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# EFFECTS OF WATER/$TIO_2$ NANO-FLUID QUENCHING MEDIA ON MICROSTRUCTURE AND PROPERTIES OF CK35 STEEL

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**Abstract**

*The effects of water/TiO2 Nano-fluid quenching media on microstructure and properties of CK35 steel were analyzed by optical microscopy, X-ray diffraction, Vickers hardness, and tensile tests. The results show that the microstructure of the CK35 steel sample quenched by water/TiO2 Nano-fluid and tempered are the mixture of tempered martensite and retained austenite with best mechanical properties. Whereas the microstructure of the CK35 steel sample quenched by water and tempered is mostly tempered martensite.*

**Keywords***:* *Nano-fluid, thermal conductivity, microstructure, CK35 steel, mechanical properties.*

## Introduction

Quenching of a medium carbon steel is one of the key factors governing its application in auto industry [1,2]. The vehicle components (fasteners spindles, hydraulic rams, crankshafts, torsion bars, sockets, ratchets, worms, light gears, guide rods and dies) are made of medium carbon steels that are quenched with oil and water mediums [3,4]. Water and oil are a conventional medium for quenching process. Therefore, there is a need to study the behaviour of medium carbon steel in a new quenching medium and it is called Nano-fluid. Research on Nano-fluids includes a wide range of application on science and engineering fields. Nano-fluid is known as a colloidal suspension is a mixture of insoluble particles smaller than 100 nm suspended in the base fluid as shown in Fig. 1. There are mainly two methods to produce a nanoparticle-fluid (Nano-fluid), including (i) one-step method (ii) two-step method [5].

Two-step method is the simplest way to produce nano-fluid with mass fraction of nanoparticle is variable [6]. This involves adding the exact amounts of nanoparticles to the base fluid. Then, agitation was induced by using an ultrasonic bath or ultrasonic processor. Over the past few years, noticeable investigations have been directed toward thermal properties (Thermal Conductivity k), mechanical properties (Tensile Strength Rm and Hardness Hv) and microstructural development behaviour of CK35 steels which quenched in Nano-fluid media. Das et al., Chandrasekar and Suresh, Ozerinc et al., Fan and Wang, [7-11] investigated the effect of volume fraction, material, size and shape of nanoparticles, the material and acidity of the base fluid, temperature of Nano-fluids, sonication power and time, and additives on thermal conductivity of Nano-fluids. They showed that enhance the thermal conductivity of Nano-fluids as shown in Fig. 2.

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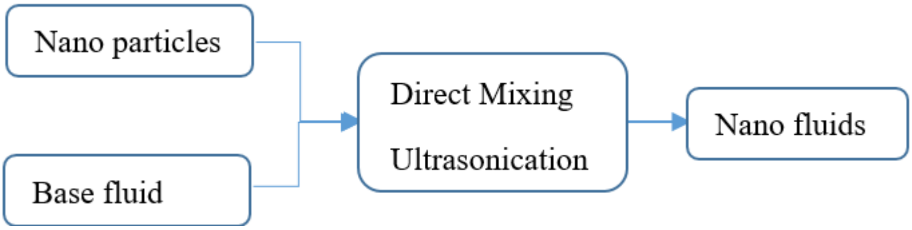


**Fig. 1.** Illustration of the Nano-fluid

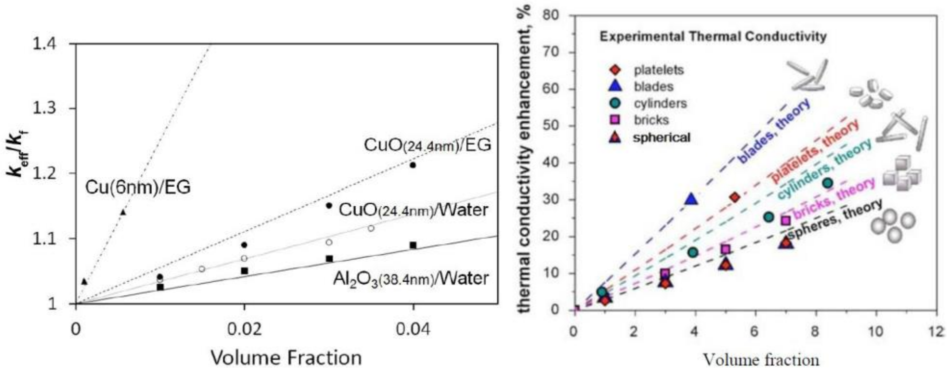


**Fig. 2.** Thermal conductivity enhancement as a function of volume fraction of particles [10]: Effect of the particle shape on the thermal conductivity of alumina Nano-fluids [11]

