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The Effect of Sodium Dodecyl Benzene Sulphonate Addition in Carbon Nanotube-Based Nanofluid Quenchant for Carbon Steel Heat Treatment

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Abstract: Nanofluid is widely researched as it has a superior thermal characteristics. It is synthesized by adding a nano-sized particle with high thermal conductivity in the base fluid. The particle addition would increase the overall thermal conductivity of the fluid. Because of its thermally enhanced characteristics, nanofluid is commonly utilized for thermal transfer applications. However, agglomeration could hinder the dispersion of the particle in the fluid. To improve the nanoparticle dispersion, surfactant is commonly added. In heat treatment, nanofluid can be used as a quench medium. This research used a Multiwalled Carbon nanotube (MWCNT) as the nanoparticle, ranging from 0.1, 0.3, and 0.5% w/v. Sodium Dodecyl Benzene Sulphonate (SDBS) was added as the surfactant with a 0 – 30% w/v concentration. Higher concentrations of SDBS create better stability, as shown in Zeta Potential at -71.2 mV. The thermal conductivity was increased to 0.68 W/mK by adding 0.5% MWCNT and 3% SDBS. S45C medium carbon steel was quenched in the nanofluid and achieved a maximum hardness of 40 HRC at the addition of 0.3% MWCNT and 5% SBDS. Excessive addition of SDBS surfactant could decrease the thermal conductivity and steel hardness, even though the stability was better.

Keywords: Heat Treatment, MWCNT, Nanofluid, SDBS, S45C Carbon Steel

1. Introduction

A solid particle commonly has much higher thermal conductivity than a fluid. Hence, adding solid particles into a fluid could enhance thermal conductivity[1)]. In nanofluid, a nanoscale particle is added into a base fluid to further increase its thermal conductivity due to the particle's vast surface area[2)]. The use of nanofluid is essential in heat transfer applications. One example of nanofluid application in heat treatment is as a quench medium [3–5)].

Several types of particles have been studied, such as metal, metal oxides, and carbon-based particles to be used as nanoparticles in the nanofluid[6–8)]. Advanced carbon-based particles like graphene, graphene oxide and carbon nanotube have significantly higher thermal conductivity than metal or metal oxide particles[9)]. Therefore, a dispersion of a small amount of this advanced carbon particle could potentially increase the thermal conductivity more significantly[10)].

The problem with nanoparticle dispersion in a fluid is agglomeration. In the fluid, nanoparticle tends to clump together[11,12)]. If agglomeration occurs, the effectiveness of the particle for heat transfer is reduced. Hence, particle stability is vital to prevent agglomeration and sedimentation. Mixing some surfactant in the nanofluid is a common practice to improve stability[13,14)]. Surfactant addition can modify the surface tension of the nanoparticle, allowing it to disperse better in the fluid[15)]. There are several types of surfactants based on the charge type, called cationic, anionic, amphoteric, and non-ionic. Better nanoparticle dispersion would retain their heat transfer ability in the nanofluid.

Nanofluid can be used as a quench medium in the heat treatment field, especially for metal materials[16,17)]. Quench medium is used to rapidly cool a steel from elevated temperature to enhance its mechanical characteristics, especially the hardness. The hardness of the steel is an essential characteristic of tribological performance [18,19)]. The steel needs to resist friction or wear from external force[20,21)]. The mechanism of the steel hardening is by phase transformation from the Austenite phase into the Martensite phase through rapid cooling. Another advantage of using nanofluid as a quench medium is the cooling rate adjustment[22]. In theory, the thermal conductivity and the cooling rate can be controlled by adjusting the particle concentration in the fluid. A quench medium with a controlled cooling rate can be helpful, as inappropriate cooling could result in many problems[23,24]. A slower cooling rate might reduce the amount of Martensite phase formation and increase the Bainite phase instead[25,26]. On the other hand, cooling too quickly could risk distortion and crack formation. Excessive Martensite phase in the steel would also unnecessarily increase the materials' hardness, producing a problem during subsequent component fabrication[27,28].

In this study, the effect of surfactant addition on thermal conductivity of the nanofluid quench medium was observed. The thermal conductivity of the quench medium would affect the quenched steel characteristic, especially its hardness due to the change of the fluid's cooling rate. The relationship between surfactant concentration, thermal conductivity, and steel hardness was elaborately discussed.

