



MEASUREMENT OF QUENCHING INTENSITY, CALCULATION OF HEAT TRANSFER COEFFICIENT AND GLOBAL DATABASE OF LIQUID QUENCHANTS

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Resume

This paper explains the need for a database of cooling intensities for liquid quenchants, in order to predict the quench hardness, microstructure, stresses and distortion, when real engineering components of complex geometry are quenched. The existing laboratory procedures for cooling intensity evaluation, using small test specimens, and Lumped-Heat-Capacity Method for calculation of heat transfer coefficient, are presented. Temperature Gradient Method for heat transfer calculation in workshop conditions, when using the Liscic/Petrofer probe, has been elaborated. Critical heat flux densities and their relation to the initial heat flux density, is explained. Specific facilities for testing quenching intensity in workshop conditions, are shown.

The two phase project of the International Federation for Heat Treatment and Surface Engineering (IFHTSE), as recently approved, is mentioned.

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1. Introduction

When hardening real engineering components, modern quenching technology uses different quenchants (different kinds of oils, different polymer-solutions and other water-based solutions, molten salt-baths, fluidized beds, and fast moving compressed gases). Any of these quenchants may be used in different conditions (bath temperatures and agitation rates), thus contributing to the immense number of possible combinations.

There are also different quenching techniques: direct immersion quenching; intensive quenching; interrupted quenching; delayed quenching; martempering; austempering; spray quenching.

Yet there is no generally recognized method and technique for the measurement, recording and comparison the relative cooling intensities of different quenchants. From the other side computer modeling and simulation

enable today scientifically based prediction of quench-hardness, microstructure, stresses and distortion in quenched workpieces. An indispensable prerequisite for any computer model is adequate heat transfer data for the entire duration of the quenching process, for actual quenching medium and quenching conditions. Currently, for measuring and recording the cooling intensity of liquid quenchants, the tests used are predominantly laboratory type (ISO 9950 and ASTM D 6200 for oils, and ASTM D 6482 and ASTM D 6549 for polymer-solutions), using a small specimen of 12.5 mm diameter × 60 mm.

The results of these tests (temperature/time and temperature/cooling rate plots – see Fig. 1) are not directly applicable to real engineering components, because they do not provide heat transfer data for the complete quenching process of a real workpiece, and because the workshop conditions at quenching widely differ from those in laboratory tests.

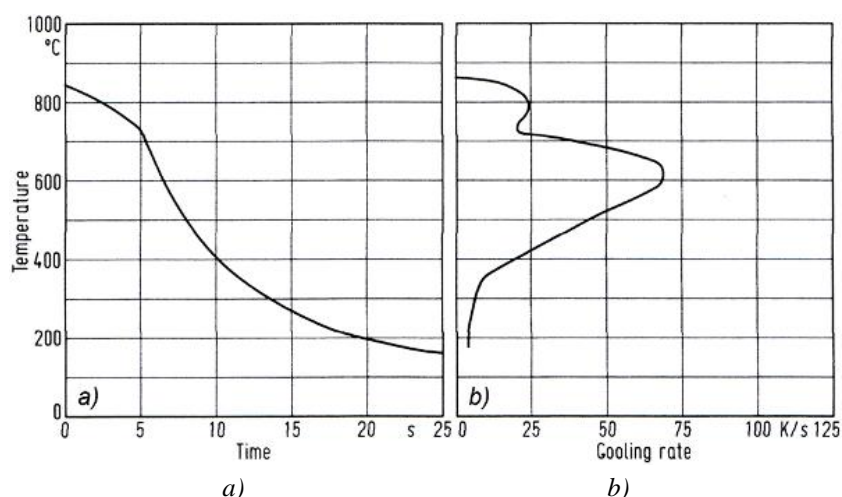


Fig. 1. Typical a) temperature/time and b) temperature/cooling rate plots for test specimen 12.5 mm diameter \times 60 mm quenched in oil, according to ISO 9950 – Industrial quenching oils – Determination of cooling characteristics – Laboratory test method

There is need to have a database of cooling intensities, also in workshop conditions, for different kinds of liquid quenchants, as a tool for designers and workshop engineers, available worldwide.

The absence of adequate heat transfer data results in many cases of less than optimal selection of quenchant and quenching conditions. This leads to poor quality of manufactured workpieces, rework or even scrap. Hence, millions of dollars could be saved in manufacture, if designers and engineers in the workplace had adequate comparative data for cooling intensities of different quenching media with specified conditions.

Given such a database, as a tool for computer programmers, computer modeling and prediction of quench-hardness, microstructure, stresses and distortion can become a normal practice when quenching real engineering components of complex geometry.

The foreseen project for development of the database is two phase:

Phase 1: Compiling the experimental results from different investigators, and establishing the database.

