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Quenching Media Effects on Carbide Microstructure and Microhardness of Locally Manufactured AISI D2 Steel during tempering process

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تأثيرات وسائط التبريد على البنية المجهرية للكربيد والصلابة المجهرية لصلب العدة المصنع محليا أثناء التلدين (*AISI D2*)

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Abstract:
Cold work tool steel is essential in manufacturing and suitable for many applications, including punching, forming, and cold rolling. This steel distinguished by a high carbon and chromium content and exhibits mechanical properties significantly influenced by heat treatment processes. It contains higher hard phases like carbides and plate martensite than other steel alloys, posing a continual challenge in balancing strength and toughness to prevent immediate fracture. The primary problems of hardening treatment of this steel include the risk of crack distortion and formation in various media. In this study, the effect of different quenching media on the microstructural evaluation and microhardness of locally manufactured AISI D2 tool steel was investigated to possibility improving microstructure and mechanical properties, where the AISI D2 tool steel was austenitized at a temperature of 900°C, quenched in different media (water and oil), and finally tempered. Microstructural analysis was conducted using optical microscopy along with quantitative image assessment through ImageJ software, and Vickers microhardness was carried out. The results showed that oil-quenched specimens exhibited coarser carbides and higher retained austenite content, whereas water-quenched specimens showed finer and more uniformly distributed carbides. Quantitative image analysis confirmed significant variation in carbide size and area fraction depending on the quenching medium. The measured Vickers microhardness values were 197.9, 248.2, and 225.9 HV for the locally manufactured base steel and for the water- and oil-quenched samples, respectively. Statistical analysis indicated a weak positive correlation between carbide distribution and microhardness (R = 0.213, 0.123, 0.169). These findings emphasize the importance of optimizing heat treatment in mechanical applications.

Keywords: AISI D2 tool steel, Quenching media, Carbide microstructure, Microhardness, ImageJ software.

الملخص:
يُعد فولاذ الأدوات للعمل على البارد من المواد الأساسية في مجال التصنيع، حيث يُستخدم في العديد من التطبيقات مثل عمليات التثقيب، والتشكيل، والدرفلة على البارد.، حيث يتميز هذا النوع من الفولاذ بمحتوى عالٍ من الكربون والكروم، وتُظهر خواصه الميكانيكية تأثراً كبيراً بعمليات المعالجة الحرارية. كما يحتوي على نسب أعلى من الأطوار الصلبة مثل الكربيدات والمارتنسيت الصفائحي مقارنةً بسبائك الفولاذ الأخرى، مما يفرض تحدياً مستمراً لتحقيق التوازن بين المتانة

والصلادة لتجنب حدوث الكسر المفاجئ. ومن أبرز المشكلات المرتبطة بعمليات التقسية لهذا الفولاذ خطر التشوه والتشقق الناتج عن استخدام أوساط تبريد مختلفة. في هذه الدراسة، تم التحقيق في تأثير أوساط التبريد المختلفة على البنية المجهرية والصلادة المجهرية لفولاذ الأدوات AISI D2 المُصنَّع محلياً، بهدف تحسين البنية المجهرية والخواص الميكانيكية. حيث تم إجراء الأوستنة للفولاذ عند درجة حرارة 900 درجة مئوية، تلتها عملية التبريد السريع باستخدام أوساط مختلفة (الماء والزيت)، ومن ثم إجراء عملية المراجعة الحرارية (المراجعة/التخمير). تم تحليل البنية المجهرية باستخدام المجهر الضوئي، إلى جانب تقييم كمي للصور باستخدام برنامج) (j, Image)كما تم قياس الصلادة المجهرية باستخدام اختبار فيكرز، أظهرت النتائج أن العينات المبردة بالزيت احتوت على كربيدات أكثر خشونة ونسبة أعلى من الأوستنيت المحتجز، في حين أظهرت العينات المبردة بالماء كربيدات أدق وأكثر تجانساً في التوزيع. كما أكد التحليل الكمي للصور وجود اختلافات ملحوظة في حجم الكربيدات ونسبة مساحتها تبعاً لوسط التبريد المستخدم. وقد بلغت قيم الصلادة المجهرية بطريقة فيكرز 197.9 و248.2 و225.9 HV لكل من الفولاذ الأساسي المُصنَّع محلياً، والعينات المبردة بالماء، والعينات المبردة بالزيت على التوالي. وأشار التحليل الإحصائي إلى وجود ارتباط طردي ضعيف بين توزيع الكربيدات والصلادة المجهرية (0.213=R ، 0.123، 0.169). وتؤكد هذه النتائج أهمية تحسين عمليات المعالجة الحرارية في التطبيقات الميكانيكية.

