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D. Scott MacKenzie*

Modern quenching oils offer a wide range of capability and performance. The selection of the proper quench oil is critical for proper heat treating operations. Improper selection of quench oil can result in short oil life, or soft parts. This paper will illustrate the selection factors in choosing a quench oil to ensure proper quality parts.

Introduction

Regardless of the product, it is likely that it is heat treated and quenched. Engine components are heat treated for wear and durability. Aircraft components are heat treated for strength and fracture toughness. Even bicycle frames are heat treated for strength, lightness and durability. To meet these needs, it is necessary to expand the knowledge of heat treating and quenching to consistently produce a quality product capable of being manufactured in a cost effective manner.

In metallurgy the definition of quenching is 'the controlled extraction of heat'. The most important word in this definition is 'controlled'. The quenchant is any medium that extracts heat from the part. The quenchant can be a liquid, solid, or gas.

Mechanism of quenching

When a hot component comes in contact with a liquid quenchant, there are normally three stages of quenching. There are exceptions to this, which will be explained as we cover each stage. The three stages of quenching are:

(i) vapour stage (stage A or vapour blanket stage)

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(ii) boiling stage (stage B or nucleate boiling stage)
(iii) convection stage (stage C).

The vapour stage is encountered when the hot surface of the heated component first comes in contact with the liquid quenchant. The component becomes surrounded with a blanket of vapour.

In this stage, heat transfer is very slow and occurs primarily by radiation through the vapour blanket. Some conduction also occurs through the vapour phase. This blanket is very stable and its removal can only be enhanced by agitation or speed improving additives. This stage is responsible for many of the surface soft spots encountered in quenching. High pressure sprays and strong agitation eliminate this stage. If it is allowed to persist undesirable microconstituents can form.

The second stage encountered in quenching is the boiling stage. This is where the vapour stage starts to collapse and the liquid in contact with the component surface erupts into boiling bubbles. This is the fastest stage of quenching. The high heat extraction rates are due to the vapour bubbles carrying away heat from the hot surface and transferring it further into the liquid quenchant allowing cool liquid to replace it at the surface. In many quenchants, additives have been added to enhance the maximum cooling rates obtained by a given fluid. The boiling stage stops when the temperature of the component's surface reaches a temperature below the boiling point of the liquid. For many distortion prone components, high boiling temperature oils or liquid salts are used if the media is fast enough to harden the steel, but both of these quenchants see relatively little use in induction hardening.

The final stage of quenching is the convection stage. This occurs when the component has reached a point below that of the quenchant's boiling temperature. Heat is removed by convection and is controlled by the quenchant's specific heat and thermal conductivity, and the temperature differential between the component's temperature and that of the quenchant. The convection stage is usually the slowest of the three stages. Typically, it is this stage where most distortion occurs. An example showing the three stages of quenching is shown in Fig. 1.

Obtaining the required properties and low distortion is usually a balancing act. Often, optimal properties are obtained at the expense of high residual stresses or high distortion. Low distortion or residual stresses are usually obtained by sacrificing properties. Therefore, the optimum quench rate is one where properties are just met. This usually provides the minimum distortion.
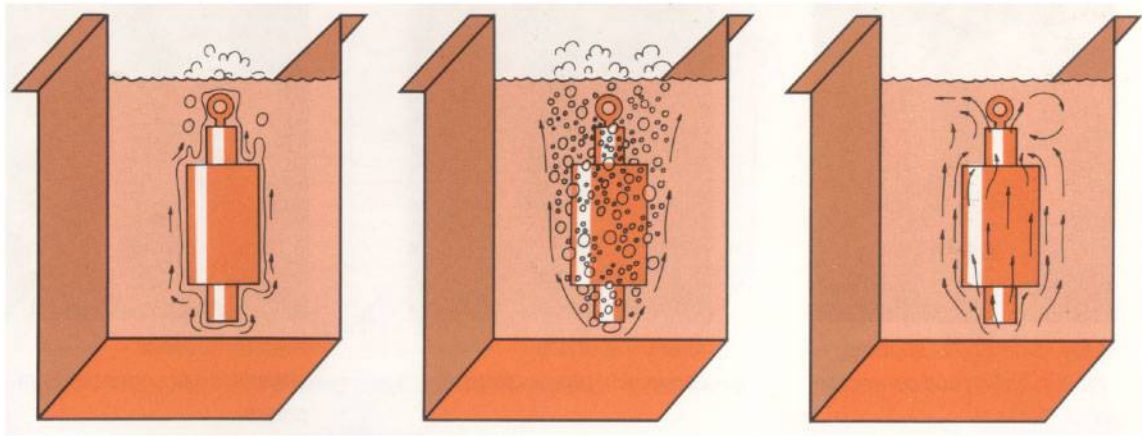
Hardenability

