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Analysis of the rapid tempering treatment on a high-strength boron steel in the martensitic state through physical simulation

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In recent years, high-strength boron steels have become increasingly important in the automotive industry, particularly for structural components such as the B-pillar. These steels, when in a martensitic state, provide excellent safety performance while enabling the use of thinner sheets, which contributes to reducing the overall weight of vehicles. However, the inherent brittleness of martensite poses difficulties during the mechanical assembly process with other car body parts. One potential solution to enhance ductility is the application of rapid tempering, which is also valued for its lower energy consumption when compared to conventional tempering treatments. This study focuses on analyzing the softening behavior and associated microstructural evolution of martensitic 37MnB4 steel when subjected to rapid tempering cycles. Three distinct heating rates (10K/s, 100K/s, and 250K/s) and two holding times (0 s and 8 s) were considered. The findings indicate that rapid tempering leads to notable softening of the steel, with the highest degree of softening observed at tempering temperatures close to, specifically a few dozen degrees higher than, the eutectoid point at equilibrium, depending on both the heating rate and the holding time applied.

KEYWORDS: RAPID TEMPERING, ADVANCED HIGH-STRENGTH STEEL, MARTENSITE, PHYSICAL SIMULATION;

INTRODUCTION

The automotive sector, driven by the growing demand for energy efficiency, is increasingly utilizing advanced high-strength steels, particularly for the production of structural components. These materials provide excellent mechanical strength at acceptable ductility, effectively meeting safety requirements. Additionally, their high strength allows for the use of thinner blanks, contributing to the overall weight reduction of vehicles. This weight reduction is essential for both internal combustion engine vehicles, as it can help lower CO, emissions, and for new electric vehicles, where it can extend battery life. Press-hardened martensitic steels are among the advanced high-strength steels commonly employed in the industry. Recent advancements have led to the development of martensitic steels capable of reaching up to 2000 MPa. These steels present superior strength levels as compared to previous generations thanks to an increased carbon content [1-3]. However, despite their high performance, these steels pose significant challenges due to the inherent brittleness of the (medium C) martensitic structure, making mechanical joining

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Department of Civil and Mechanical Engineering, Technical University of Denmark, Produktionstorvet, Denmark during processes like riveting, which is essential in the assembly phase, particularly problematic [4-5]. A strategy to overcome these issues involves applying a targeted, localized, tempering treatment, which can significantly improve the material's toughness, though at the cost of some strength reduction.

Rapid tempering is a moderately new tempering approach that is gaining increasing attention and that consists in subjecting the material to a fast heating cycle to a targeted temperature followed by direct cooling. This procedure opposes to traditional tempering that involves heating to the tempering temperatures and holding at this temperature for an extended period, which results in considerably larger energy consumption [6]. Rapid tempering achieves the targeted (rapid) thermal cycles via laser or induction heating [7-8]. In addition to reducing energy consumption, this procedure offers the advantage that it allows selective treatment of areas requiring greater ductility while maintaining high strength in the rest of the component [9]. When considering literature, recent studies have focused on testing the comparison of conventional tempering with rapid tempering in terms of achieved microstructure and mechanical properties [10-13]. Interestingly, some of these studies have highlighted that rapid tempering may enhance toughness at comparable strength level when compared to the conventional solution [10-13], which is noteworthy achievement for structural materials. This phenomenon was explained by the delayed tempering effects during rapid treatment, which limits the reduction of dislocation density, thereby favoring the nucleation of very fine cementite lamellae [10,13]. However, it is anticipated that such mechanism may change at varying the steel chemistry and process parameters. To the best of the authors knowledge, no studies of rapid tempering exist for the recently developed medium C, B containing, high strength steels, the last release in the automotive world. One of such grades is consequently selected for investigation in the present study.

The key parameters for rapid tempering treatments are considered to be tempering temperature and holding time [10,14]. However, several studies have shown that temperature is the main factor influencing the material properties [10,14]. Since rapid tempering differs from conventional methods not only due to shorter

holding times but also because of high heating rates, an experimental analysis was conducted in this work to investigate the effects of temperature and heating rate. These parameters can be adjusted by varying the process conditions applied to the tempering treatment.

