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Characterization of oil based nanofluid for quench medium

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Abstract- The choice of quench medium depends on the hardenability of the metal alloy, the thickness of the component, and the geometry of the component. Some of these will determine the cooling rate required to obtain the desired microstructure and material properties. Improper quench media will cause the material to become brittle, suffers from geometric distortion, or having a high undesirable residual stresses in the components. In heat treatment industries, oil and water are frequently used as the quench media. Recently, nanofluid as a quench medium has also been studied using several different fluids as the solvent. Examples of frequently used solvents include polymers, vegetable oils, and mineral oil. In this research, laboratory-grade carbon powder were used as nanoparticle. Oil was used as the fluid base in this research as the main observation focus. To obtain nanoscale carbon particles, planetary ball mill was used to ground laboratory grade carbon powder to decrease the particle size. This method was used to lower the cost for nanoparticle synthesis. Milling speed and duration were set at 500 rpm and 15 hours. Field Emission Scanning Electron Microscope (FE-SEM), and Energy Dispersive X-Ray (EDX) measurement were carried out to determine the particle size, material identification, particle morphology, and surface change of samples. The carbon nanoparticle content in nanofluid quench mediums for this research were varied at 0.1%, 0.2%, 0.3%, 0.4, and 0.5 % volume. Furthermore, these mediums were used to quench JIS S45C or AISI 1045 carbon steel samples which annealed at 1000°C. Hardness testing and metallography observation were then conducted to further examine the effect of different quench medium in steel samples.

1. Introduction

One of the process of steel hardening is quenching. Quenching refers to the fast cooling of a steel. In quenching, an austenized steel is immersed in a liquid, called quench medium or quenchant [1]. Commonly used quench medium are water, brine, polymers, oils, salts, and gases which have their own cooling characteristics. Brittle materials, geometric distortion, and high undesirable residual stresses on the components that lead to cracks could occur from improper use of quench media. The result of quenching, is highly effected by the cooling rates of quench media. In quenching, to get a good result, a cooling rate needs to be equal or higher than the critical cooling rate for martensite formation [2]. On the other hand, the geometry of the part is another factor that inluence the result of quenching. For components that has variation on their thicknes, quench medium with low cooling rates was used to avoid excessive residual stress, since a rapid cooling has a high possibility of the occurance of cracks, especially on the critical parts of the components. The example for selecting a quench medium is using oil as quench medium instead of water to get more evenly distributed cooling and to avoid cracking. However, the slow cooling rates of oil will result in lower hardness than the hardness achieved by water as quench medium.

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 thermal performance of heat transfer of the base fluid[3]. Studies on the performance of nanofluids with various types of particles has been done by many researchers[1, 4-15].

The present study aims to observe the quenching of carbon steel S45C using oil-based nanofluid with laboratory-grade carbon powder as nanoparticles. The samples were heated at 1000 ^{o}C for an hour and then quenched. The effect of nanofluid concentration was investigated to illustrate an optimal ratio of nanoparticle used in achieving the desired material properties.

2. Materials and methods

S45C carbon steel bar was used in this study. The chemical composition of the carbon steel sample was determined as given in Tables 1[16].

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	

This study started by preparation of specimen of medium carbon steel about 30 samples. The samples were cut in the dimension of 15mm x 10mm x 10mm. Among this samples, non-heat treated were prepared as baseline comparison. Steel samples were pre-heated at 600 ^{o}C and then annealed at 1000 ^{o}C and holding it for an hour. Those samples were then quenched in oil-based nanofluid. A vickers hardness testing is conducted to measure the hardness of the samples. In the hardness testing, a force of 300 gf is applied to press the diamond indentor into the samples, and the force is maintained for 10 s. Finally, the hardness of the materials can be calculated by the size of the indentation. To obtain the average hardness, five different locations on each sample are selected to measure the hardness.

