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Microstructure and Mechanical Properties of CK35 Steel by Using Nano Fluid (Water/TiO_2) and Oil (SAE 10W40/TiO_2) as Quenching Media

Hasenen L. Jaber
University of Thi-Qar/College of Engineering/Mechanical Eng. Dept.

Abstract

Four different quenching media (water, oil(SAE 10W40), Water/TiO_2 nanofluid and oil(SAE 10W40/TiO_2) nanofluid were used to compare the influence of quenching media on the mechanical properties and microstructure of CK35 steel. The results have proved that the microstructure for nanofluid (water base) quenching and tempering sample is the combination of tempered martensite and retained austenite with best mechanical properties and for water quenching and tempering specimen is mostly tempered martensite. While for Nanofluid (oil base) quenching and tempering specimen the microstructure is the heterogeneous mixture of ferrite and perlite with formation of Fe_3C during the quenching, while for oil quenching and tempering specimen is an equi-axed arrangement of ferrite grains with grainy spheroidized spots of cementite at the ferrite grains and along the grain boundaries.

Keywords: Nano-fluid, Quenching heat treatment, Medium carbon steel, Microstructure, Mechanical properties, Thermal conductivity.

1- Introduction

Quenching of a medium carbon steel is one of the key factors governing its application in automobile 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 behavior of medium carbon steel in a new quenching medium, namely Nanofluid.** Investigation on nanofluids contains a wide range of application on engineering and science fields.

Nanofluid is known as a colloidal suspension is a combination of insoluble particles lesser than 100 μm suspended on the base fluid as shown in Fig. 1. There are mainly two methods to produce a nanoparticle-fluid (nanofluid), including (i) one-step method (ii) two-step method [5].

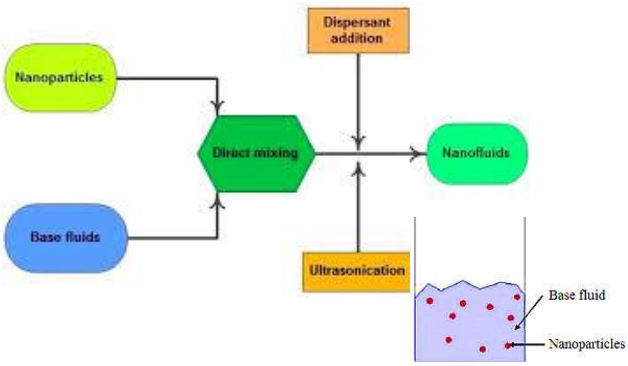
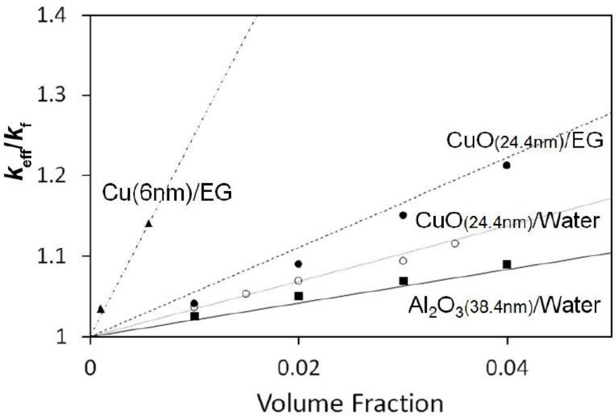


Fig. 1: Illustration of the nanofluid [6]

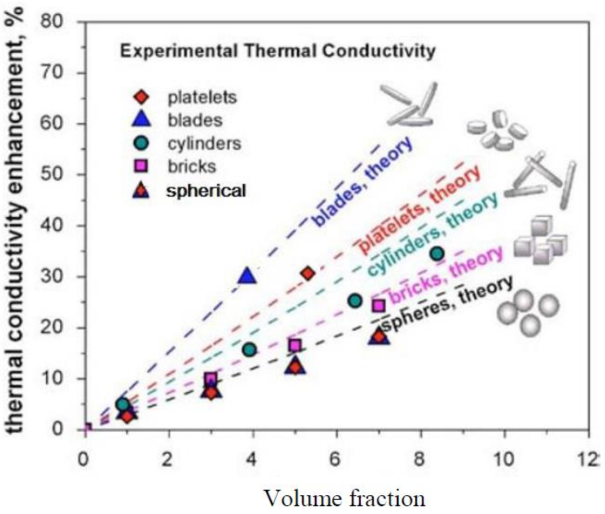
Two-step method is the simplest way to produce nanofluid with mass fraction of nanoparticle is variable [6]. This involves adding the exact amounts of nanoparticles to the base fluid. Then, agitation was made by using an ultrasonic probe or ultrasonic path.

