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Effect of carburizing time treatment on microstructure and mechanical properties of low alloy gear steels

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Abstract

Gas carburizing significantly enhances the surface properties of low-alloy gear steels, resulting in superior micro-hardness, layer thickness, carbon content, and overall mechanical properties. Unlike other thermochemical processes such as nitriding and carbonitriding, which have limitations in core properties and hardening depth, gas carburizing offers unmatched surface hardness, wear resistance, and mechanical strength. This makes it ideal for demanding applications in the automotive, aerospace, and manufacturing industries. In this research, samples were gas-carburized for 4, 6, or 8 h. The results showed significant improvements: micro-hardness increased from approximately 140 HV to over 819 HV, and the surface layer thickness grew by more than 41%, from 1166 μm to 1576 μm . Additionally, the carbon content in the surface layer increased by over 450%, reaching up to 0.94 wt%. Clear correlations were observed between the duration of heating and the mechanical properties. Longer heating times, particularly after 8 h, raised ultimate tensile strength from 427.29 MPa to 778.33 MPa, while simultaneously decreasing elongation from 26.07% to 2.88% and resilience from 180 J cm^{-2} to 6.66 J cm^{-2} . This optimization not only enhances surface hardness and durability but also improves key mechanical properties such as tensile strength, stiffness, resilience, and overall mechanical performance.

1. Introduction

The relentless pursuit of technological advancements across various industries has driven a constant demand for materials with exceptional mechanical properties. Low-alloy steels, with their cost-effectiveness and desirable mechanical properties, have become a mainstay in critical components like gears, shafts, bearings, and tools [1, 2]. However, their inherent limitations, such as low hardness, limited wear resistance, and susceptibility to fatigue, restrict their suitability for demanding applications where durability and reliability are paramount [3, 4]. While some low-alloy steels are suitable for specific applications, such as automotive clutch parts or chain components, they often lack the strength and resilience needed for high-performance applications that require resistance to wear, impact, and high stresses. These limitations highlight the need for innovative solutions to enhance the mechanical properties of low-alloy steels, particularly for applications demanding exceptional performance and durability [5–8].

Researchers and experts continuously explore advanced steel alloys and innovative heat treatment techniques to enhance steel performance for industrial applications. Studies have shown the potential of various heat treatment processes to improve mechanical properties. For instance, Calik found that heat treatment significantly enhanced hardness and mechanical properties in steels like AISI 1020, AISI 1040, and AISI 1060, particularly with increased cooling rates [9]. Similarly, Kini *et al* observed substantial improvements in hardness and impact strength in heat-treated EN 9 steel. Recent developments in heat treatment techniques have notably increased the popularity of low-carbon steel sheets, showcasing their potential as cost-effective and durable solutions. Kulkarni *et al* critically investigated the impact of different heat treatments on the mechanical properties and microstructural attributes of 0.18%–C steel [10]. Dewangan *et al* examined the altered mechanical behavior of heat-treated low carbon steel, focusing on changes in tensile strength, hardness, and microstructural properties [11]. Numerous similar studies have employed heat treatment processes to enhance steel properties, focusing on heat soaking time and temperature and analysing mechanical property variations and microstructural changes by heating steel samples at different temperatures and cooling rates [12–16].

Among these treatments, gas carburizing has gained considerable traction as an effective means of enhancing the mechanical properties of steel components [17, 18]. This heat treatment technique involves enriching the surface of the steel with carbon by exposing it to a carbon-rich atmosphere at elevated temperatures, typically ranging from 880 °C to 950 °C [19]. The diffusion of carbon into the steel's surface layer promotes the formation of a hard, wear-resistant case, while the core retains its original toughness and ductility [20, 21]. The effectiveness of gas carburizing in improving the mechanical properties of low alloy steels has been extensively studied and validated. Numerous researchers have reported significant increases in surface hardness, wear resistance, and fatigue life after subjecting steel components to this treatment [22, 23]. The carburized layer, which typically extends to a depth of 0.5 to 1.5 mm, exhibits a martensitic microstructure with a high concentration of carbon, leading to exceptional hardness values exceeding 60 HRC [24]. This hardened case effectively resists abrasive wear, corrosion, and surface deformation, thereby extending the service life of the treated components [25, 26].