2. Experiment Setup

This research used a Multiwalled Carbon Nanotube (MWCNT) as the nanoparticle. The average diameter of the MWCNT was 65nm according to the material data sheet. Further inspection to observe the MWCNT morphology was conducted under a Scanning Electron Microscope using FEI Inspect F50 with Energy Dispersive Spectroscopy (EDS) from EDAX Ametek Inc., United States. The length of the MWCNT was more than five µm, as seen in Fig. 1. To study the effect of MWCNT concentration, a variation of 0.1, 0.3, and 0.5% w/v was used to synthesize the nanofluid. The base fluid for the nanofluid in this research was distilled water. The total nanofluid volume per variation was 100 ml.

Sodium Dodecyl Benzene Sulphonate (SDBS) was used as a surfactant to improve the stability of the nanofluid. Both the MWCNT and SDBS were purchased from Sigma Aldrich.. The addition of surfactant varied from 3 – 30% w/v in each MWCNT variation. Zeta potential and thermal conductivity were conducted to observe the particle dispersion stability and thermal characteristics of the nanofluid, respectively. For zeta potential, the equipment used was the SZ-100 series from Horiba, Japan. KD2 Pro Thermal Analyzer from Meter Group, United States, was conducted for the thermal conductivity measurement.

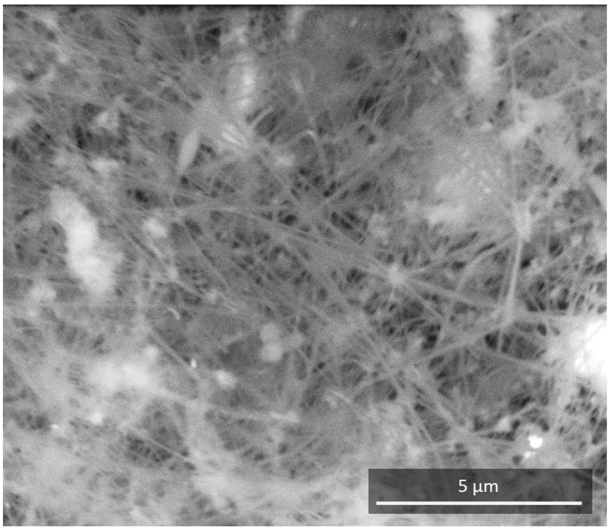

Fig. 1: Multiwalled Carbon Nanotube under Scanning Electron Microscope observation

The nanofluid was subsequently used to quench a steel sample during heat treatment. The steel used in this research was a medium carbon S45C steel. The steel's composition and dimensions are shown in Table 1 and Fig. 2, respectively. For the thermal treatment, the steel was preheated at 540°C for 10 minutes and austenized at 900°C for 60 minutes. On both preheat and austenization, the heating rate was 10°C per minute. Figure 3 shows the whole heat treatment profile. The preheat objective was to prevent any distortion or cracking due to the thermal difference between the surface and the steel core[29]. Austenization at 900°C for 60 minutes was done to ensure the steel had a complete Austenite phase[30].

