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S. R. Elmi Hosseini,[1] A. Zabett,[2] and Zhuguo Li[3]

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Reference

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ABSTRACT

Oil is one of the most common quenchants used in the heat treating industry. Since the cooling properties of a quench oil varies with the degree of aging, it is important that quenching cooling rates be monitored with respect to use time. In addition, there is insufficient general information about various oil quenchants in general and cooling rates in particular. This problem is compounded by complaints from heat treating shops that quenching oils of nominally the same type, and which are alleged to contain the same additive packages, exhibit very different quenching performance in practice. Therefore, at the request of the heat treating industry in Iran, measuring system was designed and built to determine the cooling rate of quenching oils. This system includes of a tubular furnace, oil tank, probe for measuring the temperature, a system for probe transfer from the furnace to the quenchant, and a data acquisition system. This new system is capable of measuring the cooling rate of quenching oils according to various national and international standards. In the study reported here, two types of oils were comparatively evaluated: Behran 145 Oil (New Oil) and Used Behran 145 Oil. The cooling rate of these quenching oils was measured, and the effect of their cooling rate differences on the hardness and the microstructure of a low carbon steel was determined.

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[1] Ph.D. student, School of Materials Science and Engineering, Shanghai Jiao Tong Univ., China.

[2] Associate Professor, Faculty of Engineering, Department of Materials Science and Engineering, Ferdowsi Univ. of Mashhad, Mashhad 9177948974, Iran.

[3] Professor, School of Materials Science and Engineering, Shanghai Jiao Tong Univ., China.

Keywords

quench oil, cooling curve, steel, hardness, microstructure, probe

Introduction

QUENCHANTS

Many carbon and low-alloy steels must be quenched in order to achieve the desired properties such as hardness and strength. The most common quenchants used for this purpose are typically vaporizable quenchants including water, caustic or brine solutions, aqueous polymer solutions, and petroleum oil [1]. Water, caustic, and brine solutions are most commonly used for carbon steels; however, it is often necessary to use quenchants with lower quench severity to avoid cracking and to provide the necessary distortion control. Typically, these quenchants include aqueous polymer solutions [2–4] and petroleum oils [1,5,6]. Most petroleum oil quenchants are classified as: martempering oils (hot-oils) [6–8], conventional (slow) or accelerated (fast) oils [5,9–11]. Of these quenchants, petroleum oil-based quenchants have been and continue to be the most commonly encountered quenchants in the heat treatment industry [5]. For this reason, a petroleum oil quenchant was the subject of the work discussed here.

Petroleum oils possess widely varying compositions and the amount and distribution of these components are determinant factors on wettability [12,13] thermal conductivity [13] and viscosity [12,14]; all of which are important parameters determining quenching performance [15]. However, of these, fluid viscosity is the physical property with greatest effect on heat transfer during quenching [12,13,16,17]. Asada and Fukuhara [14] reported that while cooling rates were dependent on the composition of the oil, they were especially affected by the average molecular weight and viscosity of the oil. Yokota, et al. [18] studied the effect of the boiling ranges of petroleum oil basestocks used in quench oil formulation on the hardness of a 0.45 % C carbon steel. Their results showed that hardness was dependent on cooling rates in the 300°C –350°C temperature range and that the cooling rates were closely related to boiling ranges of the petroleum oil base stocks used.

The components of a petroleum oil also impact properties such as staining [19], varnish formation but especially thermal-oxidative stability [20]. Thermal-oxidation alters the chemical composition of the oil leading changes in wettability and especially viscosity of the oil and therefore quenches severity [20–22].

Although unadditized refined petroleum can be used for quenching processes, it is more common for quenching oils to contain additives to provide the desired properties with substantially extended service life [15,23]. Generally, quenching oils are formulated with ashless-type additive packages that may accelerate cooling properties [11], enhance wettability [24], reduce potential for staining [19] and antioxidants for significantly improving thermal-oxidative stability [22,24,25]. Improved thermal-oxidative stability provides longer usage times before the quench oil must be replaced resulting in use-cost savings in addition to corresponding quality improvements due to more stable and consistent quenching performance.

QUENCHANT PHYSICAL PROPERTY AND CHEMICAL ANALYSIS

Quench bath maintenance is an essential part of the overall quality control of a commercial quenching process [21,26]. One important part of quality control process is chemical analysis. Routine tests that are typically recommended summarized in Refs. [6,10] and ASTM D6710-02 [27] and include: density, flash point and fire point, acid number, water content, carbon residue, infra-red spectroscopy, ash content, and viscosity. The experimental strategies to aid in test selection and data interpretation are discussed in detail in Refs. [6,10,21] and will not be discussed further here. Although important, they do not provide direct information on the expected impact on the quenching process. Therefore, the heat treating community typically insists that cooling time-temperature profiles (cooling curve analysis) be run at appropriate intervals on their various quench systems [21,28,29]. The remainder of this discussion will focus on cooling curve analysis.

