

# Evaluation Of Hardening Performance of Cooling Media by Using Inverse Heat Conduction Methods and Property Prediction

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*A sequential numerical method for characterization of hardening performance of quenchants applied for steel quenching is outlined here. This novel method is based on the specific processing of measured time-temperature samples performed as a result of cooling curve tests. As a function of surface temperature the heat transfer coefficient, characterises the heat transfer during cooling and is calculated using an iterative inverse algorithm. The heat transfer coefficient is used for the calculation of the microstructural constituents and the hardness profile of cylindrical samples of arbitrary diameters. The hardening performance of the media is evaluated by the estimated hardness of the specimen obtained by heat treatment.*

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## 0 INTRODUCTION

One of the most critical stages of the heat treatment process, and usually the least controllable, is the quenching operation. Improper selection or application of a quenching medium, or a drift in its cooling characteristics during its duration may result in products that do not meet required specifications and may therefore bring additional costs, e.g. reworking, rejection and delayed deliveries.

A greater awareness of the importance of the quenching process came with the introduction, in the last one to two decades, of ISO and ASTM standards for testing cooling media (hardening oils and polymers) and commercial instruments for testing compliance with these standards. Cooling curve analysis (CCA) is considered to be the best technique for the evaluation of cooling performance. Several CCA interpretation methods have been proposed recently [1] and [2], including an empirical hardening-power predictor [3], and a substantial analysis of the cooling process [4]. The method called Quench Factor Analysis, which incorporates phase transformation kinetics into quenchant characterization [5] and [6] is also commonly used.

Outlined in this paper is a complex system, based on a general concept, allowing for the

quantitative characterisation of the heat transfer of a cooling medium, as well as, the mechanical properties of the material developed as a result of hardening.

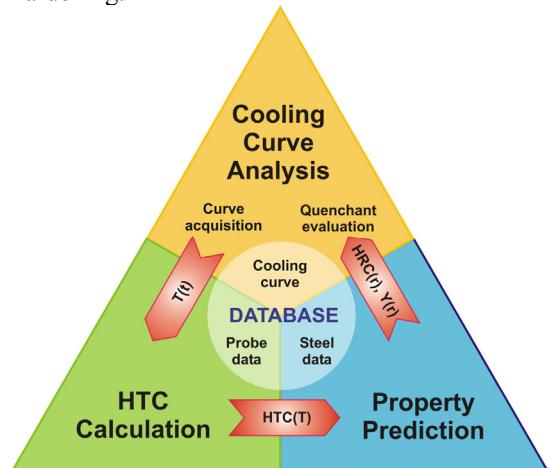


Fig. 1. Scheme of the numerical evaluation method

## 1 PRINCIPLE OF INTEGRATED METHOD

The concept of the method proposed (Fig. 1) is based on the sequential processing of the cooling curves recorded according to ISO 9950:

Step 1: The cooling curve is obtained using ISO 9950. The result of the measurement is the  $T(t)$  function.

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Step 2: The heat transfer coefficient is determined using the measured  $T(t)$  function. The output is the heat transfer coefficient as a function of the temperature  $HTC(T)$

Step 3: Distribution of microstructure and hardness in the cross-section of a cylindrical plain carbon steel (0.45% C) specimen is predicted on the bases of steel properties and calculated  $HTC(T)$

Step 4: The quenchant evaluation is made using the calculated hardness.

The software called SQintegra for the IVF SmartQuench instrument was developed to provide the functions for the integrated method proposed. SQintegra is an enhanced version of the IVF SmartQuench software which allows the user to perform an advanced evaluation of the recorded quenchant data and to carry out "virtual testing" of quenchant performance. It contains two modules: the SQ inverse module, which is used to calculate heat transfer coefficients, and the SQ property prediction module, which is used for hardenability calculations. The function applied for quenchant evaluation is detailed briefly below.

