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Effect of TiO_2 Nanofluid and Hybrid TiO_2 Nanofluid on Mechanical Properties of Steels for Automotive Applications

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Abstract. Quenching is a process involving rapid cooling in quenching media and hardening of steels. The effect of quenching process on the microstructure and mechanical properties (hardness) are studied and elaborated in this thesis. The quenching media uses including conventional fluid (distilled water and mineral oil), nanofluid (TiO_2 nanofluid), and hybrid-based nanofluid (TiO_2 hybrid nanofluid). Thermal conductivity and microstructure development are closely-linked to each other. Conventional fluids as quenching media have low thermal conductivity thus limiting the development of desired microstructure in the steels. The thermal conductivity and stability of nanofluid and hybrid-based nanofluid are studied to enhance the thermal properties of the quenching media.

1. Introduction

Quenching is a process that involves rapid cooling in medium of quenching and hardening of steels or other metal alloys. It helps to improve the mechanical and physical properties such as microstructure and hardness of steels or other metal alloys [1]. The role of quenching application can be seen in industrial areas such as nuclear reactor safety, the cooling of superconductor, metal forming, automotive and flash freezing[2].

During the past decade, the research on nanofluids has been increasing rapidly as it has beneficial value in terms of engineering applications. Metal oxides such as Al_2O_3 [2,3], CuO [3–5], Fe_2O_3, Fe_3O_4, SiO_2 [6], TiO_2[4,7,8], and ZrO_2; metal carbides such as SiC[9], metal nitrides such as AlN, and SiN; and carbon materials such as carbon nanotubes [10–12], graphite, and diamond are usually used as nanoparticles in the researches of nanofluid. In this experiment, TiO_2 nanoparticles and C-TiO_2 hybrid nanoparticles will be synthesized alongside with the conventional fluids to determine their effects on the quenching process. The mechanical and physical properties of heat treated high carbon steel and medium carbon steel will also be investigated by immersing both steels into conventional fluids (i.e. water, mineral oil, ethylene glycol, silicon oil and methanol), TiO_2 nanofluids and C-TiO_2 hybrid nanofluids.

2. Materials and Method

2.1. Synthesis of TiO_2 Nanofluid and Hybrid TiO_2 nanofluid

Different particle size of TiO_2 and C nanopowders are used for synthesis process of different volume fraction of 0.1% TiO_2 nanofluid (100 nm), 0.5% TiO_2 nanofluid (30 nm), and 0.1% TiO_2 - 0.9% C hybrid nanofluid (30 nm). Both nanopowders are weighed accordingly using the equation 1 and 2 respectively;

$$\varphi = \left[\frac{\left(\frac{W}{\rho}\right)_{TiO\ 2}}{\left(\frac{W}{\rho}\right)_{TiO\ 2} + \left(\frac{W}{\rho}\right)_{water}} \right] \tag{1}$$

$$\varphi = \left[\frac{\left(\frac{W}{\rho}\right)_{C}}{\left(\frac{W}{\rho}\right)_{C} + \left(\frac{W}{\rho}\right)_{water}} \right] \tag{2}$$

Table 1: Volume fraction of TiO_2 nanofluid and C-TiO_2 hybrid nanofluid

Volume fraction of TiO_2 (%)	Mass of TiO_2 (g)	Volume fraction of C (%)	Mass of C (g)	Mass of distilled water (g)
0.1	0.064	-	-	15.000
0.5	0.320	-	-	15.000
0.1	0.064	0.9	0.576	15.000

Table 1 shown the volume fraction of TiO_2 nanofluid and C-TiO_2 hybrid nanofluid. The table indicated the mass of TiO_2 and C are required to produce different volume fraction of TiO_2 nanofluid and C-TiO_2 hybrid nanofluid. Next, the TiO_2 nanopowder and C nanopowder are grinded using mortar and pestle to breakdown the aggregated nanopowder respectively. Then, by using magnetic stirrer, both TiO_2 and C nanopowders are poured slowly into distilled water and stirred for 3h.

