EXPERIMENTAL STUDY OF A QUENCH PROCESS

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Chapter 1 Introduction

1.1 General

Quenching is a thermal treatment process for metal alloys that must be controlled to ensure the formation of desirable transformation products. Uniform and predictable heat transfer is critical during a quenching process to minimize the potential for distortion, cracking, and nonuniform property distribution.

In order to optimize a quenching process, it is necessary to understand the thermal behavior within the quenched part as a function of time. An important step in this process is quantifying the boundary conditions at the surface of a quenched part. Unfortunately, directly obtaining accurate measurements of the surface temperatures or the heat transfer coefficient values is extremely difficult. Thus, there exists a need to develop other methods to solve this problem.

Of the various procedures that have been studied to predict the ability of a quenching process to produce desirable properties, the cooling curve test is typically acknowledged as the most effective. Cooling curves are obtained experimentally using an apparatus that primarily consists of an instrumented quench probe to monitor interior temperature response and a data acquisition system to collect and display the sampled data.
1.2 Quench Probe Development and Theory

Quench probes used for cooling curve acquisition have existed for more than 60 years (Totten et al., 1994). Some of the earliest probe development was conducted at the Max Plank Institute in the 1930's. Since that time, probes have been constructed in various shapes, including cylinders, spheres, square bars, plates, rings, and round disks. Probes have also been made in a variety of materials, such as steel, silver, nickel, copper, gold, and aluminum.

Totten et al. (1994) has discussed some of the earliest research in probe design and the investigators who made contributions to this area of study, including Liscic, Paschkis, and Luty. One of the first probes used is illustrated in Figure 1.1. The construction of this probe consisted of a 0.5 inch diameter steel tube, and two 100 x 150 mm (4 x 6 inch) sections made of SAE 5145 steel. A chromel-alumel thermocouple was hydrogen brazed to the middle of the bottom section of the probe. The other end of the thermocouple passed out the top of a 13 mm (0.5 inch) diameter hole in the top section.

![Figure 1.1 SAE 5145 steel plate probe used in early quenching research [Source: Totten et al. (1994)]](image-url)
A number of investigators (Totten et al., 1994) have used an instrumented silver ball in a testing apparatus similar to the one shown in Figure 1.2. This particular design has been relatively popular because heat transfer calculations from cooling curve data can be made fairly easily due to the symmetrical shape of the spherical probe. However, problems regarding positioning the thermocouple assembly directly at the geometric center and preserving a consistent surface finish without changing the diameter of the ball have limited their use.

**Figure 1.2** Schematic of spherical silver probe test apparatus [Source: Totten et al. (1994)]
In order to avoid the problems related to the fabrication of spherical silver probes, some investigators have used cylindrical silver probes. The particular probe assembly shown in Figure 1.3 was considered for an International Organization of Standardization (IOS) specification. After extensively testing probes made of silver, many researchers have concluded the high thermal conductivity of silver does not allow for accurate assessment of quenching processes. It has been shown that cooling curves could not be reproduced adequately using a silver probe for acquisition if certain steps were not taken to prepare the probe prior to quenching. It was also shown that it is not always possible to determine the degree of hardening over a cross section of a steel cylinder from a cooling curve acquired from a silver ball probe.

Limitations of silver probes led to the development of an austenitic stainless steel probe with a sensitive temperature measurement system (Totten et al., 1994). This probe is illustrated in Figure 1.4. The response time of the probe's thermocouple was reported to be $10^{-5}$ seconds. The benefit of using stainless steel is that it does not experience

![Figure 1.3 Cylindrical silver probe considered for ISO specification [Source: Totten et al. (1994)]]
Figure 1.4  Austenitic stainless steel probe with sensitive temperature measurement system [Source: Totten et al. (1994)]

volume changes during phase transformations that occur in plain carbon and alloy steels. Another advantage of using a stainless steel probe is the surface finish can easily be maintained.

Because the objective of a cooling curve analysis is to measure the ability of the quenching medium to extract heat, it is important that the measurement process itself does not affect the experiment. Consequently, probes must be designed with proper dimensions to ensure an accurate measurement process. Cylindrical probes are designed so the length of the cylinder is at least four times their diameter. Shorter probes may
experience significant end-cooling effects. This means the heat removed from the end of the probe will reduce the rate of temperature loss at the geometric center. Similarly, plate probes are designed with a length and width at least four times their thickness to minimize edge-cooling effects.

Another design consideration is probe size. Investigators have determined that sensitivity to quenchant characteristics increases with decreasing probe size. In addition, if a probe is too small, it simply cannot be used for accurate one-dimensional analysis. Other limitations of a small probe are that it is difficult to modify its geometry and to add features.

The majority of probes that have been previously used did not adequately model actual components used in industry. Analysis of quenching in sharp corners is necessary for several features found in production parts, such as gear teeth. Many of the probe designs mentioned do not incorporate corners. Thus, there has been a need for a new design to solve this problem.

1.3 Objective and Relevance

The main objective of the present work was to design and construct a quench system to obtain cooling rates for a series of quenching experiments. More specifically, the goals were: (1) to develop an instrumented quench probe that could incorporate features found in components used in industry, (2) to build an apparatus to quench the probe at various angles, and (3) to set up a data acquisition system to acquire and display sampled data.
The motivation for the work is to develop a practical and reliable method for determining the values of the convection heat transfer coefficient present during quenching and to use the values in future studies to properly design a quench process for optimum heat treating of materials. Evaluation of the coefficient was extremely difficult because not only does it vary within the process but it is also influenced by several process parameters. These parameters include part geometry, the angle and velocity that the part enters the quenchant, and the composition and agitation of the quench medium. Consequently, numerous experiments have been performed to determine the values of the heat transfer coefficient for specific parameters. However, there does not exist a database that includes coefficient values for every possible type of heat treatment.

The convection heat transfer coefficient values can be determined by means of fundamental analysis and experimentation. If the heat flux or the heat transfer coefficient at the probe's exterior surface are known as a function of time, then the temperature response at any interior location within the probe can be determined. In contrast, it is also possible to calculate the surface heat flux or heat transfer coefficient knowing the transient temperature measurements at an interior location within the probe. The method used to estimate the coefficient values given the temperature history at an interior location within a part is known as the inverse heat transfer method. An efficient inverse heat transfer method code is being developed at Ohio University which uses time-temperature data from cooling curve tests as input to calculate the heat transfer coefficient values.
The different stages of quenching are directly related to the conduction heat transfer within the part and the heat transfer from the part to the quenchant. Therefore, it is necessary to consider both the conduction heat transfer equation and the convection heat transfer equation in the analysis of the quenching process. The conduction heat transfer equation is given as:

$$\nabla^2 T = (1/\alpha)(\partial T/\partial t)$$

(1.1)

where $\alpha$ is the thermal diffusivity of the material, and $T$ is the temperature (Rohsenow, 1961). The convection heat transfer equation is given as:

$$q = hA(T_s - T_\infty)$$

(1.2)

where $q$ is the heat transfer rate, $h$ is the convection heat transfer coefficient, $T_s$ is the surface temperature, and $T_\infty$ is the fluid temperature (Incropera, 1990).

Analysis of the two governing equations gives information on characteristic internal and external cooling times (Alam, 1998). The internal cooling time is a function of the geometry and the thermal properties within the part, and the external cooling time is related to the thermal properties at the part-quenchant interface. The characteristic cooling times are dependent on the Fourier number and the Biot number.

