

PROTECTION OF CARBON STEEL FROM WEAR BY QUENCHING WITH NANOTECHNOLOGY TO USE IT IN DIES PARTS

Khalid Abdulhusein Hafedh ¹, Zaman Khalil Ibrahim ¹, Ahmed Ibrahim Razooqi ², Malik M. Mohammed ³, Hayder A. Alalwan ^{1,4,5,*}

¹ Middle Technical University /Technical Institute-Kut, Kut, Iraq

² Middle Technical University /Technical Engineering College, Baghdad, Iraq

³ Al-Mustaqbal University College/ Chemical Engineering and Petroleum Industries Department, Babel, Iraq

⁴ Kut University Collage, Al Kut, Wasit, Iraq, 52001

⁵ Al-Turath University College, Baghdad, Iraq

* hayder.alalwan@mtu.edu.iq

Adding nanomaterials to quenching media is an innovative method to alter alloys' mechanical properties and enhance their resistance to wear. In this work, TiO₂ nanoparticles were added to the oil as a quenching media in different mass percentages (0, 0.2, 0.4, and 0.6%) to identify the impact of adding TiO₂ nanoparticles on wear resistance (dry sliding) and mechanical properties by microstructure analysis of carbon steel CK45 before and after the addition of nanofluid. The results reveal the critical role of adding nanomaterials in altering steel's mechanical properties and increasing its resistance to wear. Specifically, increasing the TiO₂ nanoparticle percentage improves the wear resistance value under different loads (20, 30, and 40 N). Also, the homogenous microstructure and mechanical properties were enhanced after using nanotechnology. That means the nanotechnology can protect CK45 from wear to be used for dies structure parts by quenching process.

Keywords: TiO₂, nanoparticles, CK45, sliding speed, nanofluid

1 INTRODUCTION

Quenching is a standard heat treatment process that is used to control the mechanical properties of solid materials. Specifically, hardness, ductility, impact resistance, and yield tensile strength can be modified by this process [1]. Also, electrical and thermal conductivity and corrosion tendency can be slightly changed by the quenching process [2]. Quenching treatment involves controlling the heating and cooling rate of the metal to achieve the desired properties. Altering the properties of steel, an alloy of iron and carbon, with some other elements present in small percentages, has attracted much attention due to its use in numerous applications [3]. CK45 is an essential and widely used type of steel used in structural and machine parts like dies, tools, gears, rods, axles, and shafts to cast light metals, and it still attracts high development efforts to fulfill the industry requirements. One of these efforts is to enhance its hardness and wear resistance by adding nanomaterials through the quenching treatment process.

Nanomaterials with a high surface area can tailor the physical, thermal, and mechanical properties of steel and improve its wear and corrosion resistance [4, 5]. In addition, adding nanoparticles to the quenching media leads to better cooling capabilities than conventional quenching methods [6]. The heat transfer properties of the quenching medium play an essential role in controlling the mechanical properties of steel alloy. Recent investigations on nanofluid quenching media have shown that these media offer better wetting and heat transfer characteristics [7]. Furthermore, the wear resistance of steels is strongly connected to their micro-structure properties got after heat-treatments that are generally performed to achieve the best mechanical properties [8]. Thus, the heat-treatment process has an important impact on wear resistance. For this reason, in this work, the impact of adding different amounts of titanium dioxides (TiO₂) to the quenching media of CK45 steel on its hardness as well as the optimum amount of TiO₂ that should be added to the quenching media to reach the highest possible hardness value was investigated. In addition, the impact of adding TiO₂ nanoparticles in quenching fluid on wear resistance (dry sliding) was investigated to determine the optimal addition that resulted in enhancing the wear resistance.

2 EXPERIMENTAL WORK

2.1 Heating Treatment

One of the most critical steps when fabricating steel is the heat treatment which has different classifications based on the cooling method. All types of heat treatment are similar in the heating stages, but they differ in the cooling process. Quenching is usually done by immersion the manufactured piece after heating it to the desired temperature in the cooling media. The success of the quenching process in producing the desired mechanical properties depends on the heat transfer characteristics of the quenching medium. The quenching step is done by immersing specimens in the cooling media which is oil, in this work with a continuous motion to smash the steam layer that forms during the quenching process.

