

## Evaluation of C45 Steel Hardening Process Using High-Speed Videography and ISO 9950 Test

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The paper deals with the influence of various quenching media based on polymer aqueous solutions on the quenching process of carbon steel C45, connecting theoretical knowledge about heat transfer, surface phenomena and phase transformations with experimental verification. Surface phenomena at the interface of the hardened sample and the quenching medium were monitored using a high-speed camera. Cooling curves of the samples were obtained using the method according to ISO 9950 (Determination of cooling characteristics – Nickel-alloy probe test method). The paper contains practical recommendations for optimizing industrial hardening processes, especially when choosing polymer hardening baths as an alternative to water hardening baths and confirms their ability to ensure a more controlled cooling process, reduce the risk of cracks and deformations, and achieve higher hardness of hardened parts.

**Keywords:** Heat Treatment, Quenching, High-Speed Camera, Nickel-Alloy Probe Test Method, Polymer Quenchants

### 1 Introduction

The main task of the quenching environment is to remove heat from the quenched component at least at a critical or higher rate. If cooling occurs at a rate higher than the critical rate, the austenite will not undergo a pearlitic transformation, but a martensitic or bainite transformation. Other required properties of quenching media also include:

- Stability (resistance to thermal degradation, oxidation, etc.),
- Acceptable cooling capabilities over the entire temperature range (slow enough below Martensite start temperature  $M_s$  to prevent deformation),
- Non-reactivity with the hardened material, or with a protective atmosphere,
- Low toxicity and operator safety,
- Easy removal from the quenched part after the quenching process,
- Low price.

The thermal properties of the quenching medium then determine the resulting hardness and the distribution of hardness in the volume of the material. Most quenching media are liquids, less often they are gaseous or mixed. The division can be based on various parameters, but the most important division is the division into media that evaporate and those that do not evaporate. This separation criterion is significant because of the different mechanisms of

heat transfer, and thus the different effects on phase transformations. The evaporating medium consumes the so-called latent heat for the evaporation of the medium, since the evaporation process is an isothermal process at a given pressure. This heat has a source in the form of the hardened part during quenching, which means that the evaporating quenching medium has a higher cooling rate in a certain temperature range than the medium that does not evaporate [1].

The cooling effect of the quenching medium can be expressed using different values [2]:

- Grossmann H-value,
- Heat transfer coefficient  $\alpha$  [ $W/K.m^2$ ],
- Cooling time of the parts from 800 °C to 500 °C,
- Cooling curve with temperature change,
- Cooling curve with cooling rate change.

The cooling rate of a hardened part is provided by two different forms of heat transfer. On the one hand, it is the removal of heat from the surface of the part by convection. This heat removal is controlled by the heat transfer coefficient through the wall of the hardened material. The second form of heat transfer is the transient diffusion of heat from the interior of the part to the surface due to conduction. This form of heat transfer is controlled by the thermal conductivity of the given material. These two forms were taken into account in the Grossmann H-value, which describes the criticality of hardening. This value was expressed as:

$$H = \frac{\alpha}{2\lambda} [-], \quad (1)$$

Where:

$\alpha$ ...Heat transfer coefficient [W/K.m<sup>2</sup>],

$\lambda$ ...The thermal conductivity [W/K.m][2].

Thermal conductivity is related to the material itself, so the main basis here is the heat transfer value. However, the H value is not constant, but variable depending on the relative movement of the quenching medium to the quenched component. H-values are useful for detailed examination of the temperature phenomena occurring during quenching, but for the practical characterization of quenching processes, or rather for describing the cooling capabilities of quenching media, they are not informative. Cooling curves are more informative. A cooling curve with a change in temperature follows the change in the surface temperature of the quenched component in a given quenching medium over time. The greater the angle of inclination of the curve, the faster the cooling rate. The second way to illustrate the thermal capabilities of a quenching medium is a cooling curve with a change in the cooling rate [1,3,4].

## 2 Surface phenomena during quenching

Steam bubbles form at characteristic locations and their number increases with increasing heat flow at a given location. Each bubble is formed from a so-called nucleus, which is located in a local surface defect or a location with greater surface roughness. The heat from the hardened material heats the medium adjacent to its surface, where a thin layer of the medium is overheated and a thermodynamically metastable situation is created, which, however, lasts a very short time. If the nucleus is large enough or the overheating of the thin layer is high enough, a steam bubble is formed, which gains heat from this boundary layer of the medium. In order for a bubble to form, the steam pressure in it must be higher than the pressure of the surrounding liquid (medium). The surface has a variable quality and roughness, which, with increased overheating, leads to the nucleation of nuclei even in places where they would not appear with less overheating. An increase in the number of nuclei then leads to more intense mixing of the liquid on the heated surface. This leads to the fact that the greater the heat flow through the material during boiling, the greater the heat transfer coefficient [4,6].