Over the previous few years, noticeable researches have been focused in the direction of thermal properties (Thermal Conductivity K), mechanical properties (Tensile Strength and Hardness) and microstructural development behavior of medium carbon steels which quenched in nanofluid media.

Das et al., Suresh and Chandrasekar, Ozerinc et al., Wang and Fan, [7-11] investigated the effect of material, size, shape and volume fraction of nanoparticles, the material of the base fluid, sonication power and time and temperature of nanofluids. They showed that, enhancement the thermal conductivity ($K_{eff/kf}$) of Nano fluids such as shown in Fig. 2.



(A)



(B)

Fig. 2. A: Thermal conductivity (Keff/kf) enhancement as a function of volume fraction of particles [10]. B: Influence of the particle shape in the Thermal conductivity (Keff/kf) enhancement of Al_2O_3

Joseph and Ferdinand [12] studied the effect of adding different weight percentage of clay to water to procedure water/clay quenching media on the mechanical properties of 0.45%C steels. They observed that adding of 2-4wt% clay to water provides the top 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 adding of 5wt% Al_2O_3-silicate to water provides the top mechanical properties. Joshua et al. [14] found that microstructural of 0.26%C –0.83% Mn steel are quenched via water and distilled water is martensitic structures in ferrite matrix and higher level of bainite in ferrite when the steel samples quenched in palm kernel oil.

1.1 Nano-fluids Thermal conductivity.

Heat transfer is defined as the thermal energy transfer because of a temperature difference. There are three modes of heat transfer, namely convection, conduction, and radiation as shown in Fig. 3. Heat is not transferred by a one mode in nature, but single mode may be main enough that the others can be ignored. Thermal conductivity (K) with the unit of [W/m K], is a degree of the ability of a media to conduct heat and a property depending on the temperature and the material [15].

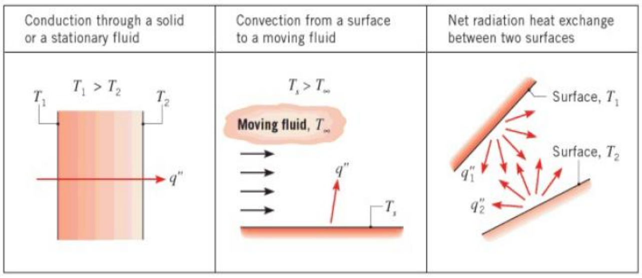


Fig. 3. Conduction, convection and radiation heat transfer modes [15].

Detecting or describing the heat transfer mode of nanofluids can be very difficult because of the dissimilar in properties between different groups and the thermal conductivity decreasing with time. Where nanofluids classified as heterogeneous fluids, because of agglomeration and sedimentation of nanoparticles. The differences in data is due to different measurement technique which is not yet standardized and diverse stability or dispersal status [16].

For pattern, Maxwell [17] proposed the model (eq 1 and 2) to estimate thermal conductivity of nanofluids, but this model is used when the volume fraction of particle is very small. The transient hot wire apparatus has been used much to measure the nano-fluids thermal conductivity [18], but this apparatus was setup at Camprage university 2014. In this work, measurement of thermal conductivity was done by theoretical (Maxwell mode) and experimental by KD2 Pro Thermal Properties Analyzer.

$$\frac{k_{eff}}{k_f} = 1 + \frac{3\phi\left(\frac{k_p}{k_f} - 1\right)}{\frac{k_p}{k_f} + 2 - \phi\left(\frac{k_p}{k_f} - 1\right)} \quad \text{-------------------------} \quad (1)$$

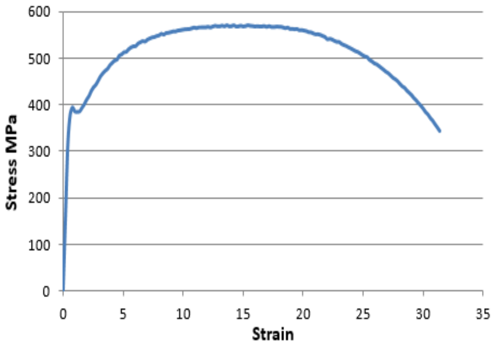
- K_{eff} : Thermal conductivity of the mixture.
- K_f : Thermal conductivity of the base fluid
- $K_{eff/kf}$:The thermal conductivity enhancement of the mixture.
- K_p : Thermal conductivity of the particle
- **ϕ** : Particle volume fraction.

$$\phi_p = \frac{\frac{m_p}{\rho_p}}{\frac{m_p}{\rho_p} + \frac{m_f}{\rho_f}} \quad \text{----------------------} \quad (2)$$

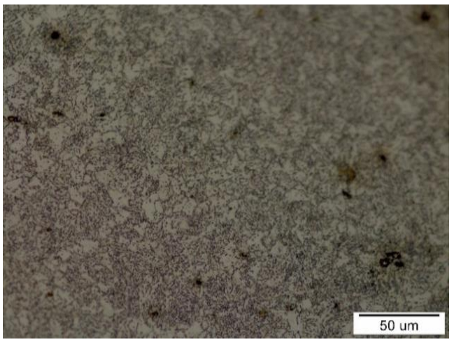
- m : mass of the particle or fluid

2-Experimental work

2.1 Material



(a)



(b)

Fig. 4. (a) Curve of Engineering stress–strain and (b) Base metal microstructure of CK35 sample.

