

The influence of heat treatment on mechanical, thermal, and structural properties of AISI D2 steel

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Abstract

This study examines the effects of air quenching and tempering at different temperatures on the mechanical, thermal, and structural properties of AISI D2 tool steel. The steel samples were austenitized at 1030°C for 30 minutes, air quenched and then tempered at various temperatures. Following each heat treatment, the mechanical properties were assessed by measuring the hardness of the samples. Thermal properties were determined using the Xenon flash method, and structural changes were analyzed using SEM-EDS. The results indicate that the hardness values were highest after quenching, while the thermal diffusivity and thermal conductivity values were lowest in this state. As the tempering temperature increased, the hardness values decreased while thermal diffusivity and conductivity values gradually increased.

Key words: AISI D2 steel, air quenching, thermal properties, tempering, hardness

1. Introduction

AISI D2 steel is a cold work type of tool steel containing high carbon and chromium concentrations. This type of steel has excellent dimensional stability during heat treatment, which is essential for manufacturing high-precision tool dies, drawing punches, lamination, stamping dies, extrusion dies, etc. [1, 2]. Standards dictate the chemical composition of steels used in part manufacturing; thus, modifying the structure and properties can be achieved through various heat treatment processes. Therefore, different properties can be influenced by optimizing the heat treatment steps [3]. The common heat treatment practice for this steel is quenching followed by tempering. The quenching process gives extremely high values of hardness but poor toughness behavior [4–6]. Under load, this behavior can cause the appearance of cracks, even tearing off the surface of the die [7]. To reduce brittleness and enhance toughness, among other properties, parts are subjected to a tempering process, preferably immediately after the quenching. In the tempering phase of this steel, significant amounts of secondary carbides form due to the high carbon content and presence of other alloying elements [8–10].

Appropriate heat treatment procedures are key to achieving a high-quality product when using cold working steels for tool and die manufacturing. Modifying quenching parameters and the temperature and time of the tempering process directly influences the dislocation density, quench stresses, and formation of said carbides, which in turn changes the values of hardness, toughness, thermal diffusivity, thermal conductivity, etc. [11]. Because of this, numerous authors have studied the impact of heat treatment on various properties of AISI D2 steel [1, 2, 4, 7, 11–15].

H. Torkamani et al. investigated the influence of quenching in KOH and NaOH molten salt baths on the mechanical and structural properties of AISI D2 steel. The authors showed that quenching in molten salt baths does not produce microcracks, as is the case with quenching in standard quenching oil. Also, after hardening in molten salt baths, a better and more uniform arrangement of carbides occurs, as well as the appearance of a smaller amount of residual austenite. Therefore, the samples quenched in molten salt baths have higher tensile strength, impact energy, and hardness values than those quenched in oil [4]. The other important parameter when quenching is austenitization. S. Salunkhe et al. [2] examined the possibility of

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applying double austenitization before quenching to obtain superior mechanical and structural properties. The authors achieved higher hardness values while almost maintaining the austenite grain size when they used the double austenitization process, compared to the conventional single austenitization process. The improvements observed after double austenitization were attributed to better carbide dissolution in the matrix [2]. V. Marušić et al. investigated the influence of tempering temperature on the hardness, toughness, and appearance of cracks in X155CrVMo12-1 tool steel. They concluded that this steel should be austenitized at 1030 °C before quenching and tempered at around 400 °C. With these heat treatment parameters, the authors obtained the highest toughness values (18 J), while the hardness values remained relatively high (56 HRC) [7].

Thermal diffusivity and thermal conductivity are the most important properties to determine when investigating tool steels. These properties dictate the heat accumulation and dissipation throughout the manufactured part. Knowledge of the changes in thermal properties, particularly with temperature variations, is crucial when considering the productivity, quality, and lifespan of different tools or dies. AISI D2 tool steel is often used to make injection dies to obtain complex shapes. The materials are injected into the die under high pressure and at elevated temperatures (operating temperatures sometimes reach 500 °C). The constant heating and cooling of the die during the production process affects its productivity cycle, i.e., its lifespan. So, it is important to know the change in thermal properties, especially at the operating temperatures.

Some researchers have focused their research on investigating the influence of heat treatment on the thermal properties of different steels. J. Wilzer et al. studied the impact of heat treatment on the thermophysical properties of martensitic steels and found that tempering leads to an increase in thermal conductivity and thermal diffusivity but causes a decrease in mechanical properties [16]. In this regard, N. A. Guru et al. investigated the mechanical and thermal properties of AISI D2 steel, which was additionally alloyed with copper to increase thermal conductivity. The authors showed that adding copper changes the structure from pure martensite to pearlite-martensite. The authors stated that due to this change, hardness values decreased while thermal conductivity values rose as much as 28 % after adding 2 wt.% copper [11].

After analyzing the literature, it was discovered that there isn't enough information on how heat treatment affects the mechanical and thermal properties of AISI D2 tool steel after the air quenching and tempering. Most researchers often focus their investigation on the influence of tempering after quenching in oil. However, due to the high hardenability of this

steel, with air quenching and tempering, it is possible to achieve the required properties. Moreover, this quenching method is the most environmentally friendly and cost-effective option.

This research aims to expand the understanding of the impact of heat treatment on the mechanical, thermal, and structural characteristics of AISI D2 tool steel. The heat treatment included normalizing, austenitizing, air quenching, and tempering at different temperatures. After each step in the heat treatment process, mechanical, thermal, and structural properties were investigated. The analysis examined how the tempering affects the thermal properties at temperatures up to 400 °C, given that this type of steel is utilized for cold work within this temperature range. Additionally, we focused on minimizing the duration of all heat treatment processes. We aimed to reduce heat treatment times while considering economic and environmental factors. This is important because shorter heat treatment processes require less energy, making them more cost-effective.