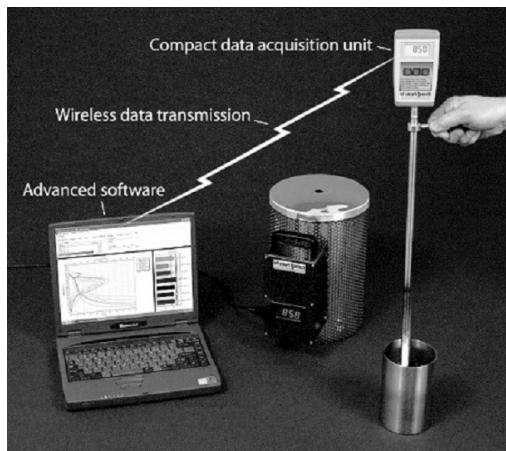


Fig. 2. The cooling curve acquisition system

### 1.1 Cooling Curve Acquisition System

The IVF SmartQuench system (Fig 2.) based on the ISO 9950 was introduced in 2003 [7]. The evaluation process of the quenching medium is performed in three steps. First, the cylindrical probe is heated up to 850 °C in the furnace. Secondly, the probe is placed to the tank

filled with the quenchant is investigated. The temperature is recorded during cooling by the thermocouple located at the centerline of the probe. As the third step, the cooling curve obtained is analysed or used for further evaluation.

### 1.2 HTC Approximation Module

In the SQ inverse module, an iterative procedure is used to estimate the temperature dependent heat transfer coefficients. The inverse numerical method is based on the following assumptions.

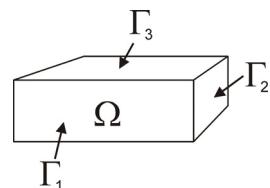


Fig. 3. The representation of the domain

The temperature distribution inside a homogeneous isotropic domain  $\Omega$  with constant material properties (Fig 3.) is governed by:

$$\nabla(k(\mathbf{r}, T)\nabla T) + Q(T, \mathbf{r}, t) = c_p(\mathbf{r}, T)\rho(\mathbf{r}, t)\frac{\partial T}{\partial t}. \quad (1)$$

where  $\mathbf{r}$  is the spatial vector and  $\mathbf{r} \in \Omega$ ,  $t$  is the time,  $k$  is the heat conductivity,  $T$  is the temperature,  $c_p$  is the specific heat,  $\rho$  is the density and  $Q$  is the latent heat. The initial condition is :

$$T(\mathbf{r}, t=0) = T_0(\mathbf{r}) \quad (2)$$

where  $T_0$  is the initial temperature of the domain. The boundary conditions are expressed by:

$$-k \frac{\partial T}{\partial \mathbf{r}} = h_i(T(\mathbf{r}, t) - T_{am}) \text{ in } \Gamma_i \quad i = 1, \dots, p \quad (3)$$

where  $h_i$  are the heat transfer coefficients corresponding to different portions of the boundary ( $\Gamma_1 \cup \Gamma_2 \dots \cup \Gamma_p = \Gamma$  and  $\Gamma_1 \cap \Gamma_2 \dots \cap \Gamma_p = \emptyset$ ) and  $T_{am}$  is the ambient temperature. Each one of these  $p$  boundary zones has a time dependent heat transfer coefficient to be optimized. The time dependence of the heat transfer coefficient can be approximated by polygonal functions, each one defined by a set of parameters  $h_i^{(r)} = (r=1 \dots p; i=1 \dots q)$ , according to Fig. 4.