Table 2. Properties and Specification of Titanium Dioxide, water and SAE 10W-40 oil at 20 °C.

Material	Type	Specification	Manufacturer	K (w/m . k) Ref[ ]		EXP	mean size	Color	Purity	Morphology
Titanium Dioxide TiO_2	powder	689 Hongwu Nanometer	China	8.4 [20]		7.9	50 nm	white	99.8 %	Spherical
Distilled Water	fluid			0.5984 [21]		0.5977				
Oil	fluid	SAE 10W-40	USA	0.12 [22]		0.11				

The steel used in this study was 6-mm in diameter of CK35 rod steel whose chemical composition is given in Table 1. Fig. 4.a shows the curve of engineering stress–strain and microstructure of base metal. The microstructure of the base metal sample consists of ferritic microstructure with perlite can be seen in the base metal microstructure Fig. 4.b. Nano particles and base fluids information that were used as quenching media is summarized on Table 2.

Table 1. Chemical composition of the CK35 steel (wt%).

Element	C	Si	Mn	P	S
Composition	0.32	0.30	0.66	0.015	0.014
Ref [19]	0.031-0.038	0.40	0.60-0.90	0.040	0.050

The quenching and tempering procedure used in this study is shown in Fig. 5. All specimens were heated to 850C° above AC3 (austenite zone) [21], and hold for 60 min in the muffely furnace then followed by various quenchants (water, water-base nanofluid, oil, oil-base nanofluid). All of the quenched specimens were heated again to be tempered at 500C° below AC3 for 60 min then air- cooled.

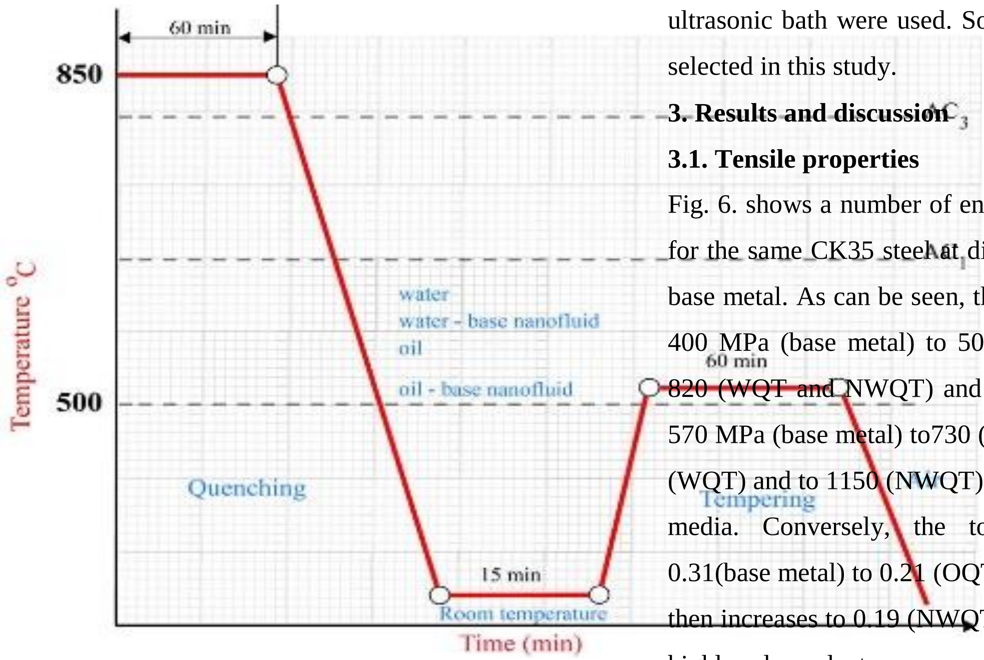


Fig. 5. Diagram illustration cycle of heat treatment used in this work.

2.2 Equipment

The samples for the tensile test were prepared according to ASTM E8M standard. The tensile tests were done on as-received and quenched specimens of (6mm) diameter and a gage length of (30mm) at a strain rate of 5 mm/min 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 nital solution (2 mL HNO_3, 98mL C_2H_5OH) were used for etching metallography sample of CK35 steel.