الكلمات المفتاحية: فولاذ الأدواتAISI D2 ، أوساط التبريد، البنية المجهرية للكربيدات، الصلادة المجهرية، برنامج. التحليل الكمي ImageJ.

Introduction:

AISI D2 is an essential component of the manufacturing industry, and it is extensively utilized for demanding applications such as molds, punches, stamping tools, and blanking dies, where the mechanical performance and service life of D2 steel are significantly impacted by heat treatment conditions. The carbide dissolution, alloying element redistribution, austenite grain formation, and phase stability prior to quenching [1, 2] are affected by austenitizing temperature. Whenever the primary component, M_3C_3 carbides, is partially dissolved, raising the austenitizing temperature enriches the austenite phase with carbon and chromium, which enhances the martensitic transformation [3, 4, 5]. Therefore, the main reason for this alloy's frequent application is its outstanding tribological properties, which include excellent wear resistance, high hardness, and dimensional stability under extreme mechanical stress [6, 7]. On the other hand, the microstructure, particularly its high chromium content and the dispersion of hard, chromium-rich carbide phases within a martensitic matrix, is inextricably tied to these mechanical properties [8].

The significance of heat treatment in carbide kinetics optimization has been highlighted in recent papers to improve microstructural uniformity. Bole and Sarkar [9] showed that high-temperature processing followed by quenching encourages the conversion of primary M_7C_3 carbides into secondary $M_{23}C_6$ carbides. It has been demonstrated that this even distribution of carbide improves toughness without sacrificing hardness and that it is important to control temperature. In addition, a final microstructure is largely determined by the quenching medium in austenitizing temperature, where the carbide precipitation behaviors, residual stress evolution, and martensitic transformation mechanisms are all directly impacted by the cooling rate determined by media like water or oil [7, 10]. While slower cooling rates may cause carbide coarsening and raise the fraction of remaining austenite, rapid cooling usually suppresses carbide re-precipitation, resulting in a refined martensitic structure [10, 11]. Therefore, attaining a balanced synergy of hardness and toughness requires optimizing quenching conditions.

Moreover, most of the studies that are now available rely on qualitative observations; only a few studies utilize quantitative image analysis to precisely measure carbide morphology and volume percentage. Although using sophisticated digital photos increases the correspondence of structure-property correlations, processing tools like ImageJ provide a reliable methodology for retrieving objective microstructural data, such as phase fractions and particle metrics [12, 13].

In the study is present the effect of austenitizing temperature and different quenching media (water and oil) on carbide morphology, volume fraction, grain size, and microhardness of AISI D2 tool steel, and optical microscopy combined with quantitative image analysis using ImageJ software is employed to characterize microstructural features, while Vickers microhardness testing is used to evaluate the mechanical response. The results aim to provide a deeper understanding of the role of heat treatment parameters in controlling carbide behavior and optimizing the performance of local AISI D2 tool steel for demanding industrial applications.

Experimental Worke:

Material and Methods:

In order to accommodate the heat treatment furnace and the ensuing metallographic preparation needs, rectangular specimens were cut to appropriate dimensions. AISI D2 high-carbon, high-chromium cold work tool steel was utilized in this investigation, the sample was a cylindrical shape with a dimensions of (50mm´25mm) As illustrated in figure (1), the samples were sourced from Libyan Iron and steel company.