At low heat flux, the main mechanism of heat transfer is natural convection. The onset of bubble formation on the surface of the heated plate, i.e. the point at which bubble boiling (nucleate boiling) begins, is manifested by a sudden change in the slope of the so-called Nukiyama graph, as the conditions for heat transfer improve significantly. With further increase in heat flux, a point is reached where bubble boiling becomes hydrodynamically unstable due to the

high bubble density and high vapor flow. At this point, a thin but continuous vapor layer, the so-called film, forms on the surface of the material, which prevents the liquid from reaching the surface of the material. Bubble boiling thus changes to film boiling. This change is called DNB (departure from nucleate boiling), and the heat flux at which this change occurs is called CHF (critical heat flux). Just before reaching DNB, the heat transfer coefficient reaches its maximum. Further increase in heat flux then results in only a significant increase in the surface temperature of the material due to limited heat transfer to the liquid. Between bubble and film boiling is a region of transitional boiling, which can occur if the surface temperature is maintained constant [4,7].

However, the film boiling period is limited in time and the breakdown of the steam film on the surface of the component occurs at the so-called Leidenfrost temperature, when the heat flow falls below the level at which it is able to maintain a stable steam layer. This temperature is not yet sufficiently described theoretically and does not have a clear definition. However, the research carried out shows that the breakdown of the film occurs when the temperature of the material surface drops to a value of at least 100 K above the saturated steam temperature at a given pressure. The surface temperature at which the steam film begins to break down, however, significantly depends on the steam pressure in the existing film. For this reason, the relevant measurements show a wide spread of values. However, this spread of values was most likely caused by different surface quality and different surface treatments [4,8].

The breakdown of the vapor film is influenced not only by the surface temperature, but also by the heat transfer conditions. In a supercooled liquid, the conditions for heat removal are better, since the condensation of vapor at the phase interface between the vapor film and the liquid produces strong turbulence, which accelerates the liquid towards the hot surface. The accelerated liquid then penetrates the film, reaches the surface of the material, and the breakdown of the film thus occurs at higher temperatures than in an uncooled liquid [8,9].

## 3 Materials and Experimental Methods

The main objective of the experimental part was to observe surface phenomena occurring on the surface of hardened parts during quenching in water and in polymer baths of various concentrations, to determine their influence on the resulting material properties. Furthermore, the objective of the experiment was to measure the cooling curves using the nickel-alloy probe test. Observation of surface phenomena took place in various environments, namely: in pure water,

in 5 %, 10 % and 15 % solutions of the quenching polymer and water. Since the influence of the quenching medium temperature on the quenching process was also investigated, the media were either used at room temperature (without heating) or were heated to 40 °C and 60 °C.

In addition to analyzing high-speed observations of surface phenomena, the experimental program also evaluated the microstructures and hardness profiles of the investigated materials. Cooling curves were also determined for the relevant cooling conditions using a nickel-alloy probe test.

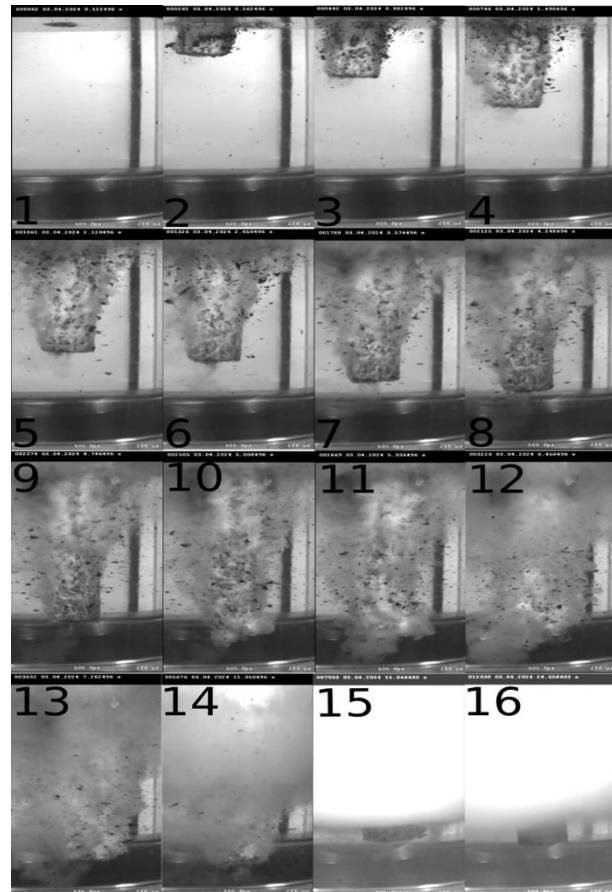
The experimental part was carried out on samples of plain carbon steel C45 in the form of cylinders with a diameter of 20 mm and a length of 150 mm. The hardening polymer medium used was the hardening polymer SERVISCOL 98 S-F1 based on modified PAG with an effective corrosion inhibitor. In practice, it is used in concentrations from 6 % to 20 %.