In most cases, the heating and quenching processes result in an oversaturated solution. Therefore, the tempering process, which is heating the metal steel to a temperature between 200 to 600 °C, is necessary to provide the appropriate environment to re-distribute the self-propagated components. The tempering process reduces internal

and residual stresses resulting from the cooling step to avoid distortion [9]. Therefore, the tempering step is a crucial factor in removing most internal stresses and accelerating the diffusion processes to disassemble the rigid solution and get a solid and balanced solution to increase the durability of the alloy without changing the hardness and resistance.

In this work, a furnace was used for heating at the specimens to a temperature of 850 °C for about 30 min then cooling followed by cooling them by either pure oil (without nanoparticles) or oil with TiO₂ nanoparticles in different percentages. The time and temperature values were computed using a digital timer and thermocouple. The quenching process was finished when the nanofluid and samples were reached the thermal equilibrium. The tempering process was carried up for one hour at a temperature of 400 °C, which falls within the tempering limits of this material which ranges between 150-600 °C. The last step was cooling the specimens at the ambient temperature.

2.2 Materials and Methods

50 nm TiO₂ (Degussa P25, purity >99.98) with a bulk and actual density of 0.25 and 3.8 (g/cm³), respectively, was used in this work after characterization by X-ray diffraction (XRD) and transmission electron microscope (TEM) to identify the bulk phase and particle sizes, respectively, and the results are presented in previous work [10]. TiO₂ was dispersed in oil (Dykolume 2300 L), and to gain homogeneous suspensions magnetic mixer was used for (10 minutes) at room temperatures. Sonication for two hours by ultrasonic wave (POWERSONIC410) at room temperature was performed before each test to prevent the nanoparticle sedimentation and minimize the agglomeration in the bottom surface of the container. Four cooling media were prepared by mixing a specific weight of TiO₂ in oil. This addition of nanoparticles was done by following equation (1)[11],

$$\varphi v = \left[\frac{1-\varphi_m}{\varphi_m} \frac{\rho_p}{\rho_f} + 1 \right]^{-1} \quad (1)$$

where φ_m is the mass concentration of nanofluid, φ_v is the volume concentration of nanofluid, ρ_f is the fluid density, and ρ_p is the nanoparticle density. Figures 1-a is a schematic diagram of the steps of preparing nanofluid and Figures 1-b is the actual image of the prepared nanofluid during the sonication process.

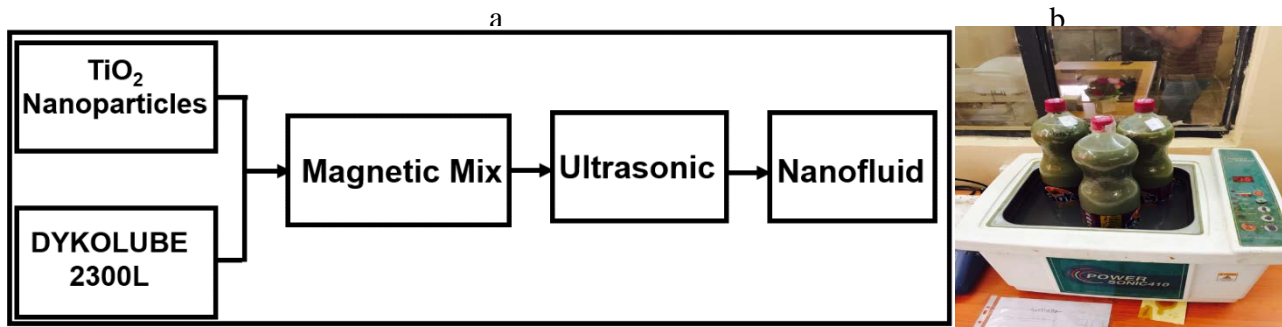


Figure 1. (a) Steps for preparation of nanofluid and (b) Sonication of prepared nanofluid

The base metal used in this study was medium carbon steel CK45, and its chemical composition it is shown in table 1. The shafts used for this process are shown in Figure (2-a). The cylindrical shape of specimens was gained by using turning machine type TX32 automated with Computer Numerical Control (CNC) to form specimens with 15mm length and 10mm diameter according to ASTM (G 99 – 04) as shown in Figure (2-b). After that, all specimens were grinding and polishing. The specimens for the test were classified into four groups named A, B, C, and D, where they resulted from the treatment with 0, 0.2, 0.4, and 0.6% of the nanofluid solution, respectively. Table 2 shows the conditions for each group.