For nanofluids preparation, laboratory-grade carbon powder was used to synthesized oil-based nanofluids by two-step method. The nanofluids of 0.1%, 0.2%, 0.3%, 0.4% and 0.5% volume fraction of particles were prepared. The nanoparticles were first produced by high energy milling. The second step involves dispersing this nanopowder into the base fluid with the help of intensive ultrasonic agitation. The two-steps method was more economical than the one-step method to produce nanofluids commercially [15]. This method was suitable for wide range of particles such as oxide particles and carbon nanotubes and it is attractive to industry because it is simple for nanofluid preparation[17].

Nanoparticles synthesis was carried out at 500 rpm for 15 hours in the high energy ball mill. The nanoparticles were weighted according to the weight of each quenching medium based on % volume. After this step, oil was slowly added to 100 ml beaker glass which is filled wirh nanoparticles powder. SAE 5W-40 oil was used for this particular study. In order to increase the stability of solutions, each nanofluid suspension was sonicated in an ultrasonic apparatus for 280 seconds before the quenching experiments.

Microstructure examination were carried out for both treated and untreated samples. Each sample was prepared using standard metallography procedure. The phase of the specimens were made visible by etching using nital containing 2% Nitric acids and 98% methylated spirit on the polished surfaces.

Then the specimen was immersed into etching media for 3 seconds. Microscopic examination of the etched surface of various specimens was taken using a metallurgical microscope.

3. Results and discussion

Field Emission Scanning Electron Microscope (FE-SEM) imaging was conducted to examine the everage particle size of the laboratory-grade carbon powder before milling (Figure 1). From this observation, the average size of the powder used in this study was 15.04 µm.

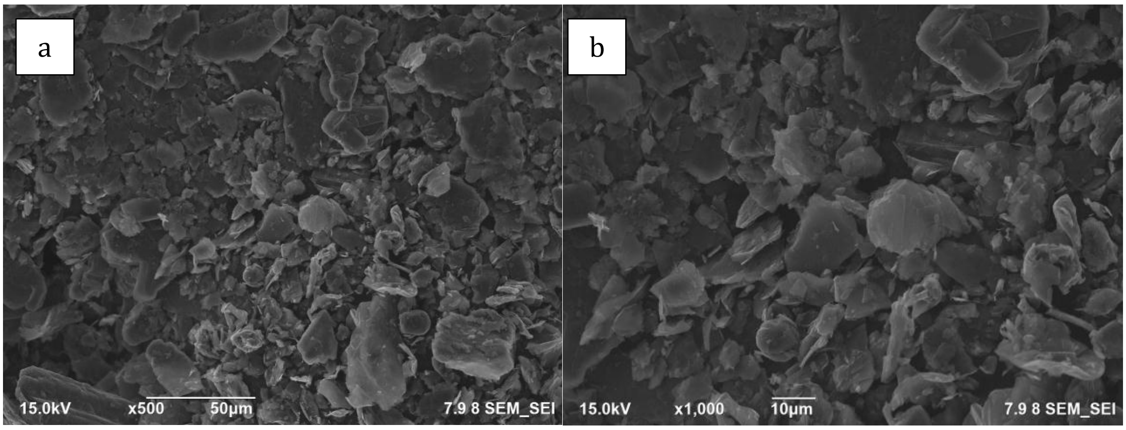


Figure 1. FE-SEM image of laboratory-grade carbon powder with (a) 500x magnification and (b) 1000x magnification.

The laboratory-grade carbon powder consist of 99.9 wt% carbon and some impurities of 0.1% Cu. This result confirms that the powders was mainly consist of carbon. The microstructure of the non-heat treated sample was shown in Figure 2[16]. Figure 2 showed that the non-heat treated sample mainly consist of ferrite and pearlite. The ferrite structure is the white area, while the darker area is pearlite.