2. Experimental part

A hot extruded round bar of AISI D2 tool steel was used for this experiment. The chemical composition complies with standard EN ISO 4957-2:2018 [17].

In the first step of the heat treatment process, all samples were placed in the Vims Elektrik LPŽ-7,5 S electric resistance furnace at 700 °C and gradually heated to 900 °C. The samples were then held at 900 °C for 20 minutes and slowly cooled to room temperature in the furnace. This step was done to remove the original structure of the samples. The resulting annealed condition was used as a reference state and marked on all graphs appropriately. In the second step, the samples were placed in a furnace at 1030 °C and austenitized for 30 minutes. They were then quenched to room temperature using forced (compressed) air. After quenching, the samples were tempered at 50 to 700 °C for 20 minutes. Finally, the samples were cooled in the open air.

After each step of the heat treatment process, samples were extracted and investigated using various experimental techniques. These techniques included thermal diffusivity and conductivity measurements, hardness measurements, and microstructure investigation.

2.1. Measurements of thermal diffusivity and thermal conductivity

The thermal diffusivity was measured using the Xenon flash method. This process included exposing disc-shaped samples (12.7 mm in diameter) to a flash of the Xenon lamp in a nitrogen environment. The

Table 1. Values of hardness, thermal diffusivity, and thermal conductivity after the annealing heat treatment

Hardness, HV ₃₀	Thermal diffusivity (mm ² s ⁻¹)	Thermal conductivity (W m ⁻¹ K ⁻¹)
241	8.37	31.31

thermal conductivity was subsequently determined using the provided equation:

$$\lambda(T) = \rho(T) \times c_p(T) \times \alpha(T), \quad (1)$$

where λ is thermal conductivity (W m⁻¹ K⁻¹), ρ is density (kg m⁻³), c_p is specific heat capacity (J kg⁻¹ K⁻¹), α is thermal diffusivity (m² s⁻¹), and T is temperature (K).

Thermal properties were measured in two ways:

1. The quenched sample was continuously heated in a protective atmosphere from room temperature (25 °C) to 400 °C, with thermal properties recorded at various elevated temperatures (25, 50, 100, 200, 300, and 400 °C).

2. Thermal properties were measured at room temperature (25 °C) after the samples underwent various heat treatment processes and were subsequently cooled to room temperature for measurement.

2.2. Hardness measurement

Vickers hardness was determined using a VEB Leipzig hardness tester under a 30 kg load and a 15-second dwelling time by the ASTM E384-22 standard [18]. Multiple measurements were conducted, and the average value was determined.

2.3. Microstructure investigation

Following the heat treatment, the samples were prepared for microscopy by grinding on waterproof SiC papers and polishing using two differently granulated alumina slurries (0.3 and 0.05 µm). Samples were etched with a 4 % Nital solution to reveal the microstructure. The microstructures were examined using light optical microscopy (LOM) on a Carl Zeiss Jena EpyTip 2 microscope. Additionally, certain samples were further analyzed using a Tescan Vega scanning electron microscope with an X-ray EDS detector from Oxford Instruments.

3. Results and discussion

3.1. Results after annealing

Table 1 shows the hardness, thermal diffusivity,

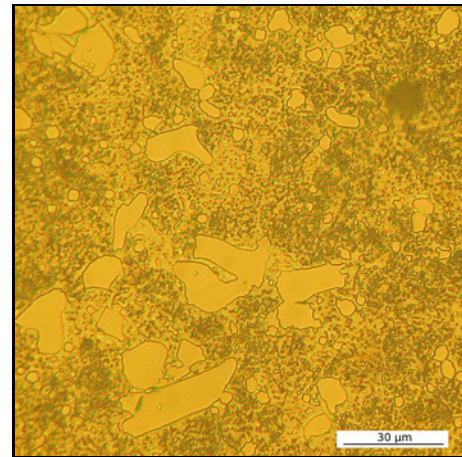


Fig. 1. The microstructure of annealed sample of the AISI D2 tool steel.

and thermal conductivity values after the annealing heat treatment.

From the presented results, it can be concluded that the hardness values are relatively low but expected to be obtained after this kind of heat treatment. Hardness values around 250 HV are also reported by other authors [12, 14]. On the other hand, the values of thermal diffusivity and thermal conductivity are relatively high. This is expected because the structure is composed of eutectic and secondary carbides distributed evenly in the ferritic matrix [12]. When the matrix is relaxed (due to the annealed state upon slow cooling), the flow of electrons, as heat carriers, is not disrupted, i.e., values of thermal properties are high.

The microstructure analysis in Fig. 1 confirms the presence of dispersed primary and secondary carbides in the ferritic matrix. Eutectic carbides (ECs) appear as large, irregularly shaped, bordered white areas, while secondary carbides (SCs) are usually far smaller in size than ECs and rounder, as shown. Other authors obtained similar microstructures after annealing [12, 14, 15].