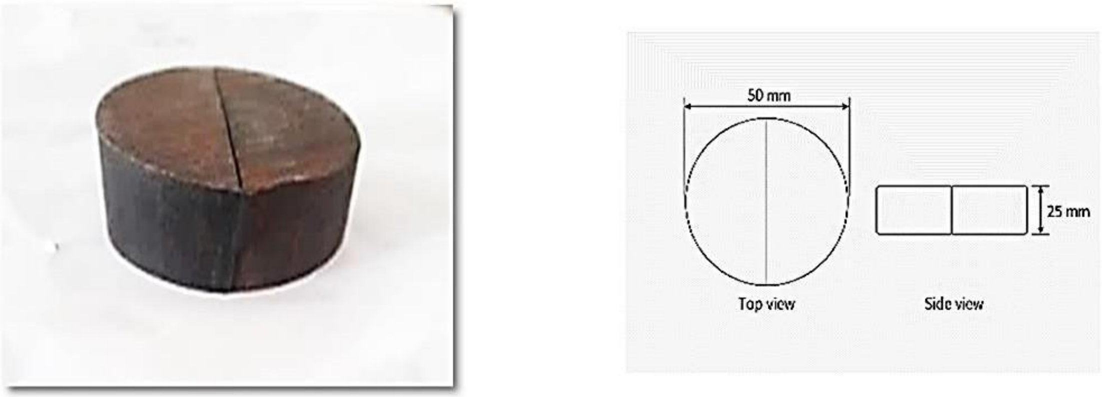

Figure (1): Locally specimen AISID2 dimensions that are designated for heat treatment.

Optical Emission Spectroscopy (FLOOR MODEL SPECTROMETER) (OPT-5800) was used to ascertain the chemical composition of the steel under investigation. Table 1 displays the Local AISI D2 tool steel's determined chemical composition. This work was conducted in Materials Characterization Laboratory at Welding Centre, Tajora, Libya. It founded the material's compliance with the standard chemical range of AISI D2 steel.

Table (1): Chemical composition of the D2 tool steel.

Sample	C%		Si%	Mn%	Cr%	Mo%	V%	P	S	Cu	Ni	Fe
Stander AISI D2	max.	1.6	0.6	0.6	13	1.2	1.1	0.03	0.03	0.25	0.3	Bal
	min.	1.4	non	Non	11	0.7	Non	Non	Non	Non	Non	
Local AISI D2	1.55		0.3	0.4	11.8	0.8	0.8	Non	Non	Non	Non	

Treatment Trial:

The aim of this heat treatment process was to investigate the effects of the austenitizing temperature and the quenching media on the microstructure of local AISI D2 tool steel. which included preheating, austenitizing, quenching, and tempering, was carried out on four specimens. In an attempt to provide an equal temperature distribution from the surface to the core and to avoid observed in Figure 2(a.b),the austenitizing process was carried out at 900 °C for one hour after preheating.

After austenitization, the specimens were immediately quenched to room temperature with two distinct quenching media: water and oil. The use of various cooling medium was designed to produce varied cooling rates and assess their impact on carbide shape, distribution, and matrix modification Figure(2c).

After quenching, all specimens were tempered at 580 °C for 1 hour to release the internal stresses, stabilize the microstructure, and alter the final mechanical characteristics. After tempering, the samples were air-cooled to room temperature.