The Fourier number and the Biot number measures how a body responds to temperature changes. The Fourier number is given as:

$$Fo = \alpha t/L^2$$

(1.3)

where $\alpha$ is the thermal diffusivity, $t$ is the time, and $L$ is the characteristic length. The Fourier number is a dimensionless time parameter that characterizes transient conduction problems. The definition of the Fourier number shows that bodies with high diffusivity
respond faster than those with a low diffusivity, and large bodies respond slower than small bodies. The Biot number is given as:

\[ \text{Bi} = \frac{hL}{k_s} \]  

(1.4)

where \( h \) and \( L \) are the same as before, and \( k_s \) is the thermal conductivity of the solid. The Biot number is a dimensionless parameter that plays a fundamental role in conduction problems that involve surface conduction effects. The importance of the Biot number can be more easily understood if it is rewritten as:

\[ \text{Bi} = \frac{(L/k_s)/(1/h)} \]  

(1.5)

The Biot number may also be interpreted as a ratio of thermal resistances. The temperature response of bodies with a low Biot number are dominated by surface resistance while those with a large Biot number are dominated by internal resistance.

Also related to this discussion is the Nusselt number. The Nusselt number is defined as:

\[ \text{Nu} = \frac{hL}{k_f} \]  

(1.6)

where \( h \) is the convection heat transfer coefficient, \( L \) is the characteristic length, and \( k_f \) is the thermal conductivity of the fluid. The Nusselt number is equal to the dimensionless temperature gradient at the surface exposed to the fluid, and provides a measure of the convection heat transfer at the surface. Although similar in form, the Nusselt number and the Biot number differ in definition and interpretation. Whereas the Biot number is defined in terms of the thermal conductivity of the solid, the Nusselt number is based on the thermal conductivity of the fluid.
In order to understand how the characteristic cooling times dependent on the Fourier number and the Biot number, a nonsteady, one-dimensional conduction problem with constant properties will be discussed (Chapman, 1984). It is desired to determine the temperature within the body at any time. The general conduction equation for constant properties and no internal heat generation is given as:

\[
\frac{(1/\alpha)(\partial t/\partial \tau)}{\partial^2 t/\partial x^2} = 0
\]  

(1.7)

where \( \alpha \) is the thermal diffusivity, \( t \) is the temperature, and \( \tau \) is the time. The body is initially at a uniform temperature \( t_i \), and the temperature of the surrounding fluid is changed to \( t_f \). Therefore, the boundary conditions for equation (1.7) are

At \( \tau = 0 \):

\[ t = t_i \text{, for all } x \]  

(1.8)

For \( \tau > 0 \):

\[ -k(\partial t/\partial x)_s = h(t - t_f) \]  

(1.9)

The linear variable will be nondimensionalized by defining new variable \( \xi \), which is defined as:

\[ \xi = x/L \]  

(1.10)

where \( x \) is the one-dimensional coordinate in the problem, and \( L \) is the characteristic length of the body. The temperature may also be nondimensionalized by the definition:

\[ T = (t - t_f)/(t_i - t_f) \]  

(1.11)

After defining the dimensionless parameters, \( \text{Bi, Fo, } \xi, \text{ and } T \), the differential equation and the boundary conditions reduce to:

\[ \partial T/\partial \text{Fo} = \partial^2 T/\partial \xi^2 \]  

(1.12)

At \( \text{Fo} = 0 \):

\[ T = 1 \text{, for all } \xi \]  

(1.13)

For \( \text{Fo} > 0 \):

\[ (\partial T/\partial \xi)_s = -\text{Bi}T \]  

(1.14)
The solution to equation (1.12) is of the form $T = f_n (\zeta, F_0, B_i)$. Therefore, geometrically similar bodies with identical Biot numbers will have the same dimensionless temperature response according to the time measured as Fourier numbers.

Consequently if the analysis of the quenching experiments were based on these dimensionless heat transfer numbers, the quantity of experimental data could be reduced substantially. It is believed that the behavior of numerous heat treatment processes can be determined with relatively few probes. In order to achieve the highest level of confidence, the dimensionless parameters of the probe should be identical to the parameters of the part being modeled.

1.4 Present Work

An experimental approach was used to determine the heat transfer behavior during quenching. The information obtained from previous research and present studies provided the necessary background to design and construct the quench system. The experimental method used in the present work is based on similar experiments performed by Beck et al. (1992). Eight cooling curve tests were performed during the study. The first test was carried out to show that the heat transfer was one-dimensional. The probe was then quenched at every 10-degree interval between 0 and 60 degrees from an initial temperature of 400 °C. The temperature of the bath was approximately 22 °C. The time-temperature data from cooling curve tests is shown in the results section. In addition, the cooling rates during the different cooling stages are given.
Chapter 2 Quenching

2.1 Theory

Many parts that are currently being manufactured are heat treated before being used (Totten et al., 1994). Parts are heat treated to improve certain properties, such as hardness, strength, toughness, ductility and corrosion resistance, and to increase uniformity of the properties. A heat treatment depends on the composition and microstructure of the alloy and the specific application for which the part is intended. Quenching is a particular type of heat treatment that involves rapidly cooling metal alloys for the purpose of hardening.

Generally speaking, heat treatment is a process involving heating a metal part to a high temperature followed by controlled cooling. The principal steps of any heat treatment the heating stage, the holding time, and the cooling stage. A heating cycle and three different cooling cycles are shown in Figure 2.1. The amount of time a part is held at the holding temperature and the rate at which heat is removed from the part are the most critical factors. It is necessary for the holding time to be sufficiently long to complete the required microstructural phase transformations. The cooling rate is equally important because different microstructural phases and their inherent properties are obtained at different cooling rates.

Steel alloys are used extensively in industry because their properties can be changed with relative simplicity by controlled heating and cooling. Therefore, the following discussion will focus on the heat treatment of steel.
2.2 Transformations of Steel during Heating

Totten et al. (1994) and Neely (1989) have described in detail the transformations of steel. In order to understand how to enhance particular properties of carbon steels and low-alloy types of steel during a heat treatment process, it is first necessary to comprehend how the microstructure of steel changes as a function of temperature and carbon content. The iron-carbon phase diagram illustrates the various phases of steel and cast iron that are present at specific temperatures and carbon concentrations. The region of the diagram that is significant to engineering applications is illustrated in Figure 2.2. Therefore, this discussion is limited to irons with carbon contents below 6.67%.

Figure 2.1 The steps in the heat treating of steel [Source: Totten et al. (1994)]
At carbon concentrations less than about 0.8 % and at temperatures below the eutectoid value of approximately 721 °C (1330 °F), the microstructure of steel consists of alpha ferrite and pearlite (Figure 2.3). At carbon concentrations greater than 0.8 % and below the eutectoid temperature, the phases present are pearlite and cementite (Fe₃O). Alpha ferrite, or simply ferrite, is a body-centered cubic (bcc) phase that is relatively soft and ductile. In contrast, cementite is fairly hard and brittle. The structure of pearlite consists of alternating layers (lamellae) of ferrite and cementite (Figure 2.4). Therefore, the mechanical properties of pearlite are intermediate between ferrite and cementite.

**Figure 2.2** The iron-carbon phase diagram [Source: Neely (1989)]
Figure 2.3  Microstructures of ferrous metals: (a) ferrite, (b) pearlite [Source: Neely (1989)]

Figure 2.4  The lamellar pearlite microstructure of steel [Source: Neely (1989)]
If a carbon steel with a carbon content below 0.8 % is heated to a temperature above the eutectoid temperature, pearlite undergoes a transformation to become face-centered cubic (fcc) austenite (Figure 2.5). The resulting microstructure consists of ferrite and austenite phases. If heating is continued, the ferrite will eventually begin to transform to the austenite phase. At a sufficiently high temperature, the ferrite will completely transform and a fully austenitic microstructure will be produced. The heating process just described is called solution treating or austenitizing.