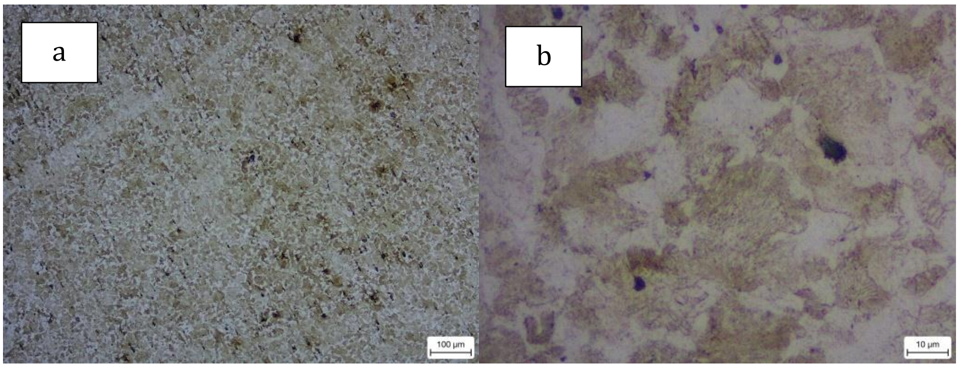
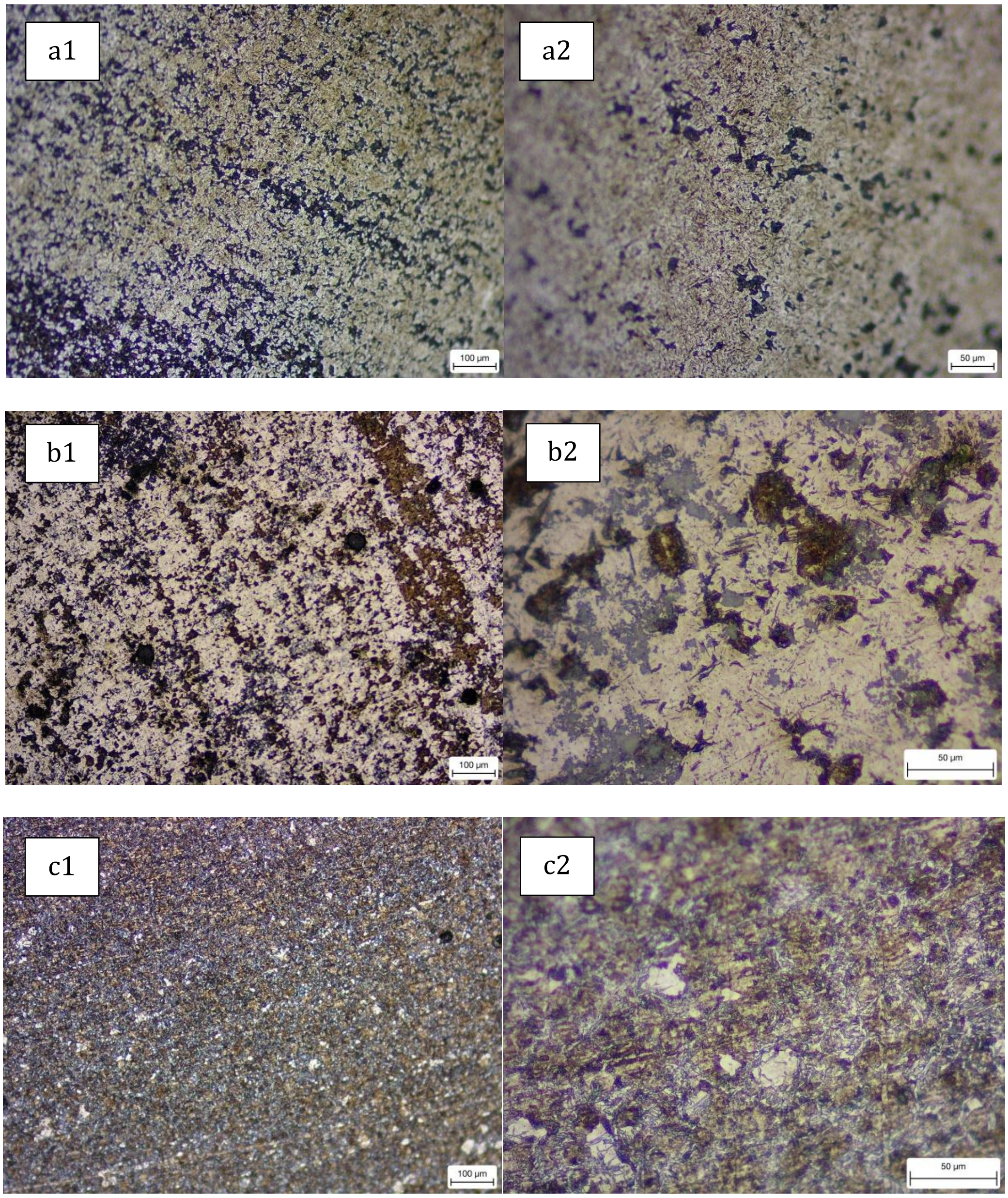