To achieve proper strength and toughness, it is necessary to convert austenite to martensite, which is then tempered to form the proper tempered martensite microstructure. To achieve this conversion of austenite to martensite, a rapid quench rate is required. This quench rate must be fast enough to avoid the formation of upper transformation products like bainite and pearlite, and convert all austenite to martensite. This critical quench rate just misses the 'knee' of the time–temperature–transformation (TTT) curve. The rate of the critical quench rate is dependent on the steel chemistry. A typical TTT curve is shown in Fig. 2.

In practice, when a steel component is quenched, the surface cools much more rapidly than the centre. This means that the surface could cool at the critical cooling rate and be fully hardened, but the centre cools more slowly and forms a soft pearlitic or bainitic microstructure.

Hardenability is the ability of steel to through harden. It is not the ability of the steel to get hard. In a sense, it is a measure of the critical cooling rate on the TTT curve. Increasing the hardenability decreases the critical cooling rate necessary to fully transform austenite to martensite.

1 Schematic illustration of three phases of quenching in liquid

Increasing the hardenability of steel is accomplished by increasing the alloying content of the steel. Carbon, manganese, chromium and molybdenum are all effective alloying elements that increase the hardenability of the steel. These alloying elements cause a delay in transformation, by shifting the transformation curve to the right. This reduces the critical cooling rate for martensitic transformation. However, alloying elements may be expensive and not always beneficial to other processes, such as machining, forging, etc.

Increasing the alloying content is not a simple panacea. Increasing the carbon content and alloying content can also have deleterious effects, by lowering the martensite-start transformation temperature (M_s). Increasing the carbon content, while shifting the TTT curve to the right, significantly lowers the M_s temperature (Table 1). Alloying elements also increase the 'effective carbon' content according to the formula[1]

$$C_{eq} = C + \frac{Mn}{14} + \frac{Mo}{10} + \frac{Cr}{10} + \frac{Ni}{30} + \frac{V}{6} + 3N + 20B$$

Cracking and distortion increase as the 'effective carbon' content increases. Alloys become prone to distortion and cracking if the 'effective carbon' exceeds 0·52%. This tendency is decreased by the proper application of quenchants. A fast enough quenchant is used that will achieve the desired properties, but slow enough that cracking or excessive distortion will not occur.

Other than increasing the hardenability of the material, the lack of through hardening can be overcome by increasing the quenching speed so that the cooling rate at the centre of the part exceeds the critical cooling rate. This can be achieved by changing from a high speed accelerated quenching oil or, if using a polymer quenchant, by reducing the concentration of the quenchant solution.

The shape or geometry of the part can also influence the hardness obtained (as well as the distortion of a part). This is illustrated in Table 2.

In this table, the influence of geometry on the part configuration is shown. By taking the characteristic thickness *T*, the equivalent round size can be determined. This table can also be used to determine proper soaking times at temperature.

To summarise, the steel composition, component section thickness, and the type of quenchant all have a major influence on the properties obtained in the heat treated condition.