Phase 2: Further development of computer models (3-D) for engineering components

with complex geometry, using the results from Phase 1.

Compiling the database, a reasonable number of selected quenchants will be included, every of them with specified conditions as they are normally used. In [8] Kobasko suggests:

- To use small silver probe for evaluation of critical heat flux densities [1].
- To calculate the heat transfer coefficient based on testing by the Liscic/Petrofer probe and solving the inverse problem [2].
- To apply the noise control system for investigation of transient boiling processes [3].

Taking into account very complicated cooling mechanisms from the one side, and the complex shape of the quenched workpiece (having thin and thick cross-sections, many holes and non-symmetric cavities), from the other side – one has to be aware how difficult task is to provide real heat transfer data at quenching in workshop practice. This situation becomes even more complicated because, as it is known, the heat flux and consequently the heat transfer coefficient (HTC) vary around the workpiece's surface, which in some cases means different duration of the vapour-film phase at different points, and this leads to distortion.

Because we cannot measure the temperature at so many different points on the surface, the question is: what is to be done to obtain real heat transfer data using an appropriate temperature measuring and recording method? The possible solution should incorporate:

- a) The probe itself should be of similar mass and shape as the workpiece to be quenched. This means that for cylindrical workpieces a cylindrical probe, for plate like workpieces a plate like probe, and for ring like workpieces a ring like probe, should be used.
- b) To calculate the HTC in different points on the surface of workpieces having complex geometry, a new advanced 3-D inverse heat conduction model, combined with local heat flux densities for different points, would probably be a possible solution.

For the most frequently used liquid quenchants the database will give the following information:

- Description of chemical and physical properties;
- Specification of test conditions;
- Incorporate own testing methods;
- Laboratory test ISO 9950 with resulting cooling and cooling rate curves, and heat transfer coefficient as function of surface temperature, calculated using a uniformly predetermined mathematical procedure;
- Workshop test using the Liscic/Petrofer probe of 50 mm dia. x 200 mm with three resulting cooling curves and calculated heat transfer coefficient as function of surface temperature and of time, respectively;
- Critical heat flux densities determined by e.g. Japanese New Silver Probe, according to JIS 2242- method B;
- Noise test to determine transition film and nucleate boiling processes.

By applying the newest theory and methods, the foreseen database would supply comprehensive information about cooling characteristics of different quenchants, some of

them not yet widely known, and enable an accurate comparison among them.

Development of contemporary quenching technologies and testing methods, is taking place at many institutions/companies. Some of them from Europe, USA and Japan are widely known. From the other side we do not know enough about relevant developments in fast growing economies – Brasil, Russia, India, China, which actually constitute the major part of potential database users. Because nowadays the industrial production is often transferred from one country to another, the database should be global, i.e. available to everyone in the world.

During the 19th Congress of the International Federation for Heat Treatment and Surface Engineering (IFHTSE) held on 17-20 October 2011 in Glasgow, U.K. the “Liquid Quenchants Database Project” has been officially launched, and Dr. Imre Felde from Hungary was named as its leader. The immediate activity is to establish the project Consortium and its Core Group which will be responsible for:

- General operational management;
- Detailed planning;
- Communications within IFHTSE, public information dissemination and progress reporting.

2. The lumped-heat-capacity method for calculation of the heat transfer coefficient

Fundamental concept of this simple method, according to [4] is the following: If the probe temperature is uniform, the heat loss from the probe Q is equal to the decrease in the internal energy of the probe

$$Q = hA(T_p - T_1) = -c\rho V \frac{dT_p}{dt} \quad (1)$$

where h is the heat transfer coefficient on the probe surface, A is the surface area of the probe, T_p the probe temperature, T_1 the quenchant

temperature, c the specific heat of the probe material, ρ the density of the probe material, V the volume of the probe, t time, and dT_p/dt the cooling rate of the probe.

If the quenchant temperature around the probe T_1 is uniform, the next relation is derived from equation (1):

$$q = h(T_p - T_1) = -\left(c\rho\frac{V}{A}\right)\frac{dT_p}{dt} \quad (2)$$

where q is the heat flux on the probe surface, and

$$h = -c\rho\frac{V}{A}\left(\frac{dT_p/dt}{T_p - T_1}\right) \quad (3)$$

The heat transfer coefficient can be directly calculated from the cooling rate dT_p/dt , therefore the preciseness of q and h values depends on the accuracy of the cooling rate calculated from measured cooling curve data.

The *Lumped-Heat-Capacity Method* can be used only if we can justify the assumption of uniform probe temperature during the cooling process. Temperature distribution in a probe depends on the thermal conductivity of the probe material and the heat transfer from the surface of the probe to the quenchant. In general, the smaller probe size and the higher the thermal conductivity of the probe material, the more realistic the assumption of uniform temperature of the probe.