Figure 2.5 Microstructure of austenite [Source: Neely (1989)]
Likewise, heating a carbon steel with a carbon content above 0.8 % to a temperature above the eutectoid value, again causes the pearlite to transform into austenite. Thus, the resulting microstructure consists of cementite and austenite phases. If heating is again continued to a sufficiently high temperature, a homogeneous austenitic microstructure will be produced.

2.3 Transformations of Steel during Cooling

If a solution treated steel consisting of less than approximately 0.8 %C is cooled very slowly from the austenite region, ferrite begins to precipitate from the austenite phase (Figure 2.6). At the eutectoid temperature of about 721 °C (1330 °F), the remaining austenite phase completely transforms into lamellae of ferrite and cementite called pearlite. The resulting microstructure produced consists of the proeutectoid ferrite phase, which was formed at a temperature higher than the eutectoid temperature, and the pearlite phase (ferrite and cementite). The spacing of the lamellae in the pearlite phase depends precisely on the rate of cooling through the eutectoid temperature.

Phase transformations such as the ones previously described cause a change in the carbon concentrations of the phases present because different microstructural constituents have different solid solubility limits to carbon in iron. Ferrite has a maximum solid solubility of 0.022 %C and austenite has a solubility limit up to 2.11 %C. Consequently, the concentration of carbon in austenite must increase during a cooling process. Below the eutectoid temperature, the extra carbon causes the formation of cementite.
Figure 2.6 Microstructures of SAE 1020 steel at various temperatures [Source: Neely (1989)]
Slowly cooling a solution treated steel with more than 0.8 %C is similar to cooling a lower carbon content steel, except that the first phase to precipitate from the austenite is cementite (Figure 2.7). At temperatures below the eutectoid value, the austenite phase again completely transforms into lamellae of ferrite and cementite, producing a microstructure consisting of the proeutectoid cementite and pearlite.

Figure 2.7 Microstructures of SAE 1095 steel at various temperatures [Source: Neely (1989)]
Continuous cooling steel with a carbon concentration of approximately 0.8% from the solution treating or austenitizing temperature, suppresses the formation of either proeutectoid ferrite or proeutectoid cementite. Thus, the microstructure produced would consist exclusively of the pearlite phase.

At higher cooling rates, different microstructural constituents are obtained with other desirable properties. Bainite and martensite are two such microstructures that can be formed. Bainite is produced during the decomposition of austenite between the temperatures of 200°C (400°F) and 540°C (1000°F) if the cooling rate is higher than that required for transformation to pearlite. Like pearlite, the microstructure of bainite consists of ferrite and cementite. However, the ferrite and cementite are not positioned in a lamellar manner. The bainite microstructure has a feathery or acicular appearance. Bainite is typically stronger and more ductile than pearlite microstructures that have a similar hardness level.

At temperatures below approximately 200°C, the fcc microstructure of austenite transforms into a body-centered tetragonal (bct) microstructure called martensite (Figure 2.8). Martensite is extremely hard and brittle and its microstructure has needle-shaped grains. Unlike other transformations, martensite transformations happen almost instantly because they do not involve the time-dependent carbon diffusion process.

Phase transformations involve volume changes because microstructural constituents have different densities. The density of the martensite phase is less than that of austenite, which causes martensite to occupy larger volumes than austenite. Thus, the transformation of austenite to martensite causes internal stresses that may lead to
dimensional changes in parts. In certain circumstances, the level of stress may be raised high enough to induce cracking. A similar but smaller volume expansion takes place during the transformation of austenite to pearlite.

If the temperature to which heat-treated steel is quenched is not low enough, a portion of the austenite phase does not transform to martensite during cooling. When this happens, the martensite phase is located in a matrix consisting of austenite (Figure 2.9). Retained austenite lowers the hardness and strength in parts, which may eventually lead to distortion and cracking in service.
2.4 Transformation Diagrams

There are two widely used diagrams that depict the time dependence of microstructural phase transformations (Totten et al., 1994; Callister, 1991). These diagrams are called time-temperature-transformation (TTT) diagrams or isothermal transformation (IT) diagrams and continuous cooling transformation (CCT) diagrams.

The time-temperature-transformation (TTT) diagrams shows the approximate percentages of the microstructural constituents present in the final microstructure of a specimen subjected to time-temperature treatments. A complete TTT diagram for an iron-carbon alloy of eutectoid composition is shown in Figure 2.10. This particular
Figure 2.10 The complete time-temperature-transformation diagram for an iron-carbon alloy of eutectoid composition: A, austenite; B, bainite; M, martensite; P, pearlite [Source: Callister (1991)]

diagram shows what the final microstructure of the alloy would be if it was subjected to one of three different isothermal heat treatments. It is apparent from studying these diagrams that there is a minimum time for a transformation to begin and each transformation occurs in a specific amount of time for a given temperature below the eutectoid value.
By comparing Figure 2.11 and Figure 2.12, it is apparent that higher carbon concentrations shift the start of transformations to later times. It can also be seen that it is extremely difficult to produce fully martensitic microstructures in low-alloy carbon steels because the transformation of austenite to ferrite and pearlite takes place very quickly. This rapid transformation of austenite to ferrite and pearlite makes it nearly impossible to produce fully hardened microstructures except in parts with relatively thin cross-sections. However, the addition of particular alloying elements can improve a part's ability to be hardened at lower cooling rates.

**Figure 2.11** TTT diagram for SAE 1021 carbon steel [Source: Totten et al. (1994)]
The second method that was developed to provide transformation data involved continuously cooling steel samples at specified rates. Continuous cooling transformation (CCT) diagrams depict the temperature range of each phase transformation and the amount of transformation that takes place at a given cooling rate and time. Two cooling curves that correspond to relatively fast and slow rates are illustrated in Figure 2.13. The transformation starts at the point where the cooling curve intersects the beginning reaction curve and ends where the cooling curve crosses the completion reaction curve.

A sufficiently high rate of cooling, such as the one represented by the curve in Figure 2.14, prevents the formation of any ferrite, pearlite, and bainite and creates a fully hardened martensitic microstructure. This cooling rate is commonly called the critical
cooling rate. The transformation to martensite begins at a temperature known as the martensite start (Ms) temperature and continues to a lower temperature called the martensite finish (Mf) temperature. A cooling rate lower than the critical value will produce some bainite or pearlite. Therefore, a fully hardened steel will not be achieved.
Figure 2.14 Continuous cooling transformation diagram for a eutectoid iron-carbon alloy and the microstructures obtained as a function of cooling rate [Source: Callister (1991)]

It should now be apparent that particular microstructures and properties can be achieved in steel by controlling the composition and the cooling rates from the austenizing temperature to room temperature. In hardening by quenching, steel is heated above the lower critical temperature of 721 °C (1330 °F) and then cooled fast enough that pearlite and ferrite do not have time to form. The maximum achievable hardness of a quenched steel is determined primarily by the carbon content and is obtained by cooling at a rate equal to or greater than the critical cooling rate for the particular alloy.
The CCT diagrams provide only the approximate cooling rates that are necessary to produce desired microstructures and properties. The true rates depend on the specific composition and microstructure of the alloy being heat-treated.

2.5 Heat Transfer with Boiling

The conversion of a liquid into a vapor is of considerable importance in numerous engineering applications. If the phase change to vapor occurs within a liquid, the process is called boiling. Chapman (1984) and Bejan (1995) have provided a detailed explanation of boiling heat transfer. Boiling is classified as a convective process because fluid motion is involved. Analyses of boiling is significantly more complex than other convective processes in which the fluid medium remains in a single phase. In addition to the fluid properties related to convection, such as thermal conductivity, density, specific heat, fluid viscosity, and the coefficient of thermal expansion, other properties—including surface tension, the latent heat of vaporization and pressure—are important in boiling analyses. Because of the large number of variables involved and the complexity of the process, general equations fully describing boiling do not exist. Therefore, the discussion to follow will be descriptive in nature.