Oil quenchants

It is not known how long oils have been used for the hardening of ferrous alloys. Many types of oils have been used including vegetable, fish and animal oils, and particularly sperm whale oil have been used for quenching operations. The first petroleum based
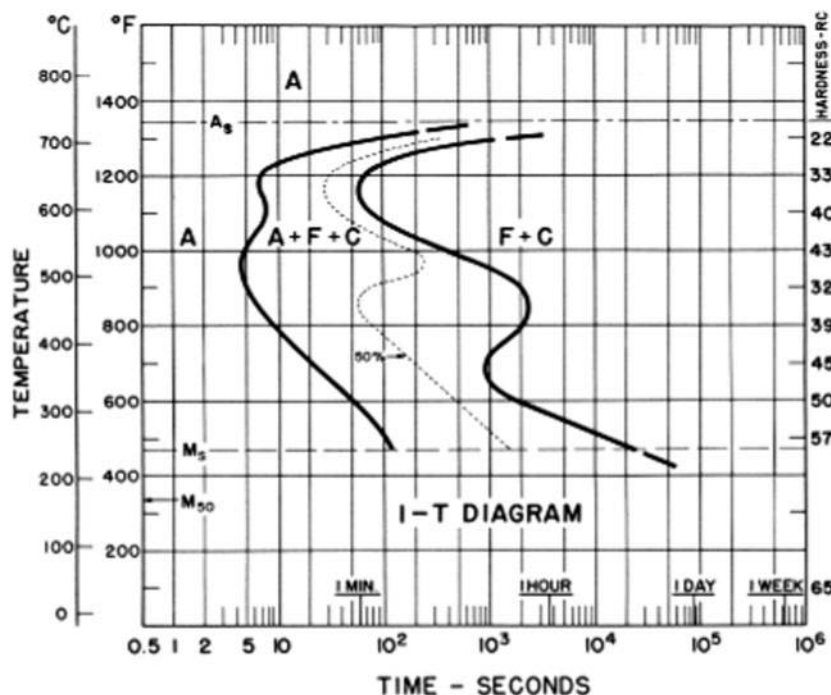
2 Typical TTT curve for AISI 5160 steel

Table 1 Martensite start transformation temperature (M_s) as function of carbon content

Carbon content/%	M_s temperature/°C
0·2	430
0·4	360
1·0	250

quenching oils were developed around 1880 by E.F. Houghton in Philadelphia. Since that time, many advances have been made in the development of quenching oils to provide highly specialised products for specific applications.

A wide range of quenching characteristics can be obtained through careful formulation and blending. High quality quenching oils are formulated from refined base stocks with high thermal stability. Selected wetting agents and accelerators are added to achieve specific quenching characteristics. The addition of complex anti-oxidant packages is included to maintain performance for long periods of continued use – particularly at elevated temperatures. Emulsifiers may be added to enable easy cleaning after quenching.

Petroleum-based quench oils can be divided into several categories depending on the operational requirements. These requirements include quenching speed, operating temperatures and ease of removal.

The quenching speed is important because it influences the hardness and the depth of hardening. This is probably the most common method of classifying quench oils. Quench oils can be classified as normal, medium or high speed quench oils.

Normal speed quench oils have relatively low rates of heat extraction, and are used in applications where the material being quenched has a high hardenability. Highly alloyed steels such as AISI 4340 or tool steels are typical examples of steels quenched in normal speed oils.

Medium speed quench oils provide intermediate quenching characteristics and are widely used for medium to high hardenability applications where dependable, consistent metallurgical properties are required.

High speed quench oils are used for applications such as low hardenability alloys, carburised and carbonitrided components, or large cross-sections of medium hardenability steels where high rates of cooling are required to ensure maximum mechanical properties.

Mar-quenching oils are a special case where the part is quenched into a quenchant at elevated temperature, typically 100–200°C. The workpiece is held in the quenchant until temperature equilibrium is established throughout the section, and then air-cooled to ambient temperature.

During mar-quenching, components are quenched to an intermediate temperature close to the M_s temperature, and held at this temperature. This eliminates the temperature gradients across the surface, and consequently, during subsequent slow cooling after removal from the hot oil, transformation to martensite occurs uniformly throughout the section. This minimises the generation of internal stresses and reduces distortion.

Since mar-quenching oils are used at relatively high temperatures, their formulation and physical properties are different from cold quenching oils. They are formulated from very carefully selected base stocks with high oxidation resistance and thermal stability. They have high flash points and viscosities, and contain complex anti-oxidant packages to provide long life. Selection of the mar-quenching oil is based on the operating temperature and quenching characteristics. A minimum of 50°C should be maintained between the operating temperature of the oil and its flash point.

The most widely used quenchant for ferrous alloys are petroleum based quenchants due to their favourable heat extraction characteristics. The dependence upon imported oil, price vulnerability, and contamination potential has caused suppliers to investigate alternative sources of quench oil base stock.

There are many reasons why vegetable based oils have seen increased interest. The primary reasons are:

(i) increased environmental regulation on ground and water contamination
(ii) need to reduce the reliance on imported petroleum crude oils
(iii) increased environmental awareness.