According to Kobasko it is assumed that a temperature field in a section of the silver probe is uniform, if the Biot number $B_i = hR/\lambda < 0.2$, where R is the radius of the probe and λ is thermal conductivity (for silver λ is 16 times higher than for stainless steel or for Inconel).

Fig. 2a shows the Japanese New Silver Probe of 10 mm diameter and 30 mm length, according to JIS 2242-method B; Fig. 2b shows some cooling curves of different quenchants measured by this silver probe, and Fig. 3 shows the heat transfer

coefficients calculated from these cooling curves, using the *Lumped-Heat-Capacity Method*.

Due to the small size of this probe, and the very high heat conductivity of silver, as it can be seen from Fig. 2b, when quenched in oils this probe cools down to 200 °C in 15 to 20 seconds, and when quenched in brine, water or polymer-solution of low concentration, in less than 2 seconds. Such probe is well suited for laboratory evaluation of the critical heat flux densities of a quenching fluid in the very beginning of the cooling process.

When quenching real workpieces of bigger size, made of steel, the temperature field across its section is not uniform, and the cooling time to 200 °C in the core (depending on the cross-section size and the quenchant's cooling intensity) takes many hundreds seconds, or even several tens of minutes. It is obvious, that the heat transfer coefficient calculated for the small silver probe does not represent the real heat transfer data on the surface of real engineering components, not only because of this vast difference in cooling time, but also because the workshop conditions (fluid flow and direction) greatly differ from those in laboratory tests.

3. Temperature gradient method for heat transfer calculations when real workpieces are quenched

The method itself, described in detail in [6] is based on the known physical rule that the heat flux at the surface of a body is directly proportional to the temperature gradient at the surface, multiplied by the thermal conductivity of the material of the body being cooled:

$$q = \lambda\frac{\partial T}{\partial x} \quad (4)$$

where: q is the heat flux density (W/m²) that is the quantity of heat transferred through a surface unit per unit time, λ is the thermal conductivity of the body material (W/mK), $\partial T/\partial x$ is the temperature gradient inside the body at the body surface, perpendicular to it (K/m).

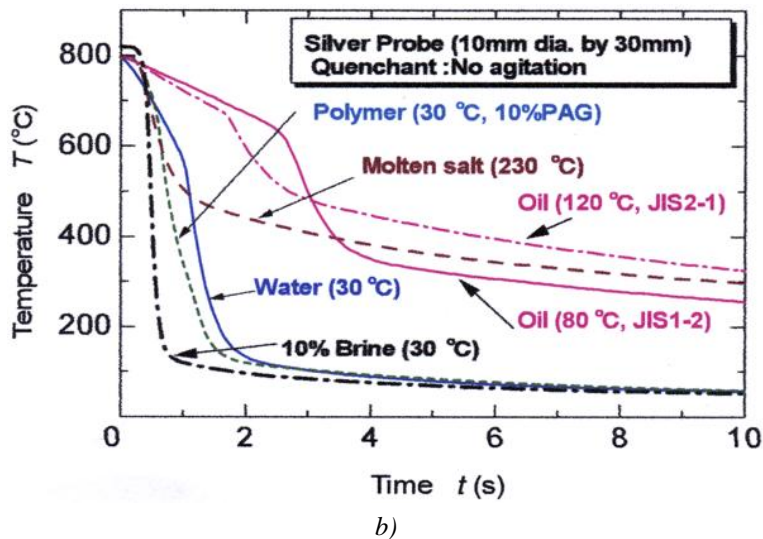
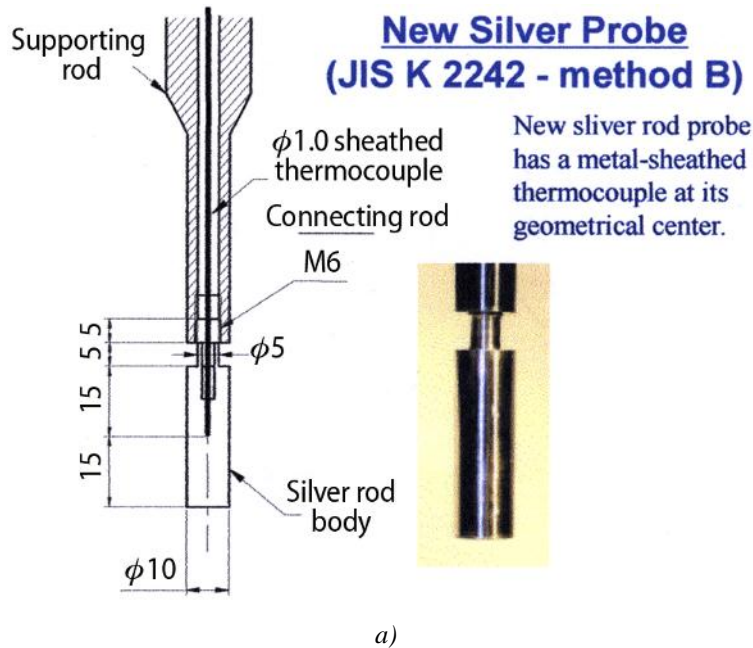


