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# MODELING STEEL TRANSFORMATION BEHAVIOR FROM COOLING BEHAVIOR BY THE KUYUCAK METHOD[1]

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**Abstract**
Heat treatment is performed to achieve the desired as-quenched and tempered mechanical properties of steel. Heat treatment of steel involves heating the steel to an elevated temperature, typically the austenitizing temperature, often with subsequent quenching into a vaporizable liquid medium such as water, oil or aqueous polymer quenchant. In the laboratory, steel quenching is studied by inserting thermocouples into a probe or an actual part to determine the time-temperature profile (cooling curve) during the quenching process. However, this is not always possible. Recently, Kuyucak has described a process involving the measurement of the temperature increase of the quenchant in the vicinity of the cooling metal surface as a function of time. From this time-temperature data, cooling curves of the metal being quenched may be calculated which are then used to predict microstructure and hardness. This paper describes the successful utilization of this process to predict the as-quenched properties of AISI 4140 steel under laboratory quenching conditions when using water as the quenchant.
**Keywords:** Kuyucak; Cooling curve; Quenching; Hardness.

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## INTRODUCTION

Quenching is a critically important process during the heat treatment of steels since it determines both the metallurgical quality of the part and the potential formation of defects such as cracking, poor distortion control, and excessive residual stresses. Steel hardening (either martensitic or bainitic hardening) requires preheating of the steel to the austenitizing temperature; 750 – 1100 °C from which the steel is quenched to obtain the desired microstructure and mechanical properties such as hardness and yield strength. Most liquid vaporizable quenchants used for this process exhibit boiling temperatures between 100 – 300 °C at atmospheric pressure. When parts are quenched into these fluids, wetting of the surface is usually time-dependent which influences the cooling process and hardness.[1]

Figure 1 shows the three primary heat transfer cooling mechanisms most commonly observed during conventional immersion cooling in water. Upon initial immersion, the part is surrounded by a vapor film where full-film boiling (FB) or vapor-blanket cooling occurs. When the temperature decreases to the Leidenfrost temperature, first identified by J.G. Leidenfrost about 250 years ago,[2] the vapor film (vapor blanket) collapses and surface wetting by the liquid occurs by a nucleate boiling (NB) process due to lateral heat conduction relative to the surface. When the surface temperature decreases to a temperature less than the boiling point, nucleate boiling ceases and convective cooling (CONV) begins.

Each of these cooling mechanisms, which coexist on the steel surface during the quenching process,[3] is associated with different heat transfer processes as illustrated in Figure 1.[4] This is significant in view of the magnitude of the different heat transfer coefficients corresponding to these cooling processes. For example, when water is used to quench steel, typical heat transfer coefficients are: full-film boiling $\alpha_{FB}$ (100–250 W/m$^2$K), nucleate boiling $\alpha_{NB}$ (10–20 kW/m$^2$K) and convective cooling $\alpha_{CONV}$ (ca. 700 W/m$^2$K). The simultaneous presence and relative stability of these widely varying heat transfer conditions are an important factor influencing non-uniform cooling and increased stresses during a water quenching process.[5-6]

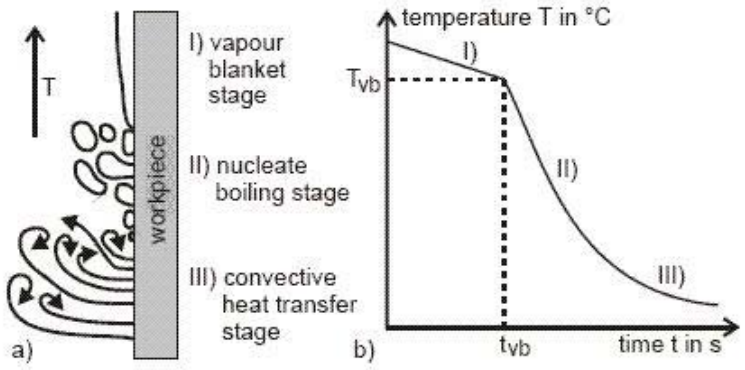



**Figure 1.** a) Different cooling stages on a workpiece surface (increasing workpiece surface temperature from bottom to top); b) Variation of the surface temperature during quenching.

Quenching steel into water has been practiced for thousands of years and the effect of varying water purity and temperature on quenching performance was studied in great detail by Rose[7] and others.[8,9] More recently, the quenching properties of water as a function of water temperature has been examined in detail by cooling curve analysis.[10,11] Generally, increasing water temperature, decreases cooling rate and the cooling rate reduction is significant at a water temperature of

50 °C – 60 °C. Uzlov and Danchenko studied the effects of cooling rate on microstructure, fracture toughness, fatigue and yield strength of carbon steels.[12] Zhang[13] and Vyshkovskii[14] showed that potential for quench cracking and unacceptable distortion control increased at lower water temperatures but the potential for either spotty hardness or insufficient hardness increased with increasing water temperature. Therefore, it would be desirable to simulate as-quenched performance especially when water is used as the quenchant.