Joseph and Ferdinand [12] studied the effect of adding different weight percent of clay to water to form clay/water quenching media on the mechanical properties of 0.45%C steels. They observed that addition of 2-4wt% clay to water gives the best mechanical properties. Oghenevweta et al. [13] analyzed mechanical properties and microstructures of medium carbon steel. They observed that the quenchant produces predominantly lath martensite. And addition of 5wt% alumino-silicate to water gives the best mechanical properties. Joshua et al. [14]. found that microstructural of 0.26%C – 0.83% Mn steel are quenched by using water, distilled water is martensitic structures in ferrite matrix and higher level of bainite in ferrite when the steel samples quenched in palm kernel oil.

## Thermal Conductivity of Nano-Fluids

Heat transfer is defined as the thermal energy transfer due to a temperature difference. There are three modes of heat transfer, conduction, convection and radiation. Heat is not transferred by a single mode in nature, but one mode may be dominant enough that the others can be neglected. Thermal conductivity λ with the unit of [$Wm^{-1}K^{-1}$], is a measure of the capability of a medium to conduct heat and a property depending on the material and the temperature [15].

Detecting or describing the heat transfer mode of Nano-fluids can be very difficult because of the discrepancies in properties between different groups and the thermal conductivity degradation over time. Where Nano-fluids categorized as heterogeneous fluids, due to aggregation and sedimentation of particles. The discrepancies in data is because of different measurement method which is not yet standardised and different stability or dispersion status [16]. For example, Maxwell [17] proposed the model eq. 1 to estimate thermal conductivity of Nano-fluids, but this model is used when the particle volume fraction is very small. The transient hot-wire apparatus has been used most frequently to measure the thermal conductivity of Nano-fluids [18], but this apparatus was setup at Cambridge university 2014. In this work, measurement of thermal conductivity was done by theoretical (Maxwell mode) and experimental by KD2 Pro Thermal Properties Analyzer.

$$\frac{K_{eff}}{K_F} = 1+\frac{3\emptyset(\frac{K_p}{K_f}-1)}{\frac{K_p}{K_f}+2-\emptyset(\frac{K_p}{K_f}-1)};\ \emptyset = \frac{\frac{m_p}{p_p}}{\frac{m_p}{p_p}+\frac{m_f}{p_f}} \tag{1}$$

Where:

$K_{eff}$ : Thermal conductivity of the mixture
$K_f$ : Thermal conductivity of the base fluid
$K_{eff/kf}$ :Thermal conductivity enhancement of the mixture
ϕ: Particle volume fraction
$K_p$: Thermal conductivity of the particle
m: mass of the particle or fluid.

## Experimental Work

### *Material*

The steel used in this study was 6-mm in diameter of CK35 rod steel whose chemical composition was (0.32% C, 0.30% Si, 0.66% Mn, 0.015% P, 0.014% S). The mechanical properties of the base metals were determined using a standard tensile test in accordance to ASTM E8M [19]. The yield stress, Tensile stress, and Total strain resulted 400 MPa, 570 MPa and 31% respectively. Information on Nano particles, base fluids that were used as quenching media is summarized in Table 1.

**Table 1.** Properties and Specification of Titanium Dioxide and water at 20 °C

| Material | Type | Specification | K(w/m·k) | | Mean size | Color | Purity | Morphology |
|---|---|---|---|---|---|---|---|---|
| | | | Ref [] | EXP | | | | |
| Titanium Dioxide $TiO_2$ | Powder | 689 Hongwu Nanometer | 8.4 [20] | 7.9 | 50nm | White | 99.8% | Spherical |
| Distilled water | Fluid | | 0.5984 [21] | 0.5977 | | | | |