COOLING CURVE ANALYSIS

The cooling curve is a depiction of the various cooling mechanisms involved upon immersion of austenitized steel (typically at ~850°C) into a vaporizable quenchant. **Figure 1** provides a schematic illustration of the different boiling transitions, film-boiling (or "vapor blanket" cooling) to nucleate boiling, and nucleate boiling to convective cooling. The temperatures and times where these occur as well as cooling rates, especially neat the martensite start (Ms) temperature of the steel are very important characteristic points which define the ability of a quenchant to harden steel [10].

When steel at its austenitizing temperature is immersed into a vaporizable quenchant, at least three heat transfer mechanisms occur; each with very different heat transfer rates [9,10]:

Full-Film Nucleate Boiling (Vapor Blanket Stage)

In this stage (also designated as A-Stage in older heat treating literature), as shown in **Fig. 1**, a stable vapor layer is formed around the steel. This will occur when the

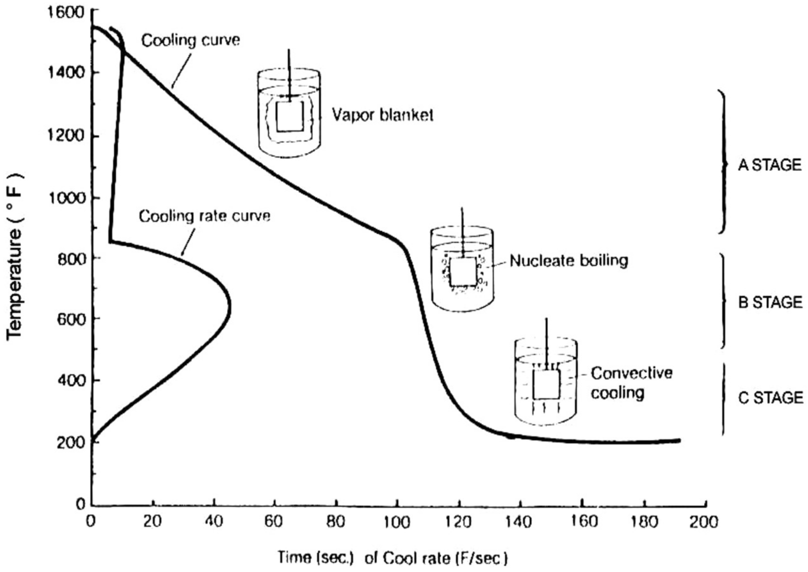


FIG. 1 Schematic illustration of cooling time-temperature curve and cooling rate curve [30].

heat per surface area is greater than that required for vapor formation. The cooling rate is very slow in this stage because the vapor layer acts as an insulator. Heat transfer occurs by radiation [30].

Nucleate Boiling Stage

This characteristic cooling stage (also known as B-stage cooling) is shown in **Fig. 1** and typically exhibits the greatest heat transfer rates of the overall quench cooling process. Nucleate boiling initiates when the surface temperature decreases to the point where the vapor layer becomes unstable and bubble formation occurs and boiling begins. These bubbles facilitate rapid cooling of samples by absorbing the heat from the steel surface. Time duration and cooling rate at this stage are controlled by different parameters such as boiling point of liquid as well as the size and the shape of vapor bubbles [30].

Convection Cooling Stage

Convective cooling (also referred to as C-stage cooling) is shown in **Fig. 1** and begins when the interfacial temperature is less than the boiling point of the quenchant. The cooling rate is typically very slow in this region as well [30].

From this discussion, it is apparent that anything that affects the overall time–temperature cooling profile is likely to affect the quench hardening capabilities of the quenchant. Cooling curves depict what would be expected to occur when quenching a load of steel components in a production process. This is the reason why heat treaters rely heavily on the availability of such data in support of their production quality control process.

PROBES

Cooling curve analysis is not new. It has been practiced since Le Chatelier's [31] first publication of the method in 1904 where he used a 18 mm diamater by 18 mm cylindrical iron bar probe with a thermocouple inserted to the geometric center to classify the quench severity of common quenchants in use at the time. A mirror galvanometer was used for data acquisition.