3.2. Results after quenching

After the annealing heat treatment, all the samples were austenitized at 1030 °C for 30 minutes and quenched. As the first step in investigating the influence of heat treatment, the samples were subjected to DXF analysis. The quenched sample underwent continuous heating at 10 °C per minute within the temperature range of 25 to 400 °C, as shown in Figs. 2a–c. The purpose was to examine whether the sample exposed to this specific temperature range would exhibit any alterations in its thermal properties compared to the quenched state. The data will help determine whether it is reasonable to temper at this temperature

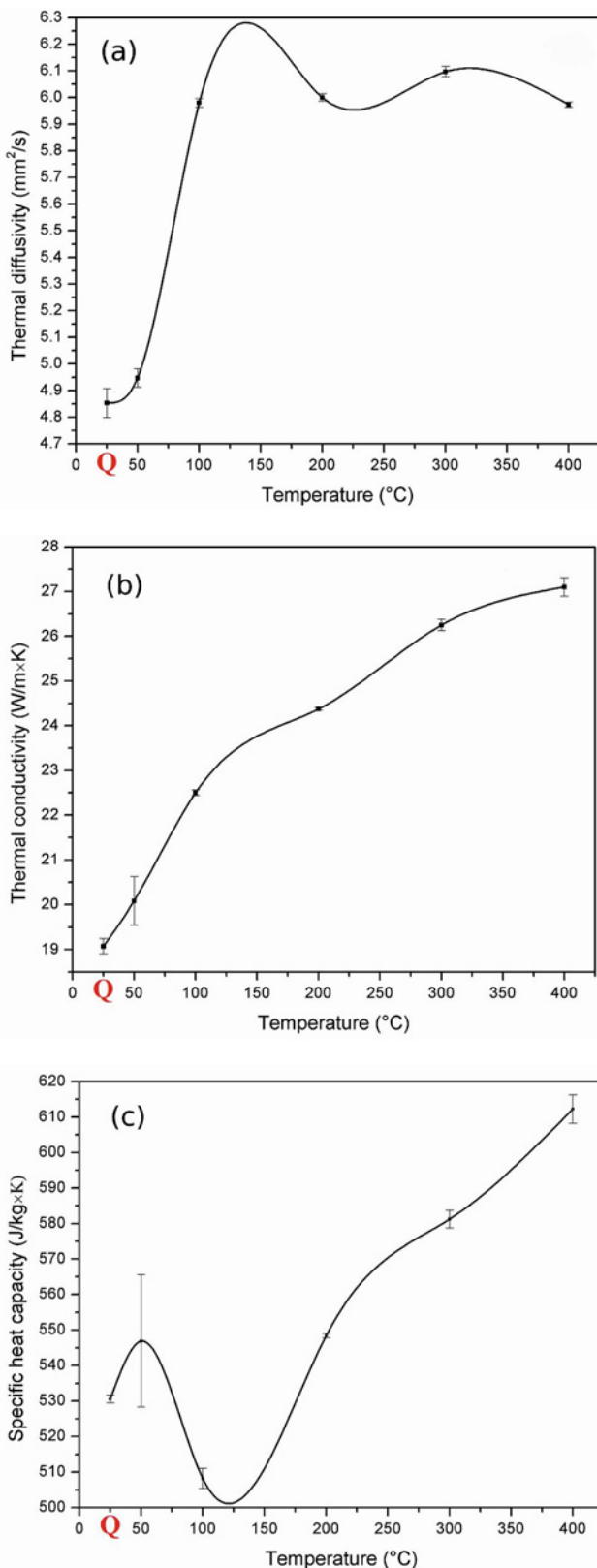


Fig. 2. The change in (a) thermal diffusivity, (b) thermal conductivity, and (c) specific heat of the AISI D2 steel sample continuously heated after quenching; Q – quenched state.

range later. Moreover, at this temperature range, tools are usually heated during the cold-forming process, so it is crucial to comprehend how tools perform at this temperature range.

The graphs presented in Fig. 2 indicate that the thermal diffusivity, thermal conductivity, and specific heat values are usually higher when compared to the quenched state (noted as Q in Fig. 2). After heating the quenched sample, the thermal diffusivity values can either increase or decrease, depending on the scattering of heat carriers, primarily electrons. Essentially, there are two processes that exhibit different behavior. On the one hand, as the temperature rises, increased vibrations in the lattice lead to greater scattering of electrons and phonons, which lowers the thermal diffusivity values. Conversely, as the sample temperature increases during testing, the precipitation of carbon atoms and alloying elements from the matrix begins. This results in the martensite lattice becoming increasingly desaturated. In a desaturated lattice, electrons can move more freely, resulting in a net increase in electron movement, which raises the thermal diffusivity values. Thus, whether the thermal diffusivity values increase or decrease depends on which of these two processes dominates at a given temperature. In this instance, thermal diffusivity values tend to increase gradually with rising temperature measurements, except at 400°C. When examining alloy steels, it is common for specific heat values to rise, as noted by S. Hafenstein et al. [19]. According to the results in Fig. 2c, specific heat values gradually increase with temperature measurements, except at 100°C, where a sudden decrease occurs. This drop can likely be attributed to measurement error.

Equation (1) states that thermal conductivity equals the product of density, thermal diffusivity, and specific heat. Therefore, the variation of thermal conductivity with temperature depends on all these factors. The graph in Fig. 2b indicates that thermal conductivity values gradually increase with temperature. This is in agreement with Eq. (1) because values of specific heat and thermal diffusivity increase, and it can be assumed that density values increase with temperature based on the data given by J. Wilzer et al. [16]. Furthermore, the desaturation of the martensite also adds to the increase in thermal conductivity values [20]. N. A. Guru et al. obtained somewhat similar results for the thermal conductivity of the quenched AISI D2 steel sample [11].

3.3. Results after tempering

The following section presents the results on how tempering affects the structural, mechanical, and thermal properties of the quenched samples. The samples were tested at room temperature after undergoing heat treatment.