Fig. 2. a) Japanese New Silver Probe of 10 mm diameter × 30 mm according to JIS K 2242 – method B; b) Examples of cooling curves measured by the silver probe [5] (full colour version available online)

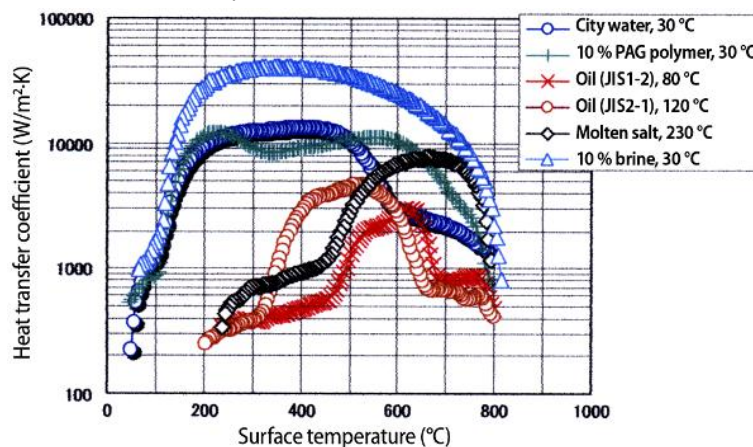


Fig. 3. Examples of calculated heat transfer coefficients from the cooling curves shown in Fig. 2b [5] (full colour version available online)

When using this method for evaluation of the cooling intensity in workshop conditions, the essential feature is the Liscic/Petrofer probe. It is a cylinder of 50 mm diameter × 200 mm length instrumented with three thermocouples placed within the same radius at the half length cross-section. One thermocouple is placed 1 mm below surface, the second one 4.5 mm below surface and the third one at the centre of the cross-section. The probe is made of Inconel 600, the structure of which does not change during heating and cooling, thus there is no latent heat because of structure changes.

When the cooling intensity is to be determined, the probe is heated to 850 °C until its central thermocouple reaches this value, then transferred quickly to the quenching bath and immersed vertically. The probe is connected to a data acquisition system including a personal computer. The data acquisition card contains three A/D converters and amplifiers with a programme enabling digital recording all three thermocouple signal outputs, and simultaneous drawing three cooling curves in real time, see Fig. 4.

Temperature Gradient Method can, of course, be used also for probes of the same design having different diameters, but always the same ratio $L/D = 4:1$. Fig. 5 shows results of such probes of a) 20 mm Dia. × 80 mm and b) 80 mm Dia. × 320 mm, quenched in low viscous accelerated oil of 50 °C with medium agitation.

Irrespective of the probe diameter and mass the *Temperature Gradient Method* exhibits two very important features:

- a) It displays clearly the *dynamic of heat extraction* during the whole quenching process.
 - b) It shows the *initial heat flux density* at the beginning of cooling.
- ad a) The probe of 80 mm Dia. × 320 mm has a mass of 13.6 kg, a ratio surface/volume of only 56 m^{-1} , and a heat capacity of 6045 J/K representing a case of great volume (and heat capacity) and relatively small surface area.

The probe of 20 mm Dia. × 80 mm has a mass of only 0.2 kg, a ratio surface/volume of 225 m^{-1} ,

and a heat capacity of only 94 J/K representing a case of small volume (and heat capacity) and relatively big surface area. The heat capacity of the bigger probe is 64 times bigger than the heat capacity of the smaller probe! Why it is then the complete cooling time to 200 °C of the bigger probe (600 seconds – see Fig. 5b) only 9.2 times longer than the complete cooling time for the smaller probe (65 seconds – see Fig. 5a)?

This can be explained by comparing the maxima of relevant temperature gradients:

max. $\Delta T_{80} = 415 \text{ °C}$ and max. $\Delta T_{20} = 114 \text{ °C}$, as the mentioned figures show.

- ad b) Two seconds after immersion the temperature gradient between 1 mm below the surface and the centre of the probe for the small probe was already 29 °C, while for the big probe the same temperature gradient was only 8 °C, as shown in Fig. 5a, and Fig. 5b respectively.