A 1.0 in. diameter by 4 in. cylindrical austenitic iron-nickel alloy Fe–Ni (32 % Ni) probe with a thermocouple inserted to the geometric center was used by Scott [32]. The thermocouple wires were passed through a pipe, also 1.0 in. diameter, which was threaded on to the probe body to prevent quenchant ingression and thermocouple contact. This Fe–Ni probe material was selected because its thermal-physical properties were approximately equivalent to the high-carbon, deep-hardening steels of interest. Furthermore, this austenitic steel did not exhibit the thermal transformation behavior typically exhibited by carbon steels. The thermocouples were connected to a portable galvanometer and the elapsed time was measured with a stopwatch.

French subsequently compared the use of spherical, cylindrical, and plate probes for quenchant characterization. For this work, the thermocouple was inserted to the geometric center and surface cooling curves were calculated from the centreline time–temperature data and a string galvanometer was used for data acquisition [33]. Presumably as a result of this work, French's subsequent publications on quenchant characterization by cooling curve analysis utilized cylindrical probes of carbon or alloy steels of interest.

Tamura developed a cylindrical silver probe with a near-surface thermocouple for quenchant characterization [34]. This probe and assembly became the basis of a Japanese national standard JIS K2242 [35]. Tamura et al. [34] compared the cooling curves obtained with the JIS K2242 silver probe quenched from 800°C with probes for a series of steel compositions (quenched from 870°C). The parameters used for cooling curve comparison were the transition temperature from full-film boiling to nucleate boiling (Leidenfrost temperature) and for nucleate boiling to convective cooling. In this study, it was reported that the Leidenfrost temperature for the steel probes was approximately 150°C greater than that obtained for the silver probes. In addition, the beginning of the convective cooling stage was somewhat higher for the steel probes than the silver probe, which was reported to be due to the differences in the surface condition and thermal diffusivity of the steel probes and that of silver [35].

Tamura et al. [34] also compared the effect of various quench oil additives on cooling curves obtained with the JIS K2242 silver probe (with a near-surface thermocouple) with the standard 12.5 mm diameter by 60 mm INOCONEL 600 probe with a thermocouple inserted to the geometric center [36]. Although the same cooling behaviours were obtained with both probes, differences in quench oils with varying additive concentrations was more pronounced with the JIS K2242 silver probe, indicating significantly greater sensitivity, which was attributed to greater temperature sensitivity of the thermocouple used in the silver probe [36].

A similar study was performed using Hajdu et al. [37] using stainless steel and copper probes with the same dimensions as the INCONEL 600 probe to evaluate ISOMAX 166, an accelerated petroleum quench oil typically used for hardening carbon and low-alloy steels. The experimental procedure was conducted according to the Wolfson test [37,38]. The copper probe exhibited a longer full-film boiling phase, less distinct transition to nucleate boiling, and a sharper transition to convective cooling. This study showed that the primary parameter that defined the boiling transition was not the surface temperature, but the heat flux which suggests that the limiting factor for heat transfer from the probe to the oil are the thermal properties of the oils [37].

Although Tamura and others reported excellent quenchant characterization results with silver probes, the use of the JIS K2242 probe has not gained global acceptance by the heat treating industry. Reasons for this non-acceptance include: thermal conductivity is considerably different from that of steel, cost and availability of the probes, problems of maintaining a clean surface, and the comparative difficulty in preparing and maintaining delicate surface thermocouple assemblies used for the silver probe [30].

At approximately the same time, there was a collaborative project sponsored by the Wolfson Heat Treatment Centre to develop an international standard for cooling curve analysis [28,38]. This development work for the corresponding international standard was led by the Quenching and Cooling Committee of the International Federation for Heat Treatment (IFHT) chaired by Professor Božidar Liščić. This work focused on the application of the 12.5 mm diameter by 60 mm probe INCONEL 600 probe developed by the Wolfson Heat Treatment Centre [38]. Some of the advantages of INCONEL 600 include: their excellent high-temperature oxidation resistance, stable and predictable heat transfer characteristics, possess thermal conductivity closer to steel than silver, no phase transformation, suitable for use at heat treatment temperatures up to 900°C, and relative ease of manufacture [28]. The INCONEL 600 probe is being used increasingly in the development of national standards for cooling curve quenchant characterization [39,40].

Wang et al. [41] performed cooling curve analysis using 10 different steels and 9 different quenchants. The probes were 50 mm diameter by 100 mm with 3 thermocouples, 50 mm, located at 2, 12.5, and 25 mm below the surface. The results showed that the differences between the cooling time-temperature curves were attributable to the thermal–physical properties of the steel used for probe construction. Kumar performed a simpler comparison of cooling curves obtained with 25 mm diameter by 100 mm cylindrical probes constructed from Ck45 carbon steel and from E19, a low-alloy steel. For this work, the Type K thermocouple was located 50 mm deep and 4 mm from the surface. The results of this study showed that the cooling curves and surface heat flux were dependent on the thermal-physical properties of the grade of steel used for probe construction [42].