Figure 2. Microstructure of non-heat treated sample (a) 100x magnification (b) 1000x magnification

Figure 3 showed the microstructure of S45C after quenching in oil based laboratory grade nanofluid. Martensite structure was observed in the samples which were quenched in 0.1% vol. and 0.2% vol. nanodluid. As for the rest of the samples, the martensite structure was few. In sample 0.3% and 0.5 %, many pearlite structures was observed. This may because of particle agglomeration occurred in the nanofluid. Sample that quenched in 0.4% vol. nanofluid shows many martensite structure. This anomaly happens because the agitation by ultrasonic is poorly done, so the nanoparticle was not dispersed fully which affect the nanofluid performance.

The hardness of the sample quenched in oil is 363 HV. The maximum hardness for oil-based nanofluid was obtained at 0,2% vol. nanofluid with 570,98 HV (Figure 4). This value was linear to its microstruture which showed the most martensite structure. From the result, it could be found that carbon nanoparticles inside the fluid can enhance the quench severity with appropriate concentration. This happen due to transformation of martensite with rapid cooling. Transformation of martensite is driven by nanoparticles in the fluid which is having higher thermal conductivity than in normal fluid. The heat transfer enhancement phenomenon can be illustrated as the formation of nanoparticle embedded surface due to scattered deposition of nanoparticles. As a result, it increases the effective surface area of heat transfer[18]. Samples quenched in water have a hardness of 675 HV. From this, we can conclude that the oil based nanofuild has a severity of quenching between that of water and oil.