Research has shown that if a liquid, such as water, contains no impurities, it will experience the liquid-vapor phase transformation without the formation of bubbles when it is heated in a clean, smooth receptacle. However, vapor bubbles normally do appear on
a heating surface when the rate of heat input is sufficiently high. The formation of these bubbles and their subsequent growth and movement substantially affects the heat transfer process.

Boiling may take place under various conditions. If a heating surface is submerged in a pool of liquid without agitation and heat is transferred to the liquid only by free convection, this process is called pool boiling. When boiling occurs simultaneously with fluid motion induced by externally created pressure differences, the process is called forced convection boiling. The following discussion will focus on pool boiling.

Under certain conditions, localized boiling may occur at a heating surface while the temperature of the bulk of the liquid remains well below the saturation temperature. This is known as subcooled pool boiling. If heating is continued, the bulk of the pool will eventually reach the saturation temperature. This condition is known as saturated pool boiling.

The heat flux through the heating surface during saturated pool boiling is a complicated function of the temperature difference, \( t_s - t_{\text{sat}} \), by which the surface temperature exceeds the saturation temperature. The different regimes of pool boiling are illustrated in Figure 2.15. The data in the figure was obtained by an electrically heated wire submerged in a pool of liquid at the saturation temperature. The wire serves the dual purpose of a heating surface and a resistance thermometer for the measurement of \( t_s \).
As the surface temperature of the wire is raised above the saturation temperature, convection currents circulate the liquid and vapor is produced by evaporation at the free liquid surface. This regime is called free convection boiling. Further increase in wire surface temperature causes the process to enter the regime of nucleate boiling. In this region, vapor bubbles form at favorable sites on the heating surface. As \( t_s - t_{sat} \) increases, additional bubbles are formed. In the lower end of this region, the bubbles that leave the heating surface may condense in the colder bulk of the fluid. As \( t_s - t_{sat} \) is further increased, the bubbles formed may rise to the free liquid surface. The upper limit in the nucleate boiling regime is called the peak heat flux or the critical heat flux. Continued increase in \( t_s - t_{sat} \) beyond the peak heat flux causes the process to enter the
regime of partial film boiling. In this region, the heating surface is partially blanketed with a film of vapor. The presence of this film causes resistance to heat transfer because the liquid phase is a better conductor than vapor. Continued heating will cause the heating surface to be completely covered with vapor. This regime is called stable film boiling. Heat transfer during this regime occurs by conduction through the vapor and then into the liquid. As $t_s - t_{sat}$ is further increased, heat transfer by radiation takes place. Eventually the temperature is reached which causes the heating surface to melt.

2.6 Theory of Quenching Heat Transfer

The cooling curve test is generally accepted as the most effective means of describing the cooling mechanisms of a quenching process (Totten et al., 1994). A cooling curve along with the critical cooling stages that occur during quenching are illustrated in Figure 2.16. If a heat-treated steel part is continuously cooled from a solution treating temperature in a quenching medium that rapidly extracts heat, four stages of heat transfer will take place. These stages characterize the four different cooling mechanisms that occur during quenching. The four cooling stages are known as the initial liquid contact or wetting stage, the vapor blanket or film boiling stage, the nucleate boiling stage, and the convective heat transfer stage.

The initial liquid contact stage lasts for an extremely brief time and is not recognizable on cooling curves acquired by practical experimental procedures. For this reason, the stage was not included in the cooling curve figure shown. This cooling mechanism is characterized by intense boiling at the metal-quenchant interface and rapid
cooling of the metal surface exposed to the quenchant. Since the duration of this stage is brief, it is frequently disregarded during cooling curve analyses.

The vapor blanket stage is initiated when an unbroken layer of water vapor bubbles form along the surfaces of the heated metal. This vapor blanket acts as a barrier to heat conduction because of the high thermal resistance of the vapor layer. Although the temperature gradients within a part are not as severe as they are during the nucleate boiling stage, the extreme temperatures may be high enough to induce plastic deformation.
The nucleate boiling stage begins once there is an insufficient amount of heat transferred from the metal surface to completely vaporize the quenchant. This stage is characterized by violent boiling at the metal-quenchant interface and rapid cooling of the metal surface in contact with the quenching fluid. Even though the temperature gradients within a part are highest in this stage, the lower temperatures at which nucleate boiling occurs generally results in lower plastic deformation. However, if this stage is initiated when temperatures are sufficiently high, quench distortion will occur.

Finally, the convective heat transfer stage is initiated when the temperature of the metal surface drops below the boiling point of the quenchant. The rates of heat removal from the metal surface are lowered to those values related to laminar convective flow. Heat transfer rates in this stage are influenced by several quenchant variables, such as viscosity, temperature, and agitation rate. Compared to the previous two stages mentioned, the convective heat transfer stage has a substantially lower affect on the performance of the quenching process.

2.7 Interpretation of Cooling Curves

Lack of standardization in probe construction has made comparisons of cooling curves acquired by different investigators extremely difficult. Totten et al. (1994) has discussed the efforts of several investigators who have simplified cooling curve interpretation, including Tamura, Wang, and Carey. Tamura introduced the method of master cooling curve calculation for cylindrical probes. This technique has also been used by Wang and others.
The master cooling curve is produced by plotting the temperature as a function of a parameter known as $k_1$. This value is expressed as:

$$k_1 = \frac{t}{D^n}$$

(2.1)

where $t$ is the time in seconds required to cool the probe from an initial temperature to any desired temperature, $D$ is the diameter of the probe in centimeters, and $n$ is a constant associated with the thermocouple position in the probe. At every temperature during the quenching process, $k_1$ is calculated by dividing the time by the probe diameter raised to the $n$th power.

Although Tamura described the method for the exact determination of $n$, the values illustrated in Table 2-1 can be used as close approximations.

Table 2-1 Approximate Values of $n$ for Construction of Master Cooling Curve

<table>
<thead>
<tr>
<th>Probe material</th>
<th>Thermocouple placement</th>
<th>$n$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silver</td>
<td>Center</td>
<td>1.34</td>
</tr>
<tr>
<td></td>
<td>Surface</td>
<td>1.14</td>
</tr>
<tr>
<td>Steel</td>
<td>Center</td>
<td>1.37</td>
</tr>
<tr>
<td></td>
<td>Surface</td>
<td>1.00</td>
</tr>
</tbody>
</table>

The materials most widely used for probe construction are silver, nickel, and steel. Each of these materials generate substantially different cooling curves. Tamura found that heat transfer within a probe is related to the product of the specific heat and
the density of the probe (Table 2-2). Once this product has been calculated, cooling curves acquired with probes of different materials can be compared by plotting temperature as a function of $t/cpD^n$, where $t$, $D$ and $n$ are the same as before, $c$ is the specific heat, and $\rho$ is the density. This relationship only holds true if the probe is cylindrical and the thermocouple is located at the geometric center.