Microscopic aQAaainspections were done by Olympus optical microscope(BH2-Japan). Vickers micro-hardness test was performed at three different points to get an average result of hardness 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 (Philips Company).

2.3 Nanofluid production

The nanofluid was produced in 2 step method, meaning nanoparticles of Titanium Dioxide (TiO_2) were added to the oil and water bases fluid. To homogenize the nanofluid ultrasonic bath were used. Sonication time of 60 min was selected in this study.

3. Results and discussion

3.1. Tensile properties

Fig. 6. shows a number of engineering stress-strain curves for the same CK35 steel at different quenching media and base metal. As can be seen, the yield stress increases from 400 MPa (base metal) to 500 (OQT) to 600 (NOQT) to 820 (WQT and NWQT) and tensile stress increases from 570 MPa (base metal) to730 (OQT) to 800 (NOQT) to 900 (WQT) and to 1150 (NWQT), depending on the quenching media. Conversely, the total strain decreases from 0.31(base metal) to 0.21 (OQT and NOQT) 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). As mentioned above, (NWQT) was the most important quenching media for get best mechanical properties of peak load and total elongation of the CK35 steels.

The thermal conductivity enhancement for four different quenching media were observed by theoretical (Maxwell mode) and experimental by KD2 Pro Thermal Properties Analyzer as shown in Table 3. The thermal conductivity enhancement of (Water/TiO2) Nano-fluid was 1.26 $[Wm^{-1}K^{-1}]$ with best mechanical properties compared with other quenching media.

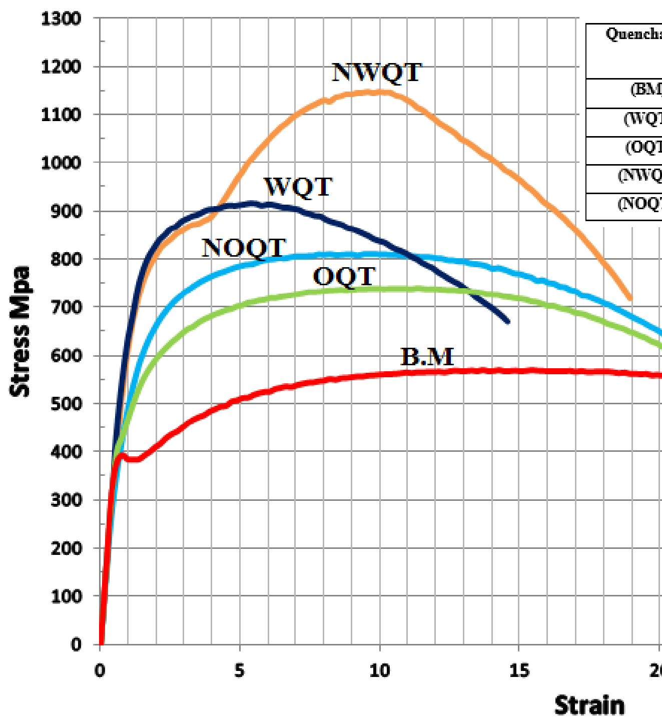


Fig. 6. Engineering stress–strain curves for CK35 at different quenching media.

Table 3. Thermal conductivity enhancement results for Quenching Media.

Quenching Media	Thermal Conductivity ($K_{eff/kf}$) enhancement of the mixture and Thermal Conductivity of water and oil		
	Ref	Theo (eq 1)	EXP
Water	K=0.5984 [22]	-	K=0.5977
Oil (SAE 10W40)	K=0.12 [23]	-	K=0.11
Nano Fluid (Water/TiO_2) 0.3 wt.%	-	$K_{eff/kf}$ =1.08	1.26
Nano Fluid (SAE 10W40/TiO_2) 0.3 wt.%	-	$K_{eff/kf}$ =0.143	0.152

3.2. Microstructural investigation

Quenchants	yield stress MPa	Tensile stress MPa	Total strain
(BM)	400	570	31
(WQT)	800	910	15
(OQT)	600	730	21
(NWQT)	870	1150	19
(NOQT)	700	800	[illegible]

XRD test of NWQT sample as shown in Fig. 7. and result of hardness in Table 4. are used to predict the microstructure after a heat treatment process. According to XRD test, the microstructure of NWQT is mainly tempered martensite and retained austenite with hardness 470 HV see Fig. 8.

The existence of (211) and (110) planes are clearly detect and show 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 [24, 25]. The analytical method [26] is used for determining the lattice planes of NWQT sample. These planes are listed in Table 5. In the water quenching media (WQT) the result of microstructure contains of martensite phase which is responsible for the high value of measured hardness (550 HV) as shown in Fig. 9.