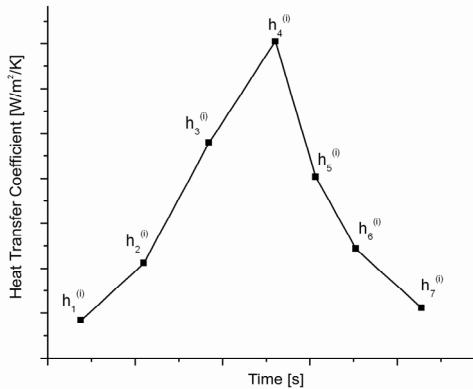


Fig. 4. Time approximations of heat transfer coefficients

The unknown design parameters can be expressed by the vector of  $m$  ( $m = p * q$ ), components  $\tau = (\tau_1, \dots, \tau_m) = (h_1^{(1)}, \dots, h_q^{(1)}, h_1^{(2)}, \dots, h_q^{(2)}, \dots, h_1^{(p)}, \dots, h_q^{(p)})$ . The temperature at different instants of time is given by measurements at  $n$  points in the solid region, located at  $r_k$ , ( $k = 1 \dots n$ ). On calling  $T_k^m$ , the measured temperatures, and  $T_k^c$ , the numerically calculated temperature at those points, one can pose the problem of obtaining the values of the heat transfer coefficients  $\tau_i$  that minimize the function:

$$S = S(\tau_1, \dots, \tau_m) = \sum_{k=1}^n (T_k^m - T_k^c)^2 = \min \quad (4)$$

being  $n$  the total number of measured temperatures, i.e. the number of points times the number of measurements at each point. A necessary condition to satisfy is that the following set of equations must be verified simultaneously:

$$F_i = \frac{\partial S}{\partial \tau_i} = -2 \sum_{k=1}^n (T_k^m - T_k^c) \frac{\partial T_k^c}{\partial \tau_i} = 0 \quad (5)$$

where  $i = 1 \dots m$ . To obtain a non-linear system of equations in the unknowns design parameters  $\tau_i$ , it is supposed that an approximated solution of this system is available  $\tau^{(0)} = (\tau_1^{(0)}, \dots, \tau_m^{(0)})$ , such that in first approximation:

$$T_k^c = G_k(\tau) \approx G_k(\tau^{(0)}) + \sum_{j=1}^m \frac{\partial G_k}{\partial \tau_j} \Delta \tau_j^{(1)} \quad (6)$$

where  $\Delta \tau_j = \tau_j^{(1)} - \tau_j^{(0)}$ . Contracting the Eqs. above, it can be written:

$$\sum_{j=1}^m \left[ T_k^m - G_k(\tau^{(0)}) - \sum_{j=1}^m \frac{\partial G_k}{\partial \tau_j} \Delta \tau_j^{(1)} \right] \frac{\partial G_k}{\partial \tau_i} = 0 \quad (7)$$

where  $i = 1 \dots m$  or after changing the summation order and rearranging terms:

$$\sum_{j=1}^m \left[ \sum_{k=1}^n \frac{\partial G_k}{\partial \tau_i} \frac{\partial G_k}{\partial \tau_j} \right] \Delta \tau_j = \sum_{k=1}^n \left[ T_k^m - G_k(\tau^{(0)}) \right] \frac{\partial G_k}{\partial \tau_i}. \quad (8)$$

Expressions are the *normal equations* of the optimization problem:

$$\mathbf{A}^{(1)} \Delta \tau^{(1)} = \mathbf{b}. \quad (9)$$

The matrix elements of this linear system are calculated with:

$$\mathbf{A}_{ij}^{(1)} = \sum_{k=1}^n \frac{\partial G_k}{\partial \tau_i} \frac{\partial G_k}{\partial \tau_j} \quad (10)$$

where ( $i = 1 \dots m$  and  $j = 1 \dots m$ ) and the components of the independent term applying:

$$\mathbf{b}_i^{(1)} = \sum_{k=1}^n \left[ T_k^m - G_k(\tau^{(0)}) \right] \frac{\partial G_k}{\partial \tau_i} \quad (11)$$

where  $i = 1 \dots m$ . The derivatives (sensitivity coefficients)  $\partial G_k / \partial \tau_i$  ( $k = 1 \dots n$ ) can be evaluated numerically and a central difference scheme is adopted here:

$$\frac{\partial G_k}{\partial \tau_i} \approx \frac{G_k(\tau_i^{(0)} + \varepsilon) - G_k(\tau_i^{(0)} - \varepsilon)}{2\varepsilon} \quad (12)$$

where the  $G_k(\tau_i \pm \varepsilon)$  are the calculated temperatures at each point  $r_k$ , increasing or decreasing the coefficient  $\tau_i^{(0)}$  in a small quantity  $\varepsilon$ .