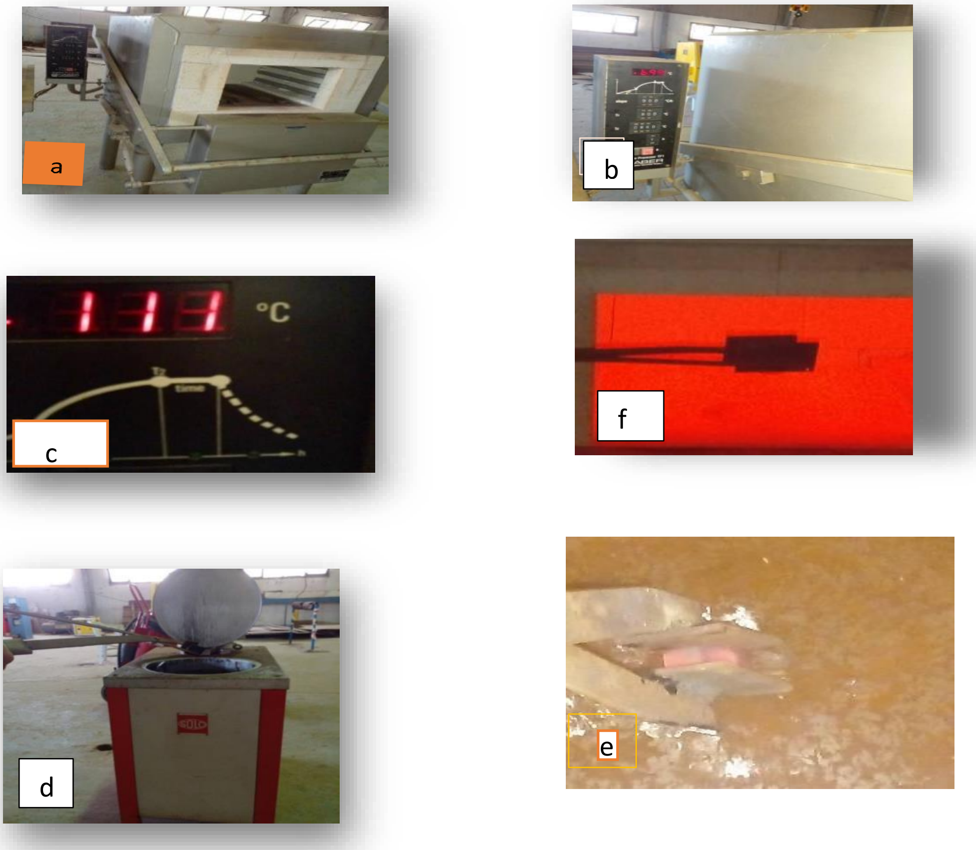

Figure (2): Shows the sequential stages involved in the heat treatment process of the specimens (a)furnace setup used for heating the sample (b) temperature control panel and monitoring system during heating; c cooling or temperature post treatment set up afterheat exposure;(f.&e) samples after cooled in different quenching media.

Morphology inspection and Quantitative Image Analysis:

For metallographic examination, heat-treated samples were grinning with 80 to 1000µ, and polished with diamond suspension (0.3 t0 0.9 µ) to a mirror finish. The polished samples were etched with a concentration of 3% Nital solution to reveal their microstructure. Observations were made with Optical Microscopes Type (HST402-AW) at various magnifications to examine grain structure, carbide morphology, and phase distribution.

After reasonable imaging, Image J software was utilized to analyze the morphology and distribution of carbides through methods such as thresholding, image segmentation, and particle analysis of optical images. Particle segmentation, contrast enhancement, and grayscale conversion were among the steps that made it possible to distinguish the carbide phase from the matrix. The analysis produced quantitative metrics such as carbide size, distribution, and volume fraction. To guarantee statistical reliability and lower measurement uncertainty, several pictures were analyzed.

Microhardness Measurements:

The mechanical properties of heat-treated samples were assessed through Vickers microhardness testing, (MODEL: HVS-1000ZT) using (ASTM E384) at a 1 kg test load and a 10 s exposure time. The tests were conducted at several locations on each sample to enhance the reliability of hardness values and reduce the effect of microstructural variations.

The test was conducted for Samples at different cooled media (oil-water - air for stander sample) at tempered in air.

Results and discussion:

The optical micrographs in figure (3) of AISI D2 tool steel reveal a heterogeneous microstructure characterized by dark regions indicating hard carbide phases. At higher magnifications, coarse primary carbides and finer secondary carbides are visible, showcasing irregular dispersion and a variety of sizes. The matrix primarily exhibits a ferritic-pearlitic structure, with carbide particles reinforcing the material.

