

ADVANCED COOLING CURVE ANALYSIS METHODOLOGY

All of the cooling curve analysis methods described thus far are essentially laboratory quenchant analysis procedure. Although they are invaluable for this use, they are of relatively limited direct value to the heat treater who needs to know if he can through-harden a part being produced in production and if he will encounter distortion or even cracking. Therefore, there are various efforts underway at the present time to advance cooling curve analysis methodology to permit modeling and simulation of the production quenching process. In this section, one cooling curve analysis methodology that has been successfully used in the heat treating shop will be described. In addition to illustrating the overall utility of modeling and simulation of production quenching conditions, this example also illustrated advanced techniques for investigating quenching behavior.

1. Temperature Gradient Quenchant Analysis

a. Background - Liscic designed a system for practical measurement, recording and evaluation of quenching (cooling) intensity under workshop conditions which expresses quenching intensity by a continuous change in relevant thermodynamic functions during the entire quenching process. This approach should be contrasted with the Grossmann H-factor concept which expresses quenching intensity with a single value and which was shown earlier to be of limited value in quantitatively represented quench severity when agitation is used.

All of the different cooling curve analysis methods discussed thus far utilize relatively small, usually 12.5 mm or 0.5 in diameter, round bar probes with a single thermocouple placed at the geometric center. Such probes, while excellent for quality control purposes, are of limited value for use under workshop conditions. The reasons include:

- Because of its relatively small mass and low heat capacity, these small probes will cool in about 10 – 30 seconds whereas in actual workpiece of 50 mm (2.0 inches) diameter will require 500-600 seconds to cool below 200°C (392°F) in the center of the workpiece when quenched into an unagitated quench oil. Therefore, to adequately model actual quench processes under production conditions, a probe of similar mass and dimensions is necessary.
- The actual heat transfer coefficient during quenching of actual production parts may be simulated using a small cylindrical probe. However, the heat transfer coefficient during nucleate boiling is heavily dependent on bar diameter [94]. This magnitude of this dependence increases with decreasing bar diameter below 50 mm (2.0 in). The dependence is less pronounced for bar diameters greater than 50 mm (2.0 in). Therefore, for the same quenching conditions, the heat transfer coefficient on the surface

of a small diameter cylinder is quite different than that expected on the surface of most production parts with diameters ≥ 50 mm (2.0 in).

Important criteria for a cooling curve analysis system to be utilized to model quench processes under workshop conditions should be applicable to: 1.) a wide variety of quenchant media including: water, brine, aqueous polymer solutions, salt baths, quench oils, fluidized beds and gas quenchants; 2.) a wide variety of quenching conditions including: different bath temperatures, agitation rates, and fluid pressures; and 3.) all quenching techniques including: direct immersion quenching, interrupted quenching, martempering, austempering and spray quenching.

The method to be reported here provides for recording of thermodynamic functions during each test to enable the user to analyze the particular quenching process of interest and quenching conditions, to evaluate quenching intensity and to compare it with previously performed tests in other facilities under different conditions. To do this, the user will establish a database of quenching intensities of different systems within the production facility. This database will provide the user with input data for subsequent computer simulation of the quenching process to determine optimal quenchant and quenching conditions for every part being produced.

In addition, for optimal simulation, it is important that the measurement method be sufficiently sensitive to reflect changes in each of the important quenching parameters (specific character of the quenchant, quenchant bath temperature, in addition to mode and degree of agitation). This criterion is addressed by recording the temperature-time history (cooling curve) at particular points within the probe used for analysis. This requires the measurement of transient temperatures within a solid body when high thermal gradients are involved which requires that the following inherent effects be considered:

- **Damping Effect** – Changes in surface temperature are damped in magnitude when sensed inside the body compared to their magnitude at the surface.
- **Lagging Effect** – Changes in the surface temperature are sensed within a finite time after they occur at the surface. The greater the distance of the temperature measurement point from the surface, the greater the damping and lagging effects.
- **Response Time** – When working with thermocouples, it is important to consider another effect that is inherent with every thermocouple – response time which is the time necessary to reach 63.2% of its total signal output when the thermal junction is subjected to a step change in temperature [95]. The time constant of a sheathed, grounded thermocouple of 1.5 mm (0.062 in) outer diameter is 1.5 seconds. To reach 99 % of its full signal output when subjected to a step change in temperature, the time constant must be multiplied by 5, which makes a 7.5 second delay. Thermocouples with an even greater diameter will exhibit an even greater time constant and delay in response time.

b. Theoretical Principles – Because of all of the above described requirements, effects and limitations, instead of recording only one cooling curve at the center of a small cylindrical test probe (as in laboratory tests) the heat flux density at the surface of the

quench probe has been selected as the main feature in measuring, recording and evaluating quenching intensity. This is because changes of the heat flux density during the quenching process best represents the dynamics of heat extraction.