Figure 2. (a) Shafts used in this work, (b) Four groups of specimens

Table 1: Chemical composition of CK45 steel

Wt%	C	Si	Mn	P	S	Cr	Mo	Ni	Fe
Standard Value	.5-0.4	0.4	-0.80.5	0.035	0.035	0.4	0.1	0.04	balance
Actual Value	0.41	1.73	0.673	0.002	0.002	0.031	0.018	0.077	balance

Table 2. Classified test for specimen's groups

NO.	Condition
Group A	Base metal heating at 850°C for time 30min then cooling in pure oil.
Group B	Base metal heating at 850 °C for time 30min then cooling in oil with 0.2 nano-fluid.
Group C	Base metal heating at 850°C for time 30min then cooling in oil with 0.4 nano-fluid.
Group D	Base metal heating at 850°C for time 30min then cooling in oil with 0.6 nano-fluid.

A dry sliding wear test tested all specimens prepared with and without nanofluid technology. The machine used for dry wear was Pin on Disk as shown in Figure (3), and the wear-rate was found by weight loss method using a sensitive microbalance with an accuracy of 0,0001g. Equation 2 was used to calculate the wear rate [12],

$$Wr = \frac{\Delta W}{SD} \quad (2)$$

where Wr is the wear rate (gm/cm), ΔW is the weight loss (gm), and SD is the sliding distance (cm). The three normal loads (20, 30, and 40 N) were applied according to ASTM standards for the wear rate test with sliding speeds of 0.5, 1, and 1,5 (cm/sec) and 10 minutes for each test at normal atmospheric conditions and room temperature.

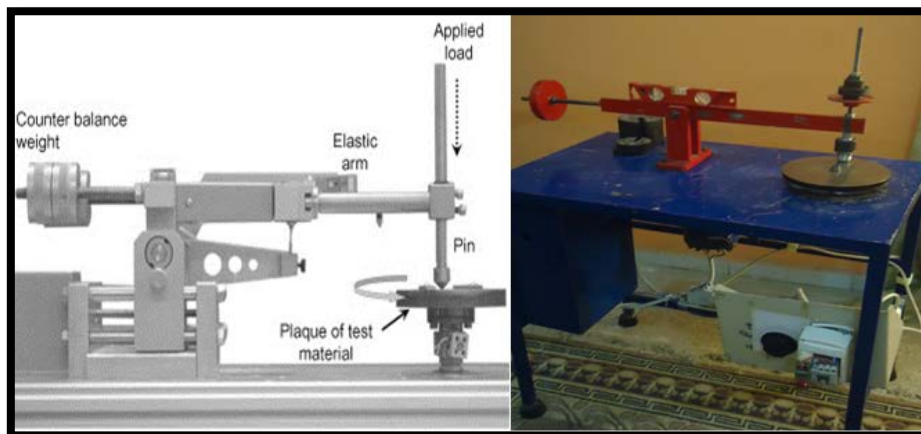


Figure 3. Pin on Disk machine was used to perform the wear resistance test

All specimens were prepared for microstructure test by grinding of these specimens by SiC emery paper of grades from 120 to 800 followed by polishing these samples with alumina (Al_2O_3) solution with a special cloth using grinding and polishing machine (Remet LS1), which is shown in Figure (4-a). Then watery treatment occurred using Nital solution, composed of 98% methyl alcohol and 2% nitric acid. Then the specimens were scanned by an optical microscope (Metallurgical Microscope L2003/L2030), which is shown in Figure 4-b.



Figure 4. (a) Grinding and polishing machine, (b) Microstructure characterization device

3 RESULTS AND DISCUSSION

3.1 Wear Resistance

The results of the wear resistance test of all specimens (A, B, C, and D) are shown in Figure 5. In general, this Figure shows that the quenching media of nanoparticles has a noticeable impact on the wear resistance and leads to improved steel's ability to wear resistance, which enables the use of this type of steel in structure parts and tools safely. Specifically, Figure 5-a shows the wear rate at 20N of medium carbon steel quenching without nanoparticles was ($15 \cdot 10^{-5}$ g/cm). But a decrease in the wear rate was observed when this steel quenching with different percentages of nanoparticles in quenching media (0.2, 0.4, and 0.6 %) at sliding speed of 0.5 cm/s to be ($14 \cdot 10^{-5}$, $11 \cdot 10^{-5}$, and $13 \cdot 10^{-5}$ (g/cm), respectively, due to the formation of rugged structure [13]. This hard structure is martensite, as will be discussed later.