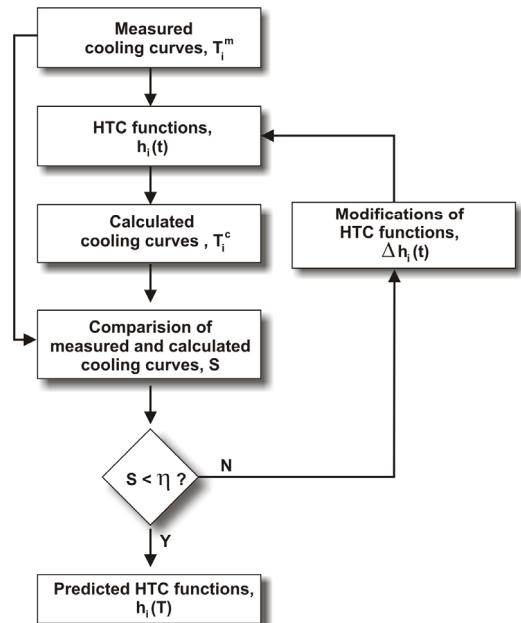


Fig. 5. The scheme of the inverse algorithm

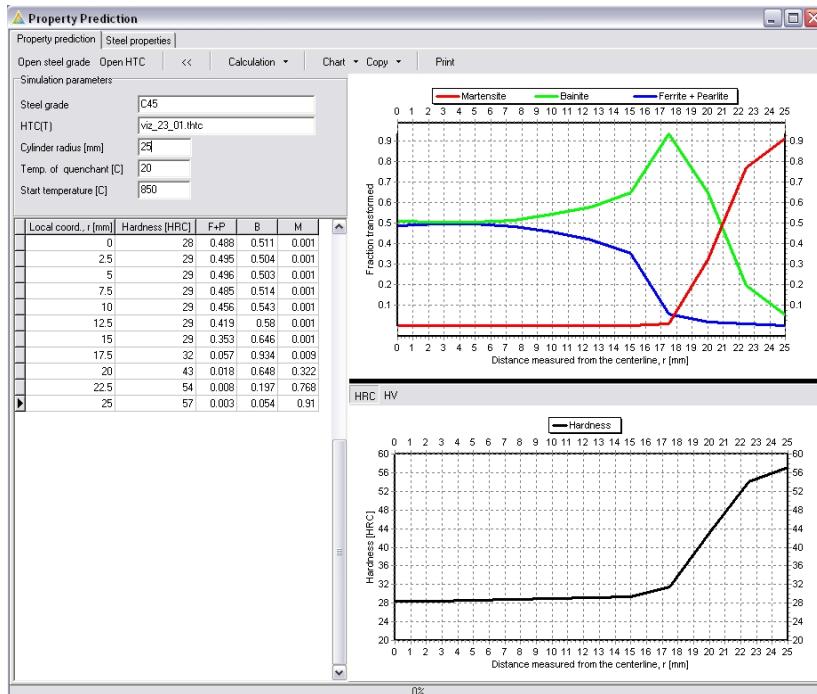


Fig. 6. Screenshot of SQi software: property prediction module

After solving the linear system, an updated approximation to the optimization problem is obtained:

$$\tau_j^{(1)} = \tau_j^{(0)} + \Delta\tau^{(1)}. \quad (13)$$

On making use of  $\tau_i^{(1)}$  ( $i = 1, 2, \dots, m$ ), an improved approximation can be obtained  $\tau_i^{(2)}$  ( $i = 1, 2, \dots, m$ ) by solving a new linear system:

$$\mathbf{A}^{(2)} \Delta\tau_i^{(2)} = \mathbf{b}^{(2)} \quad (14)$$

$$\tau^{(2)} = \tau^{(1)} + \Delta\tau^{(2)}. \quad (15)$$

This iterative procedure is repeated until corrections  $\Delta\bar{\tau}(k)$  between measured and estimated values of temperatures, satisfy certain convergence criterion (Fig. 5).