Furthermore, the carburizing process can be tailored to achieve specific mechanical properties by carefully controlling various parameters, such as temperature, time, and carbon potential. Many studies have examined the influence of temperature variations on the carbonitriding process of low-carbon steels due to their significance in the manufacturing industry [27, 28]. Additionally, studies have shown that longer carburizing times can lead to increased case depth and higher carbon content at the surface, resulting in enhanced hardness and wear resistance [29, 30]. Conversely, shorter carburizing times might not allow sufficient carbon diffusion, leading to a less pronounced improvement in surface properties [31, 32]. The impact of holding time on the resulting microstructure, particularly the formation of martensite, bainite, or retained austenite, is also critical for the overall mechanical performance of carburized steel [33, 34]. Moreover, optimizing carburizing time with subsequent heat treatments, such as tempering, can further enhance toughness and reduce brittleness, achieving a desirable balance of properties for specific applications [35, 36].

Despite the well-established benefits of gas carburizing, the underlying mechanisms and their influence on the mechanical performance of low alloy steels are not fully understood. Specifically, the relationships between carburizing parameters, microstructural evolution, and the resulting mechanical properties require further investigation. A comprehensive understanding of these interrelated factors is crucial for developing tailored heat treatment processes that can effectively address the specific requirements of various industrial applications.

This study investigated the impact of different carburizing holding times (4–8 h) on low-alloy gear steels. The primary objectives were to optimize critical parameters such as micro-hardness, surface layer thickness, and carbon content. Additionally, this study examined the relationships between mechanical properties (ultimate tensile strength, Young's modulus, elongation, resilience) and their correlation with carburizing duration. Insights were also gained into the evolution of the steel's microstructure following carburization. By elucidating the intricate connections between processing parameters, microstructural changes, and the resulting mechanical performance, the study aims to optimize gas carburizing techniques. This could enable the tailoring of low-alloy steel properties to meet the diverse needs of various industrial sectors. Furthermore, the insights gained from this work have the potential to support the development of more robust and cost-effective manufacturing strategies, facilitating the production of high-performance steel components with enhanced properties. The novelties of this work include a detailed analysis of the microstructural transformations induced by varying carburizing times and their direct impact on mechanical properties. The study's findings contribute to a deeper understanding of how specific processing conditions can be manipulated to achieve desired performance characteristics, offering valuable guidelines for industrial applications.

Table 1. The chemical composition of low alloy gear steels according to the ASTM E415-17 standard.

C	Ni	Cu	Cr	W	Mo	S	P	Ti	AL	Pb	V	N	Fe
0.23	0.129	0.0979	0.0925	0.0549	0.0316	0.0128	0.0087	0.0074	0.0056	0.005	0.0037	0.0024	98.55

Table 2. Formulation of test input parameters.

Temperature (°C) (A)	Holding time (h) (B)	Carbon flow rate (%) (C)
920	4 / 6 / 8	1.2

2. Materials and method

2.1. Materials

The material used in this investigation is a low alloy steel specifically designed for gears, and its chemical composition is shown in table 1. The samples underwent a thermochemical treatment known as gas carburizing. This process was carried out on a dedicated line controlled by Axron-Swiss Process Control software. The line was composed of several components that contributed to the overall processing of the samples.

The process began with carburizing, where the sample surfaces were enriched with carbon. Gas carburizing was employed, which involved exposing the samples to a carbon-rich atmosphere. This allowed carbon atoms to diffuse into the outer layer of the material, enhancing its hardness and wear resistance. Following carburizing, the samples underwent a diffusion step for a duration of 90 m. This step was crucial for ensuring the uniform distribution of carbon throughout the surface of the workpiece. It allowed the carbon to penetrate deeply into the material, resulting in consistent hardness and other desirable material properties.

Next, the samples were subjected to quenching treatment. Quenching is a rapid cooling process that ‘freezes’ the desired material structure achieved during carburizing and diffusion. In this case, the samples were quenched at a temperature of 830 °C for 20 m. The high temperature ensured the transformation of the material to a hardened state, significantly increasing its hardness. To further enhance the hardness and toughness of the samples, they underwent oil quenching. This involved immersing the samples in a tank of oil at a temperature of 60 °C for 18 m. Oil quenching provided a more controlled cooling rate, resulting in improved material properties such as increased hardness and reduced risk of distortion or cracking. After the oil quenching process, the samples were carefully dried for 25 m using a drying machine. This step aimed to remove any excess oil or moisture from the surface of the samples. Drying was essential for ensuring optimal cleanliness and preventing any potential negative effects on subsequent processes or evaluations.