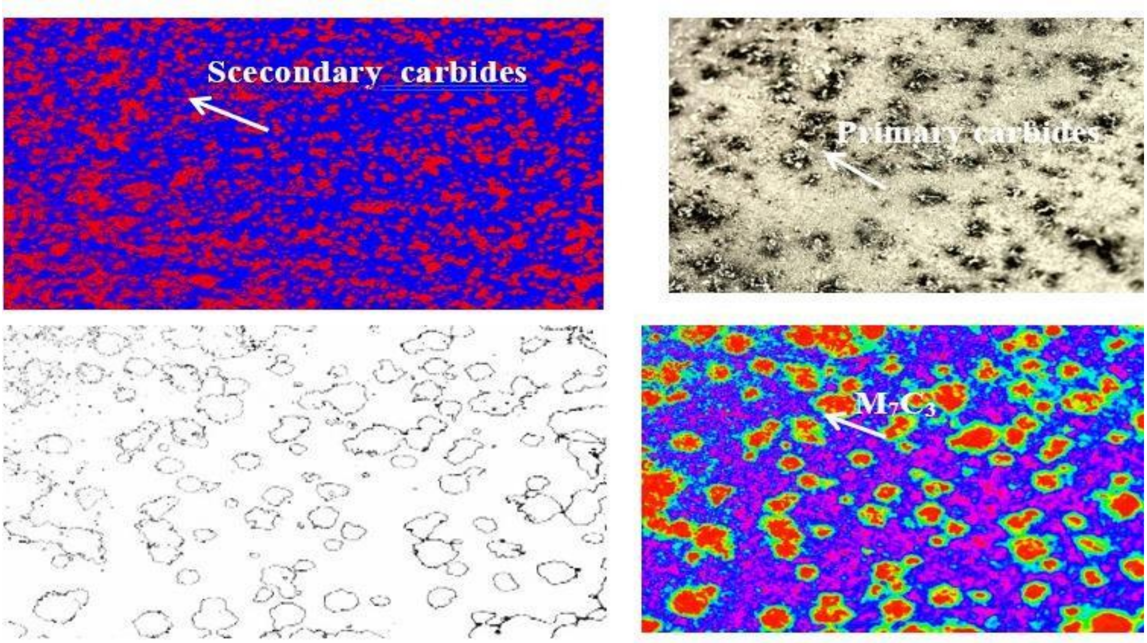

Figure (3): AISI D2 tool steel Stepwise image processing for segmentation (a)original RGB image; (b) threshold; (C)binary image; (d) segmented image

Figure (4a) illustrates specimens quenched in water after austenitization at 900 °C, revealing a martensitic matrix with uniformly distributed small carbide particles. At higher magnifications, these carbides appear as small black dots, indicating a lack of clustering and suggesting effective control over carbide coarsening due to rapid cooling when compared to the base metal.

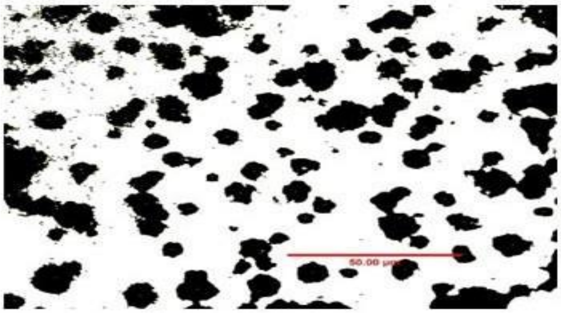

Figure (4-a): Etched metallographic image of the sample cooled in water with Mag 50x.

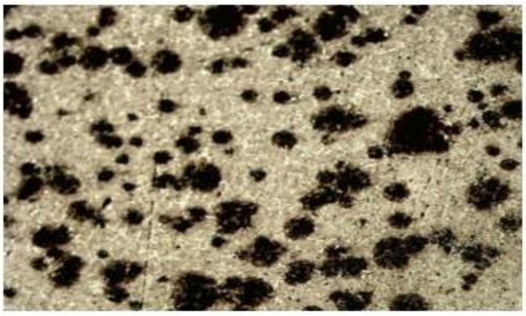

Figure (4-b): Cooled in water with detection (mask of carbides).

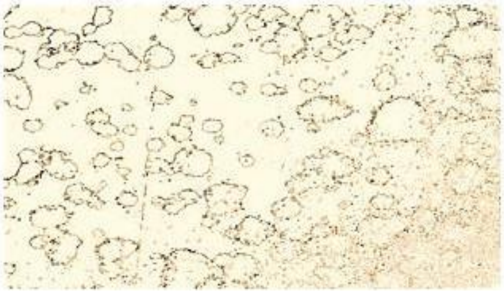

Figure. (4-c): Cooled in water with detection of carbides.

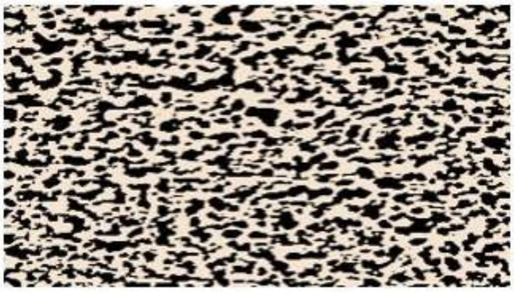

Figure (4-d): Threshold version.

The optical micrographs of the oil-quenched specimens reveal a primarily martensitic matrix with coarse carbide particles as shown in figure (5). These carbide particles are larger and less uniformly distributed compared to water-quenched samples, showing increased coarsening with darker, more prominent areas. Additionally, the matrix appears darker, suggesting the preservation of austenite.

