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Effect of Titanium Dioxide Particle Size in Water-based Micro/Nanofluid as Quench Medium in Heat Treatment Process

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Abstract. Recently, nanofluid is used to improve the thermal conductivity of the quench medium in the heat treatment industry. In this research, ball-milled micro-sized TiO_2 powder and nano-sized TiO_2 particle were used and compared for their cooling characteristic in a micro/nanofluid. The micro/nanofluids were produced by mixing 0.1%, 0.3%, and 0.5% volume of both micro- and nano-sized particle into 100 ml of distilled water. The planetary ball mill was used at 500 rpm for 15 hours to reduce the dimension of micron-sized TiO_2. Composition characterization by Energy Dispersive Spectroscopy (EDS) showed that the powder used were free from impurities. Nanofluids were then used to quench S45C carbon steel samples, which heated at 1000°C for 1 hour. The hardness test result showed that the sample quenched with 0.5%addition of the nano-sized particle in nanofluid had the highest number up to 691 HV, almost 100HV increment from a water-quenched sample where the hardness was 598 HV, showing that the cooling rate in the nanofluid was much higher. The addition of micro-size particle in fluid generally had a lower cooling rate than the addition of nano-size particle.

Keywords: Nanofluid, TiO_2, Quenching

1. Introduction

Heat treatment is a standard process used in the metal industry to improve the properties of materials, especially steels. In this process, steel properties could be adjusted to have higher hardness or more ductility, depend on the application. Quenching is a process in heat treatment, where steel parts are heated to austenizing temperature and rapid-cooled by immersing into the quenchant or quenching medium [1]. Water, brine, polymers, oils, salts, and gas are commonly used as a quenching medium which has their cooling characteristics and results. Improper quenchant can cause the material to become too brittle, suffers some geometric distortion, and undesirable residual stress.

Microstructure and mechanical properties after quenching depend on several factors such as surface temperature, thermo-physical properties of saturated liquid, size and shape of the material, the roughness of the material, and cooling rate of quench process. Typically, after the quenching process, martensite is formed in steel, and the hardness is increased. To ensure the martensite formation, cooling rate during rapid-cooling must be equal or higher than the critical cooling rate for martensite forming [2].

Studies on quenching medium that influenced cooling rate has been done by many researchers [3-5]. One of the popular quenching medium is nanofluid. Nanofluid refers to a fluid containing a small amount dispersed nano-sized (< 100 nm) solid nanoparticles. It has been proved that the nanoparticles will enhance thermal conductivity and improve the thermal performance of heat transfer of the base fluid [6]. Commonly used nanoparticles include Al_2O_3, CuO, TiO_2, and Carbon. Nanoparticles used in nanofluid are considered by their chemical stability, thermophysical properties, toxicity, and cost [7]. The thermal conductivity of TiO_2 nanofluid can increase a maximum of 38.7% than distilled water [8].

TiO_2 micro- and nanoparticle, and aqueous-distilled water were used for micro/nanofluid in this research. In this study, the effect of the particle size of TiO_2 in the quench medium after heat treatment will be compared and discussed.

2. Materials and Methods

2.1 Preparation of TiO_2 Micro/Nanofluids

TiO_2 micro/nanofluids are obtained by a two-step process, particle synthesis, and particle dispersion. The two-step method was more economical compared than the one-step method to produce micro/nanofluids commercially, and also considered the best method in dispersing the particles in a base fluid [9]. The TiO_2 powder was purchased from Sigma-Aldrich in micro-size (44 μm) and nano-size (21nm) form. This study used three particle-size variations. The mentioned size variations were micro-sized laboratory grade TiO_2, reduced micro-sized laboratory grade TiO_2, and nano-meter sized laboratory grade TiO_2. Reduced micro-sized laboratory grade milled was obtained using a high energy ball mill for 15 hours at 500 rpm. The second step was the dispersing TiO_2 in distilled water with ultrasonic agitation. Variations of TiO_2 particle addition in the fluid were 0.1%, 0.3%, 0.5%.

2.2 Material for Quenching Experiments

The material used in this experiment was S45C carbon steel bar. S45C steel is classified as a medium carbon steel material that is often used in manufacturing industries.