A high-speed camera with additional accessories IDT NX4-S3 and the Motion Studio program were used to observe surface phenomena. Due to the optimal recording length, a frame rate in the range of 400–500 fps was chosen. At higher fps values, it would not be possible to capture the entire hardening process. With regard to the type of lighting used (powerful halogen lighting), the hardening process was recorded in the exposure time range of 108–286  $\mu$ s. The cooling medium was placed in a 2000 ml container made of boiling glass. The austenitization temperature was set to 830°C. The austenitization time was uniformly 20 minutes. The heating of the quenching medium was carried out by a heating nest in which a container with the quenching medium was placed. After austenitization, the sample was manually immersed in the quenching environment. The high-speed recording was started just before the start of immersing the sample in the quenching medium. A test sample was taken from each hardened sample, on which further analyses were subsequently performed.

The following parameters characterizing the influence of quenching media on the quenching process were determined from each record: the beginning of the breakdown of film boiling, the duration of bubble boiling, the time of the transition of boiling to the convective heat transfer mode. Fig. 1 shows a sequential time example of the quenching process in a 15 % solution of quenching polymer and water heated to 60 °C.

The nickel-alloy probe test was used to measure the cooling curves. This test is performed using a test probe equipped with a type K thermocouple, which is connected to a computer via a sensing unit. This measurement is described in the ISO 9950 standard, which prescribes an outer diameter of the

thermocouple of 1.5 mm, which must be provided with an outer sheath of Inconel 600 alloy. The total length of the probe and support tube must be at least 200 mm according to the standard. The measurement begins with heating the probe to the required temperature, immersion in the appropriate cooling medium and subsequent measurement of the cooling curve using a thermocouple placed inside the probe. Temperature recording was performed with an accuracy of 0.1 s. Cooling curves were not determined in the case of a quenching environment consisting of pure water, due to possible damage to the probe due to significant thermal stresses caused by rapid heat dissipation.



*Fig. 1* Quenching process in a 15 % polymer quenching bath heated to 60 °C – 24.535 seconds elapsed from the sample touching the surface to convective transfer along the entire length of the sample

## 4 Results and Analysis

### 4.1 Hardening at a quenching media temperature 20 °C

The measured values of the hardness curves (Fig. 2) show that the greatest increase in hardness due to quenching in a quenching medium with a temperature of 20 °C occurred in the case of quenching in 10 and 15 % solutions of the quenching polymer. This fact is in accordance with the analysis of the high-speed

recording, when it was found that in these solutions the film layer collapses almost immediately after the sample is immersed in the quenching medium. In samples quenched in water and 5 % polymer bath,

film boiling was maintained on the surface of the part for a longer time, which reduced the criticality of cooling and the structure has less hardness as a result.