Ramesh and Prabhu [43] compared the effect of probes of various cross-section sizes and materials (type 304 stainless steel, INCONEL 600, nickel and silver) on heat transfer behaviour. From this study, a simple quantitative model correlating the effect of material, cross-section size, cooling rate, and quench severity was reported.

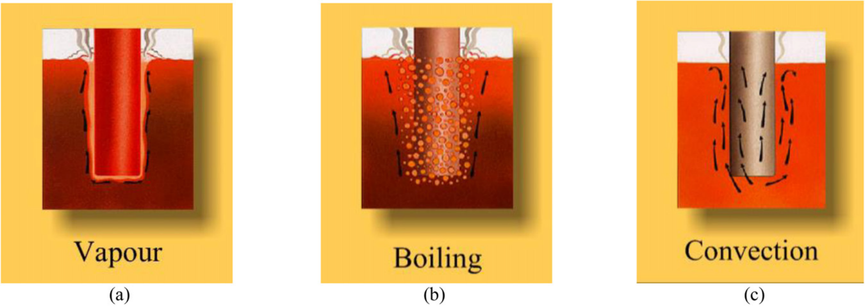
Currently, there is a wide range of commercial equipment available for performing cooling curve analysis. This equipment may be “stand-alone” with an automated probe transfer mechanism [28] or it may utilize a gravity drop mechanism [33]. Increasingly, portable quench testing units with the probe being transferred manually are being marketed throughout the industry [28,42,44–46]. However, although these are typically excellent designs, they may be prohibitively expensive for many heat treat shops and researchers. Furthermore, this equipment typically utilizes the INCONEL 600 probe, although any probe may conceptually be used. However, in Iran, there is a strong preference due to availability and cost as well as using an austenitic stainless steel probe such as type 304 stainless steel that does not undergo transformation during the quenching process. For these reasons, and also because the literature review performed prior to this study showed that a stainless steel probe is a viable alternative to other probe materials that have been reported previously, this discussion will focus on the use of a type 304 stainless steel probe. The construction and use of quenchant testing equipment for cooling curve analysis to characterize a fresh and a used petroleum quench oil will be described here.

Experimental

PROBE, THERMOCOUPLE AND PROBE TRANSFER MECHANISM

In this study, the material used for probe construction was AISI 304 stainless steel. The final dimensions of the probes used for cooling curve analysis work are shown in **Fig. 2** [47]. A 1.5 mm diameter Type K thermocouple was used. To provide a reliable contact between the thermocouple and the probe, silver nitrate powder is poured into the thermocouple/probe junction and then melted in the furnace. After soldering the thermocouple junction, the thermocouple wire is run through a hollow

FIG. 2 A schematic illustration of: (*a*) probe hole for inserting the thermocouple; (*b*) a schematic illustration of probe assembly (probe, thermocouple and handle) [47]. However, the initial bar diameter of the type 304 stainless steel used to construct the probes used for this work was 11.0 diameter. The Type K thermocouple was inserted to the geometric center as shown.



tube (handle) as shown in **Fig. 2**. The handle is used to prevent the fracturing of the thermocouple contact within the probe during movement [47–49].

The repeatability of the temperature measurement of the thermocouple-probe assembly was determined to be ±5°C. Also, the average error of the temperature measured by the probe assembly and the actual temperature in this system was not greater than ±5°C.

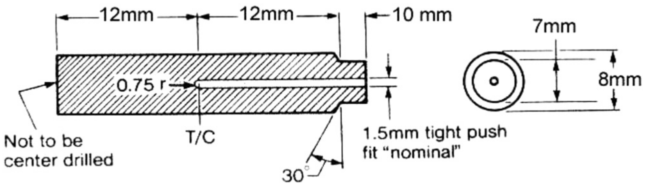
DATA ACQUISITION

When the austenitized steel probe is immersed into the quenchant, the thermocouple, which must remain in contact with the steel probe throughout the entire cooling process, emits an analog signal that is proportional to the temperature being measured at the center of the probe [50]. For personal computers, the data acquisition system utilizes an A/D converter card, which is inserted into one of the vacant slots in the computer chassis. The A/D converter card converts the analog signal to a digital signal, which may then be saved as a digital temperature file along with the corresponding time of measurement. These data may then be used for subsequent computational work and also for data display. Although data rates (data points/s) 10 000 or greater are possible, most cooling curve work with typical steel probes is generally 5–10 Hz (points/s). For this work, a data acquisition rate of 10 Hz was used. The cooling curve data were used without the use of computational smoothing methods.