The method itself, known in the literature as the Temperature Gradient Method, is based on the known physical rule that 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:

$$q = \lambda \cdot \frac{\partial T}{\partial x}$$

Where: q is the heat flux density (W/m^2), i.e. the quantity of heat transferred through a surface unit per unit time, λ is thermal conductivity of the body material (W/mK), and $\partial T/\partial x$ is the temperature gradient inside the body at the body surface, perpendicular to it (K/m).

c. Hardware – The essential feature of the method being described here is the LISCIC/NANMAC quench probe¹. It is constructed from AISI Type 304 stainless steel which is 50 mm (2.0 in) diameter x 200 mm (4.0 in) length, instrumented with three thermocouples placed at the half-length cross-section as shown in Fig. 57. One thermocouple measures the actual surface temperature of the probe (T_n), another measures the temperature at a point 1.5 mm (0.06 in.) below the surface (T_i), and the third one measures the temperature at the center of the cross-section. The thermocouples inside the body are standard sheath-type thermocouples. The thermocouple at the surface is of special design (U.S. Patent 2,829185) which allows continuous measurement of the true surface temperature of a solid body without any damping or lagging effects, in real time, because of its extremely fast response time of about 10 microseconds (10^{-5} s).

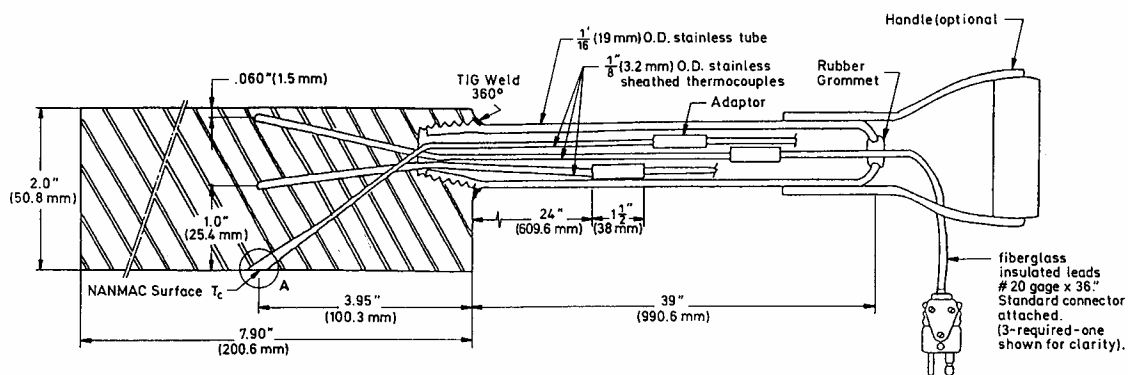


Figure 57 – The LISCIC/NANMAC quench probe.

¹ The Liscic-Nanmac probe is manufactured by the NANMAC Corporation, Framingham, MA, USA

There are two important requirements for a thermocouple used for measuring surface temperature:

1. Its thermal junction should be two-dimensional (instead of the usually encountered three dimensional geometry),
2. It should be flush with the surface.

The unique details of the sensing tip of this thermocouple, shown enlarged in Fig. 57, are as follows. In the vicinity of the hot measuring junction, the round thermocouple wires are flattened into ribbons of about $38\ \mu\text{m}$ (0.0015 in) thickness. These ribbon elements are electrically insulated from each other and from the thermowell by sheets of mica insulation of about $5\ \mu\text{m}$ (0.0002 in) thickness. This “sandwich” of ribbon elements and mica insulations, having a total thickness of about $91\ \mu\text{m}$ (0.0036 in), is placed between a split-tapered insert and pressed into the thermowell (body). The thermal junction is formed by grinding and polishing across the sensing tip. The mica insulation between the two dissimilar ribbons is so thin that metallic whiskers of one ribbon element bridges across the mica to the other ribbon element, and makes hundreds of microscopic friction welded junctions which are parallel to one another thus forming one composite measuring junction.

The microscopic burrs of the metal from the thermowell (housing) bridge the thin layers of mica thus electrically grounding the thermal junction to the thermowell at the sensing tip. The metal of the thermowell becomes the third intermediate element, and since the temperature is the same on both sides of the thermal junction, the EMF produced by the secondary junctions on both sides of the main thermal junction cancel each other out, leaving the EMF of the main thermal junction as the only observed EMF (Law of Intermediate Metals in Thermocouples).

Any subsequent erosion of the surface of the thermowell (body) simply forms new junctions while removing the old junctions, hence its name “Self Renewing Thermocouple”. Because of its unique characteristics, this is the best type of thermal junction to be used for heat transfer calculations because it registers all of the phenomenon occurring at the surface.