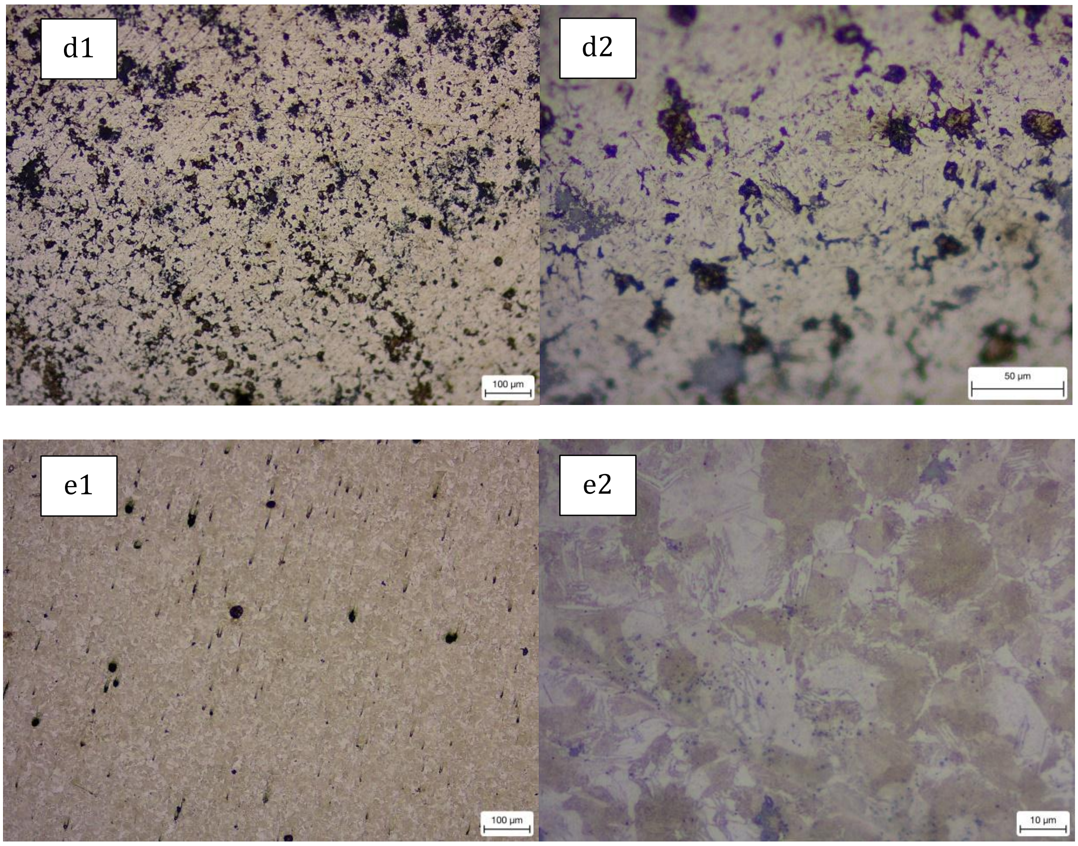


Figure 3. Microstructure of S45C after quenching in oil based laboratory grade nanofluid (a) 0.1% vol. (a1 for 100x magnification and a2 for 200x magnification), (b) 0.2% vol. (b1 for 100x magnification and b2 for 500x magnification), (c) 0.3% vol. (c1 for 100x magnification and c2 for 500x magnification), (d) 0.4% vol. (d1 for 100x magnification and d2 for 500x magnification) and (e) 0.5% vol. (e1 for 100x magnification and e2 for 1000x magnification).

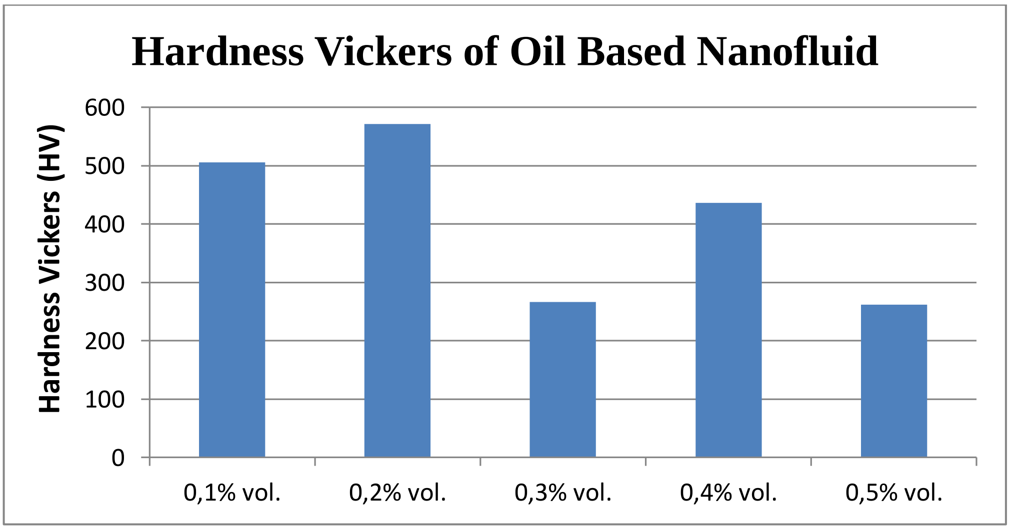


Figure 4. Result of Hardness Vickers Testing

4. Conclusion

The effect of laboratory-grade carbon in oil as quench medium was experimentally investigated using medium carbon steel, JIS S45C as specimen. For this purpose, various concentration of nanofluids were prepared with two step method. The variation of concentration is 0.1%, 0.2%, 0.3%, 0.4% and 0.5% volume. The microstucture and hardness value shows that volume fraction of nanoparticles used in nanofluids considerably affected the quenching process. Quenching with oil-based nanofluid will produce martensite structure. However, beyond 0,2% vol. the material phase mainly consist of pearlite structure. The maximum hardness is observed in 0,2% vol. with 570,98 HV. Higher particle concentration showed lower hardness which probably due to particle agglomeration. Overall, the hardness value was still higher than cooling without addition of nanoparticles. This concluded that nanoparticles inside the fluid can enhance the quench severity with appropriate concentration.

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