Table 1. Chemical composition of S45C Carbon Steel

Fe	C	Si	Mn	P	S	Cr	Mo
98.3	0.47	0.287	0.718	0.0261	0.005	0.028	0.005
Ni	**Al**	**Co**	**Cu**	**Nb**	**Ti**	**V**	**W**
0.005	0.02	0.003	0.018	<0.002	0.035	<0.002	<0.01
Pb	**Sn**	**B**	**Ca**	**Zr**	**As**	**Bi**	
0.025	<0.002	0.0013	0.0005	<0.002	0.008	<0.03	

Table 1 showed the composition of the S45C steel sample used in this research. Medium carbon steel was chosen because its microstructure is sensitive to the cooling rate during quenching.

2.3 Experimental Methodology

The TiO_2 particle was characterized by Field Emission Scanning Electron Microscope (FE-SEM) Inspect F50 from FEI, coupled with Energy Dispersive Spectroscopy (EDS) from EDAX for shape and purity checking. Cooling rate observations of the quench medium were carried out by using microstructure and hardness test of the steel sample.

The S45C steel was prepared by cutting the sample with a size of 15mm x 10mm x 10mm with a saw machine. Samples were then preheated in 600^0C for 15 minutes and then heated at 1000^0C for one hour. After that, samples were quenched in water-based nanofluid with varying particle sizes. The microstructure was obtained by metallographic observation. Structure of specimens was made visible by using 3% nital etching. An optical microscope was then used to record the microstructure observation. Hardness measurement was obtained by a Vickers Hardness Test with a 300gf load.

3. Results and Discussion

3.1 TiO_2 Particle Characterization and S45C Microstructure Observation

FE-SEM was used to observe the morphology of TiO_2 particles, while EDS was used to check any impurities present in the powder. FE-SEM and EDS examination were done at CMPFA University of Indonesia.

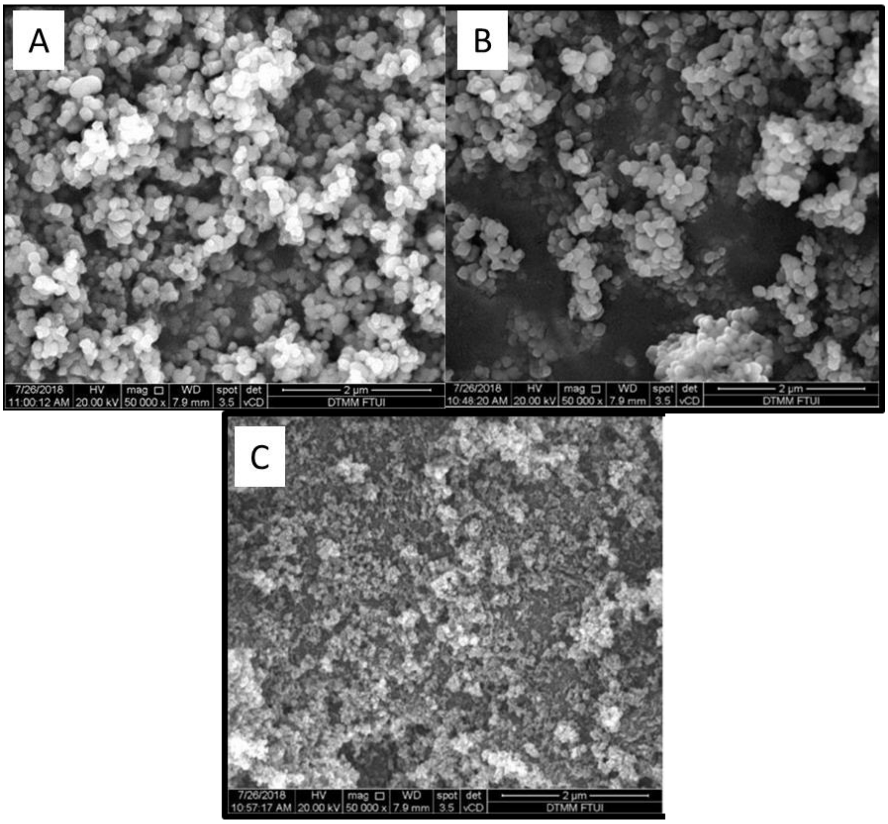

Figure 1. FE-SEM image of laboratory grade TiO_2particle. (A. micro-size, B. reduced micro-size, C. nano-size). Magnification: 50.000X

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Figures 1 showed the particle shape of micro-size, reduced micro-size, and nano-size TiO_2 respectively. Manual measurement of reduced micro-size showed an average of 29µm particle size.
Table 2 showed EDS result for micro-size TiO_2 particles. From the result, it can be seen that no significant metal impurities present on the powder. There was a tiny amount of Phosphorous and Chloride elements, probably due to contamination during EDS sample preparation. The main element on the particle was Titanium as expected.