The temperatures recorded at the surface (T_n) and at 1.5 mm below the surface of the quench probe (T_i) permit the calculation of the temperature gradient within this surface layer at each moment throughout the quenching process. The role of the center thermocouple (T_C) is to indicate the timing for heat extraction from the core of the probe and to provide a continuous measurement of the temperature difference between the surface and the core, which is essential for the calculation of thermal stresses.

Specific features of the LISCIC/NANMAC quench probe are:

1. The size of the probe and its mass ensure sufficient heat capacity and radially symmetric heat flow in the cross-sectional plane where the thermocouples are located.

2. The probe is constructed from austenitic stainless steel and does not undergo microstructural change upon heating or quenching, nor does it evolve or absorb heat because of microstructural transformations.
3. The surface condition of the probe can be maintained for each test by polishing the sensing tip of the surface thermocouple before each measurement.
4. Extremely fast response times of the surface thermocouple (10^{-5} s), and the absence of any damping or lagging effects allow any transient temperature at the surface to be measured and recorded exactly and in real time.
5. The heat transfer coefficient at the probe's surface, because of its sufficiently large diameter, may be used in computer simulation of actual production parts.

When the quenching intensity is to be determined, the probe is heated to 850°C (1562°F) in a suitable furnace, 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 enabling digital recording of all three thermocouple signal outputs.

d. Software - In addition to cooling curve data, the cooling curve analysis program (TGQAS - Temperature Gradient Quenchant Analysis System) described here permits advanced computational modeling of production quenching systems. The TGQAS consists of three modules:

D1. Module I: TEMP-GRAD (Temperature Gradient Method) - In each test, three cooling curves are obtained: T_n - for the surface of the probe, T_I for the point 1.5 mm (0.06 in) below the surface and T_C for the center of the probe. Typical cooling temperature-time profiles for each of these points is illustrated in Fig. 58. The temperature gradient between T_I and T_n is calculated from these cooling curves by multiplying the corresponding data by the temperature-dependent thermal conductivity and the heat flux density versus time, $q = f(t)$ (see Fig. 58b) and the heat flux density versus surface temperature, $q = f(T_n)$ (see Fig. 58c) are calculated.

Calculation of the differences between each thermocouple location versus time, $\Delta T - f(t)$, provides the functions illustrated in Fig. 59d. Calculation of the integral under the heat flux density curve (which represent the amount of heat extracted) from the beginning of immersion until a predetermined time, provides the functions shown in Fig. 59e. For heat extracted from the probe, the curve designated by "□" (i.e. for the surface layer of 1.5 mm thickness) is valid. In each point where thermocouples are located, the cooling rate curves versus surface temperature: $\partial T / \partial t = f(T_n)$ are calculated as shown in Fig. 59f.

Calculated functions, graphically represented in Fig. 58 and Fig. 59 permit comparison of the actual quenching intensity among different quenchant, quenching conditions and techniques. Based on these thermodynamic functions, each quenching test may be evaluated with respect to: depth of hardening, (when comparing two quenching processes), thermal stresses and possible superposition of structural transformation stresses that will occur during a particular quenching process and delayed quenching, i.e.

whether continuous or discontinuous cooling rates are occurring (with consequences on hardness distribution on the cross-section after hardening). These thermodynamic functions also provide the basis for automatic control of quenching intensity during the quenching process.