Table 1. Chemical composition of S45C carbon steel

Element	**Weight %**
Fe	98.3
C	0.47
Si	0.27
Mn	0.70
P	0.01
S	0.06

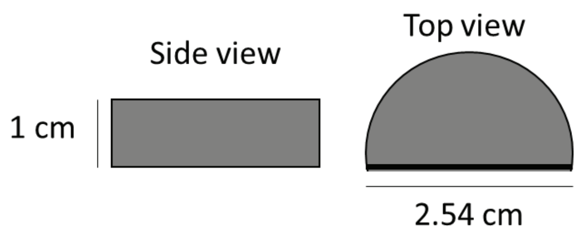

Fig. 2: Dimension of the S45C steel sample

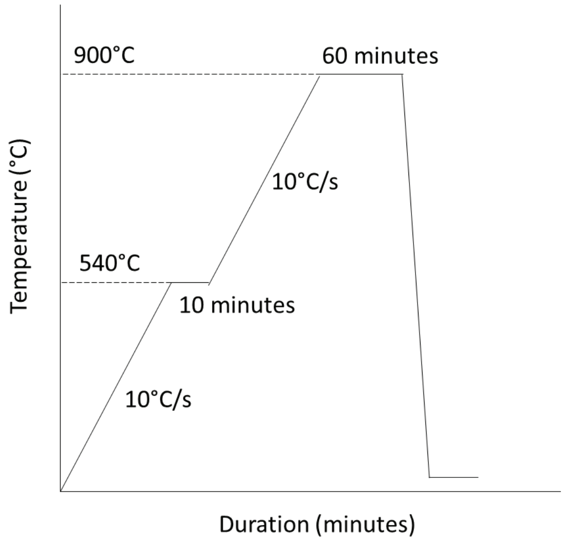

Fig. 3: Heat treatment profile

After the austenization process, the steel was quenched using the nanofluid. The quenching would transform the steel phase from Austenite to Martensite. A hardness test was conducted after quenching using a Qualirock Digital Hardness Tester from Qualitest, United States. Transformation into the Martensite phase should increase the hardness. Furthermore, microstructure observation was done to confirm the phase transformation. In this research, Olympus Inverted Metallurgical Microscope BX41M-LED from Olympus, Japan was utilized for the microstructure observation. Before the observation under the microscope, the steel sample was etched using 2% Nital solution for 3 – 10 seconds, depending on the sample.

3. Result and Discussion

3.1. Zeta Potential Measurement

From the zeta potential measurement, it seems that the addition of SDBS surfactant improves the stability of the nanofluid. Table 2 shows the zeta potential of the nanofluid with 0.5% MWCNT and SBDS at 3, 7, 10, and 30%.

Table 2. Nanofluid stability by zeta potential measurement

MWCNT (%)	SDBS (%)	Zeta Potential (mV)
0.5	3	-57.9
	7	-61.4
	10	-62.1
	30	-71.2

From the literature, a particle-dispersed liquid with a zeta potential of more than ±30 mV will have good stability[31)]. Hence, the result showed that adding only 3% of SDBS had already improved the stability of the nanofluid. The more SDBS added the zeta potential increased to -71.2 mV at 30% addition. The SDBS surfactant worked by modifying or lowering the MWCNT surface tension to better disperse in the distilled water [15)]. Therefore, agglomeration and sedimentation can be avoided. The better dispersion of MWCNT also created a much more homogeneous nanofluid and retained its thermal characteristic for a longer time.

3.2 Thermal Conductivity Measurement

The most essential thermal characteristics of the nanofluid for the heat treatment process is its thermal conductivity. Figure 4 shows the thermal conductivity test result on all variables. Without surfactant, the thermal conductivity of the MWCNT nanofluid was up to 0.63 W/mK at 0.5% MWCNT. This result was slightly increased from the distilled water, which only had 0.59 W/mK. After adding SDBS, the thermal conductivity increased to 0.68 W/mK. The higher conductivity may be because of better dispersion of the MWCNT particle in the water. Overall, adding MWCNT and SDBS can improve the thermal conductivity up to 15% higher than distilled water.

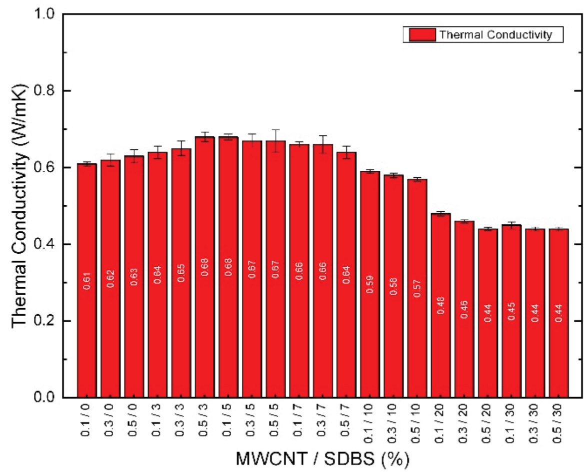

Fig. 4: Thermal conductivity measurement result

However, excessive SDBS addition seems to decrease the thermal conductivity quite significantly. The decrease was quite severe, to the point that in the 30% SDBS, the thermal conductivity was much lower than water. The viscosity of the nanofluid might be the cause of this significantly lower thermal conductivity. Several literatures mentioned that a high percentage of surfactant would increase the fluid viscosity[32–34)]. The high viscosity is not preferred in the nanofluid quench medium because it hinders the particle's Brownian motion of the particle [35)]. Brownian motion is a random motion of the dispersed particle in the fluid. The Brownian motion is crucial as it supports the heat transfer from the particle to the surrounding fluid. Reports also showed that Brownian motion contributes to the thermal conductivity enhancement in nanofluid[36,37)]. Higher Brownian motion means better heat transfer and vice versa especially in the conductive heat transfer. Therefore, it was essential to add the surfactant accordingly to improve the dispersion of the particle but not to increase the viscosity excessively.