Table 2. Chemical composition of TiO_2 powder by EDS

Element	Weight %
P	0.32
Cl	0.32
Ti	99.36

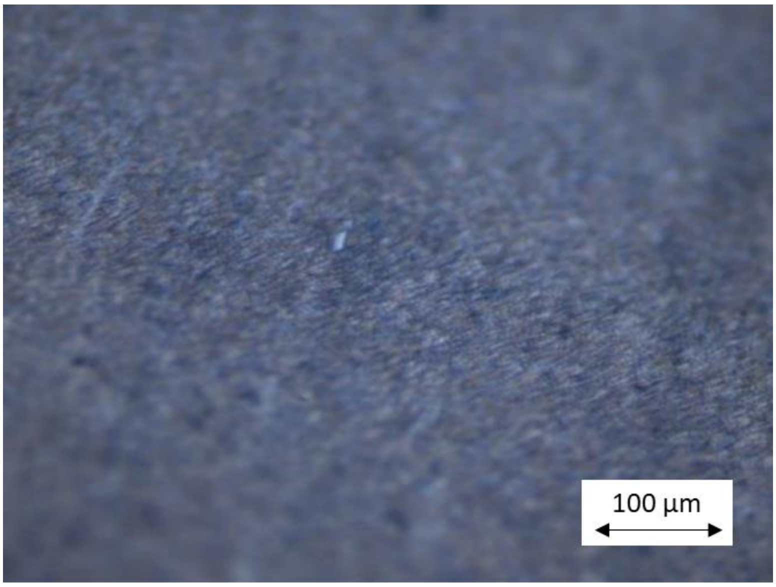
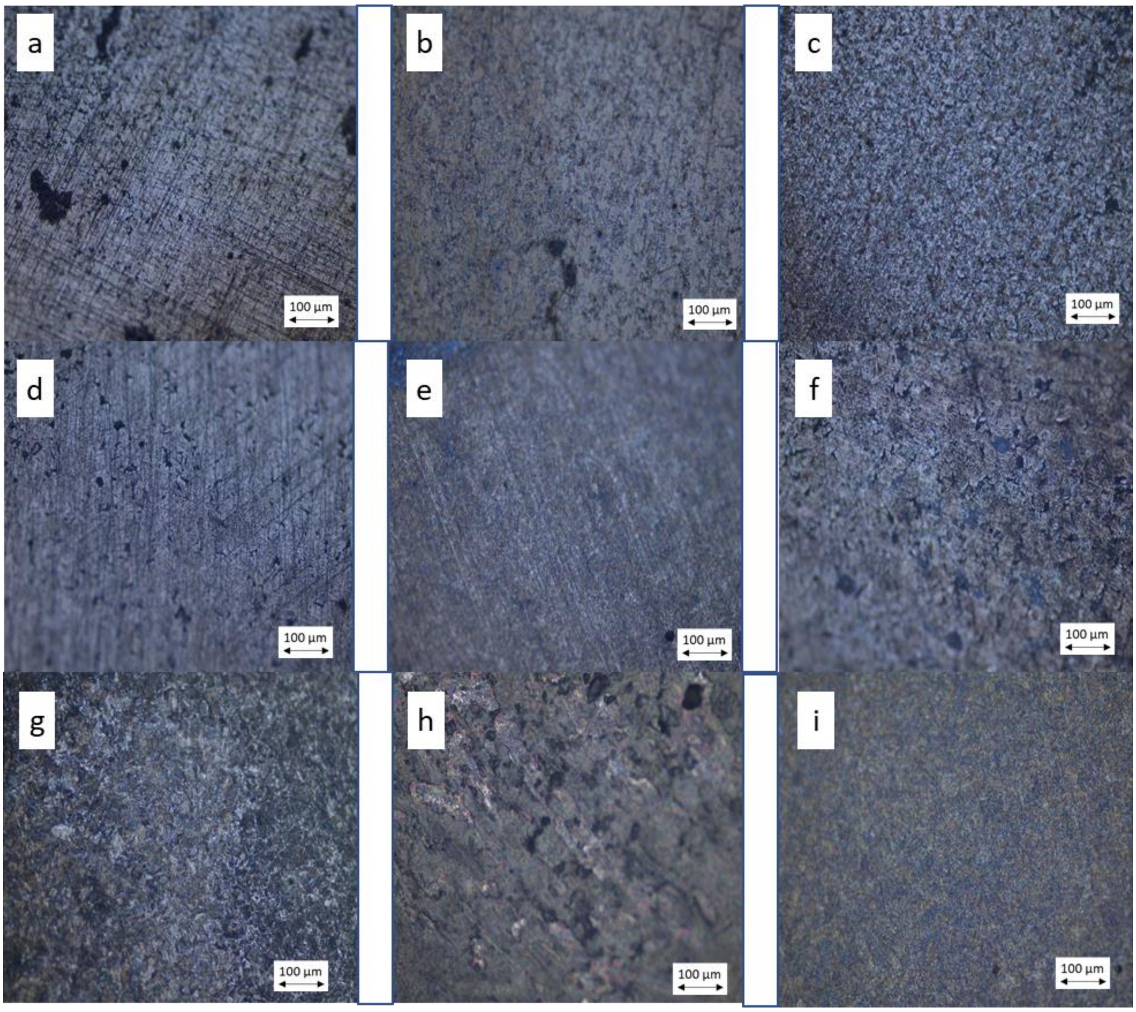