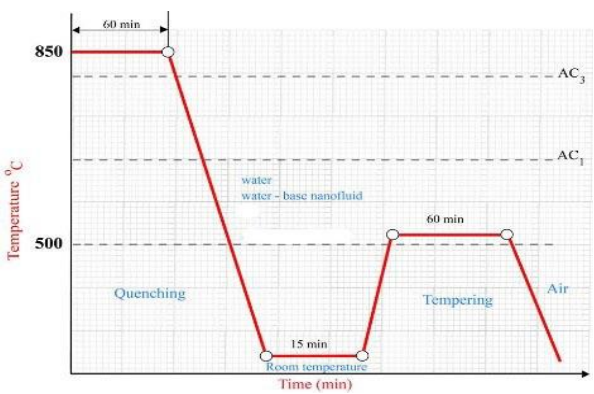


**Fig. 3.** Schematic illustration of heat treatment cycle used in this study.

The quenching and tempering which are used in this work is shown in Fig. 3. All of the specimens were heated to 850 ºC above Ac3 (austenite zone) [21]. All the specimens were hold for 60 min in muffely furnace and followed by various quenchants (water and water-base Nano-fluid). All of the quenched specimens were heated again to be tempered at 500 ºC below Ac1 for 60 min then air- cooled.

***Equipment***

The samples for the tensile test were prepared according to ASTM E8M standard. The tensile tests were carried out on as-received and quenched specimens of (6mm) diameter and a gage length of (30mm) at a strain rate of 5 mm min-1 by using an Instron testing machine at room temperature. Metallographic examination was prepared using the standard procedure from the base metal and quenched specimens and etched with (2%) nital solution. Microscopic examinations were carried out by Olympus optical microscope (BH2-Japan). Vickers micro-hardness test was performed at three different points to obtain an average hardness result by Shimadzu 341-64278 (Japan) with 300gf, time 15 sec. The structural transformation that takes place in the samples which have best mechanical properties after heat treatment is studied by using X-ray diffraction (XRD) (Philips Company).

**Table 2.** Thermal conductivity results for Quenching Media

| Quenching Media | Thermal Conductivity enhancement ($K_{eff/kf}$) of the mixture and Thermal Conductivity of water | | |
|---|---|---|---|
| | Ref | Theo (eq 1) | EXP |
| Water | K=0.5984 [22] | - | K=0.5977 |
| Nano fluid (water/$TiO_2$) 0.3 wt.% | - | $K_{eff/kf}$=1.08 | 1.26 |

Nano-fluid production

The Nano-fluid was produced in 2 step method, meaning nanoparticles of Titanium Dioxide ($TiO_2$) were added to the water bases fluid. To homogenize the Nano-fluid ultrasonic bath were used. sonication time of 60 min was selected in this study. The result of thermal Conductivity enhancement ($K_{eff/kf}$) of the mixture and Thermal Conductivity of water is shown in Table 2.

## Result and Discussion

***Tensile properties***

Fig. 4 shows a number of engineering stress-strain curves for the same CK35 steel at different quenching media and base metal as-received. As can be seen, the yield stress increases from 400 MPa (base metal) to 820 (WQT and NWQT) and tensile stress increases from 570 MPa (base metal) to 900 (WQT) and to 1150 (NWQT), depending on the quenching media. Conversely, the total strain decreases from 0.31% (base metal) to 0.15% (WQT) then increases to 0.19 % (NWQT). The properties of steel are highly dependent upon quenching produces a hard, martensitic and retained austenite structure, which is gradually softened by tempering treatments at higher temperatures (500 ◦C).

***Microstructural investigations***

XRD test of NWQT sample as shown in Fig. 5 and result of hardness in Table 3 are used to predict the microstructure after a heat treatment process. According to XRD test, the microstructure is mainly tempered martensite and retained austenite with hardness 470HV as shown in Fig. 6.