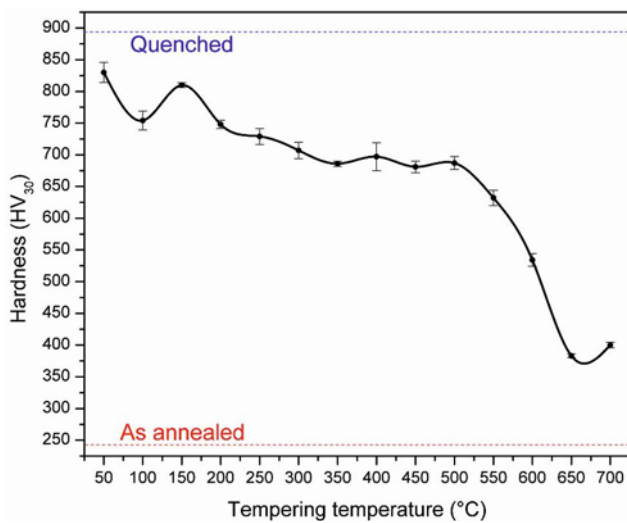


Fig. 3. Change in hardness values of AISI D2 steel after quenching and tempering at different temperatures.

The graph in Fig. 3 shows how hardness changes with tempering temperature after air quenching. The first thing to notice is the influence of the quenching process (comparison of the dotted lines). When comparing the hardness values of the annealed sample with those obtained after quenching, it is clear that the hardness almost quadruples. This demonstrates the high hardenability of this steel and shows that quenching (martensite formation) is possible using air as the quenching medium. The creation of martensite causes an increase in the number of dislocations and twins while simultaneously reducing the number of slip systems. Consequently, the hindrance of dislocations leads to significant strengthening [21]. After quenching, tempering was performed at a temperature range from 50 to 700 °C. Tempering has been found to gradually reduce hardness values as the temperature increases due to structural changes. H. Torkamani et al. [4] stated that tempering has two primary effects on the material structure. Firstly, hardness decreases with tempering temperature due to recovery caused by reduced dislocation density and quench stresses. Secondly, tempering leads to the transformation of residual austenite to martensite and the formation of SCs, which block dislocations, thereby increasing hardness [4, 8, 22–27]. The graph (Fig. 3) illustrates that high hardness values are maintained even at relatively high tempering temperatures (550 °C), after which they decrease rapidly. Other authors have reported similar results [2, 4, 7, 14, 15, 28]. The retention of high hardness values is attributed to the uneven distribution of coarse SCs in the matrix, hindering dislocation movement. Although hardness values generally decrease with increasing tempering temperature, there are some variations in hardness values up to 500 °C. As P. Jurči et al. [26] noted, the tempering of steels,

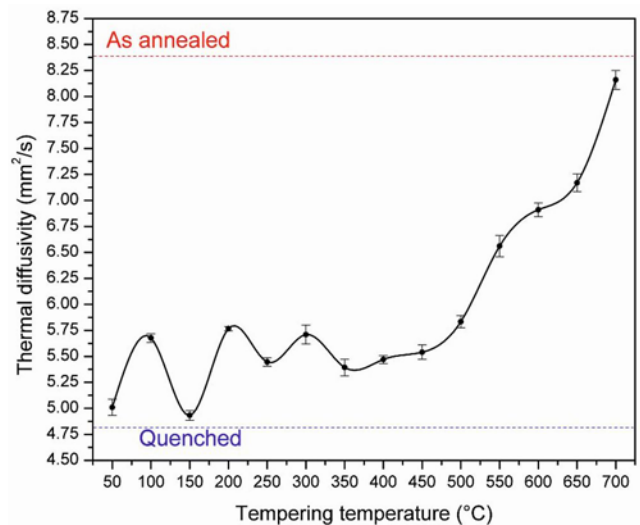


Fig. 4. Change in thermal diffusivity values of AISI D2 steel after quenching and tempering at different temperatures.

particularly ledeburitic steels, is quite complex. As a result, small variations are expected due to factors such as the uneven distribution of secondary carbides, the transformation of retained austenite upon quenching, and the amount of tempered martensite present in the microstructure. These factors can either increase or decrease hardness values at any given moment during tempering.

The peak associated with secondary hardening, expected to occur around 500 °C for this steel grade, is not visible in Fig. 3. This absence may be attributed to an uneven distribution of secondary carbides and insufficient precipitation during the heat treatment. Furthermore, inadequate temperatures and durations for the austenitization phase could have negatively impacted the development of secondary hardening due to uneven quenching of the tested samples.

With the increase in tempering temperature, SCs precipitate more from the saturated martensite matrix, resulting in a more uniform distribution due to enhanced diffusion. Consequently, dislocation movement becomes easier, leading to a reduction in hardness values.

The effect of tempering temperature on the thermal characteristics of AISI D2 steel at room temperature was also examined.

The change in thermal diffusivity and thermal conductivity values as a function of tempering temperature is given in Figs. 4 and 5, respectively. The results demonstrate that thermal conductivity and diffusivity follow a similar pattern. The presented graph shows that the lowest value for thermal diffusivity and thermal conductivity is measured after quenching, while the highest is obtained after annealing. This is expected because quenching results in the formation of

martensite, a supersaturated solid solution of carbon and alloying elements within the crystal lattice of iron. Considering that the thermal properties' values are influenced by the motion of electrons and phonons, which are responsible for carrying thermal energy, introducing alloying elements into the lattice results in their dispersion, causing a decrease in the thermal diffusivity and conductivity values [29, 30].

From the presented graphs in Figs. 4 and 5, it can also be concluded that the thermal diffusivity and conductivity values gradually increase with the rise in tempering temperature compared to the quenched sample. The samples tempered at the highest tempering temperature (700 °C) showed the highest values for thermal diffusivity and thermal conductivity. Essentially, the processes responsible for the decrease in hardness values also cause the increase in thermal diffusivity and thermal conductivity values. Desaturation of the super-saturated matrix due to tempering reduces scattering and enhances the movement of electrons, thereby increasing the thermal properties' values [19]. J. Wilzer et al. [16] also confirmed that tempering increases the thermal conductivity of carbon and alloy steels.