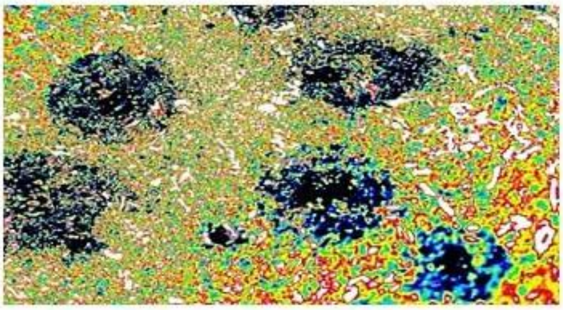

Figure. (5-a). Sample cooled in oil

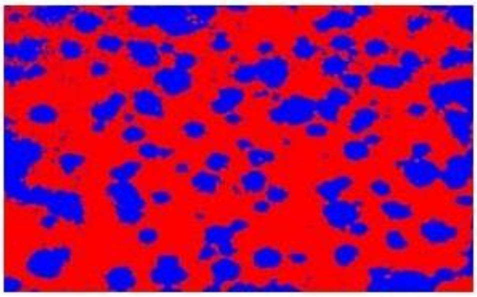

Figure (5-b): Threshold of carbides

The analysis of the findings reveals that the microstructural development and mechanical response of AISI D2 tool steel are strongly influenced by heat treatment conditions, particularly the quenching medium. Due to the rapid cooling rate, water-quenched specimens exhibit a finer and more uniformly distributed carbide morphology, which results in lower microhardness values, as reported in [14].

In contrast, oil-quenched specimens, which undergo slower cooling, display a coarser carbide morphology along with retained austenite and a martensitic matrix, leading to higher microhardness values. Although martensite is generally harder, the presence of retained austenite can enhance wear resistance. However, rapid quenching may introduce residual stresses and limit the redistribution of alloying elements, potentially reducing hardness. The complexity of achieving optimal mechanical properties in tool steels has been highlighted by the varying effects of different quenching techniques [15].

Moreover, quantitative image analysis was conducted using ImageJ software to evaluate carbide morphology, size, and area fraction in the heat-treated specimens. Representative segmented images and thresholded micrographs are shown in Figures 5 to 7.

The image analysis indicates that carbide size and distribution vary depending on the quenching medium. Water quenching results in smaller and more uniformly distributed carbides, whereas oil quenching produces larger carbides with a higher carbide area fraction, as illustrated in Figures 6a–6b. The calculated carbide area fraction values confirm that oil quenching leads to a greater fraction of visible carbides compared to water quenching.

Additionally, the size distribution histograms reveal a narrower carbide size range for water-quenched samples and a broader distribution for oil-quenched samples, reflecting the presence of both fine and coarse carbide particles, as shown in Figure 5a–5b.

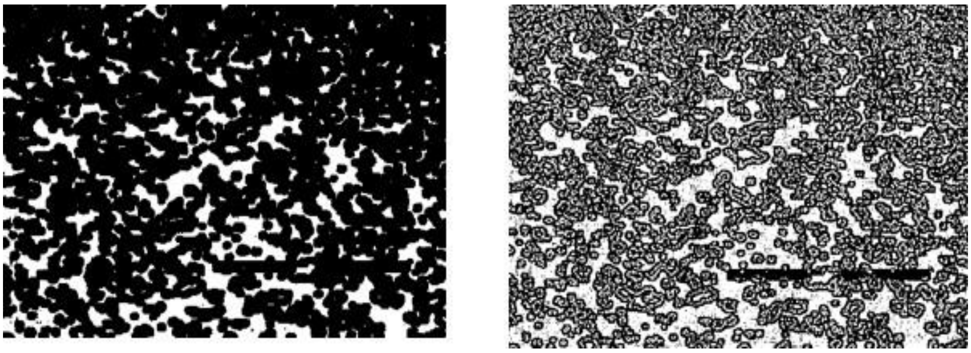

Fig (a) **Fig (b)**

Figure 6(a &b): Illustrates the pixel detection of carbides in a water-cooled sample, utilizing Lambda average J Software for bounding box detection.