It appears that the smaller probe cools from the beginning faster than the bigger one, but later the temperature gradients within the bigger probe are much bigger (i.e. the heat fluxes are bigger) than within the smaller probe. This is the reason why the cooling time of the bigger probe is only 9.2 times longer than of the smaller probe, although its heat capacity is 64 times bigger. This analysis shows how the Liscic/Petrofer probe, based on the *Temperature Gradient Method* can precisely describe the dynamic of heat extraction during the whole quenching process.

To calculate the heat transfer coefficient based on experiments with the Liscic/Petrofer probe, the 1-D inverse heat conduction method is used. Because of the length to diameter ratio of 4:1 of the probe, the heat transferred through both ends is negligible, and the probe can be considered a long radially symmetric body of a given radius R . The temperature distribution $T(r, t)$ inside the cylinder, for times > 0 , depending only on the radial coordinate r from the centre of the cylinder, is determined by the 1-D heat conduction equation:

$$\rho \cdot c \cdot \frac{\partial T}{\partial t} = \frac{1}{r} \cdot \frac{\partial}{\partial r} \left(r \cdot \lambda \frac{\partial T}{\partial r} \right) \quad (5)$$

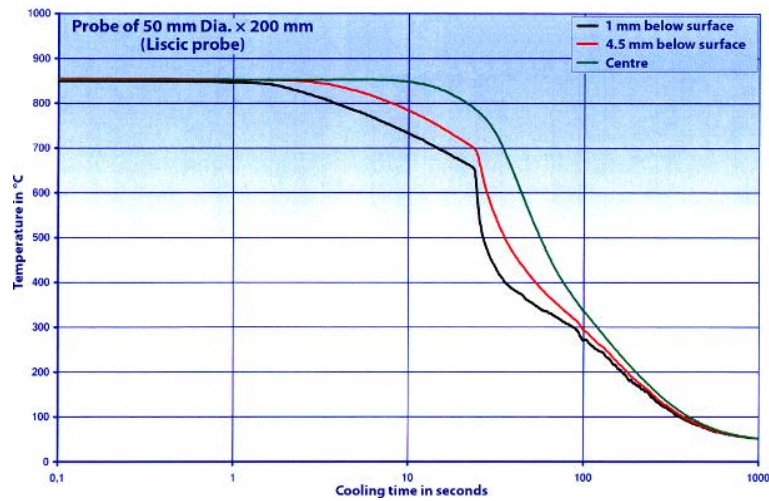
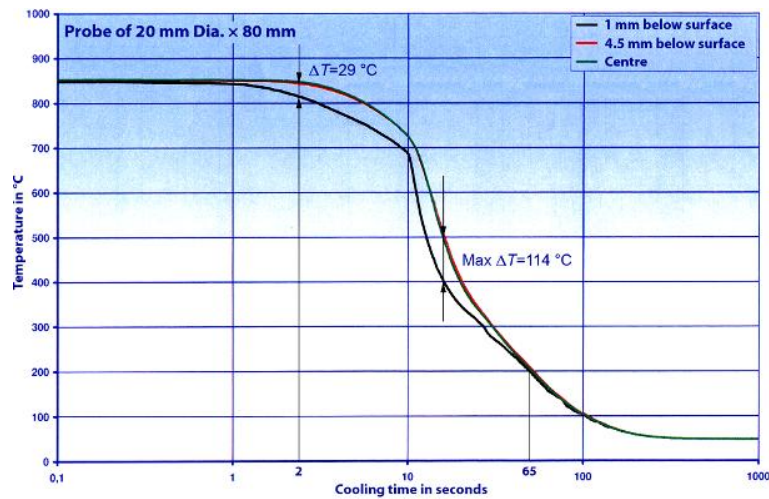
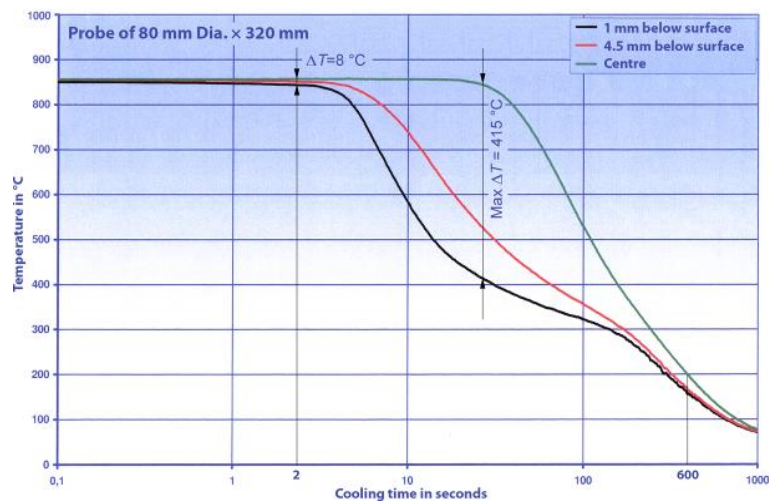