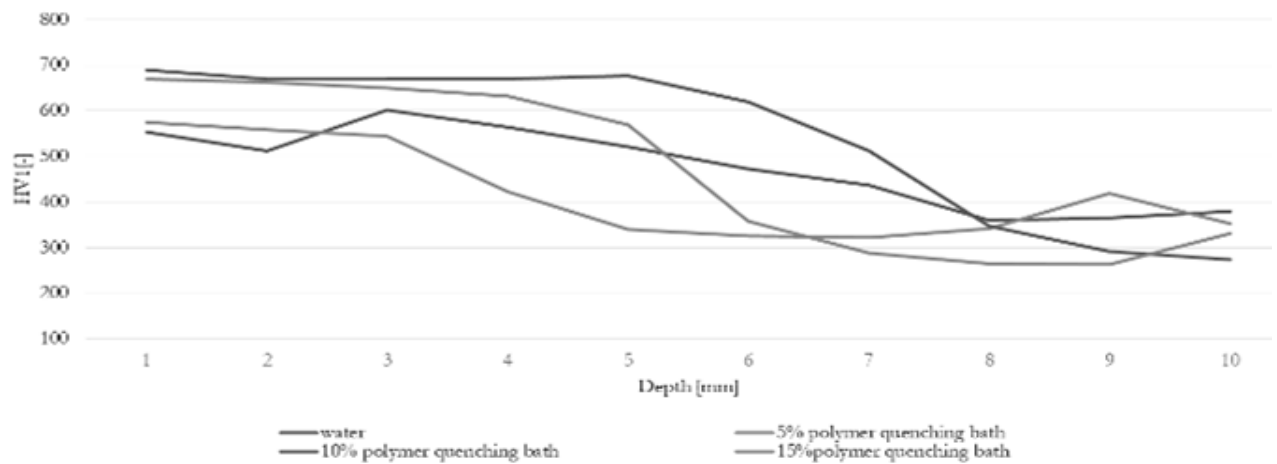


Fig. 2 Hardness HV1 curves during quenching in media at a temperature of 20 °C

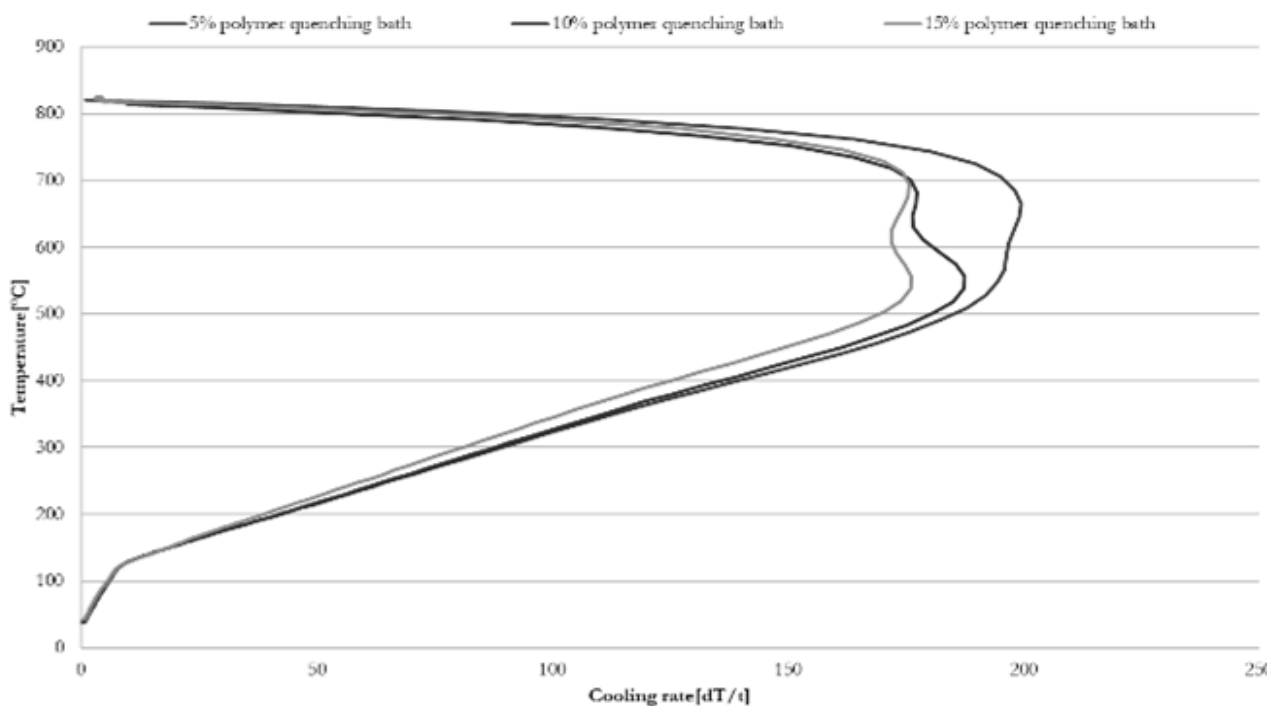


Fig. 3 Cooling curves for the case of 5 %, 10 %, 15 % polymer quenching bath at a temperature of 20 °C

Using the nickel-alloy probe test, cooling curves (Fig. 3) were obtained for individual polymer quenching media. The following findings were obtained by analyzing these curves and the high-speed recording:

- 5 % polymer quenching bath: the cooling curve shows a gradual increase in the cooling rate to 199.4 °C/s in 1.8 s, which corresponds to the peak of bubble boiling. The subsequent gradual cooling process demonstrates the absence of film boiling. Subsequently, the

- cooling rate gradually decreases to a temperature of 113 °C, when intense bubble boiling ends and convection begins,
- 10 % polymer quenching bath: the maximum cooling rate of 177 °C/s occurs in 3.3 s. A short decrease in the rate follows, and then the cooling rate increases again (188 °C/s in 4.1 s). This temperature fluctuation is related to a change in the intensity of bubble formation, which temporarily prevents heat

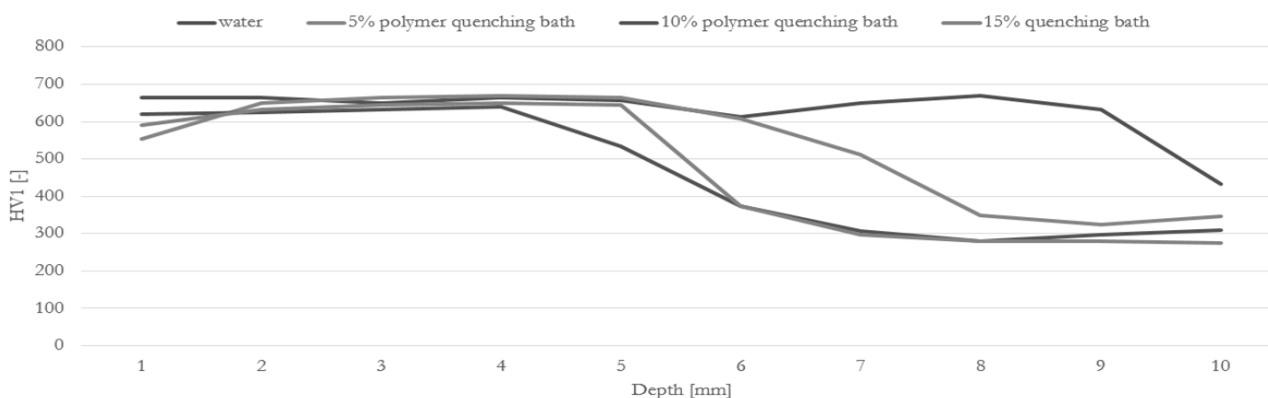
dissipation. The transition to convection is initiated at a temperature of 121 °C. The breakdown of film boiling begins very early (0.587 s) at this concentration, primarily at the edge of the sample,