### 1.3 Property Prediction Module

The calculated HTC can be used to perform hardenability calculations in the property prediction module of the SQintegra software. These calculations are based on a TTT approach published earlier [8] and [9]. The hardness is predicted using individual isothermal hardness of the microstructural elements ferrite, pearlite, bainite and martensite (Fig. 6). In the calculation, the transformed amounts of austenite on each

isothermal step and their individual isothermal hardness are taken into account.

## 2 VALIDATION OF THE METHOD

In order to verify the applicability of the integrated evaluation technique, an indirect approach has been taken. The validation is based on the comparison of hardness measured of specimen hardened in the medium evaluated and the hardness predicted using the heat transfer coefficient derived from the cooling curves in the same coolant.

Quenching and numerical experiments have been carried out in order to verify the method proposed. Oil and water quenchants at 30 and 70 °C were used for the physical experiments. Cooling curves were measured in each medium at the proper temperature and then the heat transfer coefficients were determined. The hardness as a function of distance measured from the centreline of the cylinder was predicted using the HTC (T).

Cylindrical specimens of 12.5 mm in diameter prepared from plain carbon steel (the chemical composition is 0.45 %C, 0.25 %Si, 0.65 %Mn, 0.018 %P, 0.018 %S, 0.19 %Cr, 0.13 %Ni,

0.042 %Mo, 0.03 %V, 0.15 %Cu) were quenched from 850 °C in the coolants. The hardness at the cross-section of the work pieces was measured and compared with the estimates (Figs. 7 and 8). Comparing the predicted ( $HRC^c$ ) and measured ( $HRC^m$ ) hardness profiles, the agreement is satisfactory. Based on the result of comparative tests, the applicability of the method developed is regarded as proven.

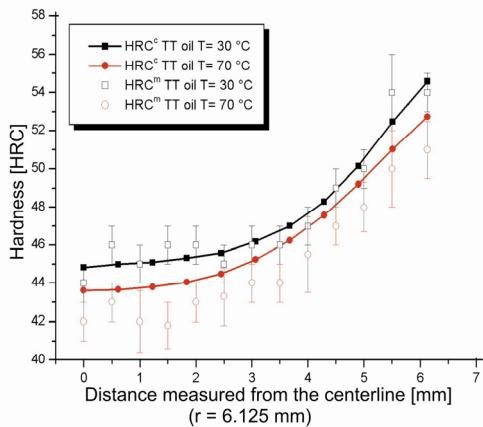


Fig. 7. Predicted ( $HRC^c$ ) and measured ( $HRC^m$ ) hardness as function of distance measured from centreline of plain carbon steel rod (12.5 mm diameter) quenched in oil

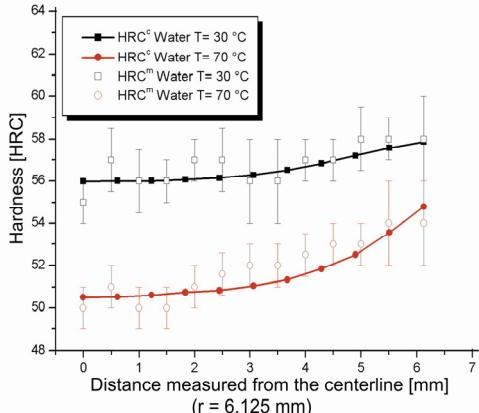


Fig. 8. Predicted ( $HRC^c$ ) and measured ( $HRC^m$ ) hardness as function of distance measured from centreline of plain carbon steel rod (12.5 mm diameter) quenched in water

### 3 CASE STUDIES

The evaluation procedure is demonstrated on characterisation of cooling power of oil and water based polymer (PVP) solutions by using

different temperatures and agitation conditions of the cooling media. The "Tensi agitation unit" has been applied for stirring the coolants [2].