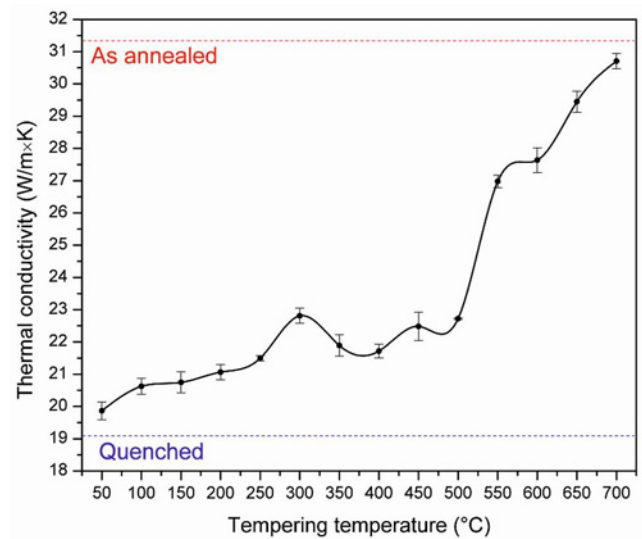


Fig. 5. Change in thermal conductivity values of AISI D2 steel after quenching and tempering at different temperatures.

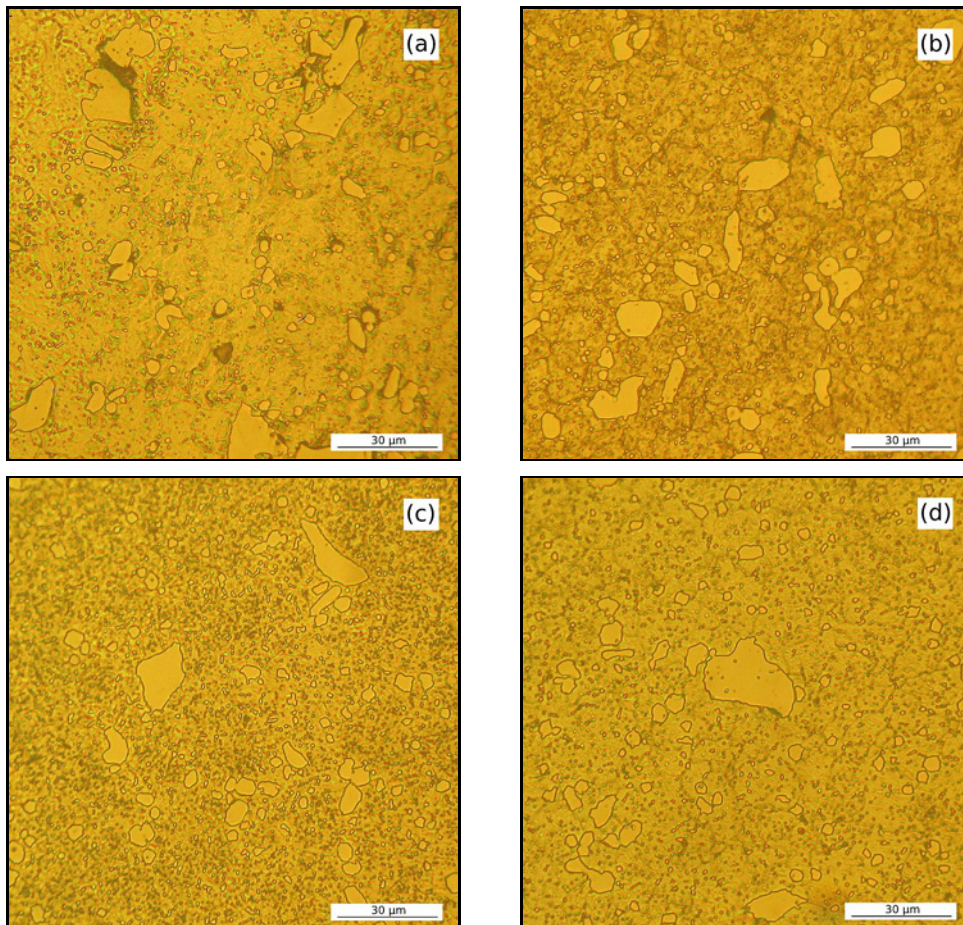


Fig. 6a–d. Microphotographs of the samples after (a) quenching and after tempering at (b) 50 °C; (c) 100 °C, (d) 150 °C.