COOLING CURVE ANALYSIS MEASUREMENT SYSTEM

The cooling curve analysis measurement system is illustrated in **Fig. 3** [51]. In addition to the thermocouple-probe assembly illustrated schematically in **Fig. 2**, the complete system contains two additional sub-assemblies; the furnace and the quench oil tank. The furnace assembly consists of a 3 kW cylindrical furnace with two doors; one at the top and one at the bottom. There is a hole in the top and bottom doors to raise and lower the probe. The probe movement and dropping speed are controlled by a rack and pinion gear drive system. The diameter and height of the furnace is

FIG. 3
Quenchant cooling curve analysis measurement system.



100 mm diameter by 300 mm height. The maximum temperature of the furnace is 1200°C. Also included is a system for closing and opening the top and bottom doors. This apparatus is equipped with a positive displacement pump for agitation of the quenchant although the work reported herein was conducted under unagitated conditions. The quenchant is heated to the desired temperature with an electrical heating element and after the quench, the quenchant is cooled in air.

EXPERIMENTAL QUENCHING PROCEDURE

Prior to conducting any quenching experiments, the first step was to polish the probe surface with emery paper to assure that it was clean and free from deposits and varnish formation. This was done before and after the evaluation of each quench oil.

Although the primary reference oil specified in ISO 9950 [52] is not locally available in Iran, a secondary standard oil was used for periodic probe calibration. When this work was done, over 100 quenching experiments were performed to show that the acquired cooling curves experimentally repeatable. Each cooling curve was essentially super-imposable on another. Thus, before each quench oil was charged to the system, the probe was calibrated by this procedure and the calibration procedure was repeated before the next quench oil to be evaluated was charged to the system.

When evaluating a quench oil, at least two cooling curves were evaluated and compared. If they were not super-imposable, a third cooling curve was obtained. The two super-imposable cooling curves were averaged and these are the average curves were used for subsequent analysis. For quench oil evaluation, it is estimated that the average overall deviation between the cooling curves was not greater than ± 8–10 %. If the difference is greater than this, a third cooling curve is necessary.

The experimental procedure used for this quenching work was essentially that outlined in ASTM D6200 [47]. The steel probe was heated to 850°C and held at this temperature for 15 min, at which point it was quenched into 2000 ml of the oil at room temperature and without agitation. It was shown previously by Guisbert and Moore [53] that the volume of the quench oil was critical in obtaining repeatable results and based on statistical analysis, it was recommended that the volume of quench oil used for this work be maintained within 2000 ± 20 ml. After quenching, the digitized time-temperature data files were then used to prepare plots of the cooling curve time-temperature and temperature-cooling rate profiles of the quench oils. By transmitting the temperature data from the probe to the thermocouple and then to the computer, it is possible to draw the curves of cooling and cooling rate for both oils.

The steel test specimens were prepared similarly. The test specimen was attached to the dropping mechanism of the quench rig shown in **Fig. 3** and then it was heated and quenched in exactly the same manner as the instrumented probe used for cooling curve acquisition. After quenching, the hardness and microstructure of the test specimen were determined for each quench oil.

COOLING CURVE ANALYSIS PROCEDURE

After the cooling curves are acquired, they are typically printed and inspected visually. At this point, the results are quantified. Cooling curve time–temperature and cooling rate data are quantified. There are numerous references describing various methods of cooling curve data quantification to facilitate subsequent comparisons some of which include: calculation of Grossmann quench severity (H-values) [54], quantification of quench severity using Q-values and, if desired, using this approach to predict as-quenched hardness [55], calculation of rewetting times [56], determination of heat transfer coefficients and critical heat flux densities [57,58], Segerberg's hardening power [59,60], and other methods of such as the Ramesh and Prabhu dimensionless cooling performance parameter [61].

While all of these methods have found utility in the heat treatment industry, the traditional cooling curve parameterization methods continue to be used extensively [40,62,63]. Typically, these parameters include [62,63]:

- Cooling temperature and time at which point the transition from full-film boiling to nucleate boiling occurs (Leidenfrost temperature);
- Cooling rate during film boiling;
- Maximum cooling rate and the time and temperature where this occurs;
- Cooling rate at defined temperatures such as 200°C and 300°C;
- Ideally, the heat transfer coefficient and heat flux density will also be calculated

For the work reported here, the cooling curve quantification parameters selected were: maximum cooling rate and the temperature where this occurs and the cooling rate at 300°C. The maximum cooling rate and the temperature where this occurs provides a measure of the ability of the quench oil to harden steel. The cooling rate at 300°C was selected because it is typical of the martensite start (Ms) temperature for many construction steels, and since it is desirable to minimize cooling rate in this region, it provides an assessment of the ability of the quenchant to minimize the potential for cracking.