2.2. *Stability Analysis*

The TiO_2 nanofluid and C-TiO_2 hybrid nanofluid prepared are sonicated using ultrasonication water bath in 3 different time (1h, 3h, and 5h) and their stability are evaluated afted sonication process. The optimum sonication time (the most stable TiO_2 nanofluid and C-TiO_2 hybrid nanofluid) is evaluated and used for quenching process.

2.3. *Thermal Conductivity Analysis*

Thermal conductivity analysis is carried out by using C-Therm equipment. Different type of quenching media such as conventional fluid (i.e. distilled water and mineral oil), different volume fraction of 0.1% TiO_2 nanofluid (100 nm), 0.5% TiO_2 nanofluid (30 nm), and 0.1% TiO_2 - 0.9% C hybrid nanofluid (30 nm) are evaluated and compared in term of thermal properties.

2.4. *Heat Treatment*

A long rod of high carbon steel is cut into same size with dimension 1 cm depth and 1 cm wide using the manual abrasive cutting machine. The various type of specimens is prepared through heating process. The specimens are heated in the furnace until 950 °C for 90 minutes and hold for 20 minutes. Then, the specimens are quenched immediately into the different type of quenching media respectively. One of the specimens heated is allowed to cool down in room temperature.

2.5. *Microstructure Analysis*

The specimens are mounted using acrylic and undergone grinding and polishing processes by using different emery paper (200, 400, 600, and 800) of roll grinder and polish disc to achieve mirror finished specimens. Then, the specimens are dipped into nital solution for 7 secs before conducting microstructure analysis. The microstructure is observed under optical microscope.

2.6. *Hardness Analysis*

Hardness analysis is carried out by Vickers hardness testing. 30 N of load are applied onto the various specimens and the hardness value at 3 different spots are evaluated including inner, middle, and outer part of the specimens.

3. Result and Discussion

3.1. *Thermal conductivity of different quenching media*

Figure 1 shown the thermal conductivity of different quenching media. The thermal conductivity of 0.5% volume fraction of TiO_2 nanofluid (30 nm) shows the highest thermal conductivity with 0.631 W/m.K based on figure 1. The result is followed by 0.1% TiO_2 -0.9% C hybrid-based nanofluid with 0.623 W/m.K and 0.1% volume fraction of TiO_2 with 0.620 W/m.K (100 nm). The thermal conductivity of the TiO_2 nanofluids and C-TiO_2 hybrid-based nanofluid are compared with the conventional fluids used during quenching process. Distilled water has a higher thermal conductivity with 0.620 W/m.K compared to mineral oil with 0.139 W/m.K. It is proven that both nanofluids and hybrid-based nanofluid have higher thermal conductivity compared to the conventional fluids. Based on the previous studies made by many researchers, hybrid-based nanofluid has a better thermal properties' performance compared to nanofluid. However, there is an anomaly found based on the data chart shown in figure 1. The thermal conductivity of 0.5% volume fraction of TiO_2 nanofluid is higher compared to 0.1% volume fraction of TiO_2 hybrid-based nanofluid. Therefore, different methods of thermal conductivity measurement should be considered and carried out to ensure the accuracy of the results.