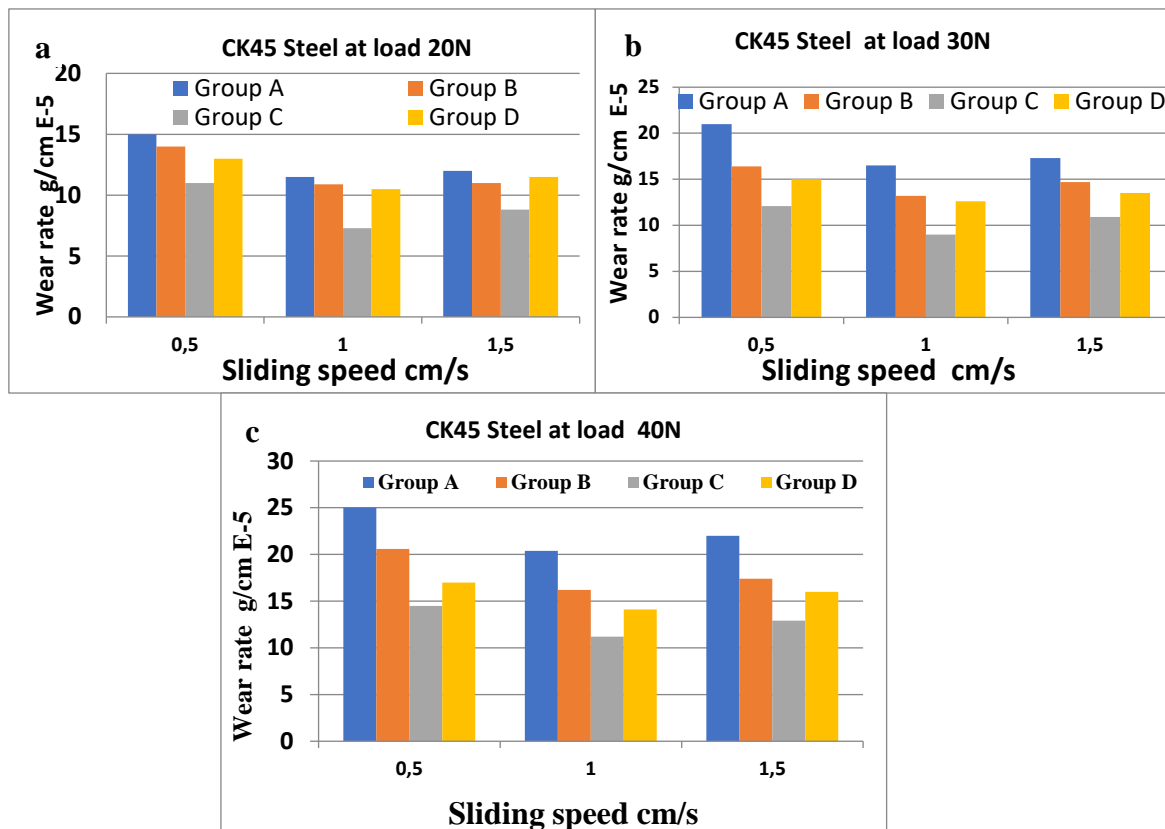


Figure 5. The effect of sliding speed on wear rate at (a) 20N, (b) 30 N, and (c) 40 N

From Figure (5-a), specimen with 0.4% nanoparticles has the minimum wear rate ($7.3 \cdot 10^{-5}$ g/cm) at 20N and (1cm/s). The comparison between this specimen and the specimen without TiO_2 nanoparticles at the same conditions shows that the decrease in the wear resistance is approximately 58%. Similarly, Figure 5-b shows that the wear rate at 30N of steel-specimen quenching in oil without nanoparticles was $16.5 \cdot 10^{-5}$ g/cm, but the wear rate decreased when this steel quenching with nanofluid. Specifically, when it quenched with media of 0.2, 0.4, and 0.6% nanofluid at a sliding speed of 1cm/s, the wear rate values became $13.2 \cdot 10^{-5}$, $9 \cdot 10^{-5}$, $12.6 \cdot 10^{-5}$, respectively. Figure 5-b shows that under this load (30N), specimen quenched with 0.4 nanofluid recorded the minimum wear rate ($9 \cdot 10^{-5}$ g/cm) at 1cm/s. The comparison between this specimen and specimen without nanoparticles at the same condition shows that the reduction in wear resistance is approximately 83%. A similar trend can be observed in Figure 5-c, which shows the wear rate at 40N. The wear rate of steel quenched without nanoparticles was $20.4 \cdot 10^{-5}$ g/cm, while the wear rate recorded for the steel quenched with 0.2, 0.4, and 0.6 % of nanofluid at sliding speed 1cm/s were $16.2 \cdot 10^{-5}$, $11.3 \cdot 10^{-5}$, and $14.1 \cdot 10^{-5}$ g/cm, respectively. Under this load (40N), specimen c also showed the lowest wear rate ($11.2 \cdot 10^{-5}$ g/cm) when testing the specimens at 1cm/s, which shows the most noticeable influence among other tested speed. The comparison here between these specimens and specimens without nanoparticles at the same condition shows decreasing in wear resistance by approximately 82%.