3.3 Steel Hardness Testing

After austenization, the S45C steel was then quenched in all nanofluid variations. Without any hardening heat treatment, the hardness of the steel was only 12 HRC. For comparison, the steel was also quenched in distilled water only, and the hardness was 40 HRC. Figure 5 shows the Rockwell hardness (HRC) testing test. The trend of the hardness testing is like thermal conductivity. Figure 6 shows the relationship and trends between thermal conductivity and steel hardness. On the nanofluid with no addition of surfactant, the hardness was lower. The low hardness may be due to the low thermal conductivity of the nanofluid. Adding a small amount of SDBS increased the hardness following the increase of thermal conductivity. The maximum hardness achieved was 40HRC after quenching in nanofluid with 0.3% MWCNT and 5% SDBS. With the excessive addition of surfactant, the hardness was also decreased. The minimum hardness was 17 HRC at the nanofluid with 0.5% MWCNT and 30% SDBS. As described earlier, excessive surfactant would increase the quench medium viscosity and hinder the Brownian motion needed for the heat transfer. Hence, the cooling rate of the quench medium was lowered and resulting in a steel with low hardness.

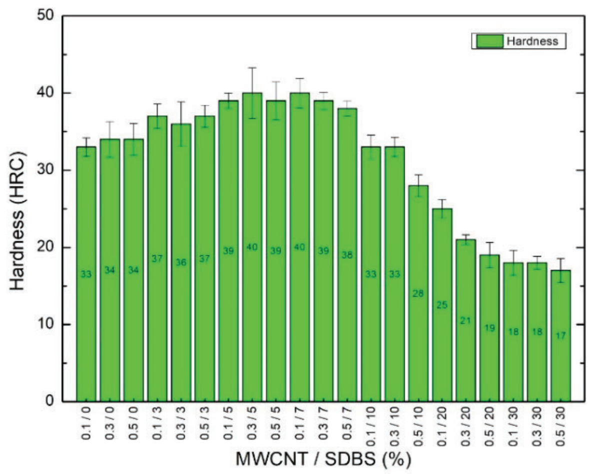

Fig. 5: S45C steel hardness testing result

As explained previously, the maximum hardness achieved by using nanofluid quench medium was not higher than quenched in distilled water. The similar hardness may suggest that the increase of 15% thermal conductivity was insufficient to increase the steel's hardness after quenching.

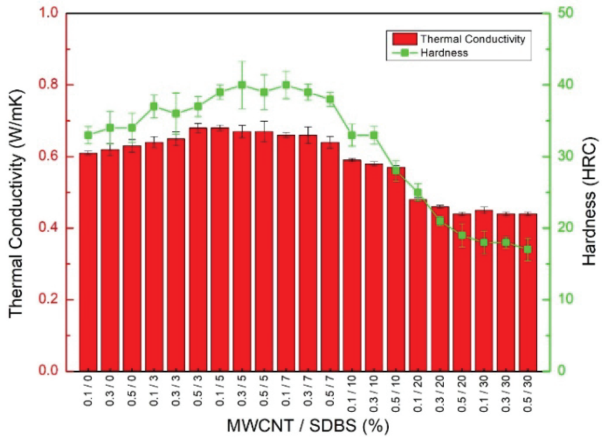

Fig. 6: Relationship between thermal conductivity and steel hardness

3.4 Microstructure Observation

Microstructure observation was done on the steel sample to support the hardness result. The microstructure of the steel without heat treatment is presented in Fig. 7a. As expected, the phases in the steel were Ferrite and Pearlite, which support the lower hardness. After quenching in the distilled water, the microstructure is shown in Fig. 7b. The phases present in this sample were Martensite and Bainite, hence the high hardness. The Martensite and Bainite phase's microstructure is similar to the report from Nishimoto [38].