Fig. 4. Cooling curves measured by the Liscic/Petrofer probe (50 mm Dia. \times 200 mm) quenched in low viscous accelerated oil of 50 °C with medium agitation
(full colour version available online)



a) 20 mm Dia. \times 80 mm



b) 80 mm Dia. \times 320 mm, of the same design as the Liscic/Petrofer probe, quenched in low viscous accelerated oil of 50 °C with medium agitation

Fig. 5. Cooling curves measured by the probes of
(full colour version available online)

All physical properties: ρ (density), c (specific heat capacity) and λ (heat conductivity) of the probe's material are temperature dependent, so the whole problem is nonlinear. The initial condition $T(r,0) = T_0(r)$ is assumed uniform for $0 < r < R$ and equal to the initial value measured at the place of the thermocouple. The problem to be solved is to determine the surface heat transfer coefficient α for the boundary condition at $r = R$:

$$\lambda \frac{\partial T}{\partial r} = -\alpha(T - T_{\text{ex}}) \quad (6)$$

where T_{ex} is the measured external temperature of the quenchant. To determine α , the measured cooling curve at 1 mm below surface at $r = r_1 = R_1$ mm is used. The inverse problem of computing α is solved by the following numerical procedure:

1. Solve the heat conduction equation (5) within the spatial domain $0 < r < r_1$ with the measured $T = T_{\text{mes}}$ as a Dirichlet boundary condition at $r = r_1$.
2. Because $r_1 < R$, extend the solution towards

the boundary from $r = r_1$ to $r = R$ and

3. Calculate α from equation (6) with measured T_{ex} by using numerical differentiation.

- Since temperatures are measured at discrete times, they have to be smoothed. This is done by cubic spline least-squares approximation [7] to get sufficiently smooth global approximation over the whole time range.
- Numerical solution of the heat conduction equation (5) is done by the nonlinear implicit method, with simple iteration per time step, to adjust all physical properties to new temperatures.
- The solution extension in step 2 is computed by local extrapolation based on low degree polynomial least-squares approximation. The same approximation is also used for the numerical differentiation needed to compute α in step 3.

Based on this calculation the heat transfer coefficient is determined as function of surface temperature – see Fig. 6a, and as function of time – see Fig. 6b.

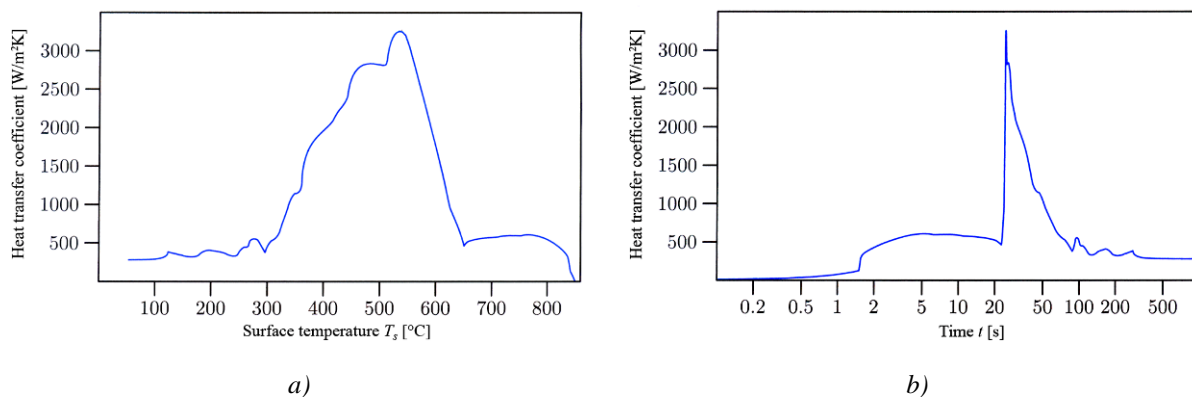


Fig. 6. a) Heat transfer coefficient for the Liscic/Petrofer probe (50 mm Dia. × 200 mm) quenched in low viscous accelerated quenching oil of 50 °C with medium agitation, – as function of surface temperature; b) Heat transfer coefficient for the Liscic/Petrofer probe quenched in low viscous accelerated quenching oil of 50 °C with medium agitation, – as function of time

4. Critical heat flux densities – their influence on distortion of the workpieces at quenching

In every quenching process there is an *initial heat flux density* which depends on the workpiece to be quenched, from the one side, and the *critical heat flux densities* q_{cr1} and q_{cr2} which depend on the quenchant, from the other side. The initial heat flux density depends on the ratio between the volume (heat capacity) and the surface of the body. At the very beginning after immersion, according to equation (4), the heat flux density depends on the temperature gradient on the surface. Bodies having a relatively small volume and a big surface, will have a bigger temperature gradient i.e. a bigger initial heat flux density than bodies having a relatively big volume and small surface, as shown in Fig. 7.