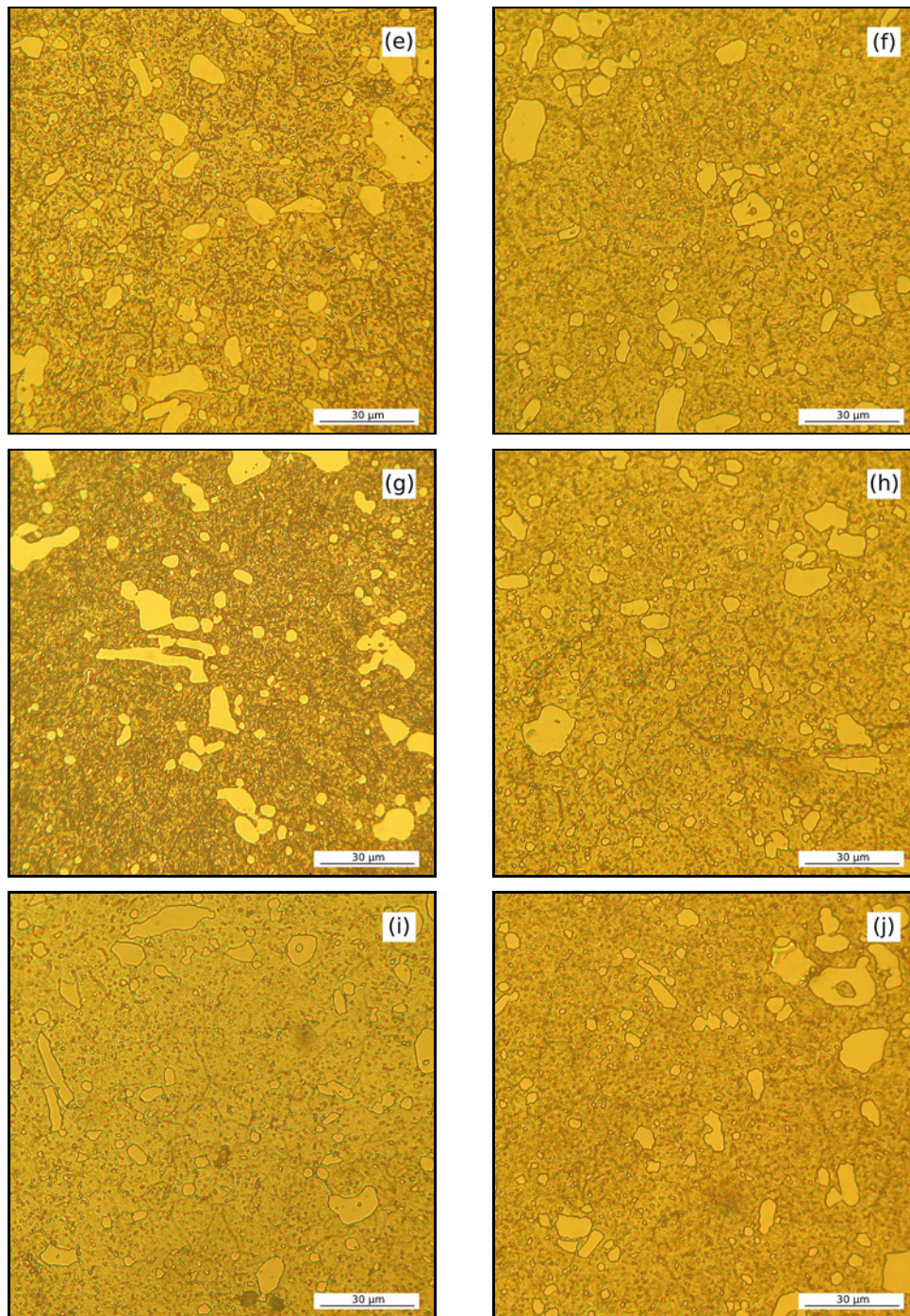


Fig. 6e–j. Microphotographs of the samples after tempering at (e) 200 °C, (f) 250 °C, (g) 300 °C, (h) 350 °C, (i) 400 °C, (j) 450 °C.

The variations in values of thermal properties due to tempering at temperatures up to 500 °C are depicted more clearly in Figs. 4 and 5. The complexities of the tempering process, which affect hardness values, are now even more apparent in the tempering diagrams for thermal diffusivity and conductivity. These properties are particularly sensitive to microstructural changes resulting from tempering. Tempering induces additional microstructural changes, including alter-

ations in mean interparticle spacing, spheroidization of carbides, and variations in the amount of carbides present, as noted by P. Jurči et al. [26]. It can be inferred that after tempering at higher temperatures (above 500 °C), these factors diminish the observed results; specifically, the values tend to rise consistently with increasing tempering temperature.

The increase in thermal diffusivity and conductivity values beyond 500 °C, without significant fluctu-