EXPERIMENTS

For this study we considered samples of 37MnB4 boron steel. The material was acquired as commercial grade under tradename USIBOR®2000. This steel belongs to the first generation of high-strength steels for hot stamping, with a carbon content in the range 0.3 %wt to 0.4 %wt and was supplied in form of a 1.85 mm thick sheet characterized by an initial ferritic-pearlitic microstructure coated with an aluminum-silicon layer to improve corrosion resistance. The samples, whose geometry is shown in Figure 1a, were austenitized at 950 °C for 4 minutes and then guenched in water to obtain a martensitic microstructure. Subsequently, these samples were subjected to tempering cycles using the Gleeble®3180 physical simulator, which included a heating phase, a holding phase at high temperature, and a rapid cooling phase. In this work, the heating temperature was set to 800 °C, which is within the intercritical range as to assure that the conditions yielding to maximum softening would be within the investigation window. Three different heating profiles were tested: two cycles featured constant heating rates of 10 K/s and 100 K/s, respectively, while the third employed a continuous (non-linear) heating profile with an average heating rate of 250 K/s, representative of a thermal history induced by industrial laser tempering processes. Moreover, two holding times at peak temperature were considered: 0 s and 8 s.

To ensure rapid cooling after the holding phase, compressed air was used, injected through the quenching system holes inside the test chamber of the physical simulator. Figure 1b illustrates the thermal cycles applied in the physical simulator for the cases with no holding time at 800 °C and the three investigated heating rates. Figure 1a also illustrates the experimental setup used for the tests. The sample is clamped between two copper grips inserted into two jaws cooled with a water-glycol solution, ensuring that the ends remain at sufficiently low temperatures. The copper grips enable the flow of current needed to heat the sample through the Joule effect. Temperature is

controlled using a K thermocouple, referred to as the pilot thermocouple, welded at the center of the sample (TC1) and connected to a proportional-integral-derivative (PID) controller. This system, comparing the set temperature with that measured on the sample, sends a signal to increase or decrease the voltage, ensuring that the sample temperature matches the set value. Three additional K thermocouples were welded at specific distances from the center of the sample: 5 mm (TC2), 15 mm (TC3), and 25 mm (TC4). These allow monitoring the temperature

distribution along the sample during the test. Due to the functioning of the physical simulator, a thermal gradient develops on the sample, with the highest temperature at the center, progressively decreasing towards the grips. Thanks to the thermal gradient, it is possible to evaluate the effects of tempering temperature over a wide range of temperatures, from the set temperature of 800 °C as measured at the position of the control thermocouple, and down to a much lower value, around 200 °C, near the grips.

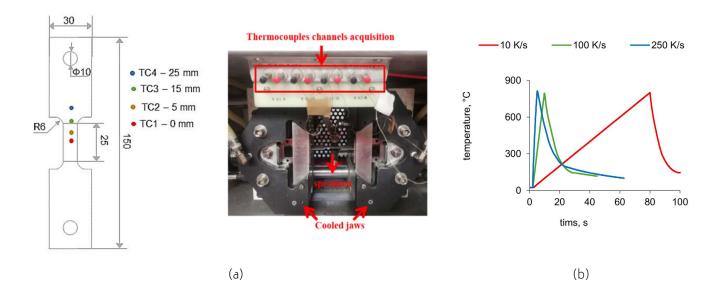


Fig.1 - (a) specimen geometry and experimental setup for physical simulation tests; (b) Thermal cycles applied in the physical simulator at varying heating rates with zero holding time.

After the physical simulation tests, the samples were ground and polished to carry out Vickers hardness tests with a 2 kgf load and a holding time of 15 seconds. These hardness tests were performed on the surface of the samples along the longitudinal axis. The distance between consecutive indentations was set to 1 mm. To obtain statistical information, 3 measurement series were carried out along the axis. The obtained hardness values were then correlated to the thermal cycle and, in particular, to the peak temperature reached during the cycle. This was estimated using a thermo-electric finite element (FE) model developed in COMSOL Multiphysics®. This model was calibrated using experimental temperature data measured with thermocouples. The model is detailed in previous works by the authors [15-16]. Thus, using the

FE approach, it was possible to accurately estimate the thermal cycle to which the sample was subjected along its entire longitudinal axis and consequently the peak temperature at each point. Finally, the microstructure obtained after the various thermal cycles was examined using a Zeiss Supra 35 scanning electron microscope (SEM) equipped with a backscattered electron detector (BSD). The samples intended for SEM analysis were previously electropolished using Struers' A2 electrolyte at 26 V for 15 seconds, employing a Struers LectroPol-5 electropolishing machine.