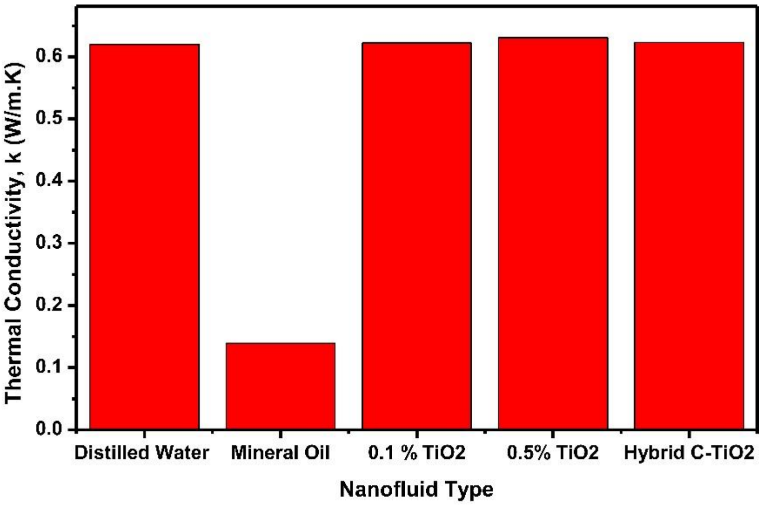

Figure 1: Thermal Conductivity of Different Quenching

3.2. *Stability of nanofluid and hybrid nanofluid*

The synthesis of nanofluids and hybrid-based nanofluid with volume fractions of 0.1% TiO_2 nanofluid (100 nm), 0.5% TiO_2 nanofluid (30 nm), and 0.1% TiO_2 - 0.9% C hybrid-based nanofluid are carried out with different sonication times of 1h, 3h, and 5 h. Figure 2 shows the most stable TiO_2 nanofluids and C-TiO_2 hybrid-based nanofluid with sonication time of 1 h.

Figure 2: (a) C-TiO_2, (b) 0.1% TiO_2 (100 nm particle) and 0.5% TiO_2 (30 nm particle) nanofluid

3.3. *Microstructure of the quenched sample*

The microstructure of the specimens is observed under an optical microscope. Figure 3 shows the microstructure of high carbon steel before heat treatment process. Based on the result shown, fine mixture of ferrite, iron carbide, and pearlite are observed on the specimen. Based on figure 3(b), martensite or needle-like structure is observed in quenched samples of distilled water. The quenched sample of 0.1% TiO_2 nanofluid (figure 3d) is made up of mostly martensite and bainite, and a few pearlite structures. Martensite and bainite structures are observed in the quenched samples of 0.5% TiO_2 nanofluid (figure 3e) and 0.1% C- 0.9% of TiO_2 hybrid nanofluid (figure 3f). The least martensite with abundant pearlite structures are observed in mineral oil quenched sample (figure 3c). It can be concluded that the thermal conductivity in different quenching media affects the changes in microstructure. The rapid cooling due to the high thermal conductivity of quenching medium allows the formation of martensite structure in the high carbon steels specimen.

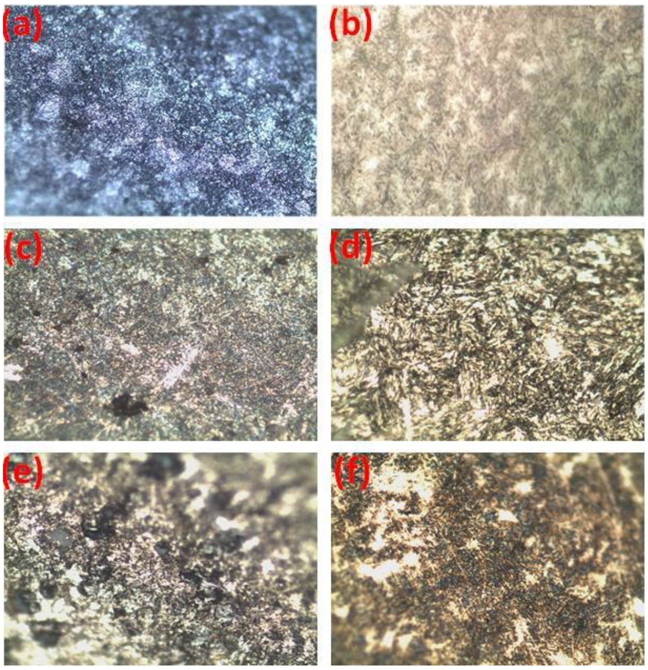

Figure 3. Microstructure of (a) As-received high carbon steel, sample of quenched sample in (b) Distilled water, (c) Mineral oil, (d) 0.1% TiO_2, (e) 0.5 % TiO_2, (f) C-TiO_2 hybrid nanofluid.