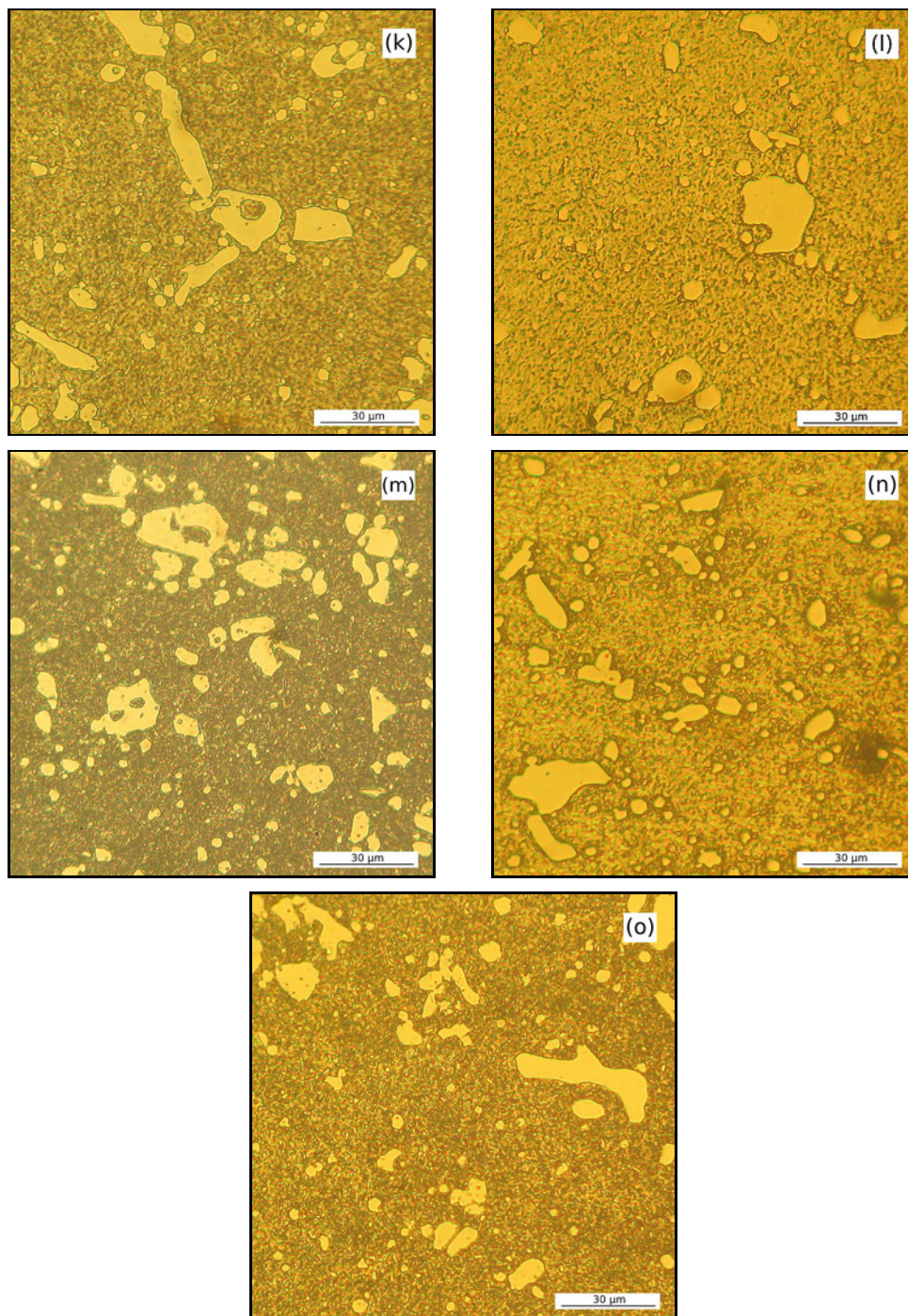


Fig. 6k–o. Microphotographs of the samples after tempering at (k) 500 °C, (l) 550 °C, (m) 600 °C, (n) 650 °C, and (o) 700 °C.

ations, can also be attributed to the increase in the volume of secondary carbides while the mean interparticle spacing probably remains constant. This stability facilitates even easier heat transfer between particles.

It is important to note that although there may be a slight decrease in hardness values due to tempering to around 500 °C, this can be more than compensated by the increase in thermal properties. This means that a manufactured part will retain its mechanical properties, such as hardness or wear resistance, while also ex-

periencing improved heat dissipation due to increased thermal diffusivity and conductivity values.

The microstructural analysis in Figs. 6a–o shows a change in the microstructures of the investigated samples and additionally confirms what was previously stated about the change in mechanical and thermal properties due to tempering.

The microphotographs reveal that all samples (quenched and tempered) still contain undissolved ECs. This is expected because a higher austenitization

