysis has been carried out for 2.5 wt. % concentration for both samples of before and after used in the microchannel heat sink as seen in Figure 3. The XRD pattern of 2.5 wt. %  $\text{Al}_2\text{O}_3$  nanofluids diffraction peaks were intense at position  $35.1^\circ$ ,  $43.4^\circ$  and  $57.5^\circ$ . Both samples have diffraction peaks existing at the same position. It is clearly shown that the structure of 2.5 wt. %  $\text{Al}_2\text{O}_3$  nanoparticles does not change after absorbing constant heat in the microchannel. However, the FWHM is also increased before it is applied into the microchannel.



**Fig. 2.** XRD pattern of 1.0 wt. % Al<sub>2</sub>O<sub>3</sub> nanofluids used as working fluid in the microchannel before and after 10 hours used in the microchannel heat sink



**Fig. 3.** XRD pattern of 1.0 wt. % Al<sub>2</sub>O<sub>3</sub> nanofluids used as working fluid in the microchannel before and after 10 hours used in the microchannel heat sink

It has been observed that the crystallite size increased from its original size as a result of being affected by the heating process inside the microchannel. The comparison of crystallite size for 1.0 wt. % Al<sub>2</sub>O<sub>3</sub> nanofluids is presented in Table I. The diffraction phase for Al<sub>2</sub>O<sub>3</sub> nanofluids at peak position of 43.40 ° was selected and the Debye-Scherrer equation was applied to find the crystallite size from the XRD data. There was a significant difference in crystallite size after used in the microchannel for both wt. % concentrations. It has been confirmed that the increment of crystallite size occurs after being in contact with constant heat input in the microchannel. The crystallite size of 1.0 wt. % Al<sub>2</sub>O<sub>3</sub> nanofluids increased from 27.6 nm to 35.0 nm.

It is shown that the crystallite size of 1.0 wt. % Al<sub>2</sub>O<sub>3</sub> nanofluids increased by 21.1 %. Table II presented the crystallite size of 2.5 wt. % Al<sub>2</sub>O<sub>3</sub> nanofluids before and after used in the microchannel. The Debye-Scherrer equation was applied to find the crystallite size from the XRD data at peak position of 43.40 °. It is shown that the crystallite size of 2.5 wt. % Al<sub>2</sub>O<sub>3</sub> nanofluids increased by 22.6 %. The present result is similar comparing to a trend study by Pa *et al.*, [14] which showed the crystallite enlarging by increasing the temperature.

**Table 1**

Crystallite size of 1.0 wt. %  $\text{Al}_2\text{O}_3$ nanofluids before and after used in the microchannel

1.0 wt. % $\text{Al}_2\text{O}_3$ nanofluids	Position [2 $\theta$ ]	FWHM [2 $\theta$ ]	D [nm]	d-spacing [Å]
Before	43.4	0.4	27.6	2.6
After 10 hours	43.4	0.3	35.0	2.6

**Table 2**

Crystallite size of 2.5 wt. %  $\text{Al}_2\text{O}_3$ nanofluids before and after used in the microchannel

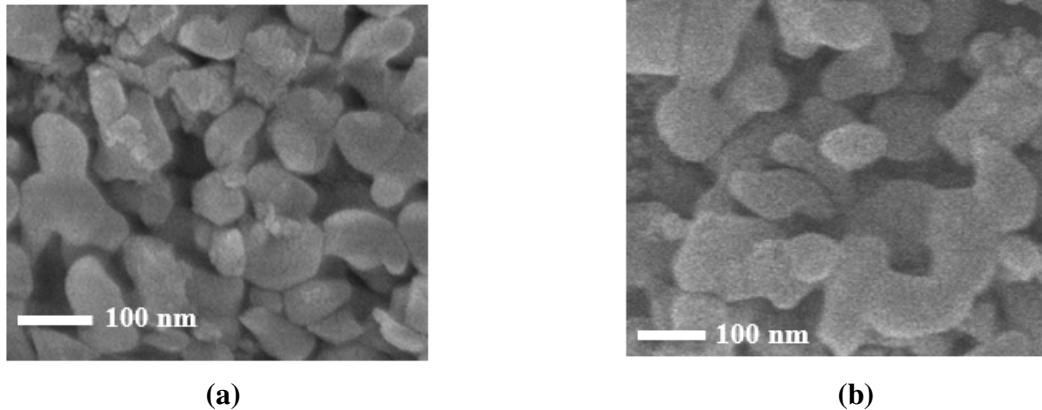
2.5 wt. % $\text{Al}_2\text{O}_3$ nanofluids	Position [2 $\theta$ ]	FWHM [2 $\theta$ ]	D [nm]	d-spacing [Å]
Before	43.4	0.2	24.2	2.6
After 10 hours	43.4	0.2	31.3	2.6