The gray value intensity distributions from selected microstructure sections that were analyzed using ImageJ software are presented in Figure 8. Significant changes in pixel intensity linked to various microstructural characteristics are reflected in the patterns. decreased levels reflect matrix- dominated regions with reduced carbide content, while higher gray values can occasionally detect. Carbide particle-rich regions are indicated by isolated peaks. Water-quenched specimens reveal smoother lines, suggesting a more uniform carbide distribution, but oil-quenched specimens show higher amplitude fluctuations, indicating stronger microstructural dispersion.

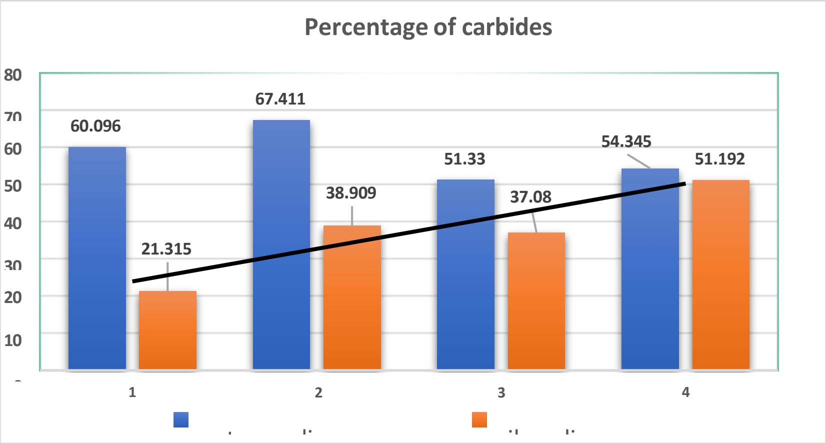

Figure (7): The effect of different quenching media on the percentage of carbides

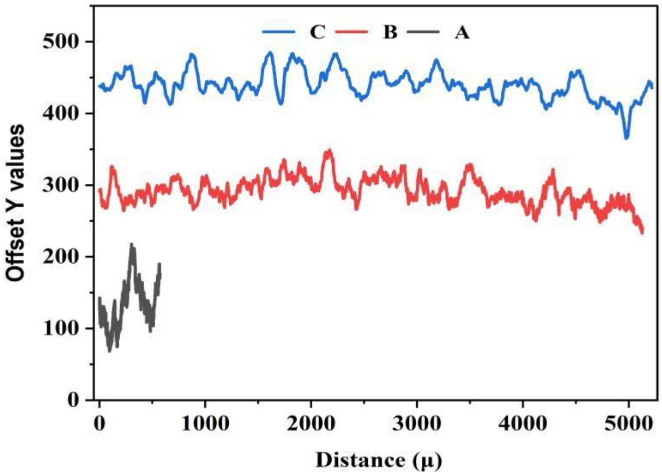

Figure (8): The gray value intensity distributions from selected microstructure sections analyzed with ImageJ software.

Figure 9 displays the gray level histogram from a digital microstructural image of D2 tool steel after heat treatment and water quenching, analyzed using ImageJ. The histogram shows pixel gray values ranging from 0 to 255, revealing a mean gray value of 154.9 and a standard deviation of 34.48, indicative of significant microstructural heterogeneity due to rapid quenching. Higher gray

values (up to 250) suggest the presence of chromium-rich carbides, typical of high-carbon, high-alloy D2 tool steel, while lower values may indicate etched areas or retained austenite. With over1.17 × 10^7 pixels analyzed, the histogram's statistical reliability is enhanced, making it a valuable tool for assessing the impact of heat treatment and quenching on microstructural evolution and associated mechanical properties like hardness and wear resistance.

