Polyethylene Glycol Addition as Non-ionic Surfactant in Water-based Carbon Microfluid for Quench Medium in Heat Treatment Process

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Abstract. The mechanical properties of a material depend on the quenching process. In this process, there is a rapid cooling from elevated to room temperature in a short time by using a quench medium. Therefore, the phase transformation from austenite to martensite occurs. The common medium used in the quenching process is water, oil, polymer, and gas. Nanofluids are started to be used as a quench medium because they offer better thermal conductivity compared with the conventional medium. Selection of carbon-based nanofluids as a quenching process medium aims to obtain high thermal conductivity values and controllable cooling rates. Thereby, the expected microstructure of the material could be relatively easier to form. In this paper, carbon particles were obtained using a top-down method with a planetary ball mill for 15 hours at 500 rpm. Based on the electron microscope and spectroscopy results, the particle dimension was average at 15 µm after milling, and the carbon purity of the powder used in this research was 99%. Carbon particles at 0.1%, 0.3%, and 0.5% with variation of non-ionic surfactant Polyethylene Glycol of 1%, 2%, 3%, 4% and 5% respectively was used in this research. AISI 1045 or JIS S45C carbon steel was used as a steel sample, and austenized at 1000°C for 1 hour and then quenched in the microfluid. The hardness obtained was up to 811 HV for the sample quenched in 0.5% carbon and 1% Polyethylene Glycol. The improvement was more than 100 HV, compared with the sample quenched in distilled water, which had a hardness only 666 HV.

Keywords: nanofluid, quenching, heat treatment.

1. Introduction
Heat treatment is a crucial process in the manufacturing industry, as it can modify the mechanical properties of a material. To increase the material hardness, for example, there is a rapid cooling from austenizing temperature to room temperature in a short time by using a quench medium, which is called quenching. Therefore, the phase transformation from austenite to martensite occurs, hence increasing the hardness. To obtain a martensite phase, the cooling rate should be equal to or higher than the critical cooling rate [1]. The standard medium used in the quenching process is water, oil, polymer, and gas.

Recently, nanofluids are started to be used as a quench medium because they offer a better thermal performance of and better thermal conductivity compared with the conventional medium [2]. Nanofluid...
is a mixture of fluids with nanoparticles (<100 nm). Many researchers have been done several research on the nanofluids performance as quench medium with different types of the particle [3-11].

However, several particles have a hydrophobic characteristic, which will reduce the performance of the nanofluid. Therefore, a surfactant is used to solve this problem. This study aims to show the effect of Polyethylene Glycol (PEG) addition as non-ionic surfactant and stabilizer for water-based quench medium with carbon microparticle. The effects of different concentrations of carbon and surfactants mixed in microfluids quench media were also discussed.

2. Materials and methods
The carbon particle used were purchased from Sigma-Aldrich with an average particle size of 21 μm. The particle size was reduced further by a top-down method with a planetary ball mill for 15 hours at 500 rpm. Observation of particle size, material morphology, and material composition were done using Field-Emission Scanning Electron Microscope (FE-SEM) and Energy Dispersive X-Ray Spectroscopy (EDS). To produce the microfluid, 100 ml water was used as the base, mixed with carbon particles at 0.1%, 0.3%, and 0.5% and variation of non-ionic surfactant Polyethylene Glycol of 1%, 2%, 3%, 4%, and 5% were also added. The micro-fluid was ultra-sonicated to increase its dispersion for 280 seconds.

JIS S45C carbon steel was used as a sample. Table 1 determine the chemical composition of the sample. This sample was the same used in the previous publication [12].

Table 1. The S45C carbon steel sample chemical composition (% weight) [12]

|     | Fe   | C    | Si   | Mn   |
|-----|------|------|------|------|
| P   | 98.3 | 0.47 | 0.287| 0.718|
| S   | 0.0261| 0.005| 0.028| 0.005|
| Cr  | 0.005 | 0.2  | 0.003| 0.018|
| Al  | Nb   | Ti   | Cr   | Mo   |
| Ni  | 0.005| 0.2  | 0.003| 0.018|
| Mo  | 0.002| 0.035|<0.002|<0.01 |
| P   | <0.002|<0.002| 0.0013| 0.0005|
| Sn  | 0.025| 0.008|<0.03 |
| B   | Bi   | As   | Ca   |
| Pb  |     |<0.002| 0.0013| 0.0005|
| Zr  |     |<0.002| 0.008|<0.03 |

In this study, eight samples were used with dimension 15x10x10 mm. Samples were pre-heated at 600°C with heating rate 13.3°C/minute, and then austenitized at 1000°C for 1 hour. Then the sample was quenched in the previously made micro-fluid.