It is worth noting that one of the main advantages of using the physical simulator for the proposed study is that with a single test it is possible to evaluate the effects of the tempering temperature over a wide temperature range. In this respect, it should be mentioned that, while setting a specific heating rate at the control point, the heating rate varies at adjacent points. Consequently, each peak temperature is associated with a specific heating rate where the two variables are interdependent. Nevertheless, the variation in the average heating rate within a single test, between the rate measured at the center point and the actual rate recorded at the point of interest (i.e., the point of maximum softening), is lower than 20%. This difference is negligible when compared to the variations applied in separate tests.

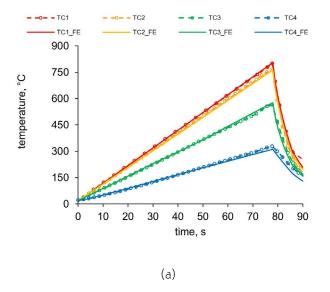
RESULTS AND DISCUSSION

In this section, the results obtained from the physical simulation tests are presented and discussed with a focus on the correlation between thermal cycle parameters and the material's response in terms of microstructure and hardness. The analysis aims to highlight the influence of both temperature and heating rate on the mechanical properties, providing insights into the material's behavior

under varying process conditions.

Figure 2a displays the thermal cycles recorded by the four thermocouples welded onto the specimen subjected to a tempering cycle with a heating rate set to 10 K/s and no holding time (0 s). It can be observed that under these test conditions, the material heats up, reaches a peak temperature, and then rapidly cools down. Moving from the center of the specimen toward the grips, the peak temperature decreases. Moreover, it is also noticeable that the cooling rates vary.

Analyzing the peak temperatures of this cycle as a function of the distance from the specimen's center, yields the curve indicated by circular markers in Figure 2b. The profile of peak temperatures as a function of distance was numerically derived and is represented in the figure with a continuous line. The numerical model accurately predicts peak temperatures in the range of interest (200–800 °C), with the difference between measured and FE model temperatures never exceeding 20 °C.



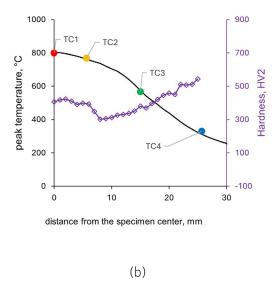


Fig.2 - a) Numerical and experimental thermal cycles with a heating rate of 10 K/s and a holding time of 0 s; (b) Numerical and experimental profiles of peak temperatures and hardness as a function of the distance from the specimen center.

The experimental hardness curve corresponding to the tempering cycle with a heating rate of 10 K/s at the control point and a holding time of 0 seconds has been superimposed onto the graph in Figure 2b. Starting from the center of the specimen, the hardness decreases, reaches a minimum value, and then increases again toward the edges.

To correlate this trend with the metallurgical phenomena occurring in the material, Figure 3a presents the hardness profiles as a function of the peak temperatures calculated using the FE model. The results indicate that the tempering temperature is the dominant parameter

in determining the hardness of the material after the treatment, while the heating rate has a minor effect. The hardness does not follow a monotonous trend with respect to temperature. Instead, as the peak temperature—and thus the tempering temperature—increases, the hardness initially decreases, reaches a minimum point, and then rises again. This indicates that near the turning point of the hardness profile, a condition of maximum softening is achieved. Specifically, the minimum hardness value is approximately 300 ± 3 HV2, independent of the heating rate. This corresponds to a softening of more than 50% compared to the initial martensitic hardness of 633 HV2. It is noteworthy that maximum softening occurs for all three cycles at temperatures close to 730 °C, which is near the eutectoid temperature for C-steels.