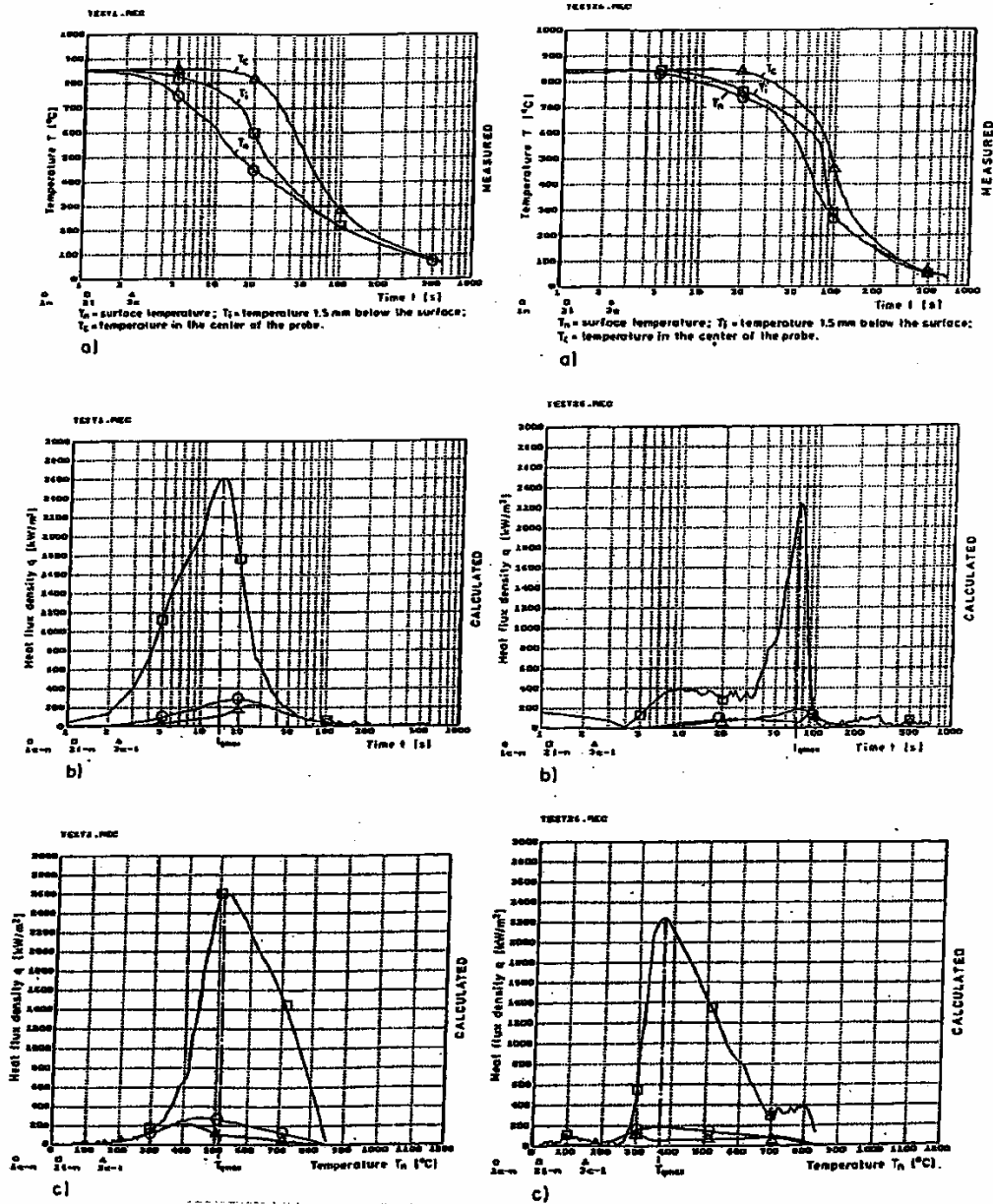


Figure 58 - Graphical Display from Module 1 (TEMP-GRAD) when quenching the LISCIC/NANMAC probe into a 20°C mineral oil bath without agitation (left row, vertically) and a 25% aqueous polyalkylene glycol quenchant solution at 40°C bath temperature and 0.8 m/s agitation rate (right row, vertically). a. Measured and recorded temperature versus time, $T = f(t)$; b. Calculated heat flux density versus time $q = f(t)$, and c. Calculated heat flux density versus surface temperature, $q = f(T_n)$.