Canola oil, with its high oleic fat content has been found to offer the best oxidation resistance of any of the common vegetable oils (soybean, sunflower seed, peanut and palm). Canola is an oil seed crop that is grown primarily in Western Canada. Each plant produces a yellow flower, which in turn produces a seedpod. These seedpods are similar to a peapod, but are approximately one-fifth the size of a peapod. Within the pods, tiny round seeds are contained. It is these seeds that are crushed to form canola oil. These seeds are 40% oil. The seed solids are filtered from the oil, and are processed to form a high protein livestock feed.

While the environmental performance of canola based quench oils is important, it still must perform as a quenchant. Cooling curves of canola based quenchants show that the cooling behaviour is equivalent and in some cases superior to mineral oil based quenchants (Fig. 3). In cooling curve tests, the canola based quench oil showed the following characteristics when compared to a mineral oil based quenchant:

(i) nearly non-existent vapour phase
(ii) faster quench rates in the 704–593°C (1300–1100°F) temperature range, where high quench rates are necessary to achieve properties

Table 2   Effect of shape on equivalent round for quenching

Shape	Equivalent round (ER)
Solids, of length *L*	
Round	ER=1*T*
Hexagon	ER=1·1*T*
Square	ER=1·25*T*
Rectangular or plate	ER=1·5*T*
Tube (any section)	
Open both ends	ER=2*T*
	When *L* is less than *D*, consider as a plate of *T* thickness.
	When *L* is less than *T*, consider section a plate of *L* thickness
Restricted or closed at one or both ends	ER=2·5T (when *D*<62 mm)
	ER=3·5T (when *D*>62 mm)
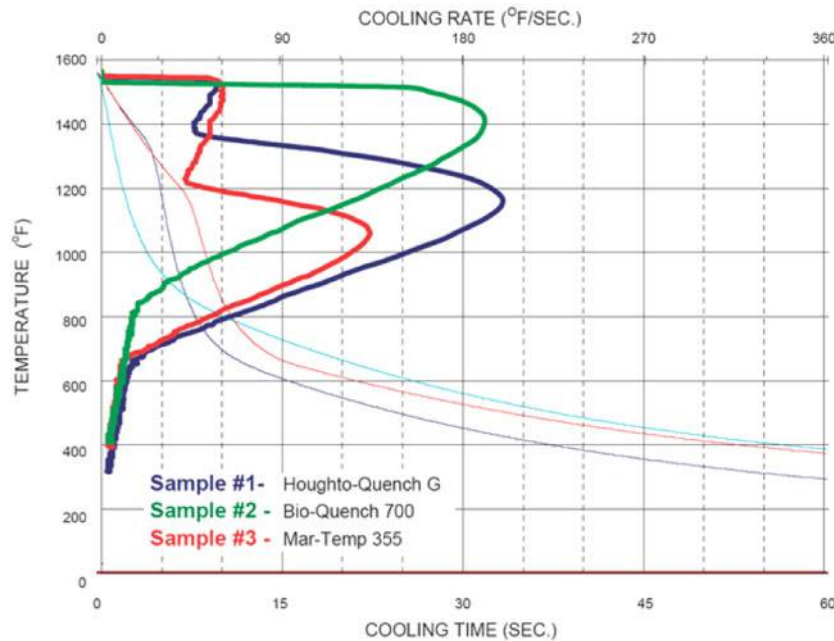
3 Comparison of cooling curve of canola based quench oil with medium speed and martempering oil

(iii) slower quench rates at low 482–121°C (900–250°F) where low rates are desirable to minimise distortion.

The high boiling temperature of the canola oil, which is almost 166°C (300°F) above the boiling temperature of most petroleum quench oils and the higher flash point of 332°C (630°F) for the canola oil versus the petroleum based quench oil flash temperature of 177–232°C (350–450°F) are both beneficial physical characteristics for a quench oil. The higher flash point gives greater safety during quenching, while the higher boiling temperature provides for an increased transition temperature between nucleate boiling and convection. This temperature is very important in reducing the temperature gradients that result in residual stresses during transformation. The virtual absence of a vapour phase contributes to a uniform heat extraction and uniform heat transfer across the surface of the part.

Methods of quench oil selection

As was discussed previously, the selection of quench oil is dependent on the part, type of furnace, and cost.

The part requirements are critical. The alloy must be considered to achieve the proper hardness and required mechanical properties. Geometry and section size is influenced by the hardenability of the alloy. This limits the section size for through hardening. The geometry of the part also influences the residual stress state and distortion. Non-uniform section sizes, sharp radii, and complex shapes can contribute to distortion and potentially part cracking. As well as the method of fixturing, the oil must be chosen to minimise the potential of distortion and quench cracking.