To observe the microstructure of the quenched sample, standard metallography procedure was used. Samples were mounted with resin to help the sample handling. Then, samples were ground gradually from 80 grit to 1500 grit, and polished. The phase of the sample was made visible in the microscope by etching using nital (3% nitric acid, 97% alcohol) for 5 seconds.

To observe the mechanical properties of the quenched sample, vickers hardness testing was conducted. The applied force to press the indentor was 300 gf into the sample for 10 seconds. Three different locations were selected to measure the average hardness on each sample.

3. Results and discussion
Based on the FE-SEM results, the particle dimension of the laboratory-grade carbon powder was average at 15 μm after milling; therefore, it was still in micro range. Figure 1 showed the morphology of the carbon particle. EDS result in Table 2, showed that carbon purity of the powder used in this research was 99% [12].
Figure 2 showed the microstructure of non-heat treated sample, which ferrite and pearlite are the mainly phase. The bright area showed the ferrite structure, and the darker area showed the pearlite structure[12].

| Laboratory-grade carbon powder | C    | Cu  |
|--------------------------------|------|-----|
|                                | 99.9 | 0.1 |

Table 2. The EDS result of the laboratory-grade carbon powder [12]

Figure 1. FE-SEM image of laboratory-grade carbon powder with 1000x magnification.

Figure 2. The microstructure of non-heat treated sample 1000x magnification

Figure 3 showed the microstructure of the sample after quenching. Martensite and bainite structure were observed in all of them but in different amount. However, it was difficult to justify the amount difference based on the microstructure only. Therefore, Vickers hardness testing was done to observe the hardness on each sample. Higher hardness could be related to a higher amount of martensite phase in the sample.

| Sample | Microstructure |
|--------|----------------|
| (a)    | 0.1% carbon, 0% PEG |
| (b)    | 0.1% carbon, 1% PEG |
| (c)    | 0.1% carbon, 3% PEG |
| (d)    | 0.1% carbon, 5% PEG |
| (e)    | 0.5% carbon, 0% PEG |
| (f)    | 0.5% carbon, 1% PEG |
| (g)    | 0.5% carbon, 3% PEG |
| (h)    | 0.5% carbon, 5% PEG |

Figure 3. Microstructure of S45C after quenching in water-based carbon micro-fluid 100x magnification

The Vickers hardness testing of the samples was showed in Figure 4. The hardness of the sample that quenched without carbon and PEG was the lowest, i.e. 666 HV. The maximum hardness is 811 HV (0.5% Carbon, 0.1% PEG). While the maximum hardness for micro-fluid with 0.1% carbon is 769 HV (0.1% PEG).
This phenomenon showed clearly that the addition of carbon micro-particle improved the hardness of steel by increasing the cooling rate during quenching. The higher cooling rate would result in more martensite phase formed in steel, hence the higher hardness number.

On the other hand, the addition of PEG as a non-ionic surfactant and stabilizer in water-based carbon micro-fluid for quench medium with an appropriate concentration can enhance the quench severity, i.e. increasing the cooling rate. Carbon is hydrophobic in nature; therefore, it tends to agglomerate in the water. It can be seen from the result that higher carbon percentage in the quench medium lower the hardness.

To avoid this, a surfactant was added to reduce the surface tension of carbon particle in water. Without agglomeration, more surface area of carbon was available and improving the heat transfer in the quench medium. Excessive addition of surfactant, however, showed to decrease the effectivity of quench medium by introducing a re-agglomeration of carbon particle.

Figure 4. The Vickers hardness testing result.

4. Conclusion
The effect of PEG addition as non-ionic surfactant and stabilizer in water-based carbon micro-fluid for quench medium was experimentally investigated using JIS S45C carbon steel. The variation of carbon concentration is 0.1% and 0.5% with addition PEG 0%, 1%, 3%, and 5% each variation of carbon concentration. The result of metallographic observation and hardness testing showed that the concentration of carbon and PEG addition considerably affect the quenching process by increasing the cooling rate.

The maximum overall hardness was 811 HV (0.5% Carbon, 0.1% PEG). Meanwhile, the maximum hardness for micro-fluid with 0.1% carbon is 769 HV (0.1% PEG). In general, the hardness of steel quenched in normal water was lower than the sample quenched with carbon micro-fluid. PEG addition as a surfactant could increase the hardness in the appropriate concentration. Excessive addition of surfactant could reduce the quench medium effectivity.

From all the result, it can be concluded that addition polyethylene glicol as a non-ionic surfactant and stabilizer in water-based carbon micro-fluid with an appropriate concentration can enhance the heat transfer for quench medium in heat treatment process.

Acknowledgement
The authors would like to thank Ms. Ghiska Ramahdita for the fruitful discussion, and gratefully acknowledged the financial support from Hibah PITTA 2018 funded by DRPM Universitas Indonesia No. 2555/UN2.R3.1/HKP.05.00/2018.
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