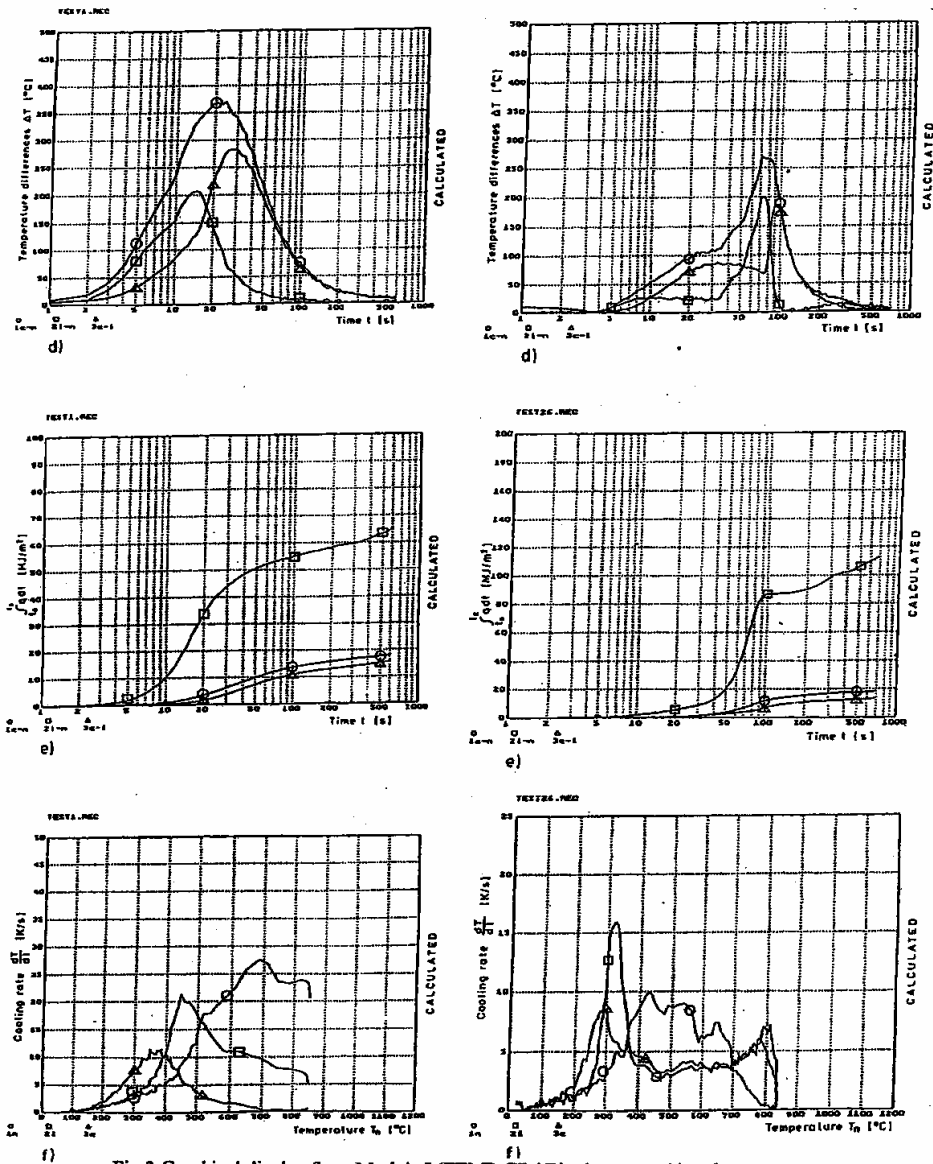


Figure 59 - Graphical display from Module 1 (Temp-Grad) when quenching the LISCIC/NANMAC probe into a 20°C mineral oil bath without agitation (left row, vertically) and a 25% aqueous polyalkylene glycol quenchant solution at 40°C bath temperature and 0.8 m/s agitation rate (right row, vertically). d. Calculated temperature difference versus time, $\Delta T = f(t)$; e. Calculated integral of the heat flux (heat extracted) versus time, $\int qdt$; and f. Calculated cooling rates versus temperature, $\frac{\partial T}{\partial t} = f(T_n)$.

D2. Module II: HEAT-TRANSF (Calculation of Heat Transfer Coefficient and Cooling Curves) - The function of this module is the calculation of the temperature distribution in the cross-section of round bars. It is based on the numerical method of control volumes where the heat conduction in the radial direction is solved as a one-dimensional problem. The software consists of two main subroutines. The first subroutine

utilizes the measured surface temperature as an input parameter to calculate the temperature distribution over the probe's cross-section versus time and the heat transfer coefficient between the surface and the quenchant versus time and versus the surface temperature as illustrated in Fig. 60a and Fig. 60b.