The microstructures of CK35 steel quenched by oil nano-fluid are illustrated in Fig. 10. The steel microstructures compose of heterogeneous mixture of ferrite and perlite with formation of Fe_3C during the quench. At the lower cooling rate of (OQT) the result is an equi-axed group of ferrite grains with grainy spheroidized particles of Fe_3C at the ferrite grains and along the grain boundaries with hardness 225 HVas shown in Fig. 11.

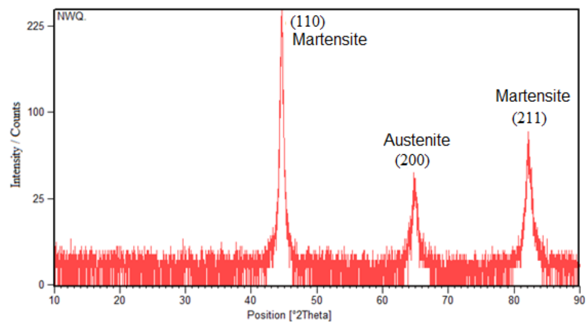


Fig. 7. X-ray diffraction profiles of CK53 after quenching at nanofluid water and tempering.

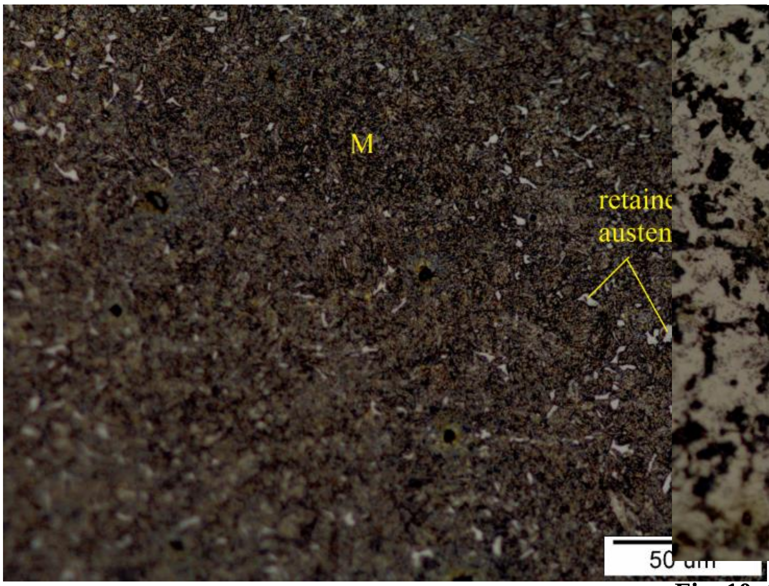


Fig. 8. Optical micrograph of microstructure of CK35 after quenching at nanofluid water and tempering.

Table 4. Micro Vickers Hardness data of CK35 after quenching and tempering.,

BM	187 HV
NOQT	241 HV
OQT	225 HV
NWQT	470 HV
WQT	550 HV

Table 5. Crystalline planes and phases of the CK35 after quenching and tempring.

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

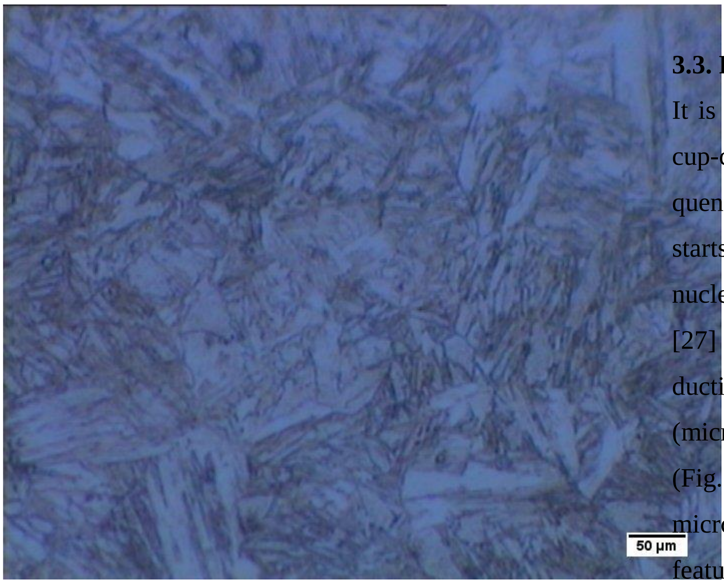


Fig. 9. Optical micrograph of microstructure of CK35 after quenching at water and tempering.