- 15 % polymer quenching bath: the cooling process is very similar to the quenching process using 10 % polymer medium. The maximum cooling rate of 176 °C/s is reached in 3.5 s. Here again, the limitation of heat dissipation due to bubble formation applies. At a temperature of 121 °C, the end of bubble boiling occurs and further heat transfer is carried out only convectively.

The breakdown of the film layer occurs almost immediately after the sample is immersed.

#### 4.2 Hardening at a quenching media temperature 40 °C

The highest hardness value was shown by the sample quenched in water (Fig. 4). Quenching in water also caused the highest depth of hardening. In contrast, samples quenched in 5, 10 and 15 % quenching solution showed lower surface hardness. In addition, the sample quenched in 10 % polymer quenching bath had the smallest depth of hardening. Samples quenched in 15 % polymer quenching bath showed a very similar hardness profile.



**Fig. 4** Hardness HV1 curves during quenching in media at a temperature of 40 °C

Unlike quenching in media at 20 °C, in this case, film boiling was maintained for a certain time in each quenching medium. The fastest cooling of the sample to the free convection region was in water – 13.867 s, followed by 5 % bath – 21.654 s, 10 % bath – 24.033 s and the slowest cooling was in 15 % bath – 24.076 s. A significant decrease in hardness is evident in samples quenched in polymer baths from the previous quenching process to 20 °C.

Using the nickel-alloy probe test, cooling curves (Fig. 5) were obtained for individual polymer quenching media. The following findings were obtained by analyzing these curves and the high-speed recording:

- 5 % polymer quenching bath: the fastest cooling (178 °C/s) occurs at 2.8 s, which corresponds to bubble boiling. Subsequently, the cooling rate decreases due to the insulation of the sample surface by bubbles. Intensive bubble boiling ends at 12.1 s when the temperature reaches 113 °C, when it passes into convection. The breakdown of the film layer is observed at 4.13 s,

- 10 % polymer quenching bath: the cooling rate increases up to 4.2 s (160 °C/s), when film boiling occurs, which reduces the cooling rate. The breakdown of film boiling occurs very unevenly up to 2.55 s. This film boiling reduces the cooling rate, after its breakdown the cooling rate increases again. The following bubble boiling, which further reduces the cooling rate, ends at a temperature of 119 °C (14.8 s). This is followed by a transition to convective heat dissipation,
- 15 % polymer quenching bath: within 2.5 s the cooling rate increases to 150 °C/s. After a short stagnation of the rate, it increases to 162 °C/s. The film layer disintegrates evenly and progresses from the beginning of the sample upwards. At a temperature of 121 °C (13.9 s) there is a transition to convection.

Lower polymer concentration in the quench bath (5 %) produces the most intense bubble boiling with

virtually no film boiling. Higher polymer concentrations (10 %, 15 %) promote the formation and later breakdown of film boiling, thereby prolonging the bubble boiling time and shifting the transition time to convection by several seconds.

The maximum cooling rate decreases with increasing polymer concentration. However, secondary cooling peaks can be achieved due to the dynamics of film layer breakdown.