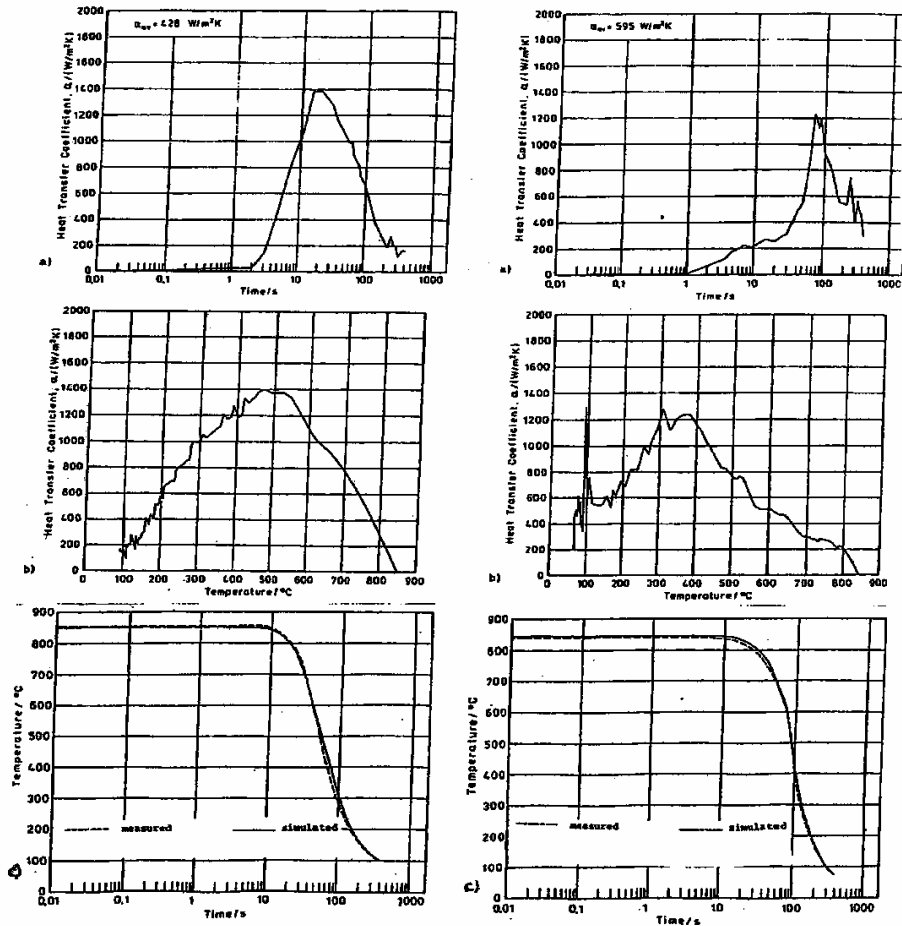


Figure 60 - Graphical display from Module 2 (HEAT-TRANSF), when quenching the LISCIC/NANMAC probe into a 20°C mineral oil bath without agitation (left row, vertically) and a 25% aqueous polyalkylene glycol quenchant solution at 40°C bath temperature and 0.8 m/s agitation rate (right row, vertically). a. Heat transfer coefficient versus time, $\alpha = f(t)$; b. Heat transfer coefficient versus surface temperature, $\alpha = f(T_n)$; and c. Comparison of measured and simulated cooling curves for the center of the quench probe (50 mm diameter).

The second subroutine utilizes the calculated heat transfer coefficient for a particular quenching test as input parameters which permits the simulation of quenching cylindrical workpieces of varying diameters under the conditions of each quenching test that are stored in the users database of quenching intensities. Physical properties of the workpiece can be selected for the desired steel grade. The cooling curve at any point

within the cross-section can be calculated as illustrated in Fig. 60c along with corresponding heat transfer coefficient versus time and versus the surface temperature.

D3. Module III: CCT-DIAGR (Prediction of Microstructure and Hardness after Quenching) - This module is used to predict the microstructure and hardness after quenching of cylindrical workpieces of different diameters. It contains an open data file of CCT (Continuous Cooling Transformation) diagrams in which the user may store up to 60 CCT diagrams of his own choice. This program enables the user to superimpose every calculated cooling curve on the CCT diagram of the desired steel. From the superimposed cooling curves (shown on the CRT monitor during analysis) the user can select the percentage of microstructural phases transformed and the hardness value at the selected point within the cross-section after hardening as illustrated in Fig. 61. For a cross-section of the selected diameter, cooling curves are calculated at three or five characteristic points (surface, 3/4R, 1/2R, 1/4R and center), using the HEAT-TRANSF module. The CCT-DIAGR module enables the user to determine hardness values at these points which will permit prediction of the hardness distribution curve.

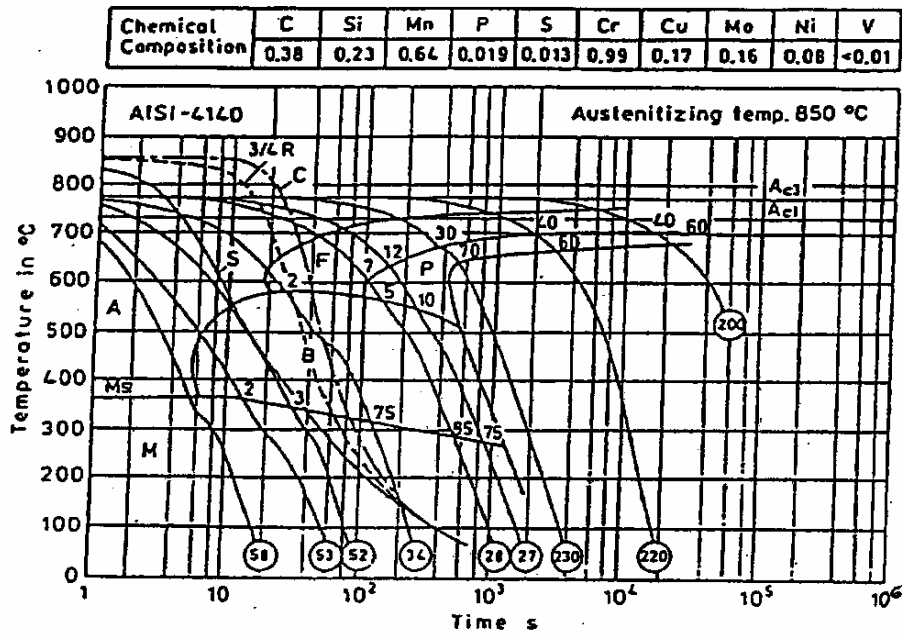


Figure 61 - CCT diagram of AISI 4140 steel with superimposed calculated cooling curves for surface (S), three-quarter radius (3/4 R), and center (C) of a round bar of 50 mm diameter.