Agitation is an important process parameter in quenching since it helps to assume a more uniform quenching process. A major source of distortion is of the presence of differential temperature gradients, whether from the center to the surface; or across the surface. This is especially critical with vaporizable quenchants such as water since all three phases of cooling, full-film boiling, nucleate boiling and convective cooling may be present on the surface which means that some surface areas may be cooled very slowly while adjacent areas of the surface may be cooling relatively rapidly at the same time.[1,2] This has the effect of creating thermal gradients on the surface of the part, which can cause distortion or even cracking. One of the purposes of agitation is to minimize the occurrence of these surface gradients.

Canale et. al. reported that parameters including directionality, flow rate, and turbulence varied significantly from system to system although the propeller rotation was the same.[15] Althernatively, H. Beitz reported at an alternating up-and-down (oscillating) movement of a part in a quench tank as an alternative to rotational agitation.[16]

The performance of a quenchant can be characterized by its ability to extract heat from the part surface. Two ways in which it can be expressed are: 1) measurement of hardness of the quenched part as a function of position beneath the surface and plotting the cross-sectional hardness to yield U-Curves; or 2) measurement of the time-temperature cooling profile at pre-specified locations in a standard test specimen. Therefore, the measurement of time-temperature of the workpiece is essential for controlled quenching. In some cases during cooling, it is possible to measure the workpiece temperature using thermocouples inserted into the surface and/or into the core of the test specimen or component to determine time-temperature cooling curves. However, this is not always possible.

An alternative method reported by Kuyucak involves the measurement of the increase of the water temperature in the vicinity of the cooling surface of the part being quenched a function of time.[17,18] From this data, cooling curves may be calculated which may then used to predict microstructure, hardness and residual stresses.[19]

This paper describes the use of the Kuyucak method to predict the as-quenched hardness of a medium alloy steel (AISI 4140) when quenched into water.

## EXPERIMENTAL

### Agitation System

The equipment used to perform the quenching experiments was constructed in the NTT laboratory of the Department of Materials, Automotive and Aeronautics, the School of Engineering of São Carlos, University of São Paulo. A schematic illustration of the equipment design and computer data acquisition system is in Figure 2.

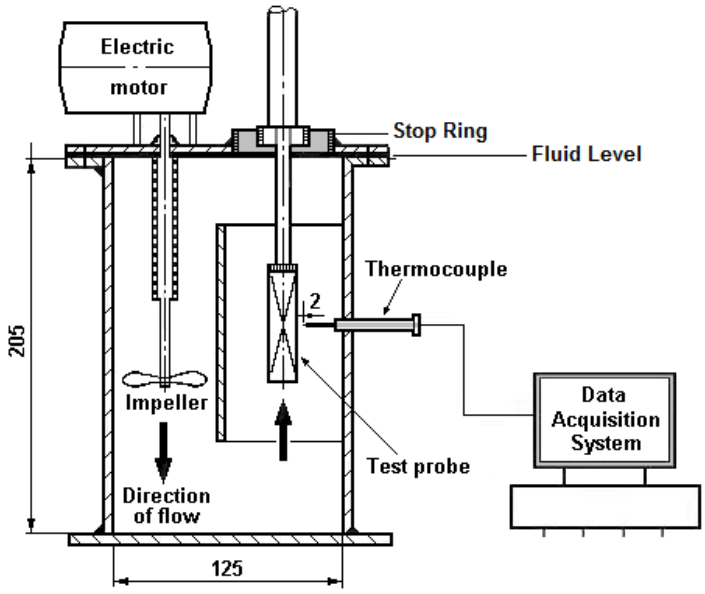



**Figure 2.** Schematic illustration of the laboratory quenching and data acquisition system.

The quench tank and agitation system is a modification of the Tensi agitation device described in ASTM D6482–99 Standard Test Method for Determination of Cooling Characteristics of Aqueous Polymer Quenchants by Cooling Curve Analysis with Agitation (Tensi Method) which consists of a rectangular acrylic tank with the following dimensions: 125 x 60 mm wide and 205 mm height, with volume of fluid of 1450 mL and weight 7.6 kg; a motor and controller to provide agitation via an impeller; computer data acquisition/processing system developed by FAC Ltda. Figure 2 shows a schematic of the agitation devise which is a modification of the Tensi agitation device described in ASTM D6482.

The agitation system consists of:

a) Electric motor with speed controller to maintain impeller rotational speed at 2000 rpm as specified in D6482.

b) A three-blade marine impeller (50 mm diameter, 3 blades, 42 mm pitch setting).