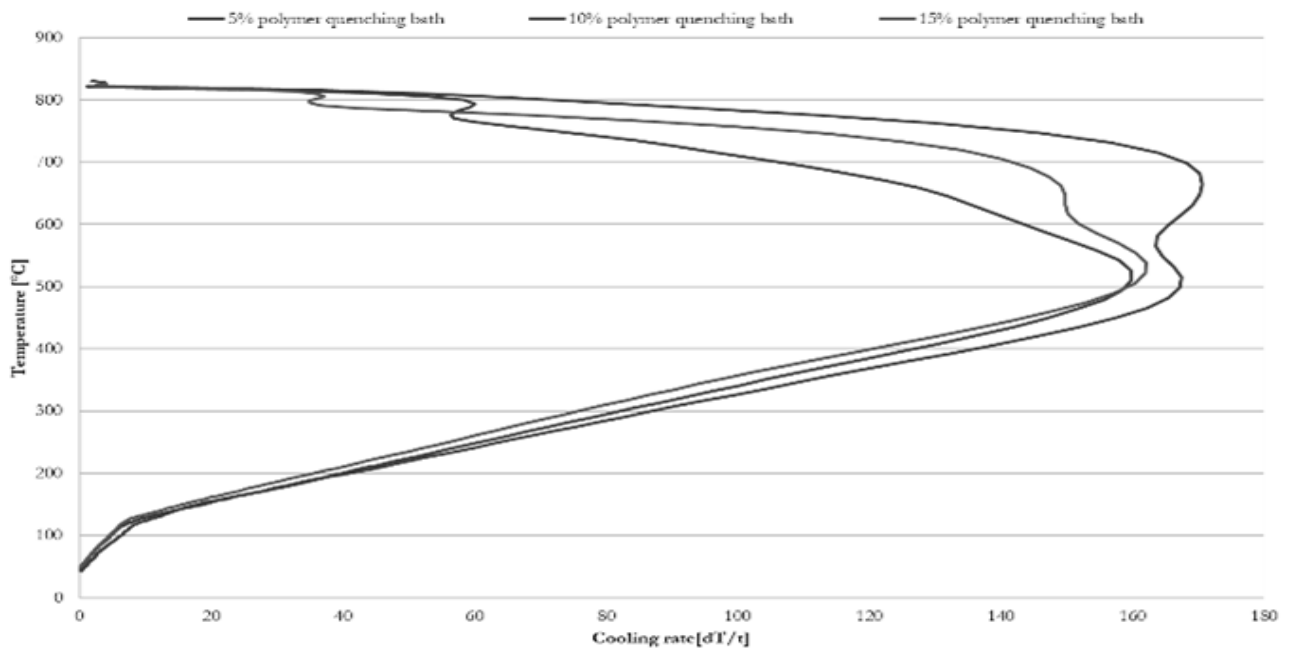


Fig. 5 Cooling curves for the case of 5 %, 10 %, 15 % polymer quenching bath at a temperature of 40 °C

### 4.3 Hardening at a quenching media temperature 60 °C

When quenching in quenching media at 60 °C, the highest hardness is shown by samples quenched in water and in a 15 % polymer bath (Fig. 6). However, in the case of a water quenching bath, the decrease in hardness is less abrupt than in a 15 % polymer bath. In the case of a 15 % polymer bath, the hardness of the samples is compared to samples quenched in polymer baths at a depth of 6.0 mm. In the case of samples quenched in 5 % and 10 % polymer baths, there was no significant quenching of the samples and these samples did not show a significant increase in

hardness. The hardness curve is constant throughout the cross-section of the samples and corresponds to the unhardened state. The cause of this state is the long time during which the film boiling was maintained in these quenching media (5 % polymer bath – 5.351 s, 10 % polymer bath – 2.996 s). As a result of the higher temperature of the media, a significantly lower criticality of cooling was subsequently achieved, which resulted in a low hardness of the structure. The high hardness value of the sample hardened to 15 % polymer bath is related to the very short time of existence of the film boiling, which is 0.599 s.