Note: In the case of delayed quenching, where a discontinuous change of cooling rates occur, the prediction of microstructural transformations and hardness values after hardening from an ordinary CCT-diagram is not correct, because the incubation time consumed (at any point on the cross-section) before the cooling rate was abruptly changed, has not been taken into account. For a detailed explanation see Reference [96].

e. Hardware Requirements - For the system being described here, the hardware requirements are relatively modest and include: IBM compatible PC (286/386/486/Pentium), 640 Kbytes RAM, a color graphic system, 2 MB free hard disk space plus additional space for files to be stored, printer port, and disk drive.

2. Example of a Quenching Process Analysis

Two different quenching processes are illustrated in Fig. 58 and Fig. 59; Case A and Case B. Case A is a quenching process in a mineral oil at 20°C (68°F) without agitation. (All associated diagrams are on the left side of the figure.) Case B is a quenching process in a 25% aqueous polyalkylene glycol (PAG) solution at 40°C (104°F) and 0.8 m/s agitation rate. (All of the diagrams for this process are on the right side of the figure.)

By comparing the diagrams of the heat flux density versus time in Fig. 58b, it is shown that Case B exhibits delayed quenching because in Case A, the time required for maximum heat flux to occur (t_{qmax}) is only 15 s, whereas in Case B it is 72 s (due to the thick PAG polymer film). In Case A (oil quenching), Fig. 59e shows that by 20 s after immersion, 34 MJ/m² has already been extracted and by 50 s, 50 MJ/m² heat has been extracted. In Case B, (quenching in a high concentration of a PAG quenchant solution), by 20 s after immersion, only 5 MJ/m² and by 50 s, only 20 MJ/m² has been extracted. However, immediately after that period, between 50 and 100 s, the heat extracted in Case A has increased only from 50 to 55 MJ/m², whereas in Case B, the heat extracted has increased from 20 to 86 MJ/m². This shows that in Case B, the thick polymer film has prevented the heat extraction for a relatively long time at which point the insulating polymer film surrounding the cooling probe burst and a sudden increase of heat extraction occurred.

By comparing the time required to decrease the heat flux density from its maximum to a low value, e.g. 100 KW/m² as shown in Fig. 58b, it is observed that in Case A, 45 s is necessary, whereas in Case B, only 28 s is necessary. This illustrates that Case B is a quenching process in which after the sudden burst of the thick polymer film surrounding the probe, there is practically no boiling, as observed in oil quenching, but an abrupt change in the convection cooling stage.

A discontinuous cooling change is inherent to such a quenching regime. It is interesting to analyze cooling rate versus surface temperature as shown in Fig. 59f. While in Case A (oil quenching), the cooling rate at the surface of the probe "o" exhibits a greater maximum cooling rate than the cooling rate at 1.5 mm below the surface (□). In Case B, the maximum cooling rate at 1.5 mm below the surface (between the period of 350°C and 300°C of surface temperature) is higher than the maximum cooling rate at the surface itself! This is also evident from the cooling curves illustrated in Fig. 58a for Case B where at 570°C, a discontinuous change of cooling curve slope of T_I occurs, and between 500°C and 300°C, this slope is greater than the slope of the cooling curve for the surface (T_n). This is an experimental proof of the theoretical calculation described in Reference [97], that in delayed quenching, cooling rates below the workpiece surface can be higher than at the surface itself.

Another analysis, with respect to thermal stresses during quenching (on which residual stresses and possible distortion depend), is possible by comparing Fig. 59d. This comparison shows that quenching in an aqueous PAG quenchant solution of high concentration (Case B) compared to oil quenching (Case A), resulted in 27% lower maximum temperature difference between the center and the surface of the probe (o). Whereas in Case A, the maximum temperature difference between the center and 1.5 mm below the surface (Δ) is higher than the maximum temperature difference between the point 1.5 mm below the surface and the surface itself (\square). In Case B, the maximum temperature difference between the point 1.5 mm below the surface and the surface itself (\square) is slightly higher than the maximum temperature difference between the center and the point 1.5 mm below the surface (Δ), which is reached about 20 seconds later. This analysis also shows an abrupt heat extraction when the polymer film bursts.

However, Figures 59d show that in oil quenching, the maximum temperature difference between the center and the surface (o) occurs 20 s after immersion when the surface temperature is 450°C (See Fig. 58a for Case A), i.e. above the martensite start temperature (M_S). In an aqueous solution of a PAG polymer quenchant at high concentration, the maximum difference between the center and the surface (o) occurs much later, i.e. after 80 s when the surface temperature has already decreased to 350°C (see Fig. 58a for Case B). Because of this, when working with steels with high M_S temperatures, there is a greater possibility of overlapping thermal stresses with those created due to austenite - to martensite transformation.

The probability of crack formation can be observed by comparing the surface temperature of the probe at the moment when maximum heat flux density occurs ($T_{q_{max}}$). As seen in Fig. 58c for oil quenching, $T_{q_{max}}$ is 515°C, while for the aqueous PAG quenchant solution at high concentration (Case B) this occurs at 380°C. The lower the value of $T_{q_{max}}$, the greater the risk of crack formation, especially with steels with high M_S temperature.