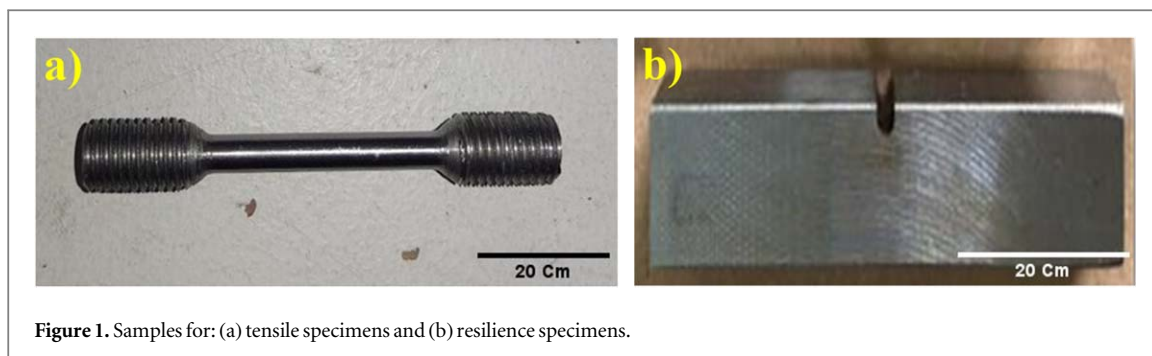
Finally, the samples were annealed. Annealing is a heat treatment process that involves heating the material to a specific temperature and holding it there for a designated period. In this case, the samples were annealed at a temperature of 180 °C for 120 m. Annealing helped relieve internal stresses, improve ductility, and enhance the toughness of the samples. It also allowed for the adjustment of the final microstructure and the development of desired mechanical properties.

Throughout the heat treatment process, a bell furnace of type 11-CG from SOLO-Swiss, Switzerland, was used. The furnace operated with a specific atmosphere consisting of a carrier gas with a composition of 39.36% H₂ (hydrogen), 0.23% CO₂ (carbon dioxide), 19.83% CO (carbon monoxide), and 0.76% H₂O (water vapor). The atmosphere was enriched with propane (C₃H₈) to create the desired conditions for the gas carburizing treatment.

The gas carburizing treatment applied to the samples was influenced by three main factors: Temperature (A), Holding Time (B), and Carbon Flow Rate (C). These factors were carefully controlled and adjusted to achieve the desired depth of carbon diffusion and ensure the desired material properties in the final samples. The specific values for these factors are likely listed in table 2. Figure 1 provides a detailed overview of the geometric shapes and dimensions of the test specimens prepared according to ASTM standards. Specifically, figure 1(a) illustrates the specimens used for tensile tests, while figure 1(b) depicts the specimens used for resilience tests.

2.2. Thickness measurement

The microstructure and dimensions of the samples under investigation were analyzed using a Leica DMI8 inverted microscope. Images of the samples were captured at various magnifications using Leica IM50 4.0 V software along with the AxioCam MRC 5 digital camera. In addition to studying the microstructure, the steel layer thickness of each sample was carefully measured at three different points. This rigorous approach was



adopted to ensure precise and dependable measurements, providing valuable insights into the samples' characteristics and properties.

2.3. Micro-hardness tester

The micro-hardness measurements were conducted in accordance with ASTM E384 standard, utilizing a Zwick Roell ZHV10-A micro-hardness tester, specially tailored for Vickers micro-hardness testing. A test load of 0.5 kgf was applied with a dwell time of 10 s, and the testing was carried out at room temperature for consistency. To accommodate variations in the material's microstructure, five hardness measurements were taken at various locations on each sample. This methodology yielded an average hardness value that faithfully represents the sample's hardness characteristics.

2.4. Chemical composition analysis

The chemical composition of the low alloy gear steels samples was identified using spark optical emission spectroscopy (S-OES) before and after the case hardening procedure. A SPECTROMAXx MX6M-BT metal analyzer from SPECTRO Analytical Instruments GmbH was used to perform the S-OES analysis. Throughout this investigation, the composition statistics are shown as a mass percentage. During the chemical analysis, the experiments were conducted at a pressure of 3 bar in an argon environment. The given values are the average of at least three measurements to assure accuracy and dependability.