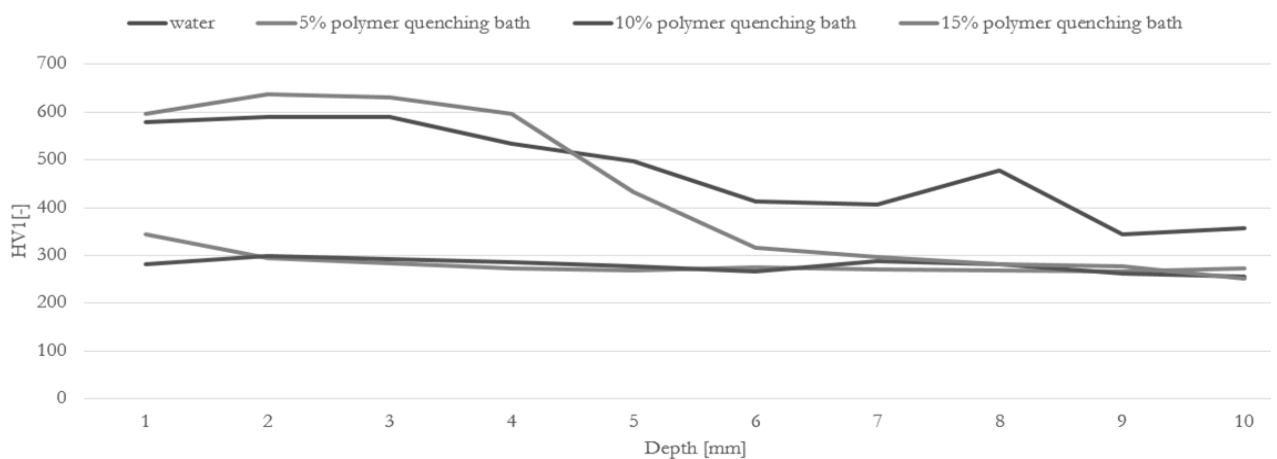


Fig. 1 Hardness HV1 curves during quenching in media at a temperature of 60 °C

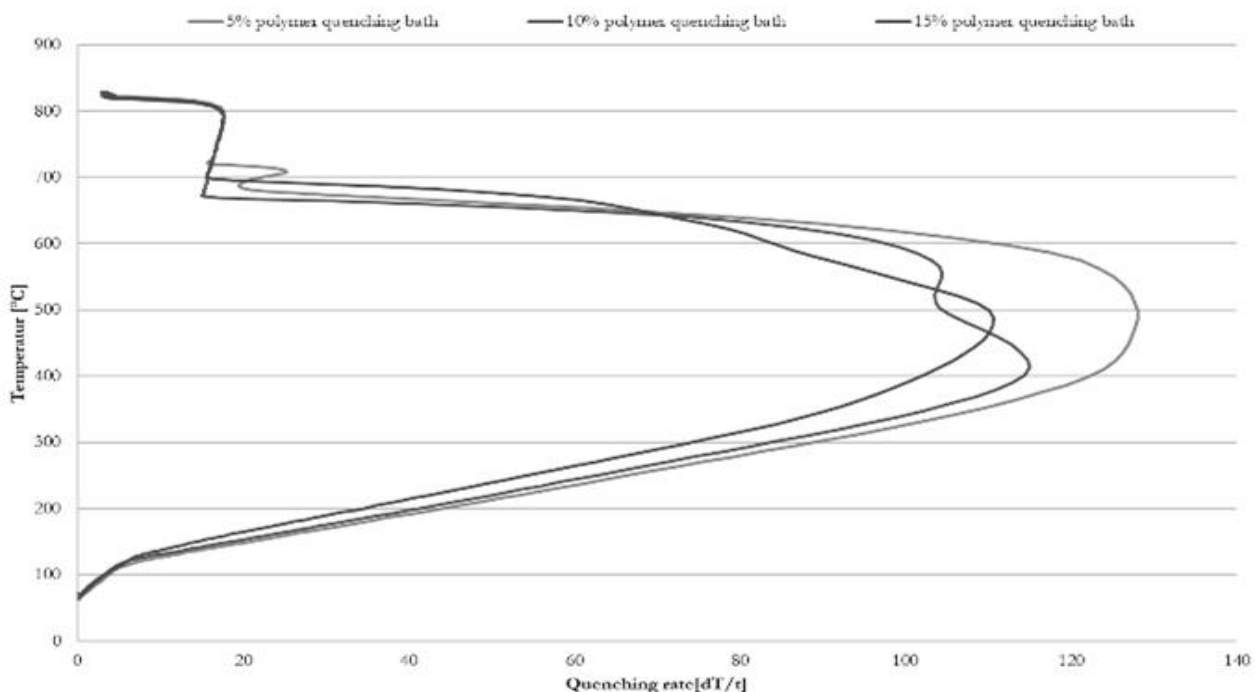
Using the nickel-alloy probe test, cooling curves (Fig. 7) were obtained for individual polymer quenching media heated to a temperature of 60 °C. The following findings were obtained by analyzing these curves and analyzing the high-speed recording:

- 5 % polymer quenching bath: the cooling rate increases until 2.9 s, when film boiling occurs, whereby the cooling rate decreases. The breakdown of the film layer is recorded at 5.35 s, when it breaks down evenly around the entire circumference of the sample. The breakdown starts from the immersed end of the sample. The maximum cooling rate of 128 °C/s is reached at 11.8 s. The transition to free convection occurs at 24.38 s,
- 10 % polymer quenching bath: increase in cooling rate up to 4.9 s, when the rate value stabilizes and then decreases due to the transition to film boiling. For this quenching bath, a secondary peak in cooling rate is detected at 15.3 s, when a cooling rate of 115 °C/s is reached. The breakdown of film boiling occurs at 2.99 s, with the breakdown

occurring uniformly from the lower end of the sample upwards. The transition to convective cooling occurs at 24.56 s,

- 15 % polymer quenching bath: the growth of the cooling rate is interrupted by the appearance of film boiling (3.1 s). The maximum cooling rate is reached at 12 s (111 °C/s). Partial breakdown of the film boiling occurs at 0.59 s, when its uneven breakdown occurs at several places of the immersed sample. Convection occurs at 24.54 s.

At a quenching bath temperature of 60 °C, film boiling is initially formed at all quenching medium concentrations used, which contributes significantly to the reduction of the cooling rate. The main maximum cooling rate decreases with increasing polymer concentration. Secondary cooling rate peaks arise from repeated breakdown and formation of the film layer. The transition to pure convection occurs around 24 s without significant differences between concentrations. The breakdown of film boiling occurs earlier at higher quenching medium concentrations (fastest at 15 %, slowest at 5 %).