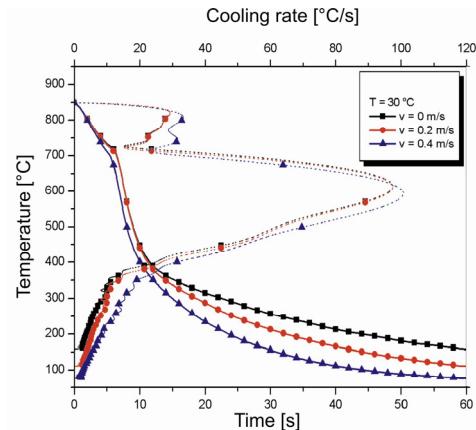


Fig. 9. Cooling curves recorded in oil

The Q8 oil from Bellini FN has been used as oil quenchant. The cooling curves were acquired in the coolant at  $T = 30$  °C using agitation rates  $v = 0, 0.2$  and  $0.4$  m/s (Fig. 9). The significant effect of stirring can be seen from heat transfer coefficients calculated according to each agitation rate (Fig. 10). The peak  $HTC$  function developed using an agitation rate  $0.4$  m/s, while there was no considerable difference noted between the static and low agitated medium rate ( $0.2$  m/s). The predicted hardness profiles illustrate the same observation (Fig. 11), the higher hardness in the centerline and the surface refers to the highest agitation rate, while the lower stirring rate has no significant hardening effect. The results of the analysis also confirm the known observations, i.e. the higher the agitation rate, the greater the hardness. The results also

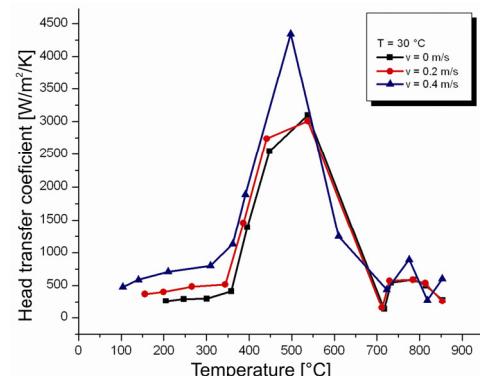


Fig. 10. Heat transfer coefficients as function of temperature

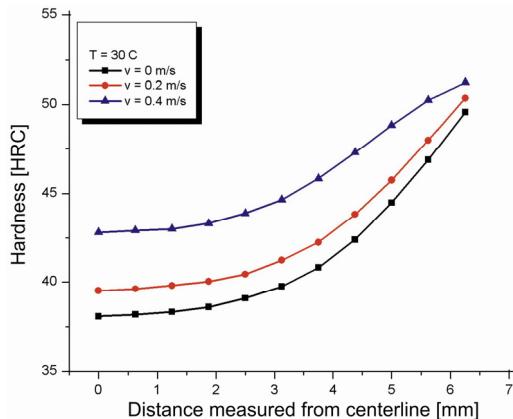


Fig. 11. Predicted hardness profiles as function of distance measured from centreline of plain carbon steel cylinder

characterise the stirring rate by a quantitative criterion (hardness profiles).

The water solutions of FeroQuench 2000 polyamide additive (producer: PETROFER-CHEMIE GmbH) medium has been analysed by applying different quenching conditions. The effects of concentration, agitation rate and the temperature of the quenchant were investigated by the analysis of the polymer solution. The cooling curves were recorded in 5, 10 and 15% polymer solutions at three different temperatures, 30, 40 and 50 °C, and at agitation rates 0, 0.2, 0.4 and 0.6 m/s.

The hardness estimated at the centreline of the plain carbon steel cylinder (12.5 mm diameter) was applied as a quantitative criterion to evaluate the hardening performance of the quenching medium. The hardness values as functions of concentration, temperature and agitation rate are demonstrated in Figs. 12 and 13.