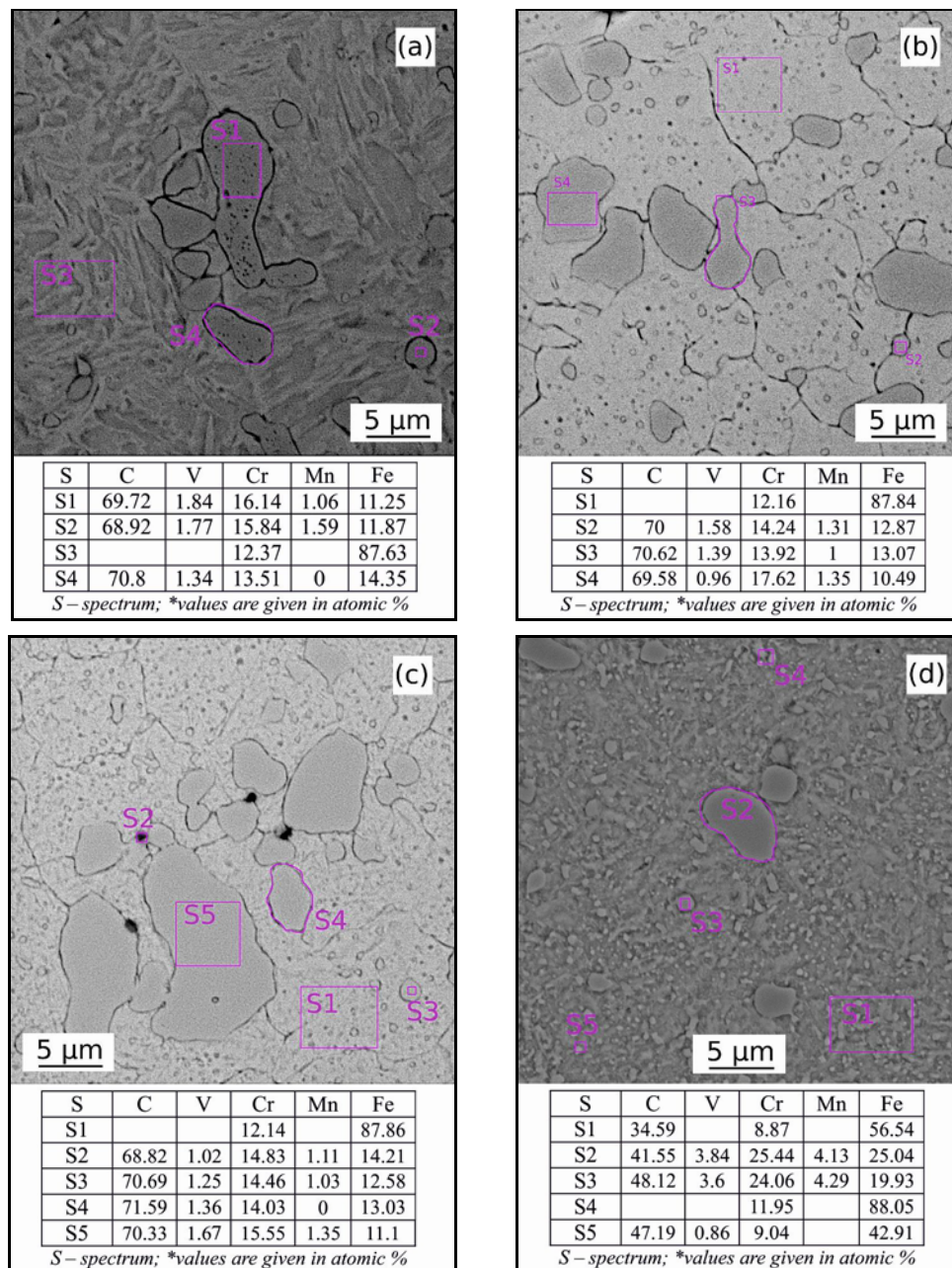


Fig. 7. SEM microphotographs with EDS analysis of samples (a) after quenching and after tempering at (b) 200°C, (c) 500°C, and (d) 700°C.

temperature and time are needed to dissolve the ECs, as noted by other authors [2, 31, 32]. The quenched sample structure (Fig. 6a) consists of martensite and dispersed carbides. The structures of the tempered samples (Figs. 6b–k) remained almost the same compared to the quenched structure [15]. When observed by optical and scanning electron microscopy, the tempering process within this temperature range leads to only subtle structural changes. For a more thorough analysis, it is crucial to examine the samples using a transmission electron microscope or by a similar investigation. These subtle changes are also reflected

in hardness, thermal diffusivity, and thermal conductivity values (see Figs. 3–5). Tempering above 550°C leads to more noticeable structure changes (Figs. 6l–o). At this temperature range, there is a greater presence of small SCs finely dispersed throughout the now finer martensite matrix. This is expected as tempering in this range leads to the formation of SCs and the transformation of coarse martensite to a finer one, as mentioned by other authors [15, 26].

To thoroughly investigate the microstructures, four samples were selected for further analysis using a scanning electron microscope: a quenched sample and sam-

ples tempered at 200, 500, and 700 °C, respectively. Figures 7a–d show SEM microphotographs with EDS analysis of the investigated samples.

The presented microphotographs further clarify the findings obtained from analyzing the optical microphotographs. The structure of the quenched sample (Fig. 7a) consists of coarse martensite, ECs, and SCs dispersed throughout the microstructure. The ECs appear large in the microstructures and contain carbon as well as alloying elements such as chromium, vanadium, and manganese (as indicated by spectra S1, S2, and S4 in Fig. 7a). The presence of SCs is minimal in the quenched sample, as they primarily appear after the sample undergoes tempering. Figures 7b–d illustrate the impact of tempering temperature on the structure. Following low-temperature tempering, the martensite appears less coarse and less visible in the microstructure (Fig. 7b). ECs remain prevalent, but there is a higher concentration of SCs, which are smaller and more rounded. Additionally, they tend to precipitate along the grain boundaries, as noted by M. D. Conci et al. as “grain boundary secondary carbide” [14]. EDS analysis confirms the presence of carbon and alloying elements, particularly chromium (Fig. 7b, spectra S2–S4). With increasing tempering temperature, the quantity of SCs should also increase, as observed in Figs. 7c and 7d. At the highest tempering temperature (700 °C, Fig. 7d), the structure most likely consists of very fine SCs within the nearly ferritic steel matrix. Due to extensive precipitation, the carbides are now even smaller and evenly distributed in the structure, while the number of ECs is minimal in this specific sample. Other authors have reported similar microstructures and conclusions [4, 14, 15].

4. Conclusions

The key findings of this research are outlined below:

- The AISI D2 steel can undergo significant changes in mechanical, thermal, and structural properties due to heat treatment.
- The annealed sample exhibited the highest thermal diffusivity and conductivity values and the lowest hardness values. The structure consisted of finely dispersed eutectic and secondary carbides in the ferritic matrix.
- Quenching with forced air has proven to be more than sufficient for this type of steel. The quenching in this way greatly influenced the structure’s mechanical and thermal properties.
- Hardness values increased significantly from 241 HV₃₀ for the annealed sample to 894 HV₃₀ after quenching, a 271 % increase. As a result, thermal diffusivity and conductivity values decreased by 42 and 39.1%, respectively.

– After quenching, an SEM investigation confirmed coarse martensite needles with dispersed carbides in the sample.

– The mechanical, thermal, and structural properties were also impacted by tempering. As the tempering temperature increased, the hardness values decreased progressively. Tempering the quenched sample at 650 °C resulted in the lowest hardness value, decreasing from 894 to 383 HV₃₀.

– As the tempering temperature increased, thermal diffusivity and thermal conductivity also gradually increased. The highest values for thermal diffusivity and thermal conductivity were achieved after tempering the quenched sample at the highest temperature. The increase in thermal diffusivity and conductivity compared to the quenched sample was 3.31 mm² s^{−1} and 11.64 W m^{−1} K^{−1}, respectively.

– After tempering, the SEM/EDS analysis revealed the presence of tempered martensite with eutectic and secondary carbides dispersed throughout the structure.

– This type of heat treatment produced positive results. When the steel samples were quenched in forced air, they achieved high hardness values of up to 893 HV₃₀. This shows that air quenching is an effective and cost-efficient way to obtain a martensitic structure. Even after tempering at 550 °C, the hardness values remained relatively high, with only a 29.31 % decrease from the quenched state. Additionally, there was a significant improvement in thermal properties, with a 35.26 % increase in thermal diffusivity and a 41.46 % increase in thermal conductivity. These improvements will enhance heat dissipation during tool operation, potentially extending the tool’s lifespan and increasing productivity. Additionally, minimizing the duration of all heat treatment processes reduces the energy requirement, making the process more cost-effective.

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