2.5. Tensile tests

The tensile tests were conducted using a LaborTech LabTest model 5.100SP1-100 kN universal electromechanical testing machine, known for its precision in material testing. The machine features advanced software that facilitates customized test procedures and in-depth data analysis. Strain measurements were obtained using an extensometer with a gauge length of 25 mm, ensuring precise and reliable data throughout the testing process. The tests were performed at 23 °C, with the samples subjected to a uniaxial load at a fixed crosshead speed of 1.5 mm min⁻¹, maintaining a strain rate of 0.001 s⁻¹. To determine the repeatability and reliability of the results, three tensile tests were performed for each sample condition, and the average values were reported. The tests were conducted in accordance with ASTM standard E8/E8M, ensuring compliance with industry-recognized guidelines.

2.6. Charpy resilience tests

The Charpy impact test was conducted to assess the impact properties of the samples. The tests were executed utilizing a Hoytom 300 J machine, which is designed specifically for Charpy testing, following the ASTM E23 standard. Testing was performed at room temperature (23 °C). Three samples were tested for each test, and the toughness measurements were averaged to provide a more precise representation of the material's impact toughness characteristics.

3. Results and discussion

3.1. Analyses of carbon content

The surface layer of the low alloy gear steel initially had a relatively low carbon content, approximately $\alpha = (0.21 \pm 0.06)$ wt%, before carburization, which was consistent with the initial carbon amount of low alloy gear steels as shown in table 1. However, after undergoing carburization for different durations, the carbon content in the surface layer gradually increased. Specifically, after 4 h of carburization, the carbon content reached $\alpha = (0.83 \pm 0.016)$ wt% (β), after 6 h it reached $\alpha = (0.88 \pm 0.012)$ wt% (γ), and after 8 h it reached $\alpha = (0.94 \pm 0.030)$ wt% (δ), as depicted in figure 2.

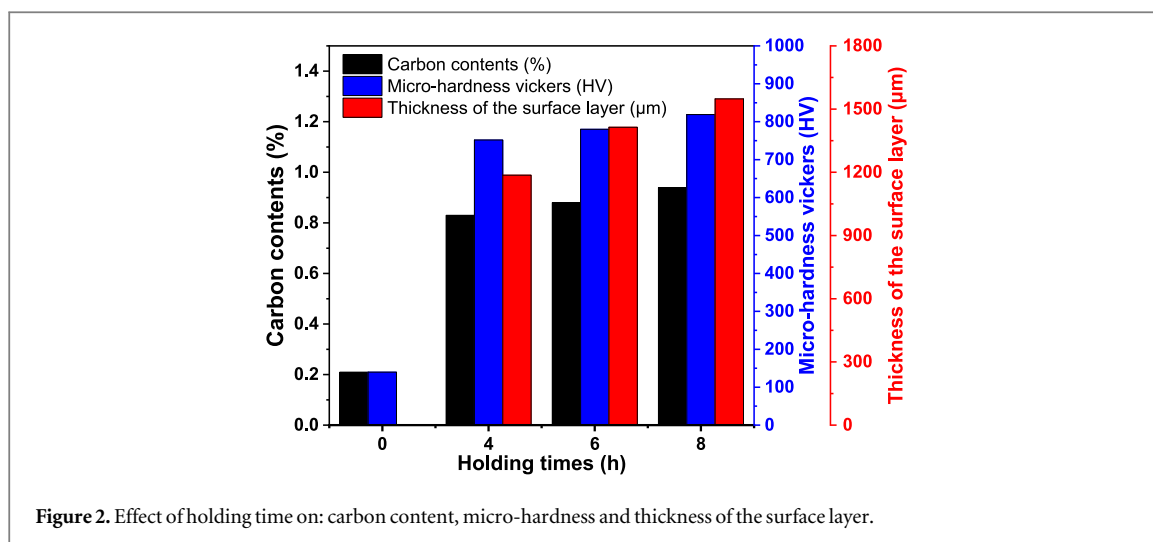


Figure 2. Effect of holding time on: carbon content, micro-hardness and thickness of the surface layer.