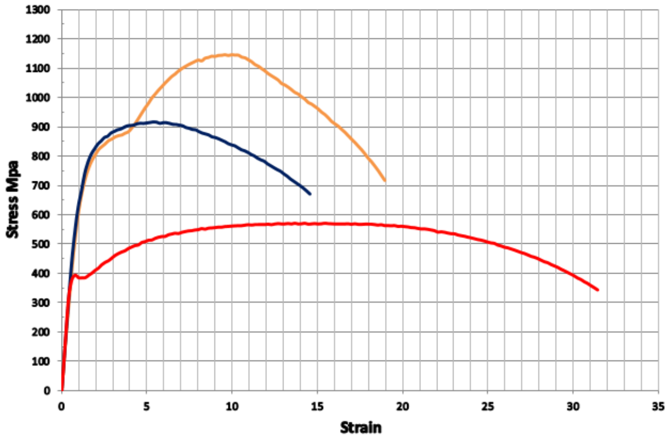


**Fig. 4.** Engineering stress–strain curves for CK35 at different quenching media.

The presence of (110) and (211) planes are clearly revealed and indicate the formation of martensite phase during heat treatment process. Additionally, the (200) plane is present characterizing the retained austenite. Similar conclusions were obtained by the other investigators [23,24]. The analytical method [25] is used for determining the lattice planes of NWQT sample. these planes are listed in Table 4. In the water quenching media (WQT) the result of microstructure consists of martensite phase which is responsible for the high value of measured hardness (550HV) as shown in Fig. 7.

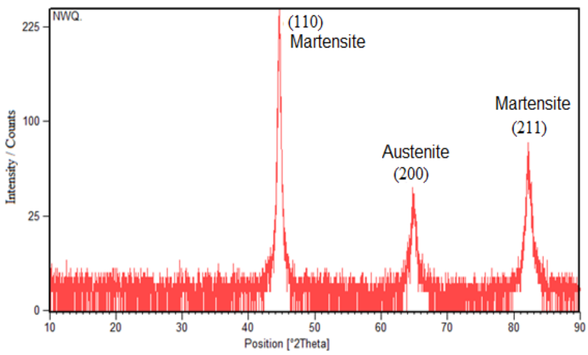


**Fig. 5.** X-ray diffraction profiles of CK53 after quenching at nano-fluid water and tempering.

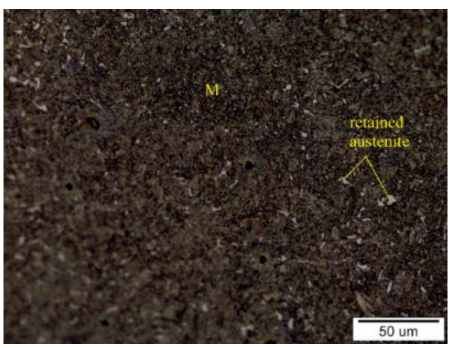


**Fig. 6.** Optical micrograph of microstructure of CK35 after quenching at nano-fluid water and tempering.

**Table 3.** Micro Vickers hardness data of CK35 after quenching and tempering

| BM | NWQT | WQT |
|---|---|---|
| 187 HV | 470 HV | 550 HV |

**Table 4.** Crystallite planes and phases of the CK35 after quenching and tempring

| 2 Theta | $h^2+k^2+i^2$ | Hki | Phase [24,25] |
|---|---|---|---|
| 44.8 | 2 | 110 | Martensite |
| 65 | 4 | 200 | Austenite or martensite |
| 82.3 | 6 | 211 | Martensite |

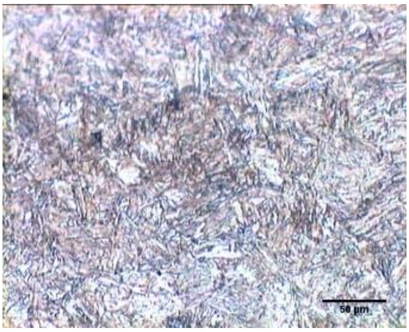


**Fig. 7.** Optical micrograph of microstructure of CK35 after quenching at water and tempering

***Failure analysis***

It is found that the ductile fracture showing the typical cup-cone geometry as shown in Fig. 8 take place at steel quenched by nano-fluid water media. This fracture mode begins in the center of the testpiece with microvoid (dimples) nucleation along grain boundaries or from interfaces [26] as shown in Fig. 9. The SEM examination Fig. 10 showed ductile fracture characterized by equiaxed dimples, a common feature of ductile tensile overload. Also Fig. 10 showed brittle fracture characterized by microvoid coalescence and quasi-cleavage, a common feature of brittle tensile.