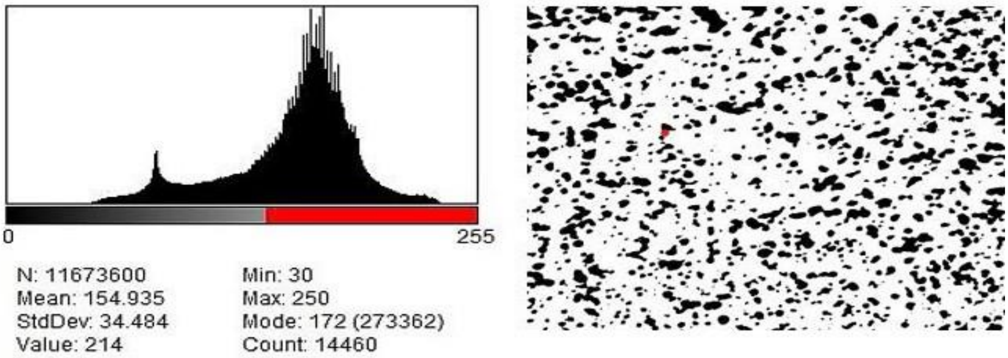

Figure (9): Illustrates the gray level histogram of D2 tool steel following heat treatment and water quenching. The histogram, generated with ImageJ, depicts the distribution of the martensitic matrix and chromium-rich carbides.

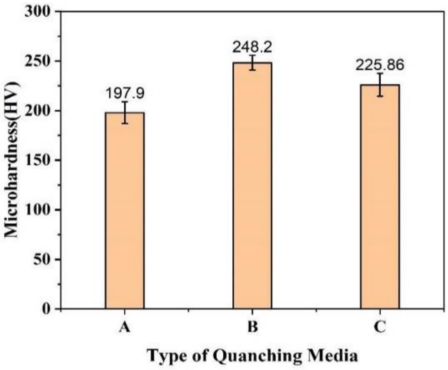

Figure (10): Microhardness at different quenching Media

Furthermore, gray value intensity analysis revealed that carbide-rich regions correspond to higher gray values due to their atomic structure, while microstructural heterogeneity influences toughness and stability. Statistical parameters derived from gray value profiles The Vickers microhardness values measured for the base material and heat-treated specimens are presented in Figure (10). The base material exhibits the lowest microhardness values among all conditions. The study examines how microhardness varies with carbide size in D2 steel, focusing on the effects of quenching methods. Water quenching yields slightly higher hardness than oil quenching due to a higher cooling rate, which enhances the martensitic fraction and limits bainitic transformation [14], [15].

The final microstructure, formed by austenitizing at 900 °C and tempering at 580 °C for 1 hour, mainly comprises tempered martensite with both primary and secondary chromium carbides. The moderate hardness is attributed to limited carbide dissolution at the relatively low austenitizing temperature and extensive decomposition at high tempering temperatures [15]. Hardness increases as carbide size decreases, reaching a peak at an intermediate size, then plateauing and eventually decreasing with further carbide coarsening due to stress concentration and reduced interfacial area [15].

The study also investigates the correlation between carbide morphology and microhardness in AISI D2 tool steel, indicating that chromium-rich carbides enhance wear resistance by hindering dislocation motion [17], [18]. It was found that oil quenching produces a microstructure with improved carbide connectivity within the martensitic matrix, resulting in higher hardness compared to water quenching [17].

Quantitative image analysis using ImageJ software provided reliable evaluation of carbide size and distribution, consistent with established metallographic methods reported in [16]. The study highlights the importance of selecting an appropriate quenching medium to optimize hardness and wear resistance for industrial applications [14]. offering a quantitative approach to correlating heat treatment conditions with carbide distribution and mechanical properties [16], [17].

Conclusion:

The present study investigated the effect of austenitizing temperature and different quenching media on the microstructure at tempered, carbide distribution, and microhardness of AISI D2 tool steel. Based on the experimental results and quantitative image analysis, the following conclusions can be drawn:

- The microstructure of AISI D2 tool steel after heat treatment consists predominantly of a martensitic matrix containing primary and secondary chromium-rich carbides, with the morphology and distribution of these carbides being strongly influenced by the quenching medium.
- Water quenching resulted in a finer and more uniformly distributed carbide population due to the higher cooling rate, leading to a relatively homogeneous microstructure with lower carbide coarsening.
- Oil quenching promoted the formation of coarser carbides embedded within a predominantly martensitic matrix, accompanied by a higher retained austenite content, which contributed to higher microhardness values compared to water-quenched specimens.
- Quantitative image analysis using ImageJ software confirmed significant variations in carbide size, area fraction, and spatial distribution between the different quenching conditions, demonstrating the effectiveness of digital image processing as a reliable tool for microstructural characterization.
- A clear correlation was established between carbide morphology, microstructural heterogeneity, and microhardness, highlighting the critical role of quenching media selection in optimizing the mechanical performance of AISI D2 tool steel.

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