The furnace also influences the choice of quenchant. If the agitation in the quench tank is poor, then fast oil must be chosen. If the part is prone to distortion, then the furnace and quench tank must be capable of heating the oil to the proper temperature for mar-tempering.

If the part is press-quenched, then the oil must be chosen that is tolerant of the presence of the inevitable infiltration of hydraulic fluid. It also should provide some lubricity to prevent excessive wear to the quench die fixtures. It also should have an excellent anti-oxidant package to satisfy provide repeatable quenching of high surface areas with limited quenchant volumes.

The cost of the quenchant is also important. Unfortunately, this is often the only variable considered. The initial cost, as well as in-use costs together with the final end-of-life costs of disposal and environmental cost must be considered.

The initial costs are influenced by the quality of the base oil stock chosen and the quality and robustness of the additive package. The additive package consists of speed improvers and anti-oxidants. The quality of the anti-oxidant package also influences the in-use cost.

The in-use cost is influenced by the maintenance costs associated with the quench oil. The cost of testing to ensure repeatable performance must be considered. Other maintenance costs such as filtering should be included in the calculation. The cost of additive packages (usually not required by quality quench oils) added to the quench tank as the quench oil ages is not usually added to the cost calculations because this usually falls under the maintenance budget. However, it should be added to understand the total cost of the quench oil selected. One additional factor not usually included is the cost of cleaning the parts. Finally, the oxidative stability of the oil should be considered. If the oil tends to oxidise rapidly, this will increase the cost of cleaning, and require more frequent replacement of the oil.

In general, there are three different situations when quench oil is selected:
(i) existing operation wanting a different quench oil because the life of the existing oil has been depleted; dissatisfaction with current supplier serviced; or cost of existing quench oil
(ii) new operation similar to existing process, such as the addition of a new line for increased capacity, or a new facility at a different location processing similar parts
(iii) new operation or new process with new or different configuration parts, or parts of a different alloy.

This is accomplished by one of three methods: comparative cooling curves; Grossman H-value or by the hardening power of the quenchant. In each case, not only must the quenching characteristics be considered, but the thermal stability of the oil should also be considered.
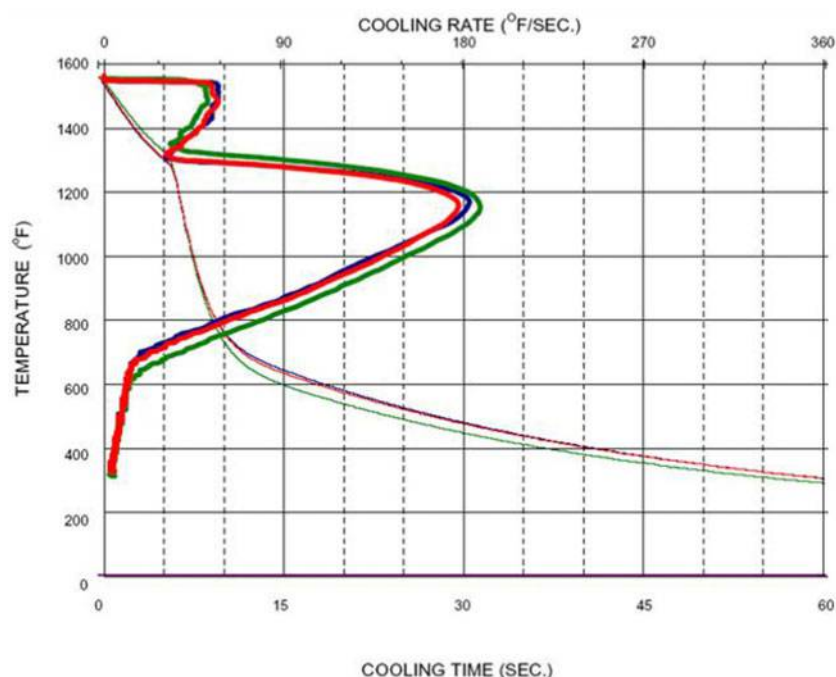
Comparative cooling curves

When an existing process requires new oil, the new quenchant must be metallurgically equivalent. This means that the cooling curves or the quenching path should be similar. The cooling curve is measured by several different methods described in Table 3. The reference fluids used in the various methods are described in Table 4.