The rotational (rpm) speed was calibrated using a commercially available optical tachometer.

The near-surface quenchant temperature was monitored with respect to time using a type K thermocouple placed in the water near the probe surface through a hollow steel tube to allow it to be clamped in place without movement, at 2 mm from the surface of the test specimen as shown in Figure 2. The time-temperature data was collected by data acquisition/processing system at a data collection frequency of 20 Hz.

Before testing, the agitation device was cleaned by filling the container with water and then agitating using the impeller stirrer for one minute. This process was repeated this twice with clean distilled water. If necessary, remove cover and clean the container with water and a cloth. After cleaning, reassemble assuring that the screws are not fastened too tightly which could lead to cracking of the acrylic tank.

After thoroughly cleaning the system, the device was filled with preheated tap water (30 and 45ºC) up to the lower distance ring as shown in Figure 2 and then

manually tilted from side-to-side to assure the release of any air which may be trapped in the shaft packing box. For this study, the rotational speed of the impeller shaft was 750 rpm. The position of the probe in the system is depicted in Figure 3b. Note: It is essential that there be no bubbles in the device during agitation. Therefore, all entrained air and air pockets must be eliminated.

The test probe body, see Figure 3a, is then positioned vertically as indicated in the Figure 3b, at half the height of the test chamber using the stop ring on the shaft of the test probe.

## Test Probe

Cylindrical test specimens, 20 mm diameter and 75 mm in length attached to a removable handle as shown in Figure 4 were used in this study. The bars were austenitized in a preheated furnace at 900°C for 4140 for 1 h and then transferred into the 1450 mL agitated quenchant tank as shown in Figure 3.

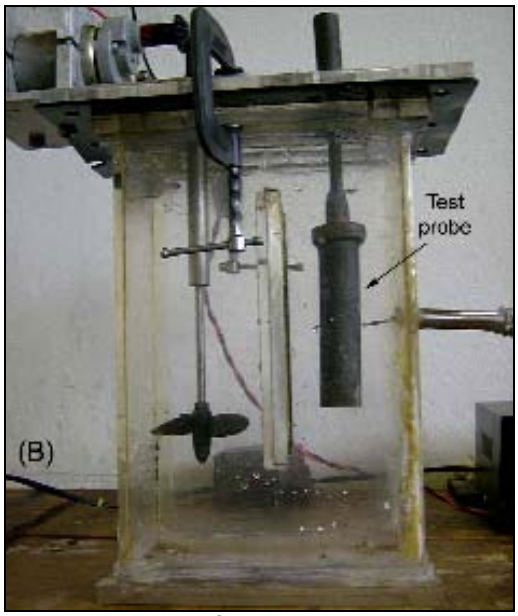



**Figure 3.** Position of the test sample in the tank.

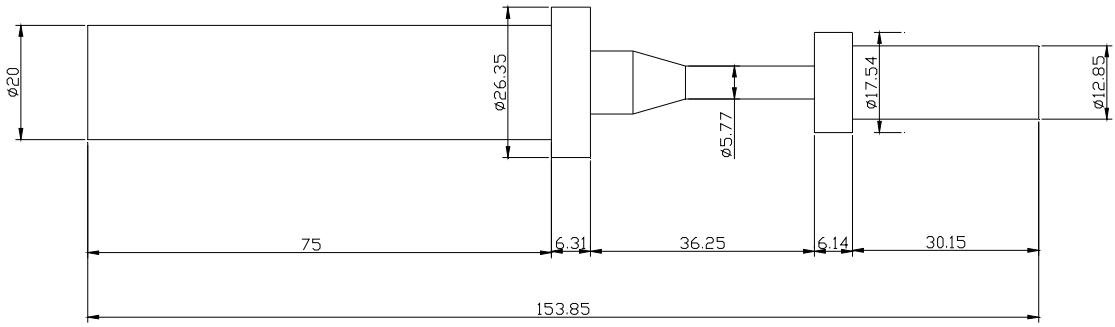



**Figure 4.** Schemic illustration of the dimensions of the test probe and the handle shown in Figure 3.

The material selected for the present study is AISI 4140 steel. The nominal chemical composition for the present study is indicated in Table 1. The accepted range of chemical composition for AISI 4140 is also displayed in Table 1.

**Table 1.** Range of chemical composition of 4140 steel (wt%) and nominal chemical composition for the probe

| Steel | C | Mn | P | S | Si | Mo | Cr |
|---|---|---|---|---|---|---|---|
| **4140** | 0.39 | 0.82 | 0.005 | - | 0.02 | 0.17 | 0.9 |
| **4140** | 0.38-0.43 | 0.75-1.00 | max. 0.035 | - | Max. 0.04 | 0.15-0.35 | 0.8-1.10 |

Hardenabily is reflected by the ideal diameter (DI). The ideal diameter is that diameter that contains 50% martensite when quenched. The DI value was 99.5 for AISI 4140.