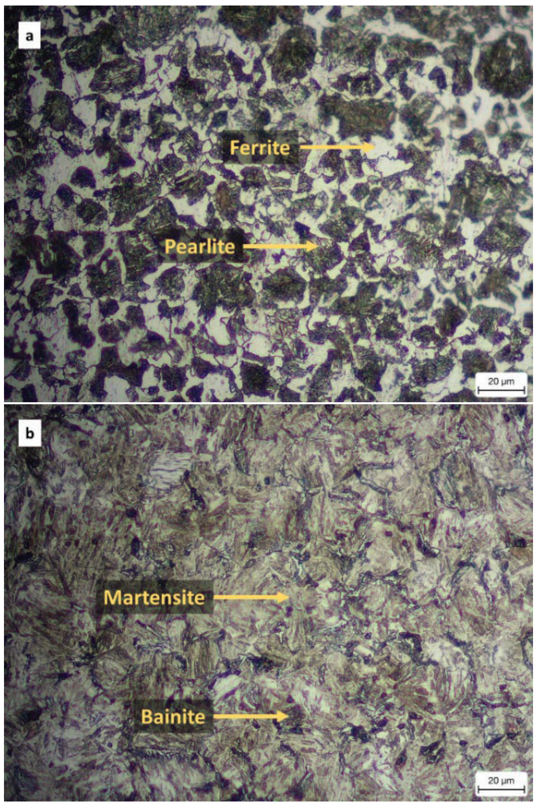

Fig. 7: Microstructure observation of S45C steel (a) before heat treatment, (b) quenched in distilled water

The microstructure of the steel sample with the lowest and highest hardness numbers after quenching in the nanofluid was also presented for comparison. Figure 8a shows the steel with the lowest hardness after quenched in nanofluid with 0.5% MWCNT and 30% SDBS. The hardness of this sample was only 17 HRC. Microstructure observation of this sample supports the low hardness as the phases were only Ferrite and Pearlite. However, due to the slightly faster cooling rate, Widmanstatten Ferrite was formed. The Widmanstatten Ferrite has a sharper grain shape which slightly increases the hardness, compared with the normal Ferrite in the steel without heat treatment. On the other hand, Figure 8b shows the sample with the highest hardness, which was quenched in the nanofluid with 0.3% MWCNT and 5% SDBS. This steel had a hardness of 40 HRC, and the phases present were Martensite and Bainite. This steel's microstructure was similar to the one quenched in distilled water. Hence, the similar hardness number.

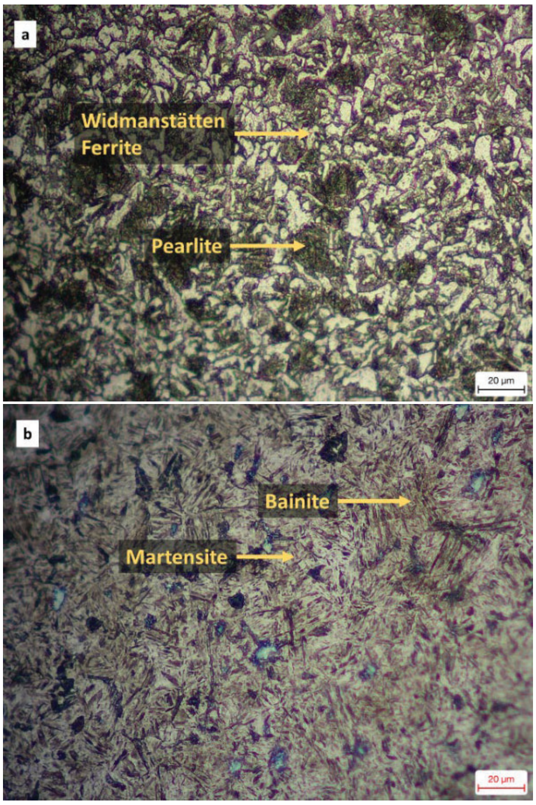

Fig. 8: Microstructure observation of S45C steel quenched in nanofluid (a) 0.5% MWCNT and 30% SDBS, (b) 0.3% MWCNT and 5% SDBS,

4. Conclusion

Adding MWCNT nanoparticles and SDBS surfactant to the nanofluid improves its thermal conductivity. Without the addition of surfactant, the thermal conductivity was increased slightly. Compared with distilled water, the thermal conductivity increased by around 15% at the optimum surfactant addition of around 3% and 5%. Excessive addition of SDBS, however, decreases the thermal conductivity. The decrease in thermal conductivity was due to the high viscosity, hindering the Brownian motion of the particle and reducing the effectivity of the heat transfer.

The trend of the steel hardness result was similar to the thermal conductivity. Without surfactant, the hardness of the steel increases slightly. At the maximum thermal conductivity, the steel hardness reached 40 HRC, the same as the one quenched with distilled water. This result suggests that the 15% increase of the thermal conductivity was ineffective enough to increase the steel hardness. The microstructure observation also supports the result where the sample was quenched in distilled water, and nanofluid had the same Martensite and Bainite phases.

This study suggests that there must be an appropriate concentration of surfactant added to the nanofluid quench medium. The added surfactant concentration must be able to improve the particle dispersion, but not too excessive for it to lower the overall thermal conductivity.

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