From a metallurgical perspective, once the eutectoid temperature is exceeded during heating, the conditions for austenite formation (re-austenitization) are met, making it possible for the steel to undergo new hardening

mechanisms during subsequent rapid cooling. For the thermal cycles considered, hardness increases because the (newly formed) austenite transforms back into fresh martensite. The extent of this phenomenon depends on the amount of austenite formed during the thermal cycle, but also on the carbon content in such austenite. In fact, the C content defined the hardness of the martensite obtained at the end of the cycle and the potential presence of retained austenite. It is anticipated that the C content is determined by the heating rate, the holding time and cooling conditions. Note that in the tests conducted, the final hardness does not reach the initial martensitic hardness level. This is because a temperature of 800 °C is not sufficient to achieve complete re-austenitization of the material. Clearly, at temperatures above the eutectoid, the martensite that does not transform back into austenite continues to temper and soften, resulting in an overall hardness lower than that of the initial fully martensitic (untempered) material.

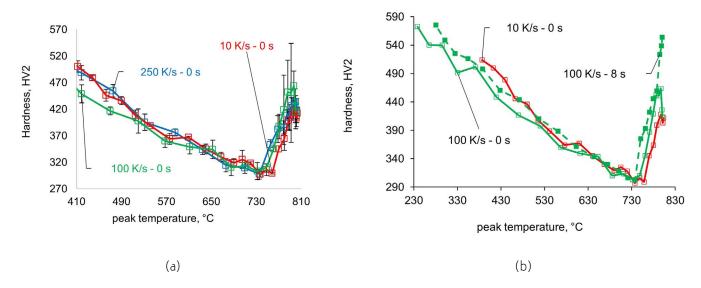


Fig.3 - (a) 2D hardness profiles as a function of peak temperature for thermal cycles with a holding time of 0 s at all investigated heating rates; (b) Hardness profiles as a function of peak temperature for thermal cycles with holding times of 0 s and 8 s at heating rates of 10 K/s and 100 K/s.

To analyze the effect of holding time, Figure 3b shows the hardness profiles as a function of peak temperature for a heating rate of 100 K/s, comparing the results for the two holding times considered: 0 s and 8 s. These data are also compared with the results for a heating rate of 10 K/s and a

holding time of 0 s. The results indicate that holding time, in this explored range, does not significantly impact the tempering phenomena in the material, as the minimum hardness value remains around 300 HV2. Similarly, the peak temperature corresponding to maximum softening

remains constant, close to 730 °C. However, the holding time has a pronounced effect on temperatures above the re-austenitization range. Specifically, for a holding time of 8 s, the final hardness increases significantly, suggesting a greater tendency of the sample to harden again after exposure to high temperatures. To fully understand this behavior, a detailed kinetic analysis of the re-austenitization process, along with an accurate assessment of carbon redistribution within the microstructure during the thermal cycle, would be required. However, such an analysis is beyond the scope of the present study.

The interpretation of the hardness results outlined above is further validated by SEM analysis (Figures 4 and 5). Figure 4 shows that for all heating rates, and no holding time at high temperature, the microstructure at the control point (800 °C) is mainly composed of fresh martensite (light gray), along with a second phase interpreted as pro-eutectoid ferrite (dark gray) and small areas of tempered martensite (darker gray with white spots, i.e. carbides). A detailed quantification of these phases and an assessment of how their characteristics vary with the applied heating

rate are beyond the scope of the present study. Moving away from the center, at the point of maximum softening (i.e., minimum hardness), tempered martensite is the predominant microstructural feature observed in all samples. Figure 5 allows for a comparison of the microstructures at the control point and at the point of maximum softening, keeping the heating rate constant while varying the holding time at high temperature. It can be observed that, regardless of the holding time, only tempered martensite is present at the point of maximum softening. In contrast, at the control point, for continuous cycles with no holding time, the microstructure is predominantly composed of fresh martensite with islands of tempered martensite and ferrite. On the other hand, for a holding time of 8 s, there is an almost complete absence of tempered martensite. This behavior is consistent with the higher hardness values obtained from the hardness tests under these conditions and shows the time dependent nature of the austenite reversion process.

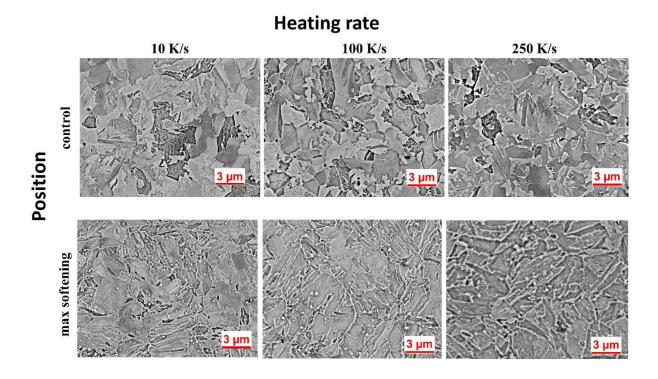


Fig.4 - BSE images presenting the microstructure of the material versus heating rate and position along the sample for holding time at high temperature equal to 0 second.