### 3.2 Morphological Study of $\text{Al}_2\text{O}_3$ Nanofluids

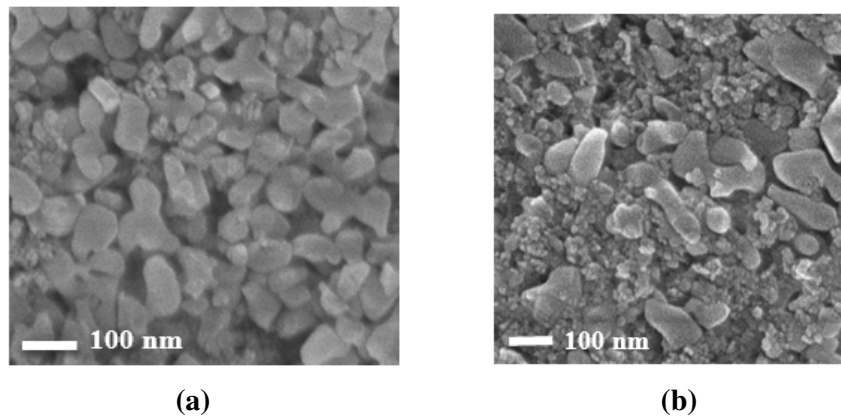
The morphological study of  $\text{Al}_2\text{O}_3$  nanofluids is obtained by using FESEM for 1.0 wt. %  $\text{Al}_2\text{O}_3$ nanofluids and 2.5 wt. %  $\text{Al}_2\text{O}_3$  nanofluids. There were two samples used to compare the difference in grain size and shape of particles. As seen in the Figure 4 (a), it is shows the images of 1.0 wt. %  $\text{Al}_2\text{O}_3$  nanoparticles in non-uniform shape. The grain size was calculated according to the average of 30 measurements of each particle. The average grain size for 1.0 wt. %  $\text{Al}_2\text{O}_3$ nanofluids is 140.9 nm. However, in the Figure 4 (b), it is clearly showing that the grain size has increased to 150.7 nm after the heating process in the microchannel. The difference in size comparing to the initial sample is 9.77 nm.

Another sample has been carried out for 2.5 wt. %  $\text{Al}_2\text{O}_3$ nanofluids according to the Figure 5 (a), where it is clearly showing the state of 2.5 wt. %  $\text{Al}_2\text{O}_3$  nanoparticles in non-uniform shape. Based on the average measurement, it is shows that the grain size was 107.7 nm before used in the microchannel. After the particles came in contact with constant heat, the grain size was increased by 12.8 nm. The Figure 5 (b) shows the grain size of 2.5 wt. %  $\text{Al}_2\text{O}_3$  nanoparticles was 129.8 nm.

The FESEM images show both shapes of 1.0 wt. % and 2.5 wt. %  $\text{Al}_2\text{O}_3$  nanoparticles have well-defined morphologies. It has been confirmed that the increasing of temperature to the nanoparticles surface is proportional to the increase in the nanoparticles size while the size distribution become narrower as the diffusion coefficient reduced [15]. The structure of the agglomerates as well as their size, weight and the interactive forces between them significantly influence the hydrodynamic behavior of agglomerating fluidization [16]. Therefore, it was tend to agglomerate to diminish the energy. The ionized particles have attracted each other on solid surfaces to form agglomeration. Thus, this has proven that the nanoparticles sizes was increased by the heating process in the microchannel heat sink and the grain size increased due to the particles starting to agglomerate.



**Fig. 4.** FESEM images of 1.0 wt. %  $\text{Al}_2\text{O}_3$  nanofluids (a) before and (b) after 10 hours used in the heat sink



**Fig. 5.** FESEM images of 2.5 wt. %  $\text{Al}_2\text{O}_3$  nanofluids in the microchannel; (a) before and (b) after 10 hours used in the microchannel

#### 4. Conclusion

As conclusion from structure and morphological study, the structures of  $\text{Al}_2\text{O}_3$  nanofluids has not changed from the effect of the heating side in the microchannel. The XRD method shows that the structures of  $\text{Al}_2\text{O}_3$  nanofluids was weaker after absorbing heat while the crystalline size of the nanoparticles became larger due to the heating process in the heat sink. It has been observed based on changes seen from the high peak position. The FESEM images confirmed that the particles were in non-uniform shape for both volume concentrations. Furthermore, agglomeration has been found after the  $\text{Al}_2\text{O}_3$  nanoparticles were used in the microchannel heat sink. It can be concluded that the performance of  $\text{Al}_2\text{O}_3$  nanofluids after 10 hours of application in the microchannel has not changed its structure but instead increased in size due to the agglomeration of the particles.

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