QUENCH OILS

There were two quench oils evaluated in this study to investigate the effect of cooling rate exhibited by heat treating oils on the hardness and the microstructure of steel. One oil was fresh, as-received, commercial quench oil. The reported physical properties for the Behran 145 quench oil are summarized in **Table 1**. The second oil was "used" Behran 145 quench oil. This oil was reported to be in use in a commercial heat treating shop for 3 years where loads of austenitized steel were quenched

TABLE 1

Physical property characterization of Behran 145 quench oil.

	Standard Test Method				
Quench Oil[a]	Viscosity (cSt @ 100 °C)	Flash Point (°C)	Fire Point (°C)	Pour Point (°C)	Density @ 15.6 °C (kg/m^3)
	ASTM D445	ASTM D92	ASTM D92	ASTM D97	ASTM D1298
Behran 145	4.8	100	190	−6	1.4

[a] The Behran 145 quenching oil was obtained from Sepahan Oil Company; No. 92, Baharan Square, Argentina Square, Tehran, Iran.

approximately 5 times per day throughout this period of time. Unfortunately, no physical or chemical properties were provided for this used oil.

MICROSTRUCTURE AND HARDNESS DETERMINATION

Test specimens, 11 mm diameter by 30 mm height for microstructure and hardness determination were prepared from AISI 1045 (also may be designated as Ck45 or DIN 1.1191) a low-hardenability, carbon steel. The chemical composition of the steel used to prepare these test specimens is shown in **Table 2**. The test specimens were quenched as described in the Experimental Quenching Procedure section.

Microstructural determination was performed using an Olympus-BX60M light microscope.

The Vickers hardness was also determined on these test specimens according to the ASTM E384-99 [64] standard using a load of 294 N load. The equipment was Model MHT.1; No: 8331 made by Matsuzawa Seiki Co Ltd of Japan.

Results and Discussion

COMPARISON BETWEEN THE COOLING RATE OF HEAT TREATING OILS

Figure 4 shows the cooling time–temperature curves for the fresh (unused) and used Behran 145 quench oils. Clearly, the used Behran 145 oil exhibited a longer and more pronounced film-boiling region. The unused Behran 145 oil which was reported to contain no cooling rate accelerators that characteristically destabilize film-boiling, in fact, behaved much more like an accelerated quench oil. A cooling rate comparison of the unused and used Behran 145 quench oils is shown in **Fig. 5**. As is often the case, the cooling rate profiles (which are the first derivative of the corresponding cooling time-temperature curves) are also revealing, showing that the unused Behran 145 oil is substantially faster than the used oil. These observations are quantified by the data shown in **Table 3**.

The cooling profiles shown in **Fig. 4** and **Fig. 5** are consistent with the aging behavior of accelerated petroleum-based quench oils [10,65,66]. The additives used to formulate these oils typically adsorb to the metal surface upon immersion of the hot steel and act to enhance surface rewetting properties during the quench and to

TABLE 2

Chemical composition of the steel used to construct the probes used for this research.

Element	C	Mn	S	P	Fe
wt. %	0.43	0.7	0.04	0.04	Rem.

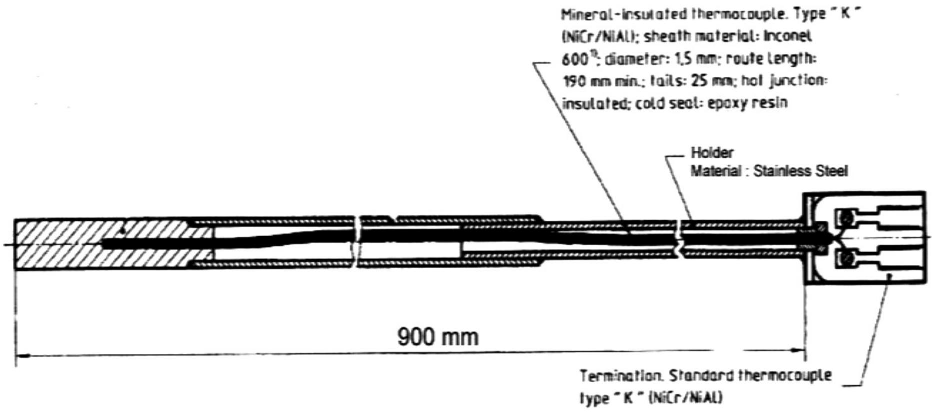


FIG. 4 Difference between the cooling curves of used and unused Behran 145 oils.

provide nucleation sites for bubble formation. Both processes will destabilize film-boiling and accelerate heat removal through nucleate boiling (the hot-vapor bubbles carry heat from the hot metal to the surface and also provide an agitation effect; thus reducing thermal gradients. However, as the steel is removed from the quench tank, the adsorbed additives remain on the surface, thus facilitating additive loss by drag-out. This may occur with antioxidants used to stabilize the oil to thermal-oxidative degradation as well as rate accelerating additives [10,67].