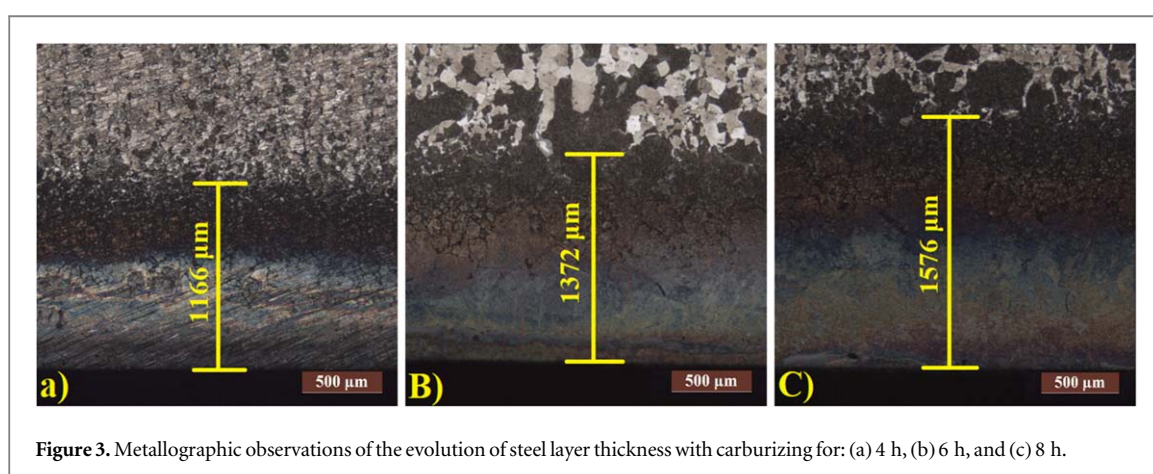


Figure 3. Metallographic observations of the evolution of steel layer thickness with carburizing for: (a) 4 h, (b) 6 h, and (c) 8 h.

These results indicate a significant increase of over 450% in the carbon content with longer holding time during the carburization process. These findings are consistent with the results reported in the literature [37, 38]. The observed increase in carbon content can be attributed to the diffusion of carbon, influenced by both the rate of absorption and the duration of carburization. This diffusion process facilitates a reaction with the iron in low alloy gear steel, leading to the formation of a new carbon-based phase. A similar trend of increased carbon content in the surface layer of Fe-C-Mn steels with prolonged carburization time was reported by Boubaaya *et al* [39].

The progressive enhancement of carbon content is significant as it allows for the customization of surface properties in low alloy gear steel. Carburization's formation of a hardened layer strongly suggests potential improvements in certain mechanical properties. Furthermore, the controlled transformation of the microstructure during carburization enables the development of desired phases such as martensite or pearlite, further enhancing the material's performance under various loading conditions. These findings are valuable in optimizing carburization parameters to achieve specific surface characteristics tailored to industrial applications.

3.2. Microstructural analysis

After subjecting low alloy gear steels to carburizing treatment, a substantial increase of over 41% was observed in the thickness of the surface layer as the holding time increased. The thickness increased from approximately 1166 μm to more than 1576 μm , as shown in figure 3. These results align with the findings reported by Abdenour *et al* and Chen *et al* [38, 40].

The increase in thickness indicates the successful diffusion of carbon into the surface layer during the carburizing process. As carbon diffuses into the steel, it combines with the iron to form a new carbon-based phase, resulting in an increased layer thickness. This increased thickness is desirable because it strongly suggests that certain mechanical properties could improve, which are crucial for low alloy gear steels used in demanding applications.