Figure 5 also shows the influence of sliding speed on wear rate for all specimens. It can be noticed that the wear rate decreased with increasing sliding speed. This decrease appears clearly when increasing the speed from 0.5 to 1 cm/s, but it is less noticeable with the further increasing of the speed to 1.5 cm/s due to reaching the steady-state. The reason for this behavior is the increase in the steel temperature. Specifically, increasing the sliding speed would lead to increasing temperature up to the melting point at asperities. That means the soften of asperities reduces the forces required to shear points and improves wear resistance [12]. This improvement of wear resistance for specimens with nanoparticles in oil at the same temperature is due to the formation of the martensite phase, a very rigid structure [13]. The quenching with nanofluid causes refined microstructure that

causes enhanced and improved dry-sliding wear resistance as will be discussed in the next section. In addition, quenching with nanofluid prevent cracks which resulted from quenching with water.

3.2 Microstructure characterization

Figure 6a-6d show the results of the microstructure test performed by microscope for all groups (A, B, C, and D) and the effect of quenching with nanofluid during heat treatment on microstructure properties before wear test. Figure 6-a, which is the microstructure scan of the specimen without nanoparticles, shows a consisted of pearlite, ferrite, and small dark dots in CK45 steel. The austenite phase completely disappeared due to quenching this specimen at a temperature (850 °C) without using a nanofluid. Thus, the appearance of the pearlite phase is due to the imperfect quenching process. Figure 6-b, which is the scan of the specimen using 0.2% of nanofluid, shows pearlite and martensite, but the absence of ferrite phase due to the homogeneity of the mixture and then use of nanoparticles (TiO_2) in the quenching fluid which leads to an increase cooling rate which resulted in the formation of the rugged (or hard) phase (martensite). Thus, using TiO_2 nanoparticles in quenching fluid resulted in the formation of the martensite phase, which is responsible for increasing the hardness of the materials and decreasing the wear rate.

Figure 6-c, which is the scan of the microstructure of sample quenched with 0.4% nanofluid, shows only martensite phase and entire absence of pearlite or ferrite due to the higher percentage of TiO_2 nanoparticles in quenching fluid which leads to a higher cooling rate that causes transformation of the austenite to martensite to avoid forming other softer phases such as retain austenite, pearlite, and ferrite. The difference in grain structures for CK45 steel with nanoparticles quenching was more apparent at the edges than at the center of the specimens because the varying cooling rates appeared across the specimens [14]. The dominant martensite phase is responsible for increasing the hardness of the materials and decreasing the wear rate. Figure 6-d, which is the scan of the microstructure of specimen quenched with 0.6% nanofluid, shows lowering in the martensite phase and formation of carbide that resulted from the addition of nanoparticles with quenching media [14] due to incomplete solubility. Adding higher than 0.4% of TiO_2 seems to cause agglomeration of the nanoparticles, which lowered their performance [15].

A more in-depth quenching process is usually done by immersing the manufactured piece after heating it to the desired temperature in the cooling media. The success of the quenching process to achieve the desired controlling of the mechanical properties depends on the heat transfer characteristics of the quenching medium. The heating of steel to a specific temperature and keeping it at the desired temperature for an appropriate period, depending on the required thickness of the section and its chemical structure, is necessary to form a homogenous chemical structure of austenite. The next step is to immerse it in the cooling media, usually water or oil, with a continuous motion for the sample to smash steam layer forms during the quenching process. If the cooling rate exceeds its critical value, the austenite transforms directly into a very rigid structure, martensite, a transitional compound between the austenite (F.C.C) and the ferrite (B.C.C). This phenomenon is because many carbon atoms in the austenite cannot escape at high cooling rates, resulting in a rigid and over-saturation solution of iron (α) dissolved in a high proportion of carbon. Specifically, the extra carbon atoms penetrate and dissolve in the iron structures due to the fast-cooling rate resulted in a crystalline prism structure for the martensite phase.