Figure 2. Microstructures of S45C after quenching in distilled water. Mag: 200X

Figure 2 showed the microstructure of S45C steel quenched by water without nanoparticle taken by an optical microscope. Figure 3 showed the microstructure of S45C steel quench with varies addition of TiO_2 particle. All of the mentioned figures showed typical quenched steel microstructure where martensite phase present in the material. However, it was challenging to differentiate the martensite percentage from the sample quenched with water and nanofluid. Hardness test was then conducted to observe the difference of hardness from these samples. The difference of hardness number will show the martensite percentage on each sample.



(a) (b) (c)

Figure 3. Microstructures of S45C after quenching in micro-size (a) 0.1 % (b) 0.3% (c) 0.5%, reduced micro-size (d) 0.1 % (e) 0.3% (f) 0.5%, and nano-size (g) 0.1 % (h) 0.3% (i) 0.5% TiO_2 particle. Magnification: 200X

3.2 Hardness Testing

Vickers hardness testing was done to support the microstructure data. In this research, higher hardness means higher cooling rate which resulted in more martensite phase formed in the steel. The hardness of the steel sample quenched in the normal water was 598 HV. Addition of micro-size TiO_2 particle into fluid resulted in higher hardness number i.e. 579 HV, 686 HV, and 628 HV for particle concentration of 0.1%, 0.3%, and 0.5% respectively. Reduced micro-size particle quench medium appeared to have generally lower hardness number, i.e. 605 HV, 608 HV, and 639 HV for the same particle concentration. This phenomenon was not expected as smaller particle size should have more surface area, thus increasing the cooling rate. However, there was no surfactant used in this research. Therefore, agglomeration may take place during particle mixing into the fluid.

Nano-size TiO_2 particle addition in the quench medium showed higher cooling rate, as seen in the higher hardness number at 611 HV, 644 HV, and 691 HV for a particle concentration of

0.1%, 0.3%, and 0.5% respectively. Increasing the particle concentration may increase the cooling rate of the quench medium as well. Figure 4 showed the Vickers hardness number trend for each sample with different quenching medium variations.