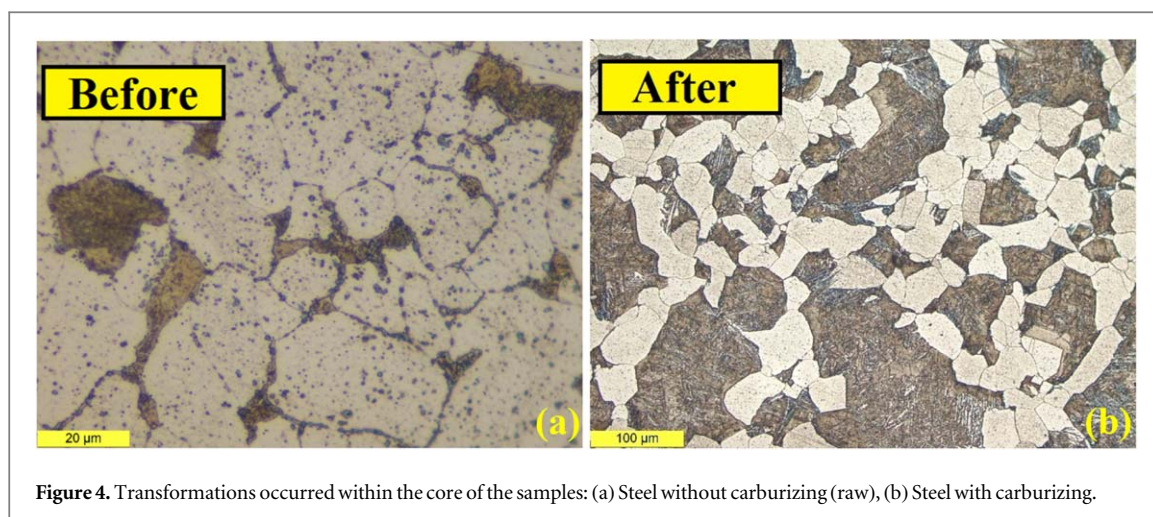


Figure 4. Transformations occurred within the core of the samples: (a) Steel without carburizing (raw), (b) Steel with carburizing.

Figure 3 depicts the enrichment of carbon content in the surface layer of the low alloy gear steels, leading to the transformation of austenite into martensite and the eventual formation of cementite (Fe_3C). This transformation in the microstructure is consistent with previous studies [41, 42], indicating the effectiveness of the carburizing treatment in altering the steel's properties.

Furthermore, significant transformations occur within the core of the samples during the carburization process. The initial ferrite and pearlite phases undergo changes, transforming into retained austenite and bainite, as shown in figure 4. These transformations in the core further enhance the mechanical properties and performance of the low alloy gear steels, making them more suitable for demanding industrial applications [43].

Indeed, the observed increase in the thickness of the surface layer and the transformations in the microstructure provide strong evidence for the effectiveness of the carburizing treatment in enhancing the properties of low alloy gear steels. These enhancements significantly improve the mechanical characteristics and overall performance of the material. The carburization process offers valuable benefits for optimizing the properties of low-carbon steels, making them more suitable for a wide range of industrial applications [44, 45].

3.3. Micro-hardness analysis

After subjecting low alloy gear steels to carburizing treatment, a remarkable increase in micro-hardness was observed with higher holding time. The micro-hardness elevated from approximately 140 HV to more than 819 HV, as shown in figure 2. The micro-hardness profiles obtained before and after different carburization times exhibited distinct patterns. For the untreated low alloy gear steels, the hardness remained constant at approximately 133 ± 2 HV both at the edge and the core. However, after carburization, a clear distinction in hardness was observed. The first zone corresponds to the carburized layer, which exhibited the highest surface hardness. The hardness values were approximately (759 ± 9) HV after 4 h of treatment, (789 ± 13) HV after 6 h, and (819 ± 14) HV after 8 h.

The elevated hardness values in the carburized layer are attributed to the quenching effect resulting from the diffusion of a substantial amount of carbon into the austenitic phase. This diffusion triggers the transformation of austenite into martensite and the formation of hard cementite (Fe_3C). These findings highlight the significant increase in micro-hardness achieved through carburizing treatment, making the surface layer more resistant to wear and mechanical stresses, according to Duan *et al* [44].

3.4. Tensile analysis

Mechanical tensile tests on low-alloy gear steel samples subjected to gas carburization treatment revealed significant correlations between mechanical properties and holding time. The ultimate tensile strength (σ), Young's modulus (E), and elongation (Δl) demonstrated distinct relationships with the duration of carburization, as depicted in figure 5. Over carburization durations of 4, 6, and 8 h, the ultimate tensile strength (σ) increased significantly from 427.29 MPa (without carburization) to 778.33 MPa (carburized for 8 h), representing an increase of approximately 82.14%. This substantial enhancement in the material's tensile strength underscores the efficacy of the carburization process. Simultaneously, Young's modulus (E) increased from 202.14 GPa (without carburization) to 207.11 GPa (carburized for 8 h), reflecting an increase of over 2.45%. This increase indicates that the carburized material has greater stiffness and rigidity, contributing to its improved mechanical performance.