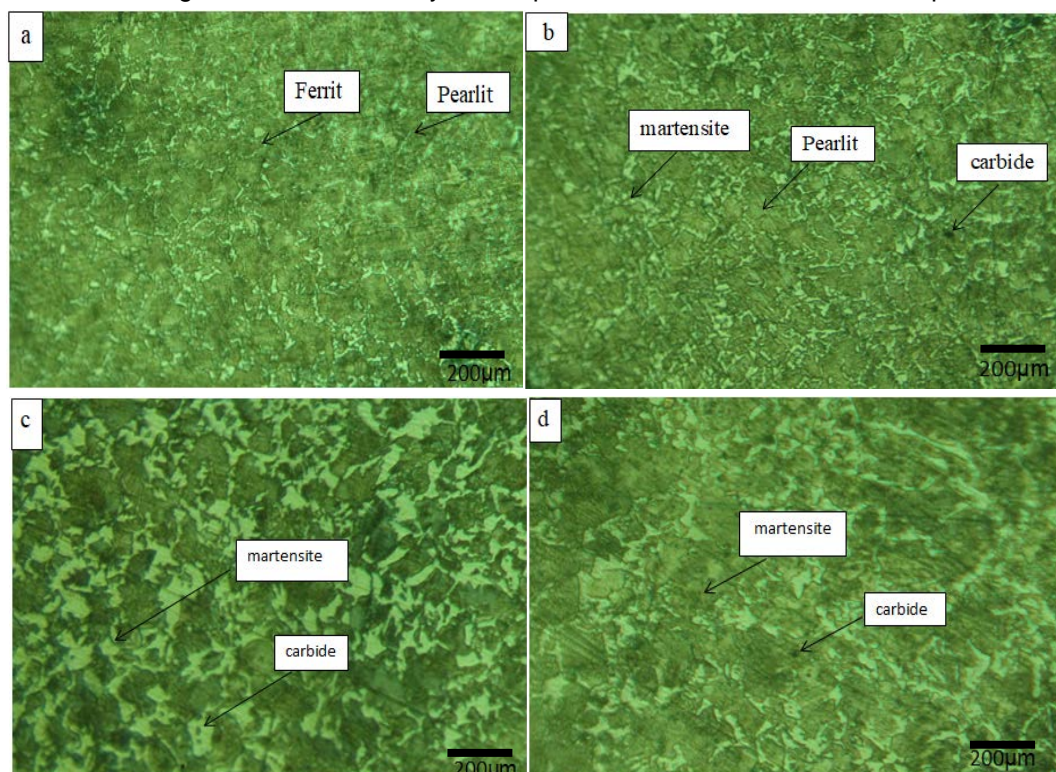


Figure 6. Microstructure of CK45 steel quenching with (a) 0%, (b) 0.2%, (c) 0.4%, and (d) 0.6% TiO_2 nanofluid

4 CONCLUSIONS

This study shows that medium carbon steel (Ck45) was successfully manufactured by quenching with TiO₂ nanofluid. The wear resistance and microstructure analysis show that using TiO₂ nanofluid notably enhanced the wear resistance. The following conclusions and observations are drawn from the experimental results. The wear rate increase with increasing load applied on the same samples to reach a maximum value at 40N. The enhancing of wear resistance for all samples occurred after using quenching media with nanofluid (oil/TiO₂) at the same temperature and procedure. The wear rate decreasing with the increasing of nanoparticles until it reaches the minimum value of wear (7.3×10^{-5} g/cm) at 20N and 0.4% nanoparticles; after that the wear rate began to increase slowly. The improvement of mechanical properties because of structural homogeneity and formation of the rugged phase (martensite) after used quenching media with nanoparticles. The addition of nanoparticles leads to increasing the cooling rate that causes the transformation of the austenite to martensite to avoid forming other softer phases such as retain austenite, pearlite, and ferrite that the developed microstructure and mechanical properties. The optimum wear resistance and microstructure were found at 0.4% of nanofluid with oil during quenching when kept constant of all condition.