## As-Quenched Property Characterization

After the quenching process, the samples were sectioned as shown in Figure 5, and the cross-sectional hardness was determined. The Rockwell C (HRC) hardness values were measured according to ASTM E92-82 - Standard Test Method for Vickers Hardness of Metallic Materials carefully measured positions across the radius of the bar.

After hardness measurements, the samples were ground with SiC sandpapers and polished with alumina of 0.3μm and 0.05μm according to ASTM E3-01 Standard Guide for Preparation of Metallographic Specimens and were etched with nital. Microstructures were determined using Philips XL -30 scanning microscope by 2000X magnification according to ASTM E883-02 Standard Guide for Reflected–Light Photomicrography.

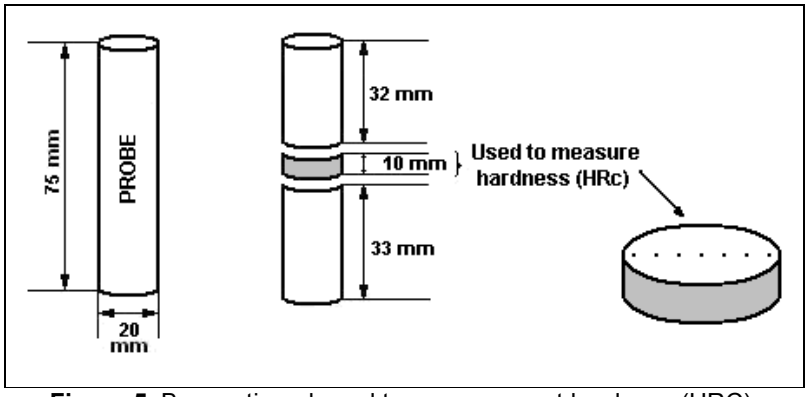



**Figure 5.** Bar sectioned used to measurement hardness (HRC).

## Modeling Procedure

The procedure for modeling the experimental results is based on the Kuyucak et. al. procedure[10,11] and was outlined in detail in a previous paper.[12] A calorimetric energy balance may be stated as:

$$m_{qu} c_{qu} \left( \overline{T}_{qu}(t) - \overline{T}_{qu}(0) \right) = \int_0^t q_p(t) A_t dt \quad , \qquad (1)$$

where $m_{qu}$ is the total mass of the quenchant; $c_{qu}$ is the specific heat of the quenchant; $T_{qu}(t)$ is the temperature of the quenchant as a function of the time $t$; $q_p(t)$ is the heat flux assumed uniform at all the surface of the probe; and $A_t$ is the total area of the probe.

From Eq. (1) an expression for the heat flux transferred from the probe to the quenchant can be derived if the evolution of the mean temperature of the bath is known:

$$q_p(t) = \frac{m_{qu} c_{qu}}{A_t} \frac{dT_{qu}(t)}{dt} \qquad (2)$$

The temperature evolution within the probe can be predicted by solving the heat conduction equation:

$$\frac{1}{r}\frac{\partial}{\partial r}\left(rk\frac{\partial T}{\partial r}\right) + \frac{\partial}{\partial z}\left(k\frac{\partial T}{\partial z}\right) + Q = c\rho\frac{\partial T}{\partial t} \quad , \qquad (3)$$

where $T$ is the temperature of the probe in (ºC); $k$ the thermal conductivity in (W/mºC); $c$ is the specific heat (J/kgºC); $\rho$ is the density in (kg/m$^3$); $Q$ the heat source by enthalpies of phase transformations (W/m$^3$). The differential equation (3) must be solved subjected to the boundary condition that results from (2) at the surface of the probe in contact with the quenchant.

## RESULTS AND DISCUSSION

### Near Surface Quenchant Temperature Variation During the Quench

The temperature of the bath was controlled to optimize the quenching process in terms of steel microstructure and hardness. Water at 30 °C and 45 °C was used as quenchant media for the 4140 steel. Although a high quench rate is essential to achieve high strength, in many cases, high quench rates with cold water ≤ 30 °C cannot be used due to formation of high thermal stresses and distortion. One way to minimize this problem is to increase the water temperature so that the temperature gradient between water and the part being quenched can be reduced. Figure 6 shows the time-temperature data of the near-surface water quenching temperature for both tests.