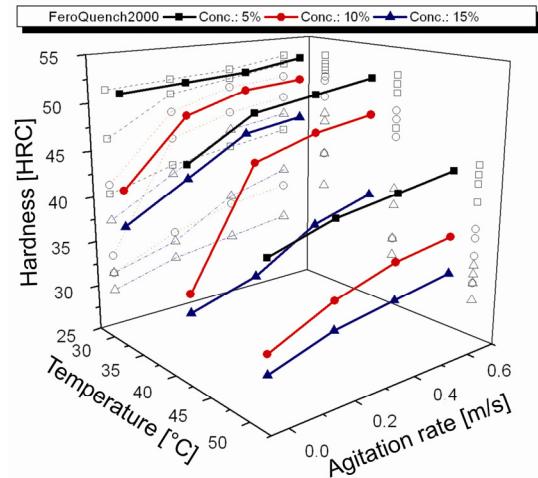


Fig. 12. Hardness predicted at centreline of plain carbon steel cylinder as functions of concentration of polymer additive, temperature of quenchant and agitation rate

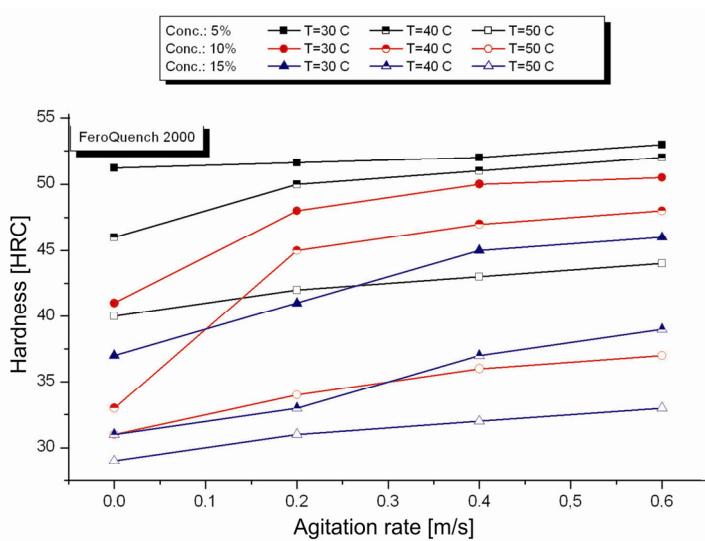


Fig. 13. Hardness predicted at centreline of plain carbon steel cylinder as functions of process parameters affecting hardening performance

The following observations can be drawn:

1. Only a limited effect of agitation rate is given using high concentration at high temperature of the coolant as well as low concentration at low temperature. So, in these cases, it is unnecessary or pointless to agitate the quenchant.
2. A sort of "compensation effect" is shown among the three parameters in the range of 30 to 50 HRC. The same hardness (i.e. similar hardening performance) can be achieved using different combinations of the three parameters. For example, 45 HRC hardness can be achieved by quenching in 5, 10 and 15% polymer solutions at 30 and 40°C using the proper agitation conditions. The predicted results imply that the set of quenching parameters investigated are "equivalent" with respect to the properties developed during the hardening process.

The practical benefits provided by the method, and the modified software result from the appropriate combination of process parameters selected for the desired hardening performance.

#### 4 CONCLUSIONS

A novel, integrated approach and software has been developed to evaluate and compare the performance of quenchants. The method is based on the calculation of a heat transfer coefficient derived from recorded cooling curves and using this data as an input for the simulation of the quenching process. The characterisation of the hardening performance of a cooling medium is based on the predicted hardness at the cross-section of a plain carbon (0.45 %C) steel cylinder. The appropriate set of process parameters effecting the cooling performance of a quenchant, i.e. temperature, concentration, agitation rate, etc., can be determined using the method and software outlined in this work.

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