5 ACKNOWLEDGMENT

Authors would like to thanks Middle Technical University for its support. They also would like to thanks Al-Mustaqbal University College for its support through the fund MUC-E-0122.

6 REFERENCES

- [1] Kandpal, B.C., Gupta, D., Kumar, A., Jaisal, A.K., Ranjan, A.K., Srivastava, A., Chaudhary, P. (2021). Effect of heat treatment on properties and microstructure of steels, *Materials Today: Proceedings*, 44 , 199-205.
- [2] Akbarpour, M., Mirabad, H.M., Azar, M.K., Kakaei, K., Kim, H. (2020) Synergistic role of carbon nanotube and SiCn reinforcements on mechanical properties and corrosion behavior of Cu-based nanocomposite developed by flake powder metallurgy and spark plasma sintering process, *Materials Science and Engineering: A*, 786 139395.
- [3] Bagherzadeh, M., Mousavi, O., Ghahfarokhi, Z.S. (2020) Fabrication and characterization of a Fe₃O₄/polyvinylpyrrolidone (Fe₃O₄/PVP) nanocomposite as a coating for carbon steel in saline media, *New Journal of Chemistry*, 44 15148-15156.
- [4] Tabish, M., Malik, M.U., Khan, M.A., Yasin, G., Asif, H.M., Anjum, M.J. , Khan, W.Q., Ibraheem,S., Nguyen, T.A. , Slimani, Y. (2021) Construction of NiCo/graphene nanocomposite coating with bulges-like morphology for enhanced mechanical properties and corrosion resistance performance, *Journal of Alloys and Compounds*, 867159138.
- [5] Alalwan, H.A., Alminshid, A.H., Mohammed, M.M. , Mohammed, M.F. (2022) Reviewing of using Nanomaterials for Wastewater Treatment, *Pollution*, 8, 995-1013.
- [6] Ravikumar, M., Reddappa, H., Suresh, R., Reddy, M.S. (2021) Experimental studies of different quenching media on mechanical and wear behavior of Al7075/SiC/Al₂O₃ hybrid composites, *Frattura ed Integrità Strutturale*, 15, 20-31.
- [7] Barai, D.P., Bhanvase, B.A., Sonawane, S.H. (2020) A review on graphene derivatives-based nanofluids: investigation on properties and heat transfer characteristics, *Industrial & Engineering Chemistry Research*, 59, 10231-10277.
- [8] Guo,W., Li, J., Qi, M., Xu, Y., Ezatpour, H.R. (2021) Effects of heat treatment on the microstructure, mechanical properties and corrosion resistance of AlCoCrFeNiTi_{0.5} high-entropy alloy, *Journal of Alloys and Compounds*, 884 ,161026.
- [9] Ba, K., Levesque, J., Gakwaya, A., Karganroudi, S.S. (2021) Residual stress investigation of quenched and artificially aged aluminum alloy 7175, *The International Journal of Advanced Manufacturing Technology*, 1-17.
- [10] Alalwan, H., Alminshid, A. (2-2-) An in-situ DRIFTS study of acetone adsorption mechanism on TiO₂ nanoparticles, *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy*, 229 , 117990.
- [11] Ciloglu,D., Bolukbasi,A., Cifci,H. (2015) The Effect of Type of Nanoparticles on the Quenching Process, *International Journal of Materials and Metallurgical Engineering*, 9 , 694-697.
- [12] Adel,K. (2014) Enhancement of dry sliding wear characteristics of CK45 steel alloy by laser surface hardening processing, *Procedia materials science*, 6 , 1639-1643.
- [13] Higuera-Cobos, O.F., Dumitru, F.-D., Mesa-Grajales, D.H. (2016) Improvement of abrasive wear resistance of the high chromium cast iron ASTM A-532 through thermal treatment cycles, *Revista Facultad de Ingeniería*, 25, 93-103.
- [14] Mohmmmed, J. (2017) Effect of Addition CuO Nanoparticle to Quenching Media on Properties of Medium Carbon Steel, *Engineering and Technology Journal*, 35 .

- [15] Mahiswara, E., Harjanto, S., Putra, W.N., Ramahdita, G., Yahya, S. (2018) Characterization of oil based nanofluid for quench medium, IOP Conference Series: Materials Science and Engineering, IOP Publishing, 012068.

Paper submitted: 08.12.2022.

Paper accepted: 17.04.2023.

This is an open access article distributed under the CC BY 4.0 terms and conditions