Heat Transfer, Hardenability and Steel Phase Transformations during Gas Quenching

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Heat Transfer, Hardenability and Steel Phase Transformations during Gas Quenching

by

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WORCESTER POLYTECHNIC INSTITUTE

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ABSTRACT

Quenching is the rapid cooling process from an elevated temperature. Compared to water and oil quench medium, high pressure and velocity gas is preferred to quench medium and high hardenability steel, with the potential to reduce distortion, stress and cracks. Currently, no standard test exists to characterize the gas quench steel hardenability and measure the performance of industrial gas quench furnaces.

In this thesis, the fundamental difference between the liquid and gas quenching, heat transfer coefficient, was emphasized. It has been proven that gas quenching with constant HTC cannot generate the similar cooling curves compared to liquid quenching. Limitations on current gas quench steel hardenability tests were reviewed. Critical HTC, a concept like critical diameter, was successfully proved to describe the gas quench hardenability of steel. An attempt to use critical HTC test bar and measure the HTC distribution of gas quench furnace was made.

Gas quenching, usually with slow cooling rate, may reduce hardness and Charpy impact toughness, compared to water and oil quenching. Lattice parameter and c/a ratio of as-quenched martensite in steel was measured using high resolution X-ray diffraction and Rietveld refinement. For AISI 4140, Charpy impact toughness decreases when the cooling rate decreases after quenching and tempering. Austenite percentage and carbon content in austenite is proposed as the dominated mechanism.
Executive Summary

Quenching is the rapid cooling process from an elevated temperature. Water, gas, petroleum, vegetable oil and forced air are selected as quench medium in different circumstances. Compared to other quench medium, high pressure and velocity gas is preferred to quench medium and high hardenability steel. It has several advantages, such as clean part surfaces after quenching, environmentally friendly, full flexibility to control quench intensity, potential to reduce distortion, stress and cracks and possibility to integrate heat treatment into the production line. Currently, no standard test exists to characterize the gas quench steel hardenability and measure the performance of industrial gas quench furnaces.

In this thesis, the fundamental difference between the liquid and gas quenching, heat transfer coefficient, was emphasized. A quenching model was developed and verified using DANTE. Using quenching experiment and model, it has been proven that gas quenching with constant HTC cannot generate the similar cooling curves compared to liquid quenching.

Current gas quench steel hardenability tests were reviewed. Several limitations were found, such as unsteady gas flow and not proper to characterize high hardenability steel. Critical HTC, a concept like critical diameter, was successfully proved to describe the gas quench hardenability of steel. The critical HTC of AISI 4140 steel is 430 W/m²°C and the critical HTC of AISI 52100 steel is 820 W/m²°C, which reveals that the gas quench hardenability of AISI 4140 is better than AISI 52100. A standard, “Method for determining hardenability of steel during gas quenching” was proposed.

An attempt to use critical HTC test bar and measure the HTC distribution of gas quench furnace was made. Based on modeling and experiment, 0.5” diameter and 4” length AISI 4340 bar can be used to evaluate 2bar nitrogen gas quench furnace. The 2bar nitrogen gas quench furnace has obvious uneven HTC distribution, which may cause microstructure and mechanical properties variations during gas quenching. A standard, “Measuring the heat transfer coefficient distribution of gas quench furnace”, is proposed.

When replacing liquid quenching to gas quenching, microstructure and mechanical properties should be addressed. Gas quenching, usually with slow cooling rate, may reduce hardness and Charpy impact toughness, compared to water and oil quenching. Lattice parameter and c/a ratio of as-quenched martensite in steel was measured using high resolution X-ray diffraction and Rietveld refinement. The modified equation can be used to estimate carbon content in martensite after liquid and gas quenching, which is essential to model mechanical properties afterwards. For AISI 4140, Charpy impact toughness decreases when the cooling rate decreases after quenching and tempering. Austenite percentage and carbon content in austenite is proposed as the dominated mechanism. For Pyrowear53, Charpy impact toughness decreases when the cooling rate decreases after quenching and tempering. Carbides is proposed as the dominated mechanism.
Acknowledgments

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CONTENTS

CHAPTER 1  Introduction ............................................................................................................. 1
  1.1 Quenching and gas quenching of steel .............................................................................. 1
  1.2 Goals and objectives .......................................................................................................... 2

CHAPTER 2  Literature review ................................................................................................ 5
  2.1 Heat transfer during quench process ................................................................................ 5
    2.1.1 Heat transfer during water and oil quenching ............................................................ 5
    2.1.2 Heat transfer during gas quenching ............................................................................ 6
  2.2 Steel hardenability .......................................................................................................... 7
    2.2.1 Hardenability definition ............................................................................................. 7
    2.2.2 Jominy end-quench test and Grossmann critical diameter test ................................. 7
    2.2.3 Influencing factors on hardenability .......................................................................... 8
    2.2.4 Characterizing gas quench steel hardenability ......................................................... 11
  2.3 Martensite phase transformation during quenching process ............................................ 13
    2.3.1 Martensite transformation in quenching .................................................................... 13
    2.3.2 Microstructure characterization in phase transformation .......................................... 14
    2.3.3 Martensite and retained austenite structure ............................................................. 15
    2.3.4 Relationship between as-quenched structure and mechanical properties ............. 17
    2.3.5 Cooling rate effect on microstructure and mechanical properties ......................... 18
    2.3.6 Model and simulation of steel phase transformation ................................................. 20

CHAPTER 3  Paper I: A comparison on heat transfer coefficient in liquid and gas quenching ................................................................. 25

CHAPTER 4  Paper II: The critical heat transfer coefficient method for characterizing hardenability of steel during gas quenching ................................................. 41

CHAPTER 5  Paper III: Evaluation on heat transfer coefficient distribution in gas quench furnace ........................................................................................................... 63

CHAPTER 6  Paper IV: Lattice parameter and the tetragonality of as-quenched martensite in steels ............................................................................................................ 75

CHAPTER 7  Paper V: Microstructure and mechanical properties comparison on AISI4140 and Pyrowear53 after gas and liquid quenching .................................................. 86

CHAPTER 8  Proposed standard I: method for determining hardenability of