When testing oil for comparison purposes, only new oils should be used. This is because old oil will show oxidation and appear faster than the new oil. Further, the oil must be tested by the same method, and preferably at the same time by the same testing laboratory to eliminate interlaboratory testing variation.

As a general rule, oils are considered to be metallurgically equivalent if the maximum cooling rate is within $\pm 14^\circ C\ s^{-1}$; the temperature at the maximum cooling rate is within $\pm 14^\circ C$ and viscosities and flash point are similar. This is illustrated in Fig. 4. As a general rule, if the oils meet these criteria, similar metallurgical results will be obtained.

However, similar physical properties and quenching characteristics will not mean that the oil will perform over time in a similar fashion. The thermal stability of the oil, measured by its oxidative stability must also be considered.

4 Comparison of two oils for similarity to baseline oil (red) for quenching characteristics

Hardening power

Alternatively, quenchants can be compared using cooling curve analysis, by using the hardening power of the quenchant.[3] In this method, three characteristic points of the cooling curve, measured by the method described in ISO 9950, are used in a formula derived by regression analysis to describe the hardening power of oil quenchants. This hardening power, for unalloyed steels, HP, is expressed as a single value as

$$HP = 91.5 + 1.34T_{VP} + 10.88CR - 3.85T_{CP}$$

where T_{VP} is the transition temperature between the vapour phase and the boiling phase (in $^\circ$C); CR is the cooling rate at 550°C; and T_{CP} is the transition temperature between the boiling phase and the convection phase.

This method allows oils to be selected by comparing the relative hardening power of each of the oils. However, for new installations, it is necessary to know the cooling rate required to achieve part properties.

This method was further developed for the hardening of carburising steels in hot oils.[4] Using the methodology described in ISO 9950, it was determined that only two critical points from the time–temperature and cooling rate curves had any significance for the measured hardness. The addition of the hardness of the Jominy end quench

Table 3 Comparison of cooling curve testing methods (after Totten *et al.*[2])

Variable	Method				
	ISO 9950	AFNOR NFT-60778	JIS K2242	Z8 E 45003	ASTM D6200
Country	International	France	Japan	China	USA
Probe alloy	Inconel 600	Silver 99·999% pure	Silver 99·999% pure	Silver 99·999% pure	Inconel 600
Probe dimensions	12·5 × 60	16 × 48	10 × 30	10 × 30	12·5 × 60
Reference fluid	Table 4	Table 4	Dioctyl phthalate	Dioctyl phthalate	Table 4
Vessel dimensions/mm	115±5 dia.	138 dia. × 99 high	300 ml beaker	300 mL beaker	115±5 dia.
Oil volume/mL	2000	800	250	250	2000
Oil temperature/°C	40±2	50±2	80, 120, 160	80±2	40±2
Probe temperature/°C	850±5	800±5	810±5	810±5	850±5

Table 4 Cooling characteristics of reference fluid specified in ISO 9950, AFNOR NFT-60778 and ASTM D6200[2]