Table 2-2 Correlational Factors for Variation in Probe Construction Material

<table>
<thead>
<tr>
<th>Probe material</th>
<th>Specific heat ($c$)</th>
<th>Density ($\rho$)</th>
<th>$c \times \rho$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>cal/g · °C</td>
<td>g/cm$^3$</td>
<td>lb/in.$^3$</td>
</tr>
<tr>
<td>Silver</td>
<td>0.061</td>
<td>10.21</td>
<td>0.37</td>
</tr>
<tr>
<td>Steel</td>
<td>0.108</td>
<td>7.76</td>
<td>0.28</td>
</tr>
<tr>
<td>Nickel</td>
<td>0.140</td>
<td>8.80</td>
<td>0.32</td>
</tr>
</tbody>
</table>

A variety of probe shapes have been used for cooling curve acquisition, including cylindrical, spherical, and plate. One investigator (Totten et al., 1994) produced a summary of constants that allow comparisons of cooling curves obtained with probes of various shapes (Table 2-3).
Table 2-3 Calculation of Tamura's D Factor as a Function of Probe Shape

<table>
<thead>
<tr>
<th>To convert from</th>
<th>To</th>
<th>Multiply D by</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cylinder</td>
<td>Sphere</td>
<td>1.33</td>
</tr>
<tr>
<td>Cylinder</td>
<td>Plate</td>
<td>0.66</td>
</tr>
<tr>
<td>Sphere</td>
<td>Cylinder</td>
<td>0.75</td>
</tr>
<tr>
<td>Sphere</td>
<td>Plate</td>
<td>0.50</td>
</tr>
<tr>
<td>Plate</td>
<td>Cylinder</td>
<td>1.50</td>
</tr>
<tr>
<td>Plate</td>
<td>Sphere</td>
<td>2.00</td>
</tr>
</tbody>
</table>

To simplify cooling curve analyses for oil quenchants, Tamura introduced a quantity called the "V-value". The V-value is directly related to the capability of a oil quenchant to harden steel. V-values are calculated from cooling curve data and CCT diagram data using the relationship:

\[ V = \frac{T_c - T_d}{T_s - T_f} \]  

(2.2)

where \( T_c \) is the temperature at the beginning of the nucleate boiling stage, \( T_d \) is the start temperature of the convective heat transfer stage, \( T_s \) is the start temperature of the austenite to pearlite transformation curve, and \( T_f \) is the start temperature of the martensite transformation. Thus, the V-value is the ratio of the nucleate boiling temperature range to the temperature range that is required to obtain high hardness. Without further development this method cannot be applied to cooling curves obtained with other quenchants.
2.8 Design Considerations for Quenching

The rate of cooling within the heat treated part must be as uniform as possible throughout the quenching process to minimize the potential for cracking, distortion, and nonuniform property formation (Kalpakjian, 1992). Optimal quench uniformity is particularly critical during the vapor blanket stage and nucleate boiling stage. Unfortunately heat transfer is not likely to be uniform during quenching, especially in parts with varying cross-sections. Thus, undesirable temperature gradients within heat treated parts cause variations in contraction, which creates thermal stresses. If the temperature of the part is sufficiently high, the thermal stresses due to nonuniform cooling cause plastic deformation and residual stresses. In addition, the volume expansions that occur due to microstructural constituent density changes may create residual stresses as well. A successful quench process will control these internal stresses to ensure the formation of desirable transformation products.
Chapter 3  Experimental Apparatus and Procedures

3.1 Quench Probe

The instrumented quench probe used during the experiments is shown in Figure 3.1. The structure of the probe consists of the following: an enclosure from which interior surface temperature measurements are taken, a protective tube that channels the thermocouples to the data acquisition system, and a handle for transport.

Figure 3.1  Quench probe fabricated at Ohio University
The most critical component of the probe was made from six 0.25 inch thick, 304 stainless steel plates that were arranged to form a rectangular box. Stainless steel was selected for the plate material so phase transformations do not occur during the quenching. Five sides of the box, including the backside to which the thermocouples were attached, were welded together. The front side was the cover to the box structure. The cover plate was bolted to the welded five-plate structure so access could be gained to the thermocouples. Fourteen 0.125 inch diameter socket head cap screws were used to fasten the cover plate to the box structure. A 0.032 inch thick copper plate was used as a gasket between the cover plate and the other structure to ensure the quenchant could not leak into the probe. The entire sealed rectangular box measures 6 x 4 x 1.25 inches.

The quench probe was instrumented with five chromel-alumel (type k) thermocouples. Four thermocouples were silver soldered to the inside surface of the back plate of the enclosure. The fifth thermocouple was mounted outside the front plate to determine the starting point of the cooling curve. Figure 3.2 shows the positioning of the interior thermocouples. Thermocouple #1 is located approximately 0.75 inch above the geometric center. Thermocouple #3 is positioned 90 degrees away from thermocouple #1, about 0.75 inch to the right of the geometric center. Both of these thermocouples were used to evaluate the one-dimensional approximation for the data sampled at the geometric center, where thermocouple #2 is located. Thermocouple #4 is positioned a 0.5 inch from the bottom right-hand corner of the back plate.

Each of the four interior thermocouple wires passed through a hole in the top of the box into a 0.5 inch diameter, 12 inch long, 304 stainless steel tube. One end of this
The purpose of this tube is simply to shield the thermocouples from the quenchant during quenching.

The handle attachment actually consists of two components that are welded together on their respective centerlines. The first part is a 0.75 inch diameter, 6 inch long pipe. One end of this pipe connects to the compression fitting and the other end is open and acts as an outlet for the thermocouples. The second part is a 0.25 inch thick, 17 inch long, t-shaped plate that serves two purposes. In addition to acting as a handle for conveyance, the plate has mounting holes that allows the probe to be suspended from its holding apparatus.
3.2 Quench Probe Holding Apparatus

The apparatus used to quench the probe at different angles is shown in Figure 3.3. The probe has the capability of being suspended from two 0.5 inch diameter, 1 inch long steel studs that are attached to an angle adjustment plate. The adjustment plate is made of 0.25 inch thick, steel stock and measures 10.375 inches by 8.5 inches. The angle that the probe is quenched is determined by the seven sets of mounting holes in the adjustment plate that are spaced in 10 degree increments. Therefore, the probe could be potentially quenched at any 10 degree position between 0 and 60 degrees. The probe is fixed to the adjustment plate by closing a hold down action toggle clamp that is mounted to the plate. The adjustment plate is securely fastened to the side of a 6 x 3 x 3 inch steel block by 0.5

Figure 3.3 Quench probe holding apparatus
inch diameter shoulder bolts. A square hole was precisely machined through the center of the metal block to allow a 31.75 inch long 2 inch square piece of tubing to fit through it. Together, the machined piece of metal and the square tubing act as a slide mechanism for the probe. Unpinning the metal block from the square tubing allows the probe to descend steadily into the quench tank. The rate the probe travels down the slide is determined by the amount of resistance that the person operating the experiment applies to the steel tube handle attached to the slider block. The depth that the probe submerges into the quenchant is determined by a stop device positioned near the bottom end of the square tubing. To ensure the probe experiences no undesirable motion, the slide mechanism is supported by a rigid structure made of square tubing and angle iron (Figure 3.4). Before being put into service, the entire structure was carefully leveled in the lab by adjusting 0.5 inch diameter, 2 inch long carriage bolts that are screwed into each leg.

3.3 Quenchant and Quench Tank

The quenchant used during the experiments was water. The water was held in a 100 gallon tank made of a high strength plastic. It was decided that a plastic tank should be used to ensure that a reaction does not occur between the quenchant and the tank that could potentially change the composition of the quenching medium. Looking from above, the quench tank is oval shaped and gets progressively narrower at decreasing depths. The side to side distances of the top of the tank measure 47.5 inches by 28 inches. As one moves down 24 inches to the bottom of the tank, the measurements narrow to 39.5 inches by 18 inches.
3.4 Data Acquisition Hardware

Described in this section are the basic features of the critical components comprising the data acquisition system. The name of the data acquisition board used during the experiments is the DAS-1701ST. The DAS-1701ST is one of a family of DAS-1700 series boards manufactured by Keithley Instruments that were designed for an IBM PC or a compatible computer operating in the Windows environment. The analog input features of the DAS-1701ST boards are 16 single-ended or eight differential channels. 12-bit resolution, $166.67 \times 10^3$ samples/second maximum throughput, and gains of 1, 5, 50 and 250. The EXP-1800 is the name of the expansion accessory that
was used with the DAS-1701ST board. The most important feature of the EXP-1800 is its capability to place 16 differential inputs on any single-ended input channel of the data acquisition board.