If the base oil has not undergone excessive degradation, it is possible to remove contaminants such as sludge, particulates, and water and to recharge the additive package, including cooling rate accelerators, antioxidants, dispersants, etc., to the

FIG. 5 Difference between the cooling rate of used and unused Behran 145 oils.

TABLE 3

Quantitative cooling parameters for used and unused Behran 145 quenching oil.

Quench Oil	Maximum Cooling Rate(°C/s)	Temperature at the Maximum Cooling Rate(°C)	Cooling Rate at 300°C (°C/s)
Unused Behran 145[a]	138	675	34
Used Behran 145	176	610	30

[a] The Behran 145 quenching oil was obtained from Sepahan Oil Company; No. 92, Baharan Square, Argentina Square, Tehran, Iran.

reconditioned oil, which may produce acceptable quenching properties, even if not identical to those exhibited by the fresh oil [65,66]. This is typically done by service companies who may perform this reconditioning service either on-site or off-site. Cooling curve analysis such as the results shown here are vital to the success of such a reconditioning service.

EFFECT OF THE COOLING PROFILE ON THE MICROSTRUCTURE

The effect of the cooling time-temperature and cooling rate profile of both the unused and used Behran 145 quench oils on microstructure and hardness was compared. The microstructure produced by both oils at the surface and core of the AISI 1045 steel test specimens was determined and the results are compared in **Fig. 6** and **Fig. 7**. A comparison of the microstructures obtained in the core of the test specimens is shown in **Fig. 6(*a*)** for the unused Behran 145 quench oil and **Fig. 6(*b*)** for the used Behran 145 quench oil. The cooling rate produced by the unused Behran 145 oil is higher than that obtained for the used oil; therefore, as expected, the core microstructure of steel quenched in the unused oil (**Fig. 6(*a*)**) exhibits more pearlite (darker regions) and less proeutectoid ferrite (lighter regions) than the core microstructure of the steel quenched in the used oil (**Fig. 6(*b*)**).

The surface microstructures of the AISI 1045 steel test specimens produced by the unused and used Behran 145 oil are shown in **Fig. 7(*a*)** and **Fig. 7(*b*)**, respectively. Since the cooling rate for the unused oil is higher than the used oil, the relative

FIG. 6

Core microstructure of quenched AISI 1045 steel test specimens quenched in (*a*) unused Behran oil and (*b*) used Behran oil (200X, Nital 2 % Etchant). Note that the Pearlite (darker regions) and proeutectoid ferrite (brighter regions) are indicated by the P and F symbols, respectively.

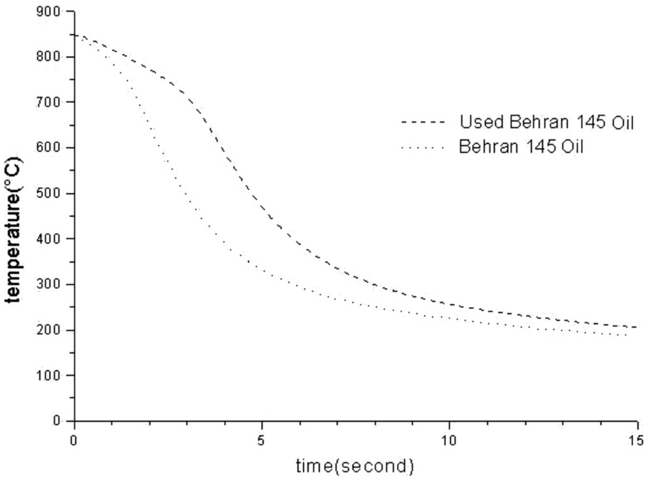
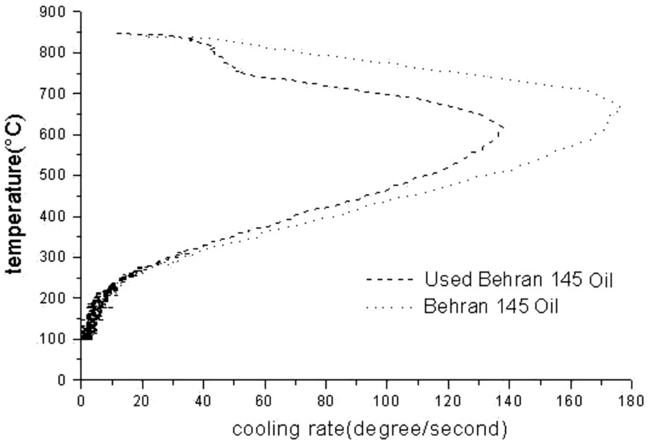


FIG. 7 Edge microstructure of quenched samples in (*a*) unused Behran oil and (*b*) used Behran oil (1000X, Nital 2 % Etchant). The symbol M depicts martensite.