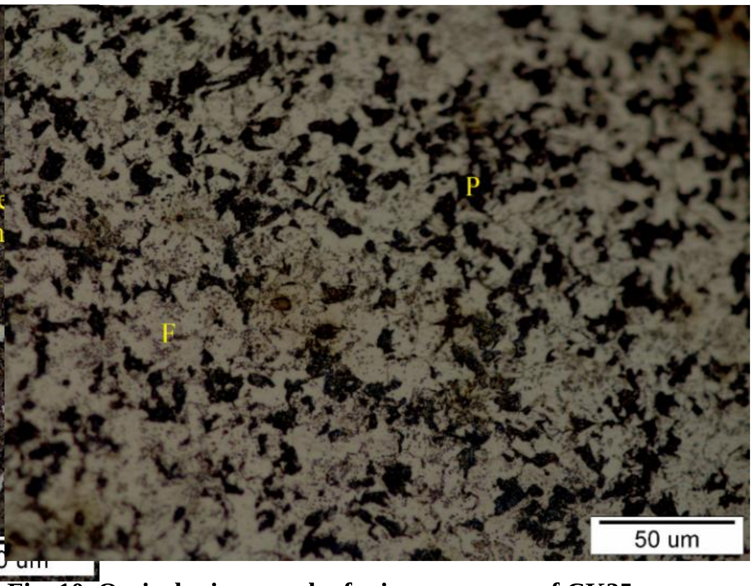


Fig. 10. Optical micrograph of microstructure of CK35 after quenching at oil nanofluid and tempering.

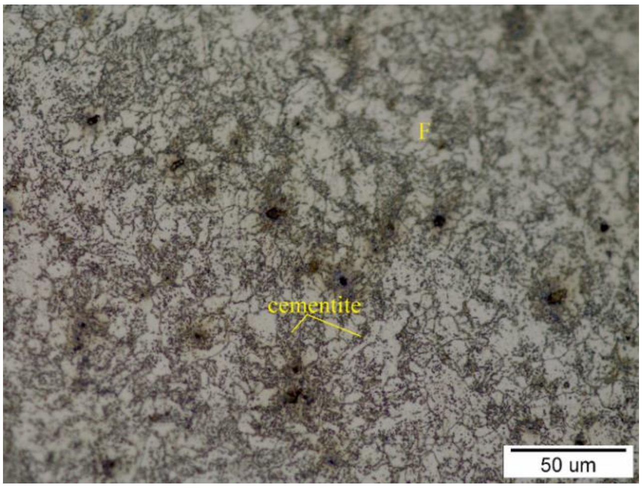


Fig. 11. Optical micrograph of microstructure of CK35 after quenching at oil and tempering.

3.3. Failure Analysis

It is found that the ductile fracture displaying the typical cup-cone geometry as shown in Fig. 12 take place at steel quenched by nanofluid water media. This fracture mode starts in the center of the sample with dimples (microvoid) nucleation along boundaries of grain or from interfaces [27] see Fig.13. The SEM examination (Fig. 14) showed ductile fracture described by equiaxed dimples (microvoid), a common feature of ductile tensile. Also (Fig. 14) showed brittle fracture characterized by microvoid coalescence and quasi-cleavage, a common feature of brittle tensile.

Fig. 15 explains in detail approximately ratchet marks create in the fracture surface of a CK35 steel, ruptured by tensile test. Ratcheting marks are another macroscopic feature that can be detected in fracture surfaces of fatigue of AISI 1045 steel [28]. These marks create when many cracks, nucleated at different points, join together, creating steps on the fracture surface. Therefore, calculation the number of ratchet marks is a good pointer of the number of nucleation sites.

The fracture surface (Fig. 16) of a CK35 steel quenched by water media have two distinct zones. Zone 1 (dark region) is roughest area on the fracture surface and characterized by dimples as shown in Fig. 17. Zone 2 is characterized by A light ring is visible around the outside circumference. In the WQ and NOQ media the fracture surface showing the typical cup-cone geometry as shown in Fig. 18.

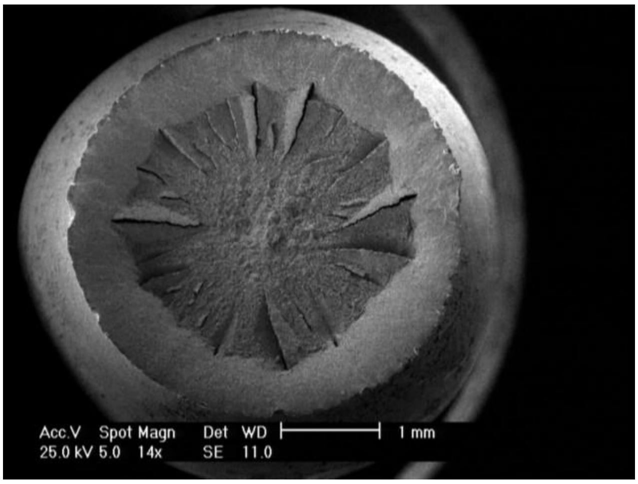


Fig. 12. Cup-cone geometry of ductile fracture at CK35 steel after quenching and tempering.