Conversely, the elongation (Δl) exhibited a marked decrease from 26.07% (without carburization) to 2.88% (carburized for 8 h), indicating a reduction of about 88.95%. This reduction in elongation suggests a significant

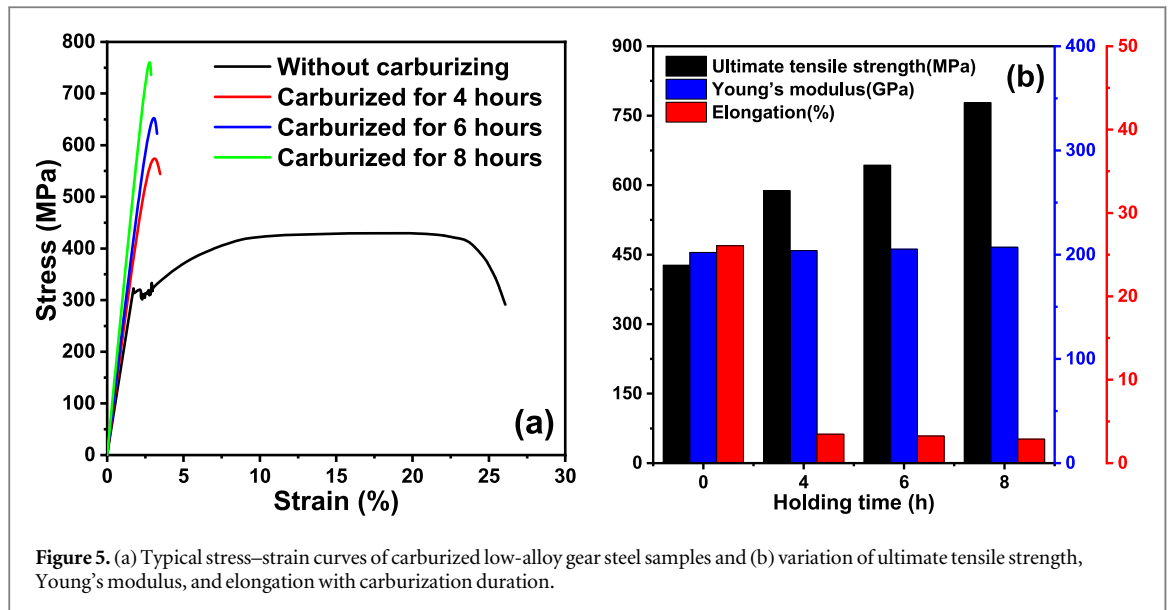


Figure 5. (a) Typical stress–strain curves of carburized low-alloy gear steel samples and (b) variation of ultimate tensile strength, Young's modulus, and elongation with carburization duration.