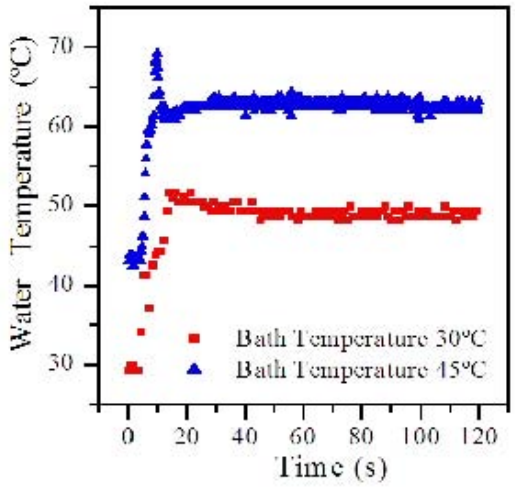



**Figure 6.** The effect of the water temperature during cooling of 4140 steel.

## Hardness Measurements

Hardness measurements were obtained to assess the influence of water temperature. Figure 7 shows the cross-sectional hardness obtained for the high hardenability 4140 steel at 30 and 45˚C bath temperatures. These two water temperatures yielded essentially the same surface hardness with relatively little decrease from the surface to the core. The 30 ˚C water quenchant did yield essentially complete through-hardening. However, the 45 ˚C water temperature did yield slightly reduced core hardness but not significantly different.

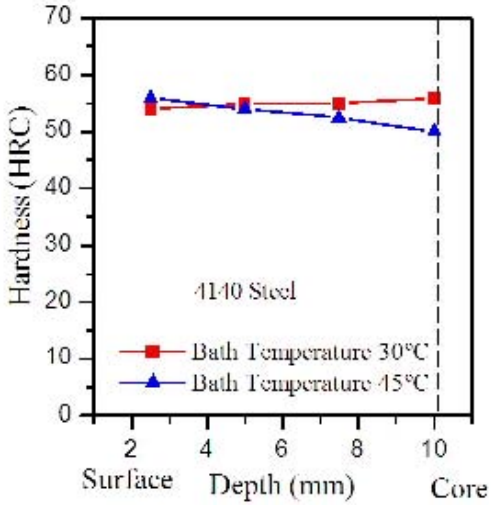
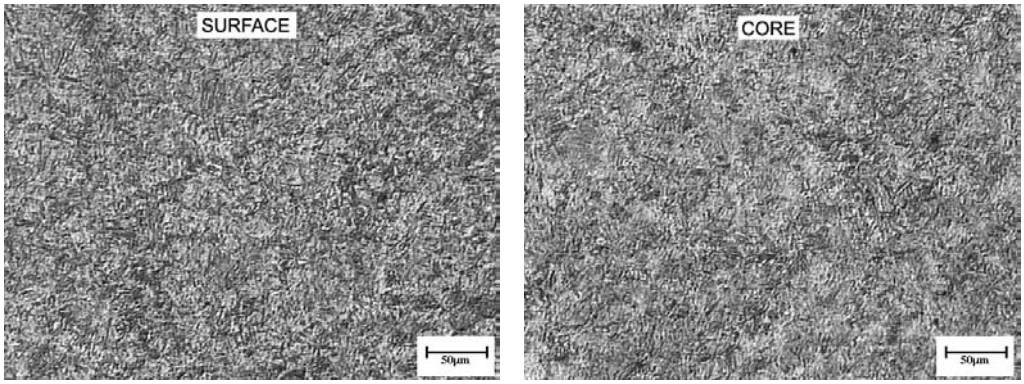



**Figure 7.** Hardness profile of 4140 steel, quenched in water, with different bath temperatures.

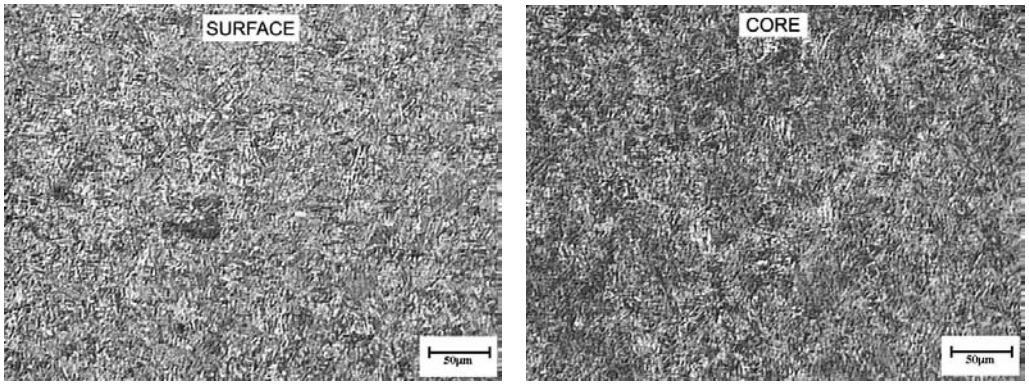
## Microstructure of 4140 Steel

From microstructural analysis, it was observed the microstructure of the test specimen were not influenced by the water temperature. The hardenable 4140 steel test specimens exhibited a martensitic microstructure at both water temperatures as shown in Figures 8 and 9.