**Fig. 7** Cooling curves for the case of 5 %, 10 %, 15 % polymer quenching bath at a temperature of 60 °C

## 5 Discussion

As the temperature of the quenching media increases, the time during which the vapor insulating layer remains on the surface of the part increases. For example, in the case of a water quenching medium, the breakdown of film boiling shifts from 0.33 s (20 °C)

to 1.89 (60 °C), in 5 % polymer baths from 2.18 s to 5.35 s. As the temperature of the quenching medium increases, the bubble boiling stage also lengthens and the convection stage begins later. From the cooling curves it was found that with increasing temperature of the media the cooling rate decreases. For 5 %

polymer bath from 199 °C/s (20 °C) to 128 °C/s (60 °C), for 10 % polymer bath from 187 °C/s (20 °C) to 155 °C/s (60 °C). The shortest time to achieve the highest cooling rate at all measured temperatures is achieved by 5 % polymer bath. A lower cooling rate of course leads to a reduction in the proportion of martensite in the structure and an increase in the proportion of other structural phases in C45 steel. For example, the hardness during quenching in a 5 % polymer bath decreases by almost half when the bath temperature is increased from 40 °C to 60 °C. In terms of the effect of temperature, it was further found that the highest and most uniform hardness is achieved at a temperature of 40 °C in water and 5 % polymer quenching baths. When using higher concentrations of quenching polymer (10 %, 15 %), the most effective quenching process is at room temperature, since at this temperature film boiling is almost absent, which guarantees a rapid onset of intensive bubble boiling. All samples hardened in media at a temperature of 60 °C achieve the lowest hardness values, especially samples hardened in a 5 % and 10 % polymer bath, which show significantly lower hardness compared to the others, across the entire cross-section of the sample. At 60 °C, the 5% and 10% polymer baths maintained film boiling for too long, resulting in very low cooling intensity and insufficient martensitic transformation. In contrast, the 15% bath showed a very short film-boiling period (0.599 s), enabling an earlier onset of nucleate boiling and a higher effective cooling rate, which allowed the samples to reach higher hardness despite the elevated bath temperature.

In the case of polymer quenching baths, the maximum cooling rate decreases with increasing polymer concentration:

- 5 % polymer solution: cooling rate 199 °C/s at 20 °C, very rapid transition to bubble boiling,
- 10 % polymer solution: cooling rate 188 °C/s at 20 °C, immediate collapse of film boiling, longer bubble boiling compared to 5 % polymer solution,
- 15 % polymer solution: cooling rate 176 °C/s at 20 °C, no film boiling, in contrast, bubble boiling takes the longest of all quenching media,
- Film boiling lasts the longest in a 5 % polymer bath (2.2 s), and almost does not occur in 10 % and 15 % polymer baths. Film boiling also disappears very quickly in a water bath (0.33 s). Bubble boiling is significantly longer in polymer baths (20–22 s) than in a water bath (12–14 s).

## 6 Conclusion

The results obtained from the high-speed camera IDT NX4-S3 recordings of surface phenomena on the C45 steel samples during immersion in the individual quenching media differ slightly from the cooling-curve data obtained using the nickel-alloy probe test. However, their results correlate well with each other in terms of the influence of the temperature of the quenching medium on the resulting properties of the quenched part. For more accurate data, it would be necessary to ensure the same dimensions and the same surface finish of the test samples as the test probe of the nickel-alloy probe test and to ensure the same immersion speed of the quenched sample for both methods.

Overall, it can be concluded that high-speed recording analysis is a useful method for comprehensively characterizing the quenching process. Since it is a visual method, high-speed recording can be used to better understand the heat transfer processes occurring between the quenched part and the liquid phase.

The obtained results confirm the assumption that the choice of the concentration of the polymer cooling medium and its temperature allows for a targeted influence on the cooling rate and the resulting hardness of C45 steel.

- If it is necessary to achieve maximum hardening and hardness with the risk of deformation, water hardening must be chosen,
- For an optimal hardening process, i.e. to achieve the optimal ratio between high hardness and structural stress in the material, a 5 % polymer solution is ideal. This concentration of the hardening bath provides a high cooling rate, adequate bubble boiling time and good depth of hardening,
- Higher polymer concentrations in the quenching bath (10 %, 15 %) are suitable where a slight reduction in the cooling rate is required (thus achieving lower stress), but a relatively hard surface still needs to be achieved,
- Increasing the temperature of the quenching medium always slows down the heat transfer, prolongs the boiling phase, reduces the cooling intensity, resulting in a decrease in hardness. To achieve optimal criticality of cooling, it is necessary to choose a quenching medium temperature that provides a suitable

balance between insulation caused by the vapor film and effective bubble boiling,

- As the polymer concentration in the quenching medium increases, the cooling intensity decreases, the bubble boiling phase is prolonged, and the depth and uniformity of quenching decreases,
- Pure water as a quenching medium is the most critical, but also the medium that causes the highest values of internal stress.

Overall, it can be concluded that high-speed recording analysis is a useful method for comprehensively characterizing the quenching process. Since it is a visual method, high-speed recording can be used to better understand the heat transfer processes occurring between the quenched part and the liquid phase. The results also confirm the assumption that the choice of the concentration of the polymer cooling medium and its temperature allows for a targeted influence on the cooling rate and thus the resulting hardness of C45 steel.

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