3.4. *Hardness analysis*

The hardness value at three different spots of heat treated and quenched samples are measured and tabulated in table 2. The hardness value of heat treated high carbon steels are taken in three different spots (i.e. inner, middle, and outer). The highest hardness value measured is the quenched sample of 0.1% TiO_2 - 0.9% C volume fraction of hybrid-based nanofluid which is 1677.77 HV. The least hardness value measured is the normalized sample (without quenching process) which is 710.37 HV. Furthermore, the outer spots of the quenched samples (i.e. distilled water, mineral oil, 0.1% and 0.5% volume fractions of TiO_2 nanofluids, and 0.1% TiO_2 - 0.9% C volume fraction of hybrid-based nanofluid) compared to the inner and middle spots of quenched samples. In essence, the hardness values in the quenched samples are influenced by the microstructure. For instance, 0.1% TiO_2 - 0.9% C volume fraction of hybrid-based nanofluid consists of martensite and bainite structure, thus it will improve the material strength as it is extremely hard and brittle. Plastic deformation may occur as well due to the slip mechanism in the quenched sample.

Table 2: Hardness value of heat treated and quenched samples at three different spots

Type of Quenching Media	Type of Spots	Hardness Value (HV)	Average Hardness Value (HV)
Distilled water	Inner	708.3	1015.43
	Middle	662.7	
	Outer	1675.3	
Normalising	Inner	483.7	710.37
	Middle	416.1	
	Outer	351.1	
Mineral Oil	Inner	880.2	839.00
	Middle	489.3	
	Outer	1147.5	
0.1% TiO_2 nanofluid	Inner	557.7	954.77
	Middle	594.4	
	Outer	1712.2	
0.5% TiO_2 nanofluid	Inner	1095.8	1275.73
	Middle	1571.4	
	Outer	1160.0	
0.1%TiO_2-0.9% C hybrid nanofluid	Inner	1750.3	1677.77
	Middle	1501.2	
	Outer	1781.8	

4. Conclusions

Throughout the experiment conducted in this project, there were four different aspects of analysis can be concluded in terms of thermal conductivity, stability, microstructure, and hardness, of the heat treated samples in different quenching media.

i) Thermal conductivity:

The thermal conductivity of TiO_2 nanofluids are influenced by the different size of TiO_2 nanoparticles. For example, the size of TiO_2 nanoparticle in 0.5% volume fraction of TiO_2 nanofluid is smaller compared to 0.1% volume fraction of TiO_2 nanofluid, resulting to a higher thermal conductivity. The C-TiO_2 hybrid-based nanofluid showed the lower thermal conductivity compared to 0.5% volume fraction of TiO_2 nanofluid. Both nanoparticles size used for synthesis are 30 nm. This could indicate that there were abnormalities of data collected from C-Therm equipment. The value of thermal conductivity of C-TiO_2 hybrid-based nanofluid should be higher than TiO_2 nanofluids. Hence, an improvement of methods of thermal conductivity analysis should be considered, carried out, and evaluated to obtain the most accurate and precise data.

ii) Stability:

The stability of the different type of nanofluids' synthesized showed that 1 h sonication time was the optimum time to achieve the stability. The high time leads to deterioration of the stability of nanofluid.

iii) Microstructure:

Microstructure of quenched samples are affected by the thermal conductivity of different quenching media (conventional fluids such as distilled water and mineral oil, and different type of nanofluids such as TiO_2 nanofluids and C-TiO_2 hybrid-based nanofluid). The martensitic structure is observed under microscope on quenched samples of both TiO_2 nanofluids, TiO_2 hybrid-based nanofluids, and distilled water. These quenching media showed the significant values of thermal conductivity that enable the transformation of austenitic structure to martensitic structure.

iv) Hardness:

The hardness value is influenced by the microstructure observed on the surface of quenched samples. The highest hardness value measured is the C-TiO_2 hybrid-based nanofluid quenched sample. There were abundant of martensite and bainite structure causing the great enhancement of the hardness of the heat treated quenched sample. The plastic deformation in the quenched sample may occur due to the slip mechanism develop because of the quenching process.

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