3.5 Data Acquisition Software

The data acquisition applications used during the experiments were created with a software package called TestPoint. TestPoint has several capabilities which include: controlling external measurement devices, creating user interface items, displaying and analyzing data, creating data files, and dynamically exchanging data with other software applications. Furthermore, TestPoint applications have the ability to support sequential execution, repeating loops, and conditional statements.

The TestPoint programming structure is different than conventional programming languages. In TestPoint, the user chooses objects that represent various components of the application, such as a user interface item or external hardware. The user then integrates the actions the objects carry out to perform a desired task. In other words, a sequence of actions is executed by the application's objects when the associated object receives a particular event to initiate a response. For example, in order to graph analog samples, a pushbutton object could carry out the action to begin sampling after being activated when the user pressed a pushbutton icon. An A/D object could then execute the action to graph the samples after being activated by the analog input arriving to the computer.
3.6 Experimental Procedures

In this section is a brief description of the experimental procedures followed during the experiments. The experimental procedures basically consisted of the following: preparing the probe and the holding apparatus for immersion quenching, heating the probe to the specified steady-state value, running the data acquisition application programs to collect and display the sampled data, and quenching the probe.

The surfaces of the quench probe were first thoroughly scrubbed with a wire brush to remove oxides and other unwanted material. The connections leading from the interior thermocouples of the quench probe to the data acquisition system were then carefully checked to ensure there were no loose connections.

In the second part of the procedure, the quench probe was placed inside the furnace in such a manner as to not disturb the furnace thermocouple. The top of the furnace was then completely covered with a plate of alumina insulation to minimize heat transfer from the furnace. Next, various control parameter values were adjusted on the furnace temperature controller to guarantee the furnace reached the desired steady-state temperature of 400 degrees.

The custom data acquisition applications used during the third segment of the procedure were created with a software package called TestPoint. The first data acquisition application we created using TestPoint was used to monitor the probe’s interior surface temperatures until they converged to their perspective steady-state values. A second application was used to measure the temperature response during the different
cooling stages of the quenching process. Basically, both programs took analog samples from the analog/digital (A/D) board, converted the measured electric voltages to temperature values, and graphed them when a button was pushed by the user.

The first application required slower sampling over a longer time period with continuous monitoring of the data. For this purpose, the "Start A/D" and the "Add point(s) to" TestPoint programming actions were executed in the application. The "Start A/D" action has parameters that describe the number of desired samples, the rate at which the samples are collected, and the input channels to be sampled. The "continuous" programming command was entered for the number of samples and 1 hertz was given as the sampling rate. The "Start A/D" command begins sampling immediately and activates the next action to be carried out before the analog samples arrive at the computer. The "Add point(s) to" action adds a data point to a continuously scrolling strip chart each time a sample arrives.

The data acquisition application used during the quenching process required a substantially higher sampling rate over a shorter time period. For this reason, the "Acquire A/D" and the "Draw Graph" programming actions were used in the second application. The "Acquire A/D" action has the same parameters as the "Start A/D" action, although the "Acquire A/D" action does not activate the next action to be executed until all of the desired samples are taken. The "Draw Graph" action simply graphs the stored data that is collected by the "Acquire A/D" command. Data acquisition for the cooling curve was initiated approximately 1 second before removing the probe from the
furnace. Each of the thermocouples were sampled every of 0.01 of a second for 2 minutes. Therefore, 12000 samples were taken per channel.

In the final part of the procedure, the probe was quickly removed from the furnace and suspended from two studs protruding through the angle adjustment plate. The probe was then rigidly fixed to the adjustment plate and the attached slider block by closing the toggle clamp fastened to the plate. Next, a pin was removed from the slide that allowed the probe to descend into the quenchant until the slider contacted a stop device at the bottom of the slide. Once data acquisition was completed, the temperature history was stored in data files.
Chapter 4  Experimental Results

4.1 Discussion of Cooling Curves

The eight cooling curve data files were opened using the spreadsheet software package – Excel. The cooling curve to show that the heat transfer is one-dimensional is illustrated in Figure 4.1. The curves for the data sampled at the center and to the right of center are directly on top of each other. The temperatures measured at the point above the center are slightly higher than those at the center and to the right of center. The higher temperatures can be explained by the fact that the top of the probe enters the quenchant last, and therefore, there is a delay in cooling. For the other seven quench tests, the temperatures measured by the outside thermocouple and the center thermocouple were plotted as a function of time.

The cooling curve for the 0-degree quench angle test is shown in Figure 4.2. The starting point of the cooling curve is determined by the time at which the outside thermocouple enters the quenching medium. This point is indicated on the cooling curve by the vertical segment of the outside thermocouple trace. The three stages of cooling that are recognizable by practical experimental procedures were observed.

After carefully examining the temperature history, the duration of the vapor blanket and the nucleate boiling cooling stages were determined. The vapor blanket stage was determined to last for approximately one second and the nucleate boiling stage lasted for approximately 16 seconds.
Figure 4.1 Temperature versus time plot to show that one-dimensional approximation is valid.
Examination of the cooling curve shows that the data has some scatter. The temperature measurements varied by as much as approximately 6 degrees between two consecutive readings. This variation is normal during cooling curve acquisition. The deviation in the temperature readings is caused by external electrical noise, internal error limits in the instrumentation and the data acquisition system, and disturbances in the quench test apparatus.

The convection heat transfer from the plate surface can be expressed as:

\[ q = hA_p(T_{p,av} - T_\infty) \]  

(4.1)

where \( h \) is the convection heat transfer coefficient, \( A_p \) is the surface area of the plate, \( T_{p,av} \) is the average temperature of the plate, and \( T_\infty \) is the fluid temperature.

The plate temperature can be approximated as:

\[ T_{p,av} = \frac{(T_{in} + T_{fin})}{2} \]  

(4.2)

where \( T_{in} \) is the initial temperature of each cooling stage and \( T_{fin} \) is the final temperature of each cooling stage. The heat transfer can also be expressed as:

\[ q = m_p c_p \Delta T_p \]  

(4.3)

where \( m_p \) is the mass of the plate, \( c_p \) is the specific heat at constant pressure, and \( \Delta T_p \) is the change in temperature of the plate. If the two expressions for the heat transfer are set equal to each other, one could solve for the convection heat transfer coefficient.

Approximate values of the convection heat transfer coefficient along with some other values that were used in the calculations are shown in Table 4-1. The values of the heat transfer coefficient for the vapor blanket stage, the nucleate boiling stage, and the convective heat transfer stage are 657.1 W/m² \cdot K, 2154.4 W/m² \cdot K, and 551.8 W/m² \cdot K,
Table 4-2  Values that were used to calculate the Convection Heat Transfer Coefficient for the Different Cooling Stages

<table>
<thead>
<tr>
<th></th>
<th>( T_{p,av} = (T_m + T_f)/2 )</th>
<th>( q = m_p c_p \Delta T_p )</th>
<th>( h )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vapor Blanket Stage</td>
<td>385.9</td>
<td>3428.7</td>
<td>657.1</td>
</tr>
<tr>
<td>Nucleate Boiling Stage</td>
<td>240.6</td>
<td>104217.0</td>
<td>2154.4</td>
</tr>
<tr>
<td>Convective Heat Transfer Stage</td>
<td>61.5</td>
<td>28529.5</td>
<td>551.8</td>
</tr>
</tbody>
</table>

respectively. The values of the specific heat used in the calculations for the three cooling stages are 565 J/kg · K, 535 J/kg · K, and 485 J/kg · K. The value of the mass of the plate used in the calculations was 0.7767 kilograms.