When comparing two different processes with continuous cooling (for the same workpiece and the same steel grade), to determine which will provide the greater depth of hardening, the following analysis may be used (see Fig. 58b). The larger the value of q_{max} , and the shorter the time ($t_{q_{max}}$), the greater will be the depth of hardening. In the case of a quenching process with discontinuous (delayed quenching) such reasoning is not valid.

The sensitivity of the method to variation of quenching parameters is demonstrated in Fig. 62 which shows the calculated heat flux densities versus time for the LISCIC/NANMAC quench probe, all experiments performed in the same aqueous PAG polymer quenchant solution of 40°C bath temperature and 0.8 m/s agitation rate. The only parameter that was changed was the polymer concentration from: a.) 5% to b.) 15% to c.) 25%. This figure illustrates how increasing polymer quenchant concentration decreases the heat flux density, i.e. quenching intensity. By shifting the heat flux density maximum to a longer time (because of increasing thickness of the insulating polymer

film surrounding the probe with increasing quenchant concentration) changes a normal quenching process to a delayed one.

This example illustrates the possibilities that are available for simulation of production quenching processes using advanced data acquisition and analysis techniques.

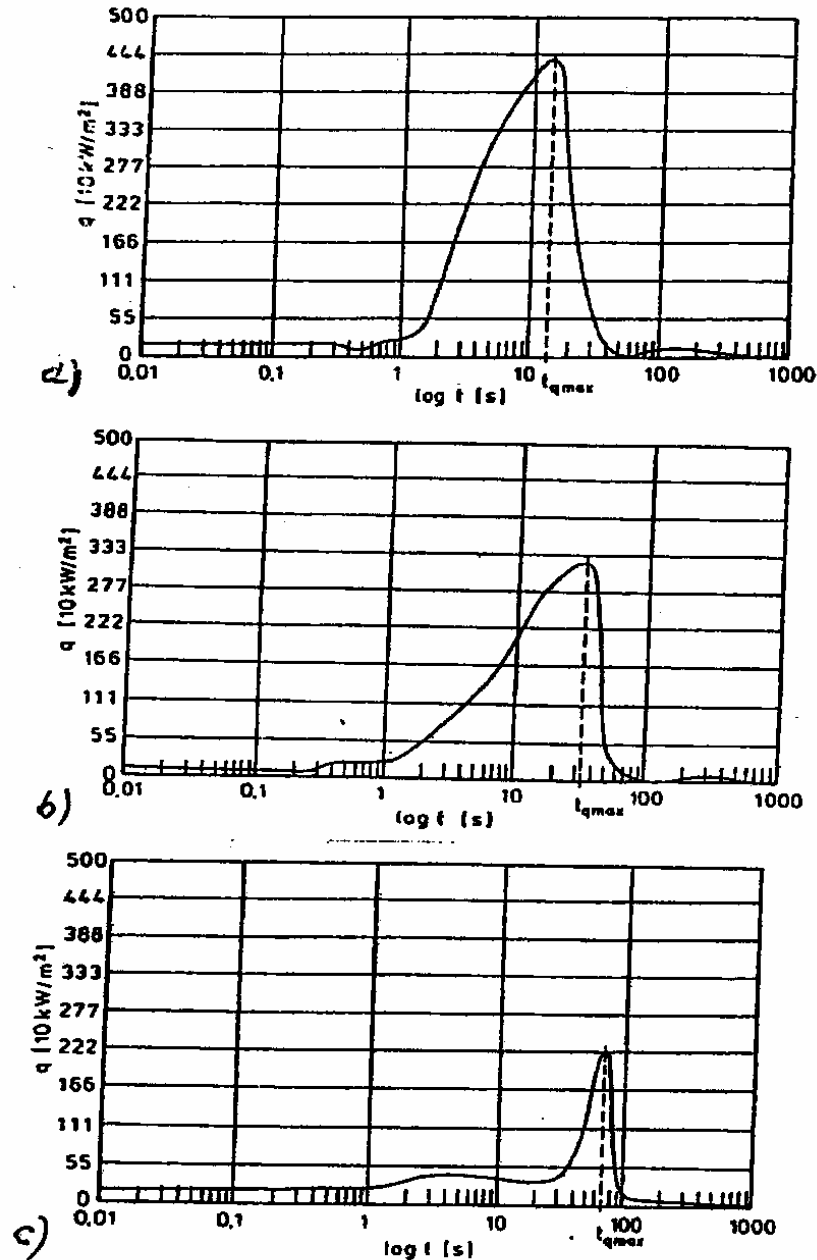


Figure 62 - Calculated heat flux densities versus time, when quenching the LISCIC/NANMAC probe into an aqueous polyalkylene polymer solution at 40°C and 0.8 m/s agitation rate, but having concentrations: a. 5%, b. 15% and c. 25%.