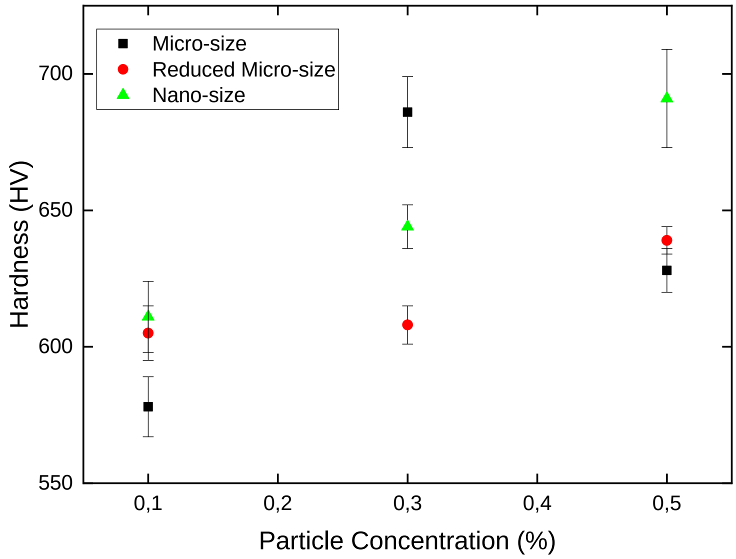


Figure 4. The hardness value of steel quenched in a different medium

This result was in accordance with the initial hypothesis where the smaller the size of the fluid particles, the heat transfer will be evenly distributed and increasing the cooling rate. The higher cooling rate will result in more martensite phase formed in the steel.

4. Conclusions

The cooling rate of the quench medium with several variations of particle size was conducted. Micro-size (44 μm), reduced micro-size (29μm), and nano-size (21nm) were used as size variation and then dispersed in distilled water as the fluid base. Vickers hardness test showed that the highest steel hardness was archived by quenching in 0.5% nano-size TiO_2 particle water-based quench medium. In this condition, the steel hardness could reach up to 691 HV.
This increment was quite significant. It was almost 100HV increment from steel quenched in normal water-based quench which only had 598 HV. The hardness improvement may occur because of the TiO_2 nanoparticle, which has higher thermal conductivity, provide better heat transfer in the medium during quenching.
In other words, nanoparticle in the quench medium produces a faster cooling rate. Thus, more martensite phase was formed. Hence, the higher the hardness number. The smaller size and a higher percentage of the particle may affect the cooling rate as well.

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6. References

[1] Babu, K., Arularasan, R., & Srinath Ramkumar, S. (2017). *Quenching performance of AISI 1010 in CNT nanofluids*. Materials Today: Proceedings, *4*(10), 11044–11049. http://doi.org/10.1016/j.matpr.2017.08.065

[2] S.S. Yahya, S. Harjanto, W.N. Putra, G. Ramahdita, Kresnodrianto, and E. P. Mahiswara. *Characterization and observation of water-based nanofluids quench medium with carbon particle content variation,* AIP Conference. Proceedings 1964, 020006(2018);https://doi.org/10.1063/1.5038288

[3] J Župan, T Filetin & D Landek (2012) *The effect of TiO_2 nanoparticles on fluid quenching characteristics*, International Heat Treatment and Surface Engineering, 6:2, 56-60, DOI: 10.1179/1749514812Z.00000000014

[4] Ramesh, G. and Narayan K. Prabhu., "*Effect of Addition of Aluminum Nanoparticles on Cooling Performance and Quench Severity of Water during Immersion Quenching*." In STP1567-EB Nanofluids, edited by K. Prabhu, (pp. 122-135). West Conshohocken, PA: ASTM International, 2012. doi:10.1520/STP156720120008

[5] Ramesh, G. & Prabhu, N.K. *Review of thermo-physical properties, wetting and heat transfer characteristics of nanofluids and their applicability in industrial quench heat treatment.* Nanoscale Res Lett (2011) 6: 334. https://doi.org/10.1186/1556-276X-6-334

[6] Dhinesh K D and Valan A A. 2016. *A review on preparation, characterization, properties and applications of nanofluids*. (India: Elsevier) 21-40.

[7] Yu, W., France, D., Choi, S., & Routbort, J *Review and Assessment of Nanofluid Technology for Transportation and Other Application.*. 2007, Argonne: Energy Systems Division, Argonne National Laboratory.

[8] Akhgar, A., & Toghraie, D. (2018). An experimental study on the stability and thermal conductivity of water-ethylene glycol/TiO_2-MWCNTs hybrid nanofluid: Developing a new correlation. *Powder Technology*, *338*, 806–818. https://doi.org/10.1016/j.powtec.2018.07.086 Developing a new correlation. *Powder Technology*, *338*,806–818. https://doi.org/10.1016/j.powtec.2018.07.086

[9] A.M. Rashidi, M.M. Akbarnejad, A.A. Khodadadi, Y. Mortazavi, A. Ahmadpour, Single-wall carbon nanotubes synthesized using organic additives to Co-Mo catalysts supported on nanoporous MgO, Journal of Nanotechnology 18 (31) (2007). No.315605