Physical characteristics	Institute of petroleum test method	Minimum value	Maximum value
Kinematic viscosity at 40°C/cSt	IP71	19	23
Kinematic viscosity at 100°C/cSt	IP71	3·9	4·4
Kinematic viscosity index	IP226	95	105
Density at 15°C/kg	IP160	0·855	0·870
Flash point/°C	IP24	190	210
5% distillation/°C	IP123	330	360
50% distillation/°C	IP123	400	420
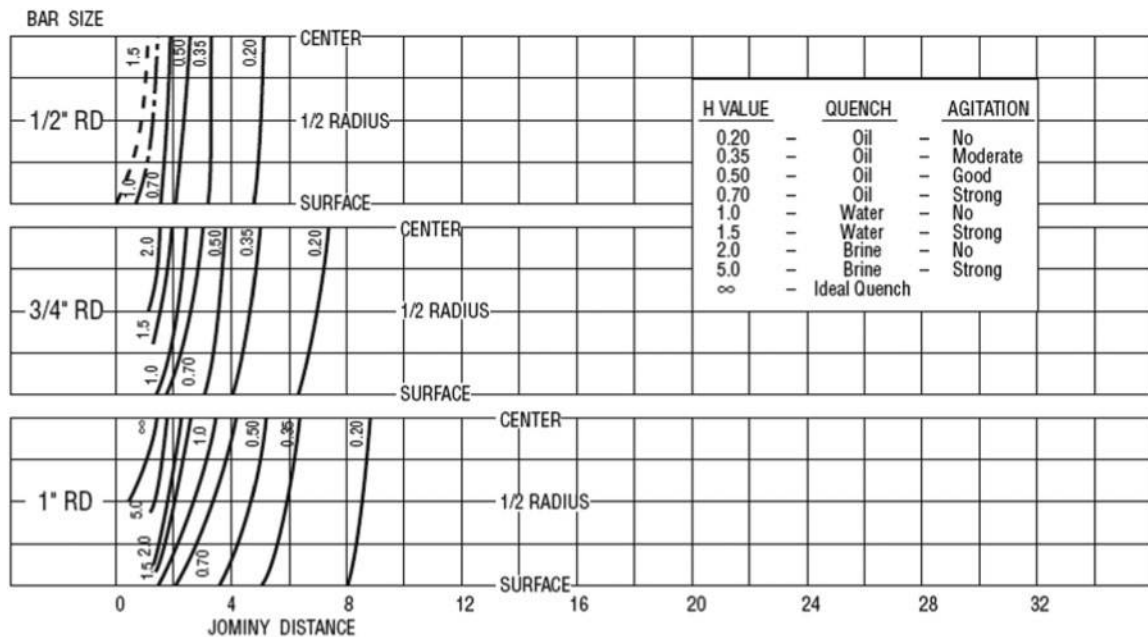
5 Chart for predicting the approximate cross-section hardness of quench round bars using Jominy test results[11]

test at 6·5 mm increased the accuracy of the hardness prediction. In this experiment, the core hardness when mar-tempering yielded the following equation

$$HV30 = 135 + 0.56 CR_{500} - T_{20} + 5HRC_{J=6.5}$$

where CR_{500} is the cooling rate at 500°C; T_{20} is the temperature (°C) after 20 s; and $HRC_{J=6·5}$ is the hardness at a Jominy distance 6·5 mm from the quenched end. It was shown that this method accurately predicted the core hardness.

It is important to realise that different steels have different cooling requirements, and that the hardening power equation was based on unalloyed carbon steels. Different alloys will require different regression constants.[5] However, this method has been shown to be effective in determining the relative ranking of oils for quenching. However, the hardening power equation should be used with caution when comparing different steels, or for general hardness predictions. Alternative equations for making hardness predictions and ranking of oil quenchants have been made by Chen and Zhou.[6]

Grossman H-value
The classic method related to the ability of a quenchant to harden steel is to determine the Grossman H-value.[7,8] The H-value is defined as[9]

$$H = \frac{\alpha}{2\lambda}$$

where α is the average heat transfer coefficient at the surface of the part and λ is the thermal conductivity of the steel. For most steels, the thermal conductivity does not change appreciably over temperature or from alloy grade to grade, so it is directly approximate to the average heat transfer of the quenchant.

To achieve equivalence to the data presented in Jominy end quench testing, the H-value only refers to the quench rate at the very specific temperature of 705°C (1300°F). To ensure that the proper cooling rate and microstructure is achieved, it is necessary to refer to continuous cooling diagrams (CCT) for the steel of interest.

The Grossman H-value is determined experimentally by quenching a series of round bars. After quenching, the bars are sectioned, polished and etched. The 50% martensite region is determined. This is readily achieved because the transition in etching between dark and light etching corresponds to 50% martensite. The location of the transition, through the use of a series of charts and calculations, determines the Grossman H-value. This is tabulated in Table 5.

This method makes possible the calculation of the critical size in terms of a standardised quench and calculation of the critical size from a single test. Using charts,[10] it is possible to predict how a known steel with a specific Jominy curve would behave, and predict the hardness distribution within the bar. Using the concept of equivalent rounds (Table 2), the

hardness distribution can be predicted. An example of charts to predict hardness distribution based on hardness correlation to round bars is shown in Fig. 5.

Although, this method has been used in the industry for many years, it is not without problems. The biggest issue with the application of the Grossmann H-value, is the difficulty in quantifying agitation rates. There is really no understanding of what is meant by 'mild' or 'violent' agitation. Further, the different methods of quenching such as spray quenching have no equivalent in the Grossman H-value. This method is only focused on the ability of the quenchant to harden steel, and gives no indication regarding distortion.