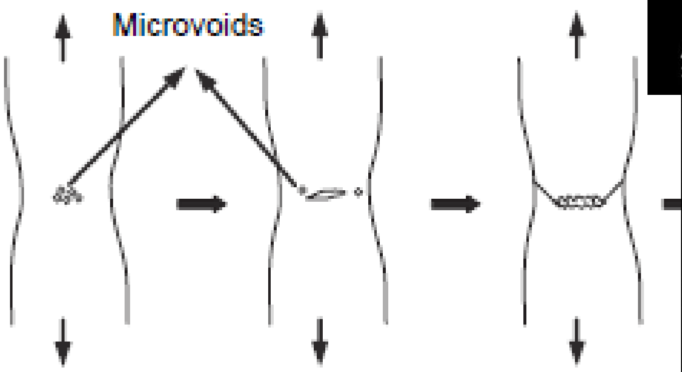


Fig. 13. Graphic illustration of the cup-cone geometry creation through the ductile fracture process [21].

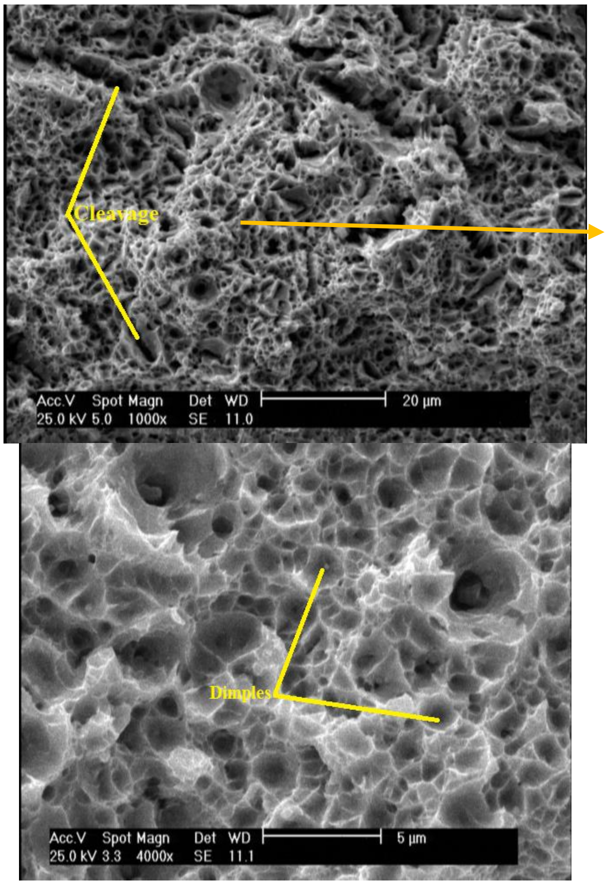


Fig. 14. Dimples (microvoid) in the fracture surface of CK35 tensile test piece.