*(a)* *(b)*

**Figure 8.** Microstructure of 4140 steel, quenched in water (30°C). a) Surface sample, b) core sample. They show martensitic microstructure.




(a) (b)

**Figure 9.** Microstructure of 4140 steel, quenched in water (45°C). a) Surface sample, b) core sample. They show martensitic microstructure.

## Numerical Results. Predicted Hardness of 4140 Steels Probes

A sigmoidal function was fitted to the measured temperature data of the quenchant shown in Figure 6 (bath temperature 30ºC and 45ºC). Results of the fitting procedure are summarized in Table 2.

$$T_{qu}(t) = \frac{A_1 - A_2}{1 + exp\left(\frac{x - x_0}{dx}\right)} + A_2 \qquad (3)$$

**Table 2.** Parameters of sigmoidal curve fitted to measured bath temperature. 4140 Steel

| Bath Temp. | $A_1$ | $A_2$ | $X_0$ | $dx$ | Chi^2 | $R^2$ |
|---|---|---|---|---|---|---|
| 30ºC | 25.493 | 49.191 | 5.6691 | 2.6692 | 1.35774 | 0.91777 |
| 45ºC | 43.21072 | 62.71488 | 5.79026 | 0.60517 | 0.73836 | 0.95402 |
| 60ºC | 56.9644 | 76.36433 | 16.22313 | 5.42118 | 0.96428 | 0.96716 |

After derivation with respect to time of the sigmoidal function the heat flux is given by:

$$q_p(t) = \frac{m_{qu}c_{qu}}{A_t}\frac{dT_{qu}(t)}{dt} = \left(\frac{m_{qu}c_{qu}}{A_t}\right)\frac{-\frac{(A_1 - A_2)}{dx}\exp\left(\frac{t - x_0}{dx}\right)}{\left(1 + \exp\left(\frac{t - x_0}{dx}\right)\right)^2} \qquad (4)$$

To calculate $q_p(t)$, $A_t$ included the bottom and lateral surface of the steel probe plus the lateral area of the support (see Figure 3).

It was assumed that at the top end; the probe and the support device were almost at the same temperature, with the heat flux mainly in radial direction. Figure 11 shows the calculation domain and boundary conditions considered for the numerical simulation.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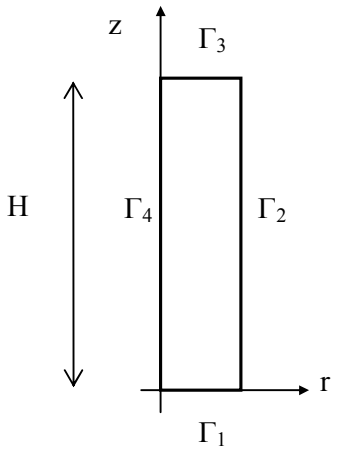


$$-k\vec{n}\cdot\nabla T = q_p(t) \quad on \quad \Gamma_1 + \Gamma_2$$

$$-k\vec{n}\cdot\nabla T = 0 \quad on \quad \Gamma_3 + \Gamma_4$$

**Figure 11.** Calculation domain and applied boundary conditions.

HT-MOD (Heat Treating Modeling)[19] was used to simulate the quenching tests and to predict the hardness distribution of the steel probe taking into account the influence of its chemical composition.[10]

Figure 12 and 13 illustrate the numerical results for the 4140 steel bars at 30ºC and 45ºC water temperature, respectively.The fitted curve to measured bath temperature is shown in figure (a); the estimated heat flux in figure (b); the temperature as a function of time at three points along the radial coordinate at the plane z = H/2 in figure (c) and the cooling rate at the center of the probe in figure (d).

Comparison between measured and predicted hardness of the steel probe quenched in water at 30ºC and 45ºC is shown in Figure 14 (a) and (b), respectively. Simulations were carried out for the nominal chemical compositions given in Table 1. Excellent agreements between calculated and experimental hardness distribution were obtained, being the differences at most 3 HRC.