Cooling curves measured by the Liscic/Petrofer probe – see Fig. 5a and 5b, prove this fact.

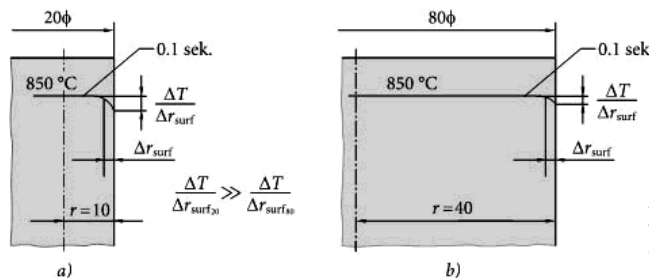


Fig. 7. Schematic presentation of the temperature gradient at the surface, in the very beginning of cooling a) for a cylinder of 20 mm Dia. × 80 mm and b) for a cylinder of 80 mm Dia. × 320 mm

Critical heat flux densities q_{cr1} and q_{cr2} are inherent properties of any vaporizable liquid. The first critical heat flux density q_{cr1} is the maximum heat flux density that causes film boiling (vapour blanket) at the very beginning of the quenching process, as shown in Fig. 8.

The second critical heat flux density q_{cr2} is the minimum amount of heat energy necessary to support film boiling, this is the point at which the surface of a hot part has cooled enough to allow the collapse of the vapour (end of film boiling), and nucleate boiling begins. There is a relation

between q_{cr1} and q_{cr2} that is true for all vaporizable liquids:

$$q_{cr1} = 5 \cdot q_{cr2} \quad (7)$$

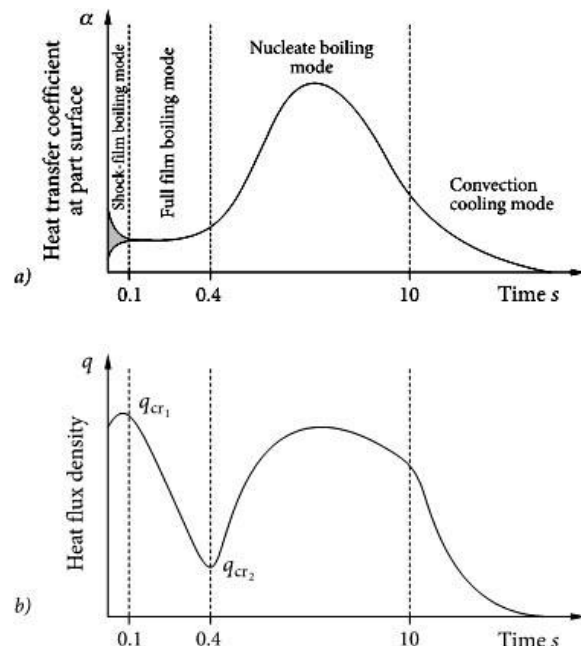


Fig. 8. Four modes of cooling at quenching a) critical heat flux densities; b) according to [8]

According to [9] upon immersion of a steel part into the quenchant, the initial heat flux density can be:

$$q \gg q_{cr1} ; q \approx q_{cr1} ; q \ll q_{cr1} \quad (8)$$

When $q \gg q_{cr1}$ full film boiling (vapour blanket) will appear. When $q \approx q_{cr1}$ transition boiling is observed. In case $q \ll q_{cr1}$ film boiling stage is absent i.e. nucleate boiling starts from the beginning. Each of these three cases will produce different values of the heat transfer coefficient. The first critical heat flux density q_{cr1} has a great effect on the cooling rate of steel parts and their distortion. It depends on the saturation temperature of the liquid, and the difference between the saturation temperature and the actual temperature of the quenchant. The more resistant a liquid is to boiling, when heat is applied, the

higher is the liquid's q_{cr1} . The more resistant a quenchant is to boiling, the more uniformly the part will be quenched (without film boiling) thus yielding less distortion.

When water is applied as quenchant the q_{cr1} value depends on the water flow rate and the water temperature [10]. It can be increased by increasing the agitation rate. Besides, a small amount (e.g. 0.1 %) of chemical additive can increase q_{cr1} by 2-3 times.

To provide for uniform cooling i.e. to eliminate distortion variation, the critical heat flux density q_{cr1} should be greater than the initial heat flux density. To achieve this, the practical know-how includes the knowledge of the additive, its concentration, and adequate water velocity.

Both critical heat flux densities q_{cr1} and q_{cr2} can be determined experimentally using a small silver probe as e.g. the Japanese New Silver Probe shown in Fig. 2. Finding the highest q_{cr1} for a given quenchant will optimize the quench system for all parts quenched in that system, minimizing distortion and maximizing the part properties after the quench.