Once the convection coefficients are known, one can determine the Biot numbers and the Nusselt numbers for the thermal boundary layer. The Biot numbers for the vapor blanket stage, the nucleate boiling stage, and the convective heat transfer stage are 0.203, 0.748, and 0.226, respectively. The values of the thermal conductivity used in the Biot number calculations for the three stages of cooling are 20.6 W/m² · K, 18.3 W/m² · K, and 15.5 W/m² · K. The Nusselt numbers for the cooling stages are 6.806, 22.318, and 5.716. The value of the thermal conductivity of the fluid used in the Nusselt number calculation was 0.613 W/m² · K.

The cooling curves for the six other quench tests performed at angles between 10 and 60 degrees are illustrated in Figures 4.3 - 4.8, respectively. Each of these cooling curves has the same characteristic shape as typical cooling curves. Comparison of the different cooling curves shows the scatter is nearly identical.
Figure 4.3 Temperature versus time for the 10 degree quench angle test
Figure 4.4 Temperature versus time for the 20 degree quench angle test.
Figure 4.5 Temperature versus time for the 30 degree quench angle test
Figure 4.6 Temperature versus time for the 40 degree quench angle test
Figure 4.8 Temperature versus time for the 60 degree quench angle test
Gur et al. (1996) experimentally investigated quench processes that were similar to these tests. Gur’s study measured the effect of bath temperature on water quenching C60 steel. More specifically, the study consisted of quenching a 30-mm diameter, C60 steel cylinder from 720 °C to 20 °C, and to 60 °C. The cooling curves obtained from the experiment are illustrated in Figure 4.9.

Examination of the plot shows the cooling curve has the same shape to the cooling curves in the study, although, the Gur’s test produced much higher cooling rates. Higher rates are expected because of the higher initial probe temperature. The cooling rates in the vapor blanket stage are close to 1 °C/second. In the nucleate boiling stage, approximate rates were calculated at 3, 6, and 10 seconds. The peak cooling rates were experienced near 3 seconds. The rates were in the 90 °C/second range. After 6 seconds, the cooling rates were in the sixties. After 10 seconds, the rates fell below 20 °C/second. The cooling rates continued to gradually decrease in the convective boiling stage.

![Figure 4.9 Cooling curves for water quenching a C60 steel cylinder. [Source: Gur et al., (1996)]]
Beck et al. (1992) has discussed the use of an inverse heat conduction method for analysis of quenching. The inverse method uses the transient temperatures measured inside the test sample to estimate the surface heat flux or the transient heat transfer coefficient. Beck’s method was not based on an experiment. However, his technique can be applied to the data collected in the study.

Beck developed and verified a computer program called QUENCH1D. The program can analyze nonlinear, composite materials of different shapes, including one-dimensional flat plates, cylinders, and spherical bodies. The results obtained shows the program can be used for accurate analyses.

Several numerical methods have been used to solve the inverse heat conduction problem. Beck’s method is sequential and involves the use of future temperatures for each calculated component of the surface heat flux. An important feature of the procedure was the ability to use a variable number of future time steps during a single experiment. This feature provided information regarding the variation of the surface heat flux. Moreover, the method involves specifying a functional form of the surface heat flux to stabilize the problem.

The function specification method was used for the analysis of a homogeneous planar body having constant thermal properties. The body was exposed to a triangular heat flux history, which is shown in Figure 4.10. The thickness of the body is given as ‘L’. The temperature sensor was attached to the interior surface at x = L. In Figure 4.10, a dimensionless heat flux ‘$q^+$’ is plotted versus dimensionless time ‘$t^+$’, which is given as:

$$t^+ = \alpha t/L^2$$

(4.4)
Figure 4.10 Heat flux versus dimensionless time plot for a homogeneous planer body. [Source: Beck et al., (1992)]

where $\alpha$ is the thermal diffusivity, $t$ is the time, and $L$ is the characteristic length. In the region of the curve below $t^+$ values of 0.6, the dimensionless heat flux $q^+$ equals the dimensionless time $t^+$. The dimensionless heat flux is given as:

$$q^+ = q/q_N$$

(4.5)

where $q_N$ is a nominal value of the heat flux determined by setting $t^+$ equal to unity in equation (4.4). The dimensionless $x$ coordinate is given as:

$$x^+ = x/L$$

(4.6)

where $L$ is the characteristic length.
The dimensionless temperature is given as:

$$T^+ = (T - T_0)/(q_N L/k) \quad (4.7)$$

where $T$ is the temperature, $T_0$ is the exterior surface temperature, $k$ is the thermal conductivity, $q_N$ is the nominal value of the heat flux, and $L$ is the same as before.

The simulated temperatures as a function of dimensionless time for $x^+ = 0$ and 1 are shown in Figure 4.11. The temperature response at the interior surface was delayed because of changes in the surface heat flux. The temperature at the exterior surface started to rise the moment $q$ is nonzero.

Inverse calculations for the surface heat flux values and temperatures were performed using temperature measurements at $x^+ = 1$ with a time spacing of $\Delta t^+ = 0.06$. Twenty-equally spaced values were used to plot the curves.

The results obtained from QUENCHID are illustrated in Figures 4.12 and 4.13. In Figure 4.12, the dots denote the calculated heat fluxes at different values of future time steps. These time steps are represented by an ‘r-value’. The r-value equal to unity is for

Figure 4.11 Exterior and interior surface temperatures for the planer body exposed to the triangular heat flux. [Source: Beck et al., (1992)]
exact matching of the experimental temperatures by the calculated values. The calculation for \( r = 1 \) is not stable, and therefore, is not shown in the figure. The estimated heat flux values near \( t^+ = 0.6 \) have the largest error. Even in this region the values are accurate, with the greatest error being approximately 10 percent for \( r = 5 \).

The calculated exterior surface temperatures for \( r = 2 \) and 5 are shown in Figure 4.13. The calculated surface temperatures are more accurate than the calculated heat flux values. Examination of the plot shows the \( r = 2 \) curve is extremely close to the exact surface temperatures except at times near \( t^+ = 0 \).
4.2 Determination of Cooling Rates

After plotting the time-temperature data, the Excel trendline function was used to calculate a linear least-squares fit through the vapor blanket stage data points. The trendline function was then used to calculate a sixth-order polynomial least-squares fit through the nucleate boiling stage and the convective heat transfer stage data points. The temperature versus time plot for the 0 degree quench angle test with the least-squares equations is illustrated in Figure 4.14. The derivatives of the least squares fit equations were then calculated to determine the cooling rate curves.

The cooling rate curve for the 0 degree quench angle test is shown in Figure 4.15. The time scale of this plot was corrected so the origin corresponds to the start point of the
Figure 4.14 Temperature versus time plot showing least-squares equations for different cooling stages.

- Convective heat transfer stage data
- Nucleate boiling stage data

Nucleate boiling stage least-squares equation:

\[ y = -0.0001x^6 + 0.0165x^5 - 0.9732x^4 + 30.202x^3 - 515.92x^2 + 4542.4x - 15497 \]

Convective heat transfer stage least-squares equation:

\[ y = 1\times10^{-9}x^6 - 5\times10^{-7}x^5 + 0.0001x^4 - 0.0115x^3 + 0.7081x^2 - 24.175x + 435.38 \]
Figure 4.15 Cooling rate versus time for the 0 degree quench angle test
cooling curve. The beginning of the cooling rate curve is considered to be the start of the vapor blanket stage. The cooling rates in this region were extremely low because of the high thermal resistance of the vapor layer. At approximately one second later, the highest cooling rates are observed. These rates correspond to the earliest moments of the nucleate boiling region. Beyond the earliest moments of nucleate boiling, the rates of heat removal gradually decrease.