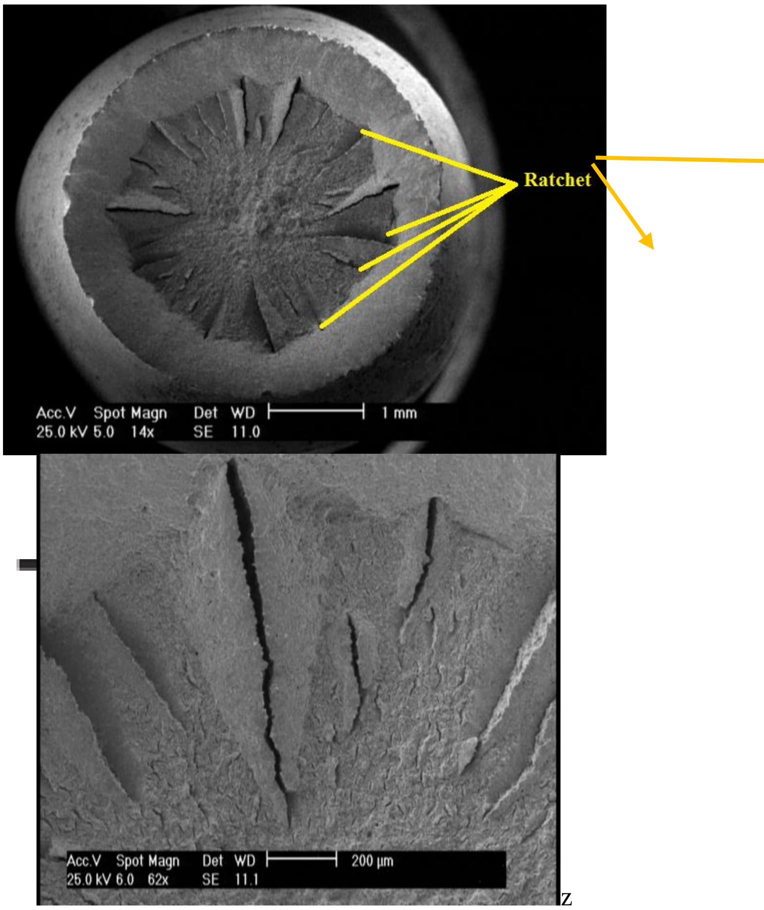


Fig. 15: Ratcheting marks observed during tensile of CK35 steel ruptured by tensile test.

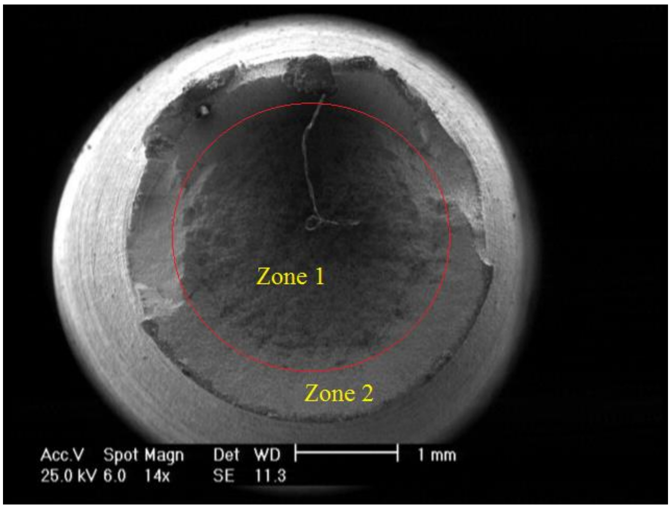


Fig. 16: fracture surface at CK35 steel after quenching at water and tempering at 500°C.

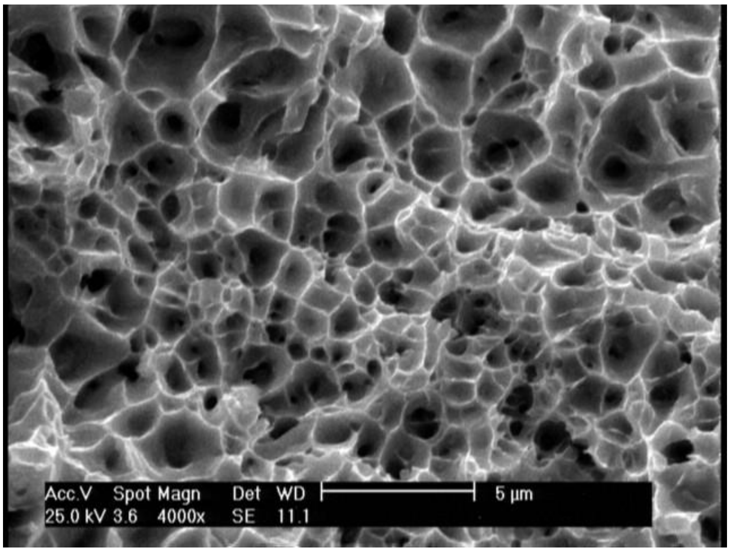


Fig. 17: Dimples (microvoid) on the fracture surface of CK35 steel tensile test piece.

Fig. 18. fracture surface at CK35 steel after quenching at oil and nanofluid oil and tempering at 500°C.

4. Conclusions

1- The (water/TiO_2) nanofluid quenched specimen of CK35 has the microstructure of tempred martensite and retained austenite with (470) HV. The (oil/TiO_2) nanofluid quenched specimen of CK35 has the microstructure of ferrite and a perlite with formation Fe_3C during quenching.

2- The water quenched and tempred specimen of CK35 has the microstructure of martensite with (550) HV. The oil quenched and tempered specimen of CK35 has the microstructure of equi-axed group of ferrite grains with grainy spheroidized particles of Fe_3C at the ferrite grains and along the grain boundaries with hardness 225HV.

3- Based on the data of mechanical properties (peak load and total elongation), it is found that the favorite final microstructure of CK35 steel is tempered martensite and retained austenite, and it could be achieved by the tempering of (water/TiO_2) nanofluid quenched specimen with thermal conductivity enhancement (K_{eff}/k_f) equal (1.26).

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Abbreviations

NWQT : Nanofluid (water base) quenching and tempering.
WQT : Water quenching and tempering.
NOQT : Nanofluid (oil base) quenching and tempering.
OQT : Oil quenching and tempering.