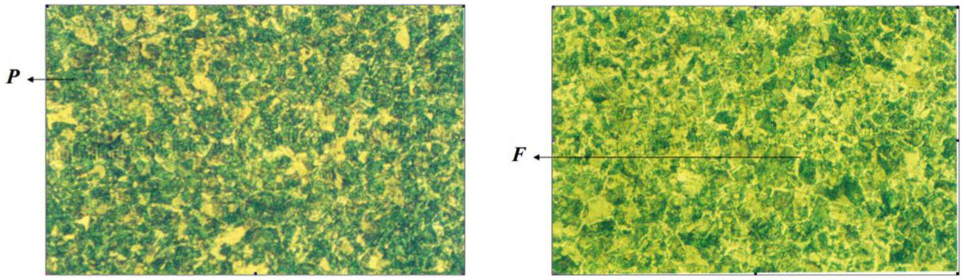
fraction of martensite at the surface of the steel test specimen is greater for the unused oil than for the used oil.

EFFECT OF COOLING RATE ON HARDNESS

Cross-sectional hardnesses were determined on the AISI 1045 steel test specimens quenched in unused Behran 145 quench oil and the used oil obtained from a commercial quench tank. These results are shown in **Fig. 8**. As expected, the unused oil, which exhibited the highest cooling rate, also produced the highest cross-sectional hardness profile. It is important to note that the hardnesses shown are averages of three determinations for each position.

The average of the hardness test results for the AISI 1045 steel test specimens quenched in unused Behran 145 and a used version of this quench oil sampled from a commercial production line and the average difference is shown in **Table 4**. This comparison shows that the unused Behran 145 oil produced >8 % higher surface hardness than the used oil. This is a significant difference in quench severity for such a low hardenability steel.

FIG. 8 The difference between the hardness of AISI 1045 steel test specimens quench in unused Behran 145 oil and a used Behran 145 sample taken from a commercial production tank.



(a) (b)

TABLE 4

The average of hardness of AISI 1045 steel test specimens quenched in unused and used Behran 145 quench oils and their difference.

The average of hardness of samples quenched	In unused Behran 145 oil	292.45 HV
	In used Behran 145 oil	269.5 HV
Difference between the average hardness of samples quenched in the oils		8.44 %

Conclusions

A review of oil quenchants and quench cooling mechanisms has been provided. In addition, the development and use of a proprietary quenchant testing system which utilizes a type 304 stainless steel probe was described. The use of this equipment and probe system was used to characterize the quenching behavior of a commercial unused quench oil and a sample of this oil after three years of use in a heat treat shop. The results showed the utility of cooling curve analysis to characterize the cooling profiles exhibited by these oils. Using the experimental approach described here, it was shown that the locally produced Behran 145 quench oil was actually an accelerated quench oil and the cooling rates significantly decreased for the used oil as would be expected if additive drag-out occurred, which would be expected for accelerated quench oils. The effect of the observed cooling rate decrease on the microstructures obtained for AISI 1045 steel test specimens quenched in the unused and used oil resulted in the expected formation of ferrite in the test specimen quenched into the slower used oil. This also was confirmed by lower superficial hardnesses produced by the used oil relative to the fresh oil.

These studies showed the importance of cooling curve analysis as part of a quality assurance program in any well-run heat treating facility. In addition, these results suggest that cooling curve analysis may be an integral part of a quench oil recycling and/or recovery process, which would yield longer use times, lower production cost, and reduced environmental impact. However, the support work to facilitate this process is yet to be completed.

Future Work

There are a number of research areas that need further development. These include:

1. The support work to readditize and recycle used quench oil using cooling curve analysis must be completed.
2. A second research area suggested by the work described here is the development of a “repeat quench” testing apparatus to evaluate rates and impact of additive drag-out, the impact of increased thermal stress on the fluid due to repeated quenching, especially on additive drag-out, and thermal-oxidative degradation on quenchant lifetimes and performance. Although some equipment for this purpose has been reported in the past [68,69], thus far, there is no commercial availability of such equipment. Therefore, the development of such equipment is being considered.

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