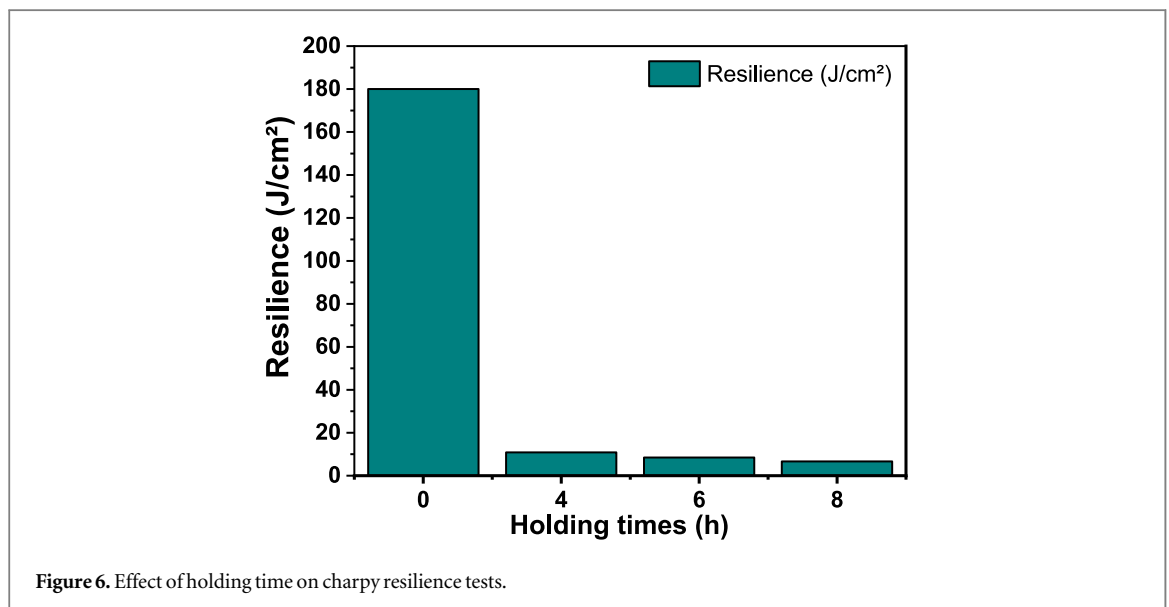


Figure 6. Effect of holding time on Charpy resilience tests.

decrease in the material's ductility, which aligns with the observed increase in hardness and strength resulting from the carburization process. These trends underscore the influence of carburization duration on the mechanical properties of low-alloy gear steels. The observed increase in ultimate tensile strength and Young's modulus indicate improved strength and stiffness, whereas the decrease in elongation reflects reduced ductility. These findings are consistent with the results reported by Sarkar *et al* and Dewangan [21, 34].

3.5. Analysis of Charpy resilience tests

Impact tests conducted on low alloy gear steels subjected to gas carburization treatment revealed an inverse relationship between resilience and holding time. As the duration of carburization increased from 4 to 8 h, the resilience of the samples decreased from 180 J cm^{-2} in the as-received state to 10.83 J cm^{-2} (carburized for 4 h), 8.43 J cm^{-2} (carburized for 6 h), and 6.66 J cm^{-2} (carburized for 8 h), and the results are displayed in figure 6. These findings are consistent with the research conducted by Saoudi *et al* and Hesham *et al* [38, 46].

The observed brittleness in the samples after carbon diffusion can be attributed to two primary factors. Firstly, the increased depth of the carburized surface layer contributes to the reduction in resilience. The deeper layer, enriched with carbon and containing solid solution-strengthening phases such as martensite and cementite, alters the material's ability to absorb and dissipate impact energy effectively. Secondly, the formation of these solid solution-strengthening phases, particularly martensite and cementite, introduces numerous sites where cracks can initiate along the interface of cementite. This microstructural change further contributes to the brittle fracture behaviour observed in the samples [38].

4. Conclusion

Gas carburizing treatment has proven to be an effective thermochemical method for enhancing the surface properties and mechanical characteristics of low-alloy gear steels. The extended holding time during carburization, ranging from 4 to 8 h, had a profound influence on the experimental outcomes:

- Surface hardness increased significantly, by approximately 485%, from around 140 HV to over 819 HV, demonstrating a substantial improvement.
- The carbon content in the surface layer augmented by over 450%, reaching up to 0.94 wt%, greatly enhancing the steel's durability.
- The thickness of the carburized layer increased by more than 41%, from 1166 μm to 1576 μm , providing a thicker, more durable hardened surface suitable for harsh and demanding applications.
- Ultimate tensile strength increased by approximately 82%, from 427.29 MPa to 778.33 MPa. Additionally, Young's modulus increased by approximately 2.45%, from 202.14 GPa to 207.11 GPa, reflecting enhanced stiffness and rigidity.
- After 8 h of treatment, elongation decreased from 26.07% to 2.88%, indicating increased brittleness with prolonged carburizing times. This change highlights the trade-off between enhanced hardness and reduced ductility.
- An inverse relationship was observed between resilience and holding time, with resilience decreasing from 180 J cm^{-2} (without carburizing) to 6.66 J cm^{-2} (after 8 h of carburizing).
- Significant microstructural transformations were observed, with the enrichment of carbon leading to the conversion of austenite to martensite and the formation of cementite. The core of the samples saw initial ferrite and pearlite phases transform into retained austenite and bainite, further enhancing the mechanical properties and performance of the steel.

These findings underscore the effectiveness of gas carburizing in significantly enhancing the performance of low-alloy gear steels. The process not only boosts surface hardness and durability but also improves key mechanical properties such as tensile strength, stiffness, resilience, and overall mechanical performance. Consequently, carburized low-alloy gear steels are well-suited for challenging applications in the automotive, aerospace, and manufacturing sectors.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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