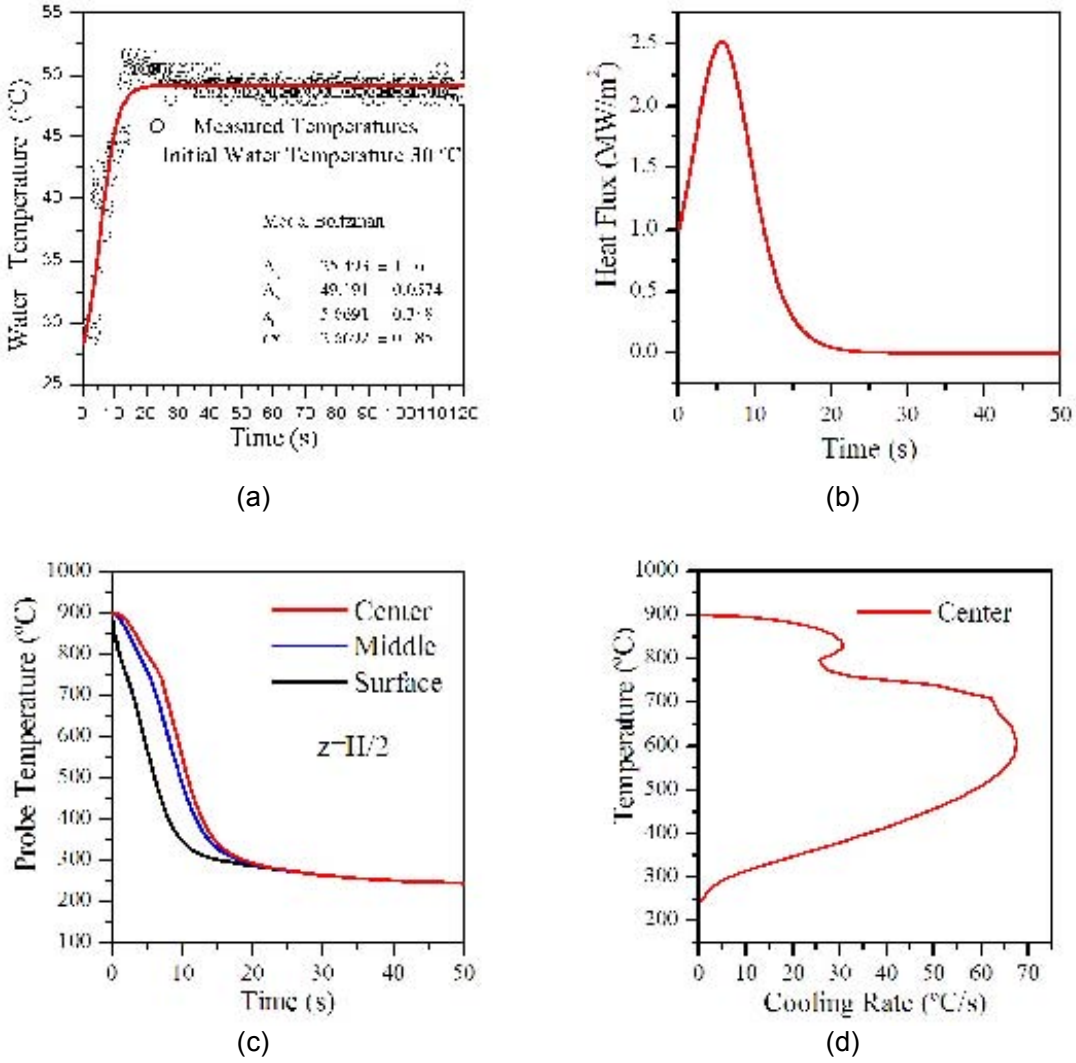


**Figure 12.** Simulation results for 4140 steel, quenched in water (30°C).

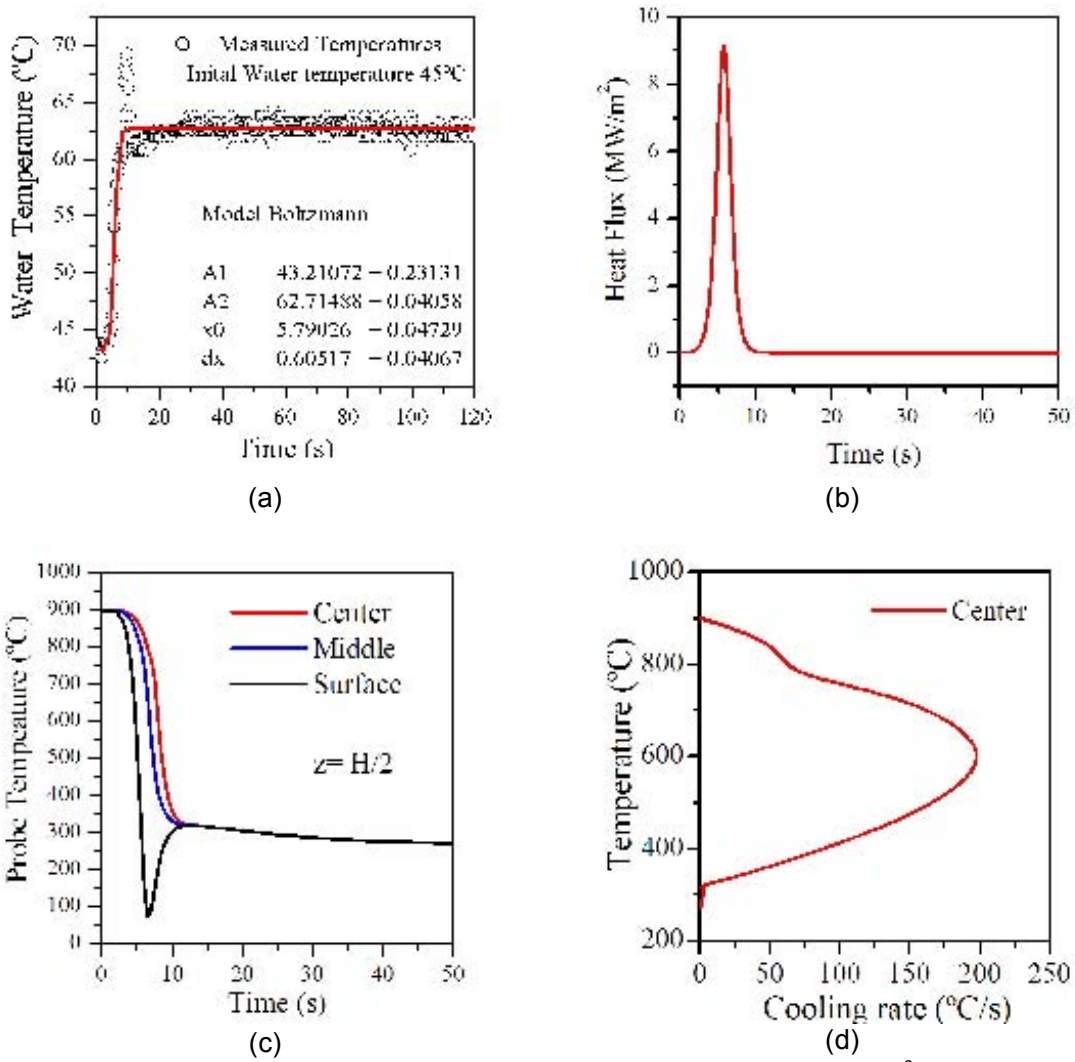


**Figure 13.** Simulation results for 4140 steel, quenched in water (45°C).

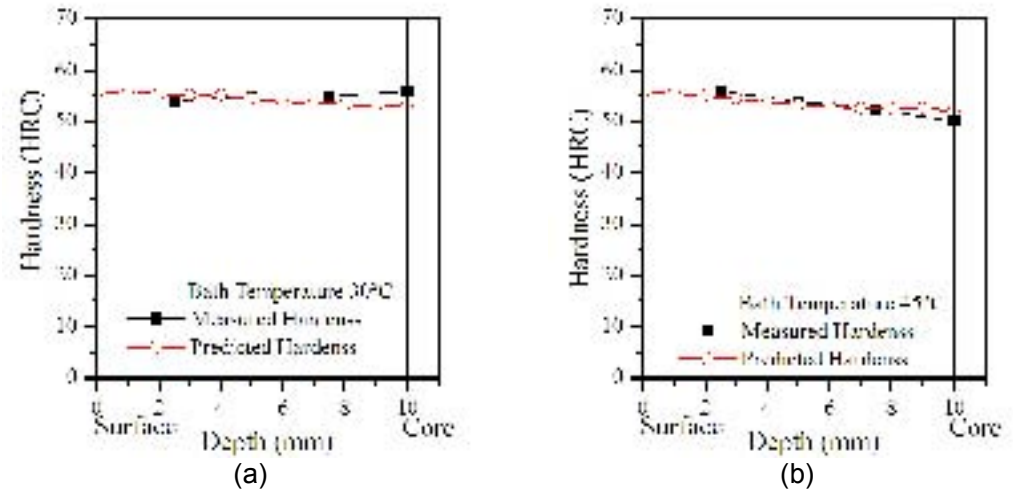


**Figure 14.** Comparison between measured and predicted hardness for 4140 steel quenched in water. (a) 30°C; (b) 45°C.

## CONCLUSIONS

Water is an agent with the highest cooling intensity. The hardness measured is, therefore, suitable high as well. A comparison of the cooling intensity of bath temperature selections can confirm the efficiency of the quenched process.

Under all the bath temperature conditions, the hardness of the quenched samples of 4140 steel showed the high hardness until depth of 7.5 mm.

A comparison of the results of hardness measurements in the transverse direction of the samples permits a conclusion that the effect of the water temperature is not relevant for 4140 steel.

From the microstructure analysis, it was observed the metallographic were not influenced by the bath temperature in the samples. The 4140 steel bars that are highly hardenable show martensitic microstructure at different bath temperatures.

The results obtained in this laboratory examination suggest that “Bath Temperature Measurement” (Macro Calorimeter Method) is a viable and favorable method for obtaining cooling curve data without the use of thermocouples inserted in the workpiece.

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