A method, proposed by Guisbert[12] offers an excellent method of precisely determining the H-value of a quench system (the quench oil and agitation system) to achieve repeatable results. In this method, small pins approximately 19 mm in diameter of AISI 1045 steel are placed anywhere within a tray or basket to evaluate quenching performance. This method is a modification of the method recommended by the American Gear Manufacturer's Association (AGMA).[13]

Since the pin diameter is fixed, the Grossman data and the Jominy hardness position can be plotted together (Fig. 6 left). When the core hardness of the test pin is determined, the actual Jominy hardenability data can be used to find the equivalent Jominy hardness position. The intersection of the Jominy hardness position with the
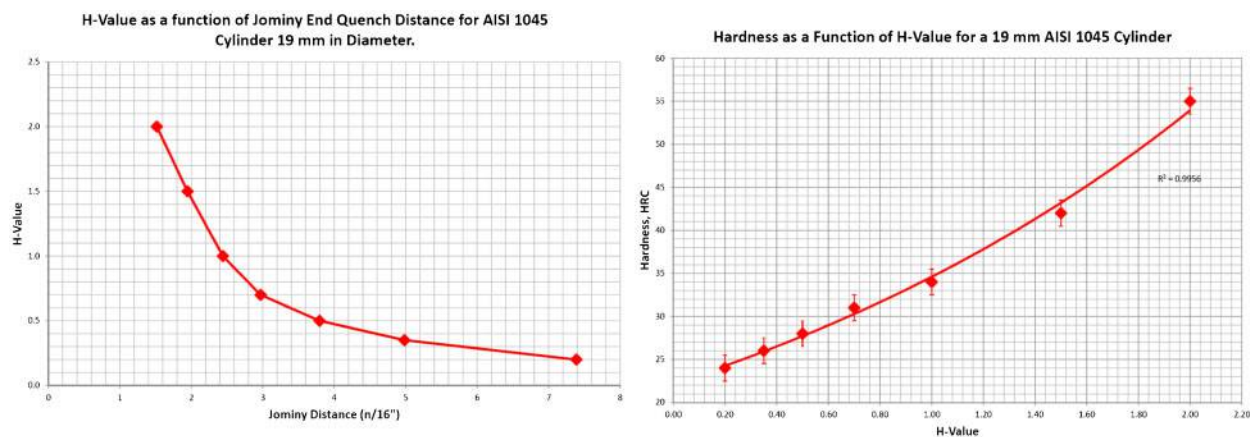
6 Correlation of hardness as function of Jominy distance (left) and hardness as function of H-value for 19 mm diameter AISI 1045 cylinder (right)

Table 5 Severity of quench (H) for various quenching media

Agitation	Air	Oil	Water	Brine
No circulation	0·02	0·25–0·30	0·90–1·0	2
Mild circulation	…	0·30–0·35	1·0–1·1	2–2·2
Moderate circulation	…	0·35–0·40	1·2–1·3	…
Good agitation	…	0·40–0·50	1·4–1·5	…
Strong agitation	0·05	0·50–0·80	1·6–2·0	…
Violent agitation	…	0·8–1·10	4	5

test pin size determines the H-value (Fig. 6 right).

This alternative method is useful for determining the H-value for specific quenchants, and would allow more precise determination of hardness in a particular quench system (oil and agitation). It would also allow two different quench systems to be compared. This method can also be used to determine the quench uniformity of a quench system by placing pins at eight corners of a quench tank, and one located at the centre of the quench tank (assuming a rectangular quench tank). Systematic monitoring of the quench system could also be achieved in this manner.

From an oil selection standpoint, the H-value could be determined using no agitation, in a similar fashion as the cooling curve, and the H-value determined. Multiple quench oils could be quenched, and rated according to the H-value.

Conclusions

There is not one perfect method to the selection of quench oils. It often involves experience, and an understanding of the entire heat treatment process. This requires a deep knowledge of the alloys used; the part geometry; and the entire quench system. Each of the methods presented have merit in the selection of quenchants used in the heat treatment

facility. However, these methods only consider the mechanical properties (hardness) and do not consider the important considerations of distortion. Further, the initial cost, operating cost and disposal cost of specific quench oils have not been considered. It is incumbent on the supplier and the customer to work together as a team to achieve optimal quenching.

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