Cooling rate curves were also calculated for three of the other quench tests performed. The cooling rate curves for quench angles of 20, 40, and 60 degrees are illustrated in Figures 4.16 - 4.18, respectively. Like the 0 degree quench angle plot, the time scale of these plots were corrected so comparisons could be made with the other quench angles.

Table 4-2 shows some of the cooling rate values that were used to plot the cooling rate curves at different quench angles. The cooling rates at 1 second occur during vapor blanket stage cooling. These rates are approximately a couple of °C/second. The cooling rates at 2, 5, and 12 seconds are experienced during the nucleate boiling stage. The peak cooling rates are observed near 2 seconds. These rates are in the range of 40 °C/second.

Table 4-2  Cooling Rates given in °C/sec at Various Times after the Starting Point of the Cooling Curve

<table>
<thead>
<tr>
<th></th>
<th>1 sec</th>
<th>2 sec</th>
<th>5 sec</th>
<th>12 sec</th>
<th>30 sec</th>
<th>50 sec</th>
<th>70 sec</th>
</tr>
</thead>
<tbody>
<tr>
<td>0°</td>
<td>0.88</td>
<td>42.09</td>
<td>26.93</td>
<td>8.17</td>
<td>1.39</td>
<td>0.75</td>
<td>0.44</td>
</tr>
<tr>
<td>20°</td>
<td>1.36</td>
<td>40.11</td>
<td>26.97</td>
<td>8.82</td>
<td>1.24</td>
<td>0.64</td>
<td>0.37</td>
</tr>
<tr>
<td>40°</td>
<td>1.73</td>
<td>40.87</td>
<td>26.96</td>
<td>8.67</td>
<td>1.34</td>
<td>0.67</td>
<td>0.38</td>
</tr>
<tr>
<td>60°</td>
<td>2.36</td>
<td>40.67</td>
<td>26.11</td>
<td>8.47</td>
<td>1.30</td>
<td>0.63</td>
<td>0.40</td>
</tr>
</tbody>
</table>
Figure 4.16 Cooling rate versus time for the 20 degree quench angle test
Figure 4.17 Cooling rate versus time for the 40 degree quench angle test
Figure 4.18 Cooling rate versus time for the 60 degree quench angle test
After 5 seconds, the cooling rates are in the mid twenties. In the lower end of the nucleate boiling stage, the rates fall below 10 °C/second. The cooling rates given at 30, 50, and 70 seconds occur during the convective heat transfer stage. The rates near 30 seconds are comparable to those given in the vapor blanket stage. After 50 seconds, the rates have fallen below 1 °C/second. Comparison of the cooling rate values at the different quench angles shows there is no significant difference in cooling behavior along the inside surface of the probe.
Chapter 5 Conclusions and Future Work

5.1 Summary

A quench system was developed to collect heat transfer data that will be used in future studies to design quench processes for optimum heat treating of materials. Development of the quench system consisted of designing and constructing critical components of a quench test apparatus and setting up a data acquisition system. The fabricated components of the quench test apparatus are a quench probe and a probe holding structure.

The main structure of the quench probe consists of a six-plate stainless steel rectangular box which is instrumented with chromel-alumel (type k) thermocouples. The probe has mounting holes, which allow it to be suspended from a holding apparatus.

The holding apparatus is used to quench the probe in a controlled manner. This apparatus has seven sets of mounting holes that are spaced in 10-degree increments. Thus, the probe has the capability of being quenched at every 10-degree interval between 0 and 60 degrees. Another important component of structure is the slide mechanism. Releasing a pin from the slide mechanism allows the probe to descend steadily into the quench tank.

5.2 Conclusions

During a quenching process, the part temperatures and the cooling rates need to be controlled within certain limits to ensure the formation of desirable metallurgical properties. Uniform and predictable heat transfer is critical during a quenching process to
minimize the potential for distortion, cracking and nonuniform property distribution. In order to predict the transient temperature distribution and the resultant stress distribution within a quench part, experimental investigation of the time-dependent heat transfer coefficient is required.

A cooling curve test was carried out at every 10 degree interval between 0 and 60 degrees from an initial temperature of 400 °C. Temperature measurements were taken at the probe's geometric center and from outside the probe. For every quench angle, the measured temperatures were plotted as a function of time to produce a cooling curve. After plotting the cooling curves, the Excel trendline function was used to calculate a least squares fit through the data points. The derivatives of the least squares fit equations were then calculated to determine the cooling rate curves.

The cooling rates at 1 second occur during vapor blanket stage cooling. These rates are approximately a couple of °C/second. The peak cooling rates are observed near 2 seconds. These rates are in the range of 40 °C/second. After 5 seconds, the cooling rates are in the mid twenties. In the lower end of the nucleate boiling stage, the rates fall below 10 °C/second. The rates near 30 seconds are comparable to those given in the vapor blanket stage. After 50 seconds, the rates have fallen below 1 °C/second. Comparison of the cooling rate values at the different quench angles shows there is no significant difference in cooling behavior along the inside surface of the probe.

Approximate values of the convection heat transfer coefficient for the vapor blanket stage, nucleate boiling stage, and the convective heat transfer stage are 657.1 W/m² · K, 2154.4 W/m² · K, and 551.8 W/m² · K, respectively. The Biot numbers for
the three stages of cooling are 0.203, 0.748, and 0.226. The Nusselt numbers for the cooling stages are 6.806, 22.318, and 5.716.

5.3 Future Work

Future experimental work is expected to be performed based on the current procedure. Moreover, modifications are anticipated to be made to experimental apparatus and to the analysis technique. As stated earlier, comparison of the cooling curves and the cooling rate curves at different angles revealed there was no discernable difference in the cooling response along the inside surface of the probe. This behavior leads to the main objective of the future work, which is to analytically and experimentally determine the cooling response at the probe's exterior surface. Given below is a complete list of modifications to be made and the additional research to be performed.

1. Calculate the values of the convection heat transfer coefficient from the current series of quenching tests using the inverse heat transfer code being developed at Ohio University.
2. Perform additional tests with the thermocouples positioned closer to cooling surface.
3. Carry out additional tests with a new probe with increased wall thickness to determine the effects of lengthening the duration of the vapor blanket stage.
4. Use the heat transfer coefficients found to properly design a quench process for optimum heat treating of materials.
5. Use fast-response thermocouples.
References


Appendix

TestPoint code
Objects in panel "Panel1":

- Acquire
- Graph1
- A/D1
- File1

Settings for Panel object "Panel1":

- X: -1
- Y: -1
- Multitasking mode: Yield at end of loops
- Disable scroll bars: 0

Settings for Pushbutton object "Acquire":

- Visible: 1
- Enabled: 1
- Exec. actions at initialize: 0
Action list for A/D object "A/D1":

1) Acquire A/D A/D1 #samples=12000, rate=100 Hz, channel(s)="1(250),2(250),3(250),4(250),5(250)"
2) Draw graph Graph1 with A/D1
3) Erase File1
4) Output to File1 with A/D1, term.=CRLF

Settings for A/D object "A/D1":

Board #: 0
Demo mode: 0
Raw data mode: 0
Log to disk: 0
Filename: data.dat
Type: ASCII
Format: width: 7
decimal pl.: 3
Auto-increment name: 1
Mode: Append
Apply scaling: 0

Graph1

Settings for Graph object "Graph1":

Mode: Line
X vs Y: 0
X step (if not XvsY): 1
Visible: 1

File1

Settings for File object "File1":

Filename initial value:
Update disk on each output: 0
'Save As' style (else 'Open'): 0
Warn on create (if 'Open'): 1
Must exist (if 'Open'): 0
Warn on existing (if 'Save As'): 1
Default extension:
Dialog title:
File name filter:
Visible: 1
Enabled: 1
Execute actions at init: 0