This clearly shows the need to systematically investigate critical heat flux densities for different liquid quenchants in the framework of the proposed database.

5. Facilities which enable measurement and recording the cooling intensity in workshop conditions

When Liscic/Petrofer probe of 50 mm Dia. × 200 mm, having a mass of 3.3 kg is used for measurement and recording the cooling intensity of any liquid quenchant (oils, water-based solutions), in workshop conditions, adequate facility is necessary. Besides the required quantity of quenchant it should enable different quenchant's temperatures, and different agitation rates. Fig. 9a shows such facility at the Quenching Research Centre of the Faculty of Mechanical Engineering and Naval Architecture, University of Zagreb, Croatia. This facility has a

range of working temperatures from 20 to 80 °C, and a flow velocity (agitation rate) from 0 to 1.4 m.s⁻¹.

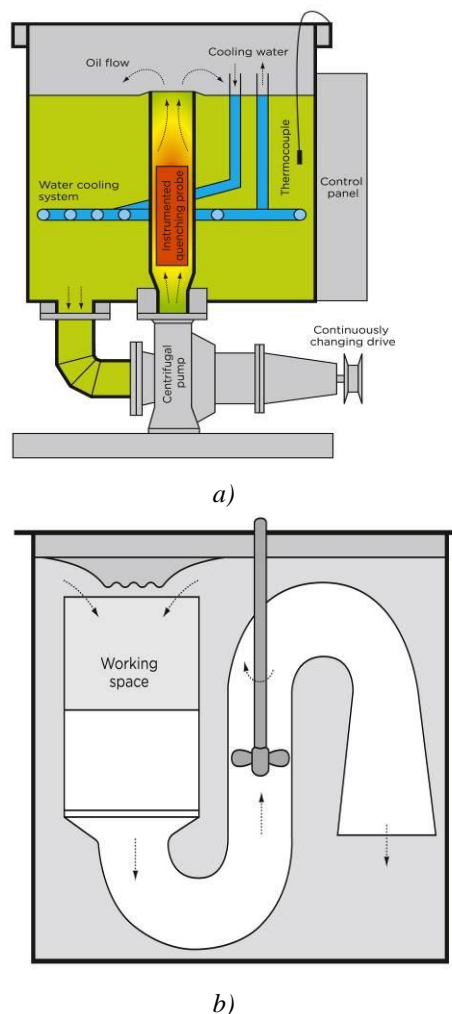


Fig. 9. a) Experimental quenching tank of 300 liter capacity for evaluation of the cooling intensity of oils, water, water-based solutions and polymer-solutions; b) Experimental salt-bath of 1 m³ capacity for evaluation of the cooling intensity of quenching salts for Martempering and Austempering [11] (full colour version available online)

For isothermal quenching in salt-bath the same centre has a proprietary salt-bath of 1 m³ salt capacity for Martempering and Austempering processes, see Fig. 9b. By the violent downward flow of liquid salt, a very effective cooling intensity is achieved which is enhanced by automatic addition of small quantities of water. This enables to martemper workpieces of up to 150 mm cross-section, and austemper workpieces of up to 30 mm thickness. The working range of this facility is: temperature 180-450 °C; agitation rate 0-0.6 m/s; water addition 0-2 vol. %.

6. Conclusions

Development of new computer aided experimental techniques enable to characterize every liquid quenchant in concrete quenching conditions, in respect of their cooling intensity, more comprehensive and accurate than ever before. The possible consequences of this achievement are twofold:

- a) Computer modeling of hardness distribution, microstructure, stress and distortion

The results of investigations during Phase 1 of the mentioned project, will serve in Phase 2 as input into new advanced 3-D software code for calculation of the heat transfer coefficients in every point of the surface, at real engineering components of complex geometry. This will enable to predict the hardness distribution, microstructure, stresses and distortion in every point of any section of the workpiece.

- b) Virtual selection of optimal quenchant and quenching conditions

Once the database will contain the mentioned comprehensive information, for sufficient number of different quenchants under specified conditions, virtual computer aided selection of optimal quenchant and quenching conditions, according to specific requirements in every concrete case, will be possible. By the virtual comparison one will gain the following important information:

1. What are the critical heat flux densities q_{cr1} and q_{cr2} of the relevant quenchant. This information is vital to know whether or not film boiling will occur, on which deformation of the quenched workpiece depends.
2. From the laboratory test: what is the cooling rate, especially in the critical temperature region of possible pearlite and ferrite formation.

3. From the test in real workshop conditions: Because the calculated heat transfer coefficient represents the heat flux during the whole quenching process, it represents best the real quenching intensity, which is directly proportional to the expected depth of hardening.

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