Fig. 11 presents in detail some ratchet marks found on the fracture surface of a CK35 steel shaft, fractured by tensile test. Ratcheting marks are another macroscopic feature that can be observed in fatigue fracture surfaces of CK45 steel [27]. These marks originate when multiple cracks, nucleated at different points, join together, creating steps on the fracture surface. Therefore, counting the number of ratchet marks is a good indicator of the number of nucleation sites.

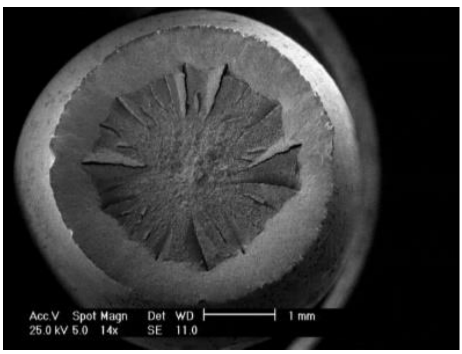


**Fig. 8.** Ductile fracture showing the typical cup-cone geometry at CK35 steel after quenching and tempering

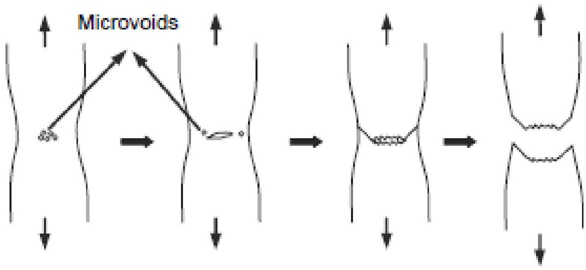


**Fig. 9.** Schematic representation of the cup-cone geometry formation during the ductile fracture process

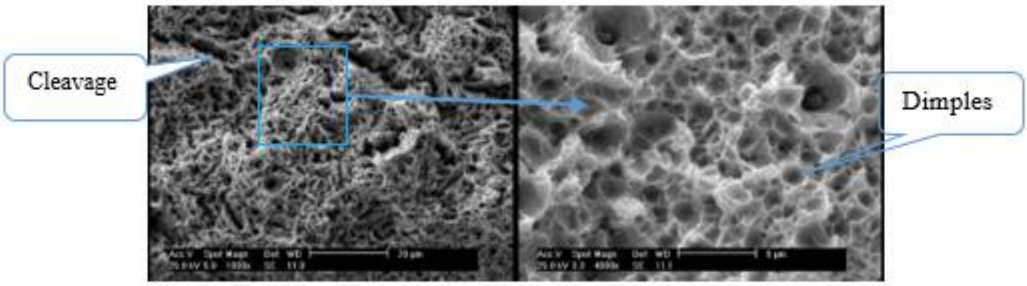


**Fig. 10.** Microvoids on the fracture surface of CK35 tensile test piece

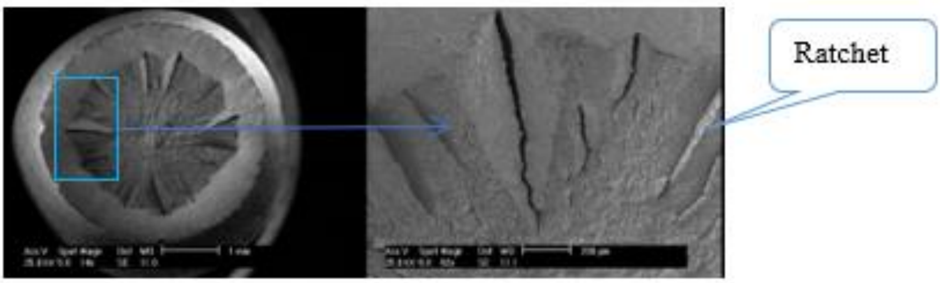


**Fig. 11.** Ratcheting marks, indicated by the arrows, in an CK35 shaft fractured by tensile

## Conclusion

The (water/TiO2) Nano-fluid quenched specimen of CK35 has the microstructure of tempred martensite and retained austenite with (470) HV. The water-quenched specimen of CK35 has the microstructure of martensite with (550) HV.

Based on the data of mechanical properties (peak load and total elongation), it's found that the desired final microstructure of CK35 steel is tempered martensite and retained austenite, and it could be obtained by the tempering of (water/$TiO_2$) nano-fluid quenched specimen with thermal conductivity enhancement ($K_{eff/kf}$) eq. 1.26.

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