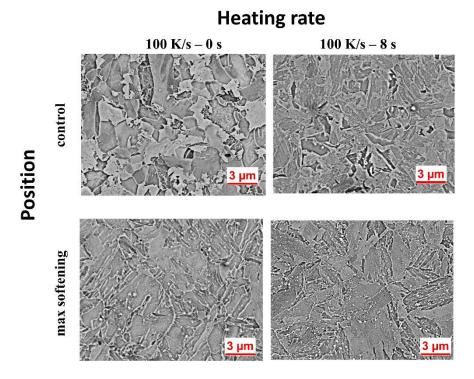


Fig.5 - BSE images as a function of heating rate and position for a holding time of 0 seconds, compared to the condition with a holding time of 8 seconds.

The results on the effects of temperature and tempering time are in line with the literature [10,14]. Specifically, temperature is the dominant factor influencing martensite softening, rather than holding time, even when the holding time is limited to just 8 s, as in this study. It is also evident that increasing the tempering temperature up to the eutectoid temperature enhances the softening effect. Data available in the literature for PHS1500 steel —a material comparable to the one used in this study, albeit with a lower carbon content— show the degree of softening achieved at 650 °C with holding times ranging from 0.2 seconds to 3 hours [17]. It was found that, at this temperature, the steel experiences a softening of 35% after 0.2 seconds and 51% after 3 hours. Although the treatment cycles in this study are continuous while those described in the literature are isothermal, the latter value is comparable to the softening achieved in the present work at the same tempering temperature but through rapid heating with no holding time. In addition to this, the present work demonstrates that the holding time becomes particularly significant when the thermal cycle temperature exceeds

that of minimum hardness.

In summary, while numerous studies have explored the effects of rapid tempering compared to conventional tempering, this work presents an efficient approach for systematically analyzing the effects of holding time, temperature, and heating rate in rapid thermal cycles. Additionally, metallurgical aspects at much higher temperatures, up to 800 °C, compared to those typically used in tempering treatments, were examined to assess their impact on hardness and microstructure. It was found that the maximum level of softening, obtained at a peak temperature of 730 °C with rapid thermal cycles and a heating rate of 250 K/s, can reduce the material's hardness by over 50% compared to the quenched condition. However, exceeding the eutectoid temperature leads to a significant increase in hardness relative to the observed minimum, and this increase becomes more pronounced with longer holding times. Thus, a rapid tempering process at a temperature below the critical value is recommended for a more stable and robust process. Exceeding such a temperature yields a reduced softening effect. Such a reduction is the most pronounced the longer the material is kept in temperature.

Further experimental activity, i.e. X-Ray diffraction, is planned to gain a deeper understanding of the metallurgical phenomena occurring in the material subjected to rapid tempering, particularly near or beyond the temperature that results in maximum softening. The findings from these studies will be presented in future publications.

CONCLUSIONS

In this study, we studied the effects of rapid tempering on the martensitic microstructure and related properties of a B containing high-strength steel medium C steel, grade 37CMnB4.

The findings of this investigation are summarized as follows:

 Rapid tempering, conducted at heating rates in the range 10 K/s to 250 K/s, is a highly effective method for reducing the hardness of martensite. Specifically, a reduction in hardness of over 50% can be achieved with this method;

- Within the range of heating rates tested, the maximum degree of softening achievable is essentially independent of the heating rate;
- In the range of holding times investigated, 0 s to 8 s, it was found that the duration of the hold does not affect the level of achievable softening but impact the hardness of the material when the temperature for maximum softening is exceeded. Specifically, at a given temperature of overheating, the effectiveness of the treatment is compromised the most, the longer the holding time;
- It was established that maximum softening is achieved by treating the material at a temperature close to
 the eutectoid temperature. This observation aligns
 with theoretical expectations regarding the behavior
 of carbon steels;
- The application of a physical simulator, combined with temperature profile simulation using finite elements, proved to be particularly useful for the fast mapping of treatment parameters.

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