Characterization of water based nanofluid for quench medium

Kresnodrianto 1, a), S. Harjanto 1, b), W.N. Putra 1, c), G. Ramahdita 1, d), S.S. Yahya1, E.P. Mahiswara 1

1 Material and Metallurgy Departement, Faculty of Engineering, Universitas Indonesia, Depok, Indonesia
a) kresnodrianto@ui.ac.id
b) sri.harjanto@gmail.com
c) wahyuaji@metal.ui.ac.id
d) ghiska@metal.ui.ac.id

Abstract - Quenching has been a valuable method in steel hardening method especially in industrial scale. The hardenability of the metal alloys, the thickness of the component, and the geometry is some factors that can affect the choice of quench medium. Improper quench media can cause the material to become too brittle, suffers some geometric distortion, and undesirable residual stress that will cause some effect on the mechanical property and fracture mechanism of a component. Recently, nanofluid as a quench medium has been used for better quenching performance and has been studied using several different fluids and nanoparticles. Some of frequently used solvents include polymers, vegetable oils, and mineral oil, and nanoparticles frequently used include CuO, ZnO, and Alumina. In this research, laboratory-grade carbon powder were used as nanoparticle. Water was used as the fluid base in this research as the main observation focus. Carbon particles were obtain using a top-down method, whereas planetary ball mill was used to ground laboratory grade carbon powder to decrease the particle size. 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. Nanofluid was created by mixing percentage of carbon nanoparticles with water using ultrasonic vibration for 280s. 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 austenized at 1000°C. Hardness testing and metallography observation were then conducted to further check the effect of different quench medium in steel samples. Preliminary characterizations showed that carbon particles dimension after milling was still in sub-micron stage, hundreds of nanometres to be precise. Therefore, the milling process parameters are needed to be optimized further.

1. Introduction
Heat Treatment and quenching have been one of the important process of steel hardening in industrial scale. 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]. Quench medium that commonly used are water, brine, polymers, oils, salts, and gases which have their own cooling characteristics and results. Improper quench media can cause the material to become too brittle, suffers some geometric distortion, and undesirable residual stress that will cause some effect on the mechanical property and fracture
mechanism of a component. 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]. Usually, for components that has variation on their thickness, we use quench medium with low cooling rates to avoid excessive residual stress, since a rapid cooling has a high possibility of the occurrence of cracks, especially on the critical parts of the components. 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, 4-11]. The present study aims to compare the quenching of carbon steel S45C using deionized (DI) water-based nanofluids with laboratory-grade carbon powder as nanoparticles. The samples were heated at 1000 °C for an hour and then quenched. The effect of nanofluid concentration is investigated to illustrate an optimal ratio of nanoparticle that is used in achieving the desired material properties.

2. Materials and methods
S45C carbon steel bar was purchased from a local steel distributor. The chemical composition of the carbon steel sample was determined as given in Table 1.

| 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 °C and then austenitized at 1000 °C and holding it for an hour. Those samples were then quenched in water-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-step method was more economical than the one-step method to produce nanofluids commercially and also considered the best method in dispersing carbon particles in a base fluid[12]. This method is suitable for a wide range of particles such as oxide particles and carbon nanotubes and it is attractive to industry because it is simple for nanofluid preparation[13]. 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, water was slowly added to 100 ml beaker glass which filled of nanoparticles powder. 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 5 seconds. Microscopic examination of the etched surface of various specimens was taken using a metallurgical microscope.
3. Results and discussion

FE-SEM imaging was conducted to examine the average particle size of the laboratory-grade carbon powder before milling (Figure 1). From this examination, the average size of the powder is 15.03767 µm.

![Figure 1. FE-SEM image of laboratory-grade carbon powder with (a) 500x magnification and (b) 1000x magnification.](image1.png)

![Figure 2. EDS layered image of laboratory-grade carbon powder.](image2.png)

**Table 2. Result of EDS for chemical composition (wt%) of laboratory-grade carbon powder**

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

Figure 2 showed the EDS layered micrograph and elemental distribution of laboratory-grade carbon powder. The elemental distribution confirms that the powders is mainly consist of carbon. The exact composition of the powder can be seen in Table 2. The laboratory-grade carbon powder consist of 99.9 wt% carbon and some impurities of 0.1 wt% Cu.

The microstructure of the non-heat treated sample is shown in Figure 3. Figure 3 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 3. Microstructure of non-heat treated sample (a) 100x magnification (b) 1000x magnification.

Figure 4. Microstructure of S45C after quenching in water-based laboratory grade nanofluid (a) 0.1% vol. 100x magnification (b) 0.2% vol. 100x magnification, (c) 0.3% vol. 100x magnification, (d) 0.4% vol. 100x magnification, and (e) 0.5% vol. 100x magnification.
Figure 4 shows the microstructure of S45C after quenching in water based laboratory grade nanofluid. Martensite structure is observed in the samples which were quenched in 0.2% vol. Nanodluid, which can be observed as the one that has the densest martensite structure. The rest also exhibit martensite structure, but in a different amount. This anomaly happens because the agitation by ultrasonic is poorly done, so the nanoparticle was not dispersed fully which effect the nanofluid performance.

The result of hardness Vickers testing of the sample is shown in Figure 5. Figure 5 show that the first samples that is quenched with DI Water exhibit the hardness number of 675 HV. Samples that quench in water-based nanofluid gives a unique behavior, it’s hardness rises until 0.2% and then it drops. The hardness The maximum hardness in water-based nanofluid is obtained by 0.2% vol. nanofluid with 885.34 HV. The hardness value is linear to it’s microstructure which shows the most martensite structure. This result shows that carbon nanoparticles inside the fluid can enhance the fluid 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[14].

![Hardness Vickers of Oil Based Nanofluid](image)

**Figure 5.** Result of hardness vickers testing.

4. Conclusion

The effect of laboratory-grade carbon in water 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 microstructure and hardness value shows that volume fraction of nanoparticles used in nanofluids considerably affected the quenching process. It is observed that after quenching with water-based nanofluid, the microstructure mainly consist of martensite. The maximum hardness is observed in 0.2% vol. which is 885.34 HV. The hardness number is decreasing in 0.3% vol. and increasing again in 0.4% vol and decreasing again in 0.5% vol. This anomaly happened because of the poor mixing process using ultrasonic vibration, that causes agglomeration between the
carbon nanoparticles. The hardness value is higher than cooling without nanoparticles added. This concludes that nanoparticles inside the fluid can enhance the fluid severity with appropriate concentration.

5. Reference

[1] Nayak, Vignesh U. Prabhu, Narayan. K. 2016. “Wetting Behavior and Heat Transfer of Aqueous Graphene Nanofluids.

[2] J. Grum, S. Bozic, and M. Zupancic, Influence of Quenching Process Parameters on Residual Stresses in Steel, J. Mater. Process. Technol., 2001, 114(1), p 57–70

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

[4] Xie H, Wang J, Xi T, Liu Y, Ai F and Wu Q, “Thermal conductivity enhancement of suspensions containing nanosized alumina particles”, (J Appl Phys, 2002) vol. 91:4568-4572.

[5] S. Lee, S.U.S. Choi, S. Li, and J.A. Eastman, "Measuring thermal conductivity of fluids containing oxide nanoparticles", (Journal of Heat Transfer, 1999) vol. 121, pp. 280-289.

[6] X. Wang, X. Xu, and S.U.S. Choi, "Thermal conductivity of nanoparticle–fluid mixture,” (Journal of Thermophysics and Heat Transfer, 1999) vol. 13, pp. 474-480.

[7] J.A. Eastman, S.U.S. Choi, S. Li, W. Yu, and L.J. Thompson, "Anomally increased effective thermal conductivities of ethylene glycol-based nanofluids containing copper nanoparticles,” (Applied Physics Letters, 2001) vol. 78, no. 6, pp. 718-720.

[8] Ramesh, Gopalan and Narayan Kotekar Prabhu, “Review of thermo-physical properties, wetting and heat transfer characteristics of nanofluids and their applicability in industrial quench heat treatment”, (Nanoscale Research Letters, 2011) (15 pages)

[9] Xuan Y and Li Q, “Heat transfer enhancement of nanofluids”, (Int J Heat Fluid Flow, 2000) vol. 21 pp 58-64.

[10] Kim, H., DeWitt, G., McKrell, T., Buongiorno, J., and Hu, L.W., “On the quenching of steel and zircaloy spheres in water-based nanofluids with alumina, silica and diamond nanoparticles”, (International Journal of Multiphase Flow, 2009) vol. 35, pp 427–438.

[11] Khoshmehr, Habibi, Ahmad Saboonchi, and Mohammad Behshad Shafii, “The quenching of silver rod in boiling carbon nano tube-water nanofluid”, (International Journal of Thermal Sciences, 2014) pp 95-104

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

[13] G. Paul, P.K. Das, and I. Manna, “Rewetting of vertical pipes by bottom flooding using nanofluid as a coolant”, (Int. J. Heat Mass Transf., 2015) vol. 137 (9 pages).

[14] N. Patra, V. Gupta, R. Singh, R.S. Singh, P. Ghosh and A Nayak, “An experimental analysis of quenching of continuously heated vertical rod with aqueous Al2O3 nanofluid”, (Resource-Efficient Technologies, 2017) (8 pages)

Acknowledgment

Authors would like to thank the Directorate of Research and Public Services, Universitas Indonesia for financial support and project administration respectively to conduct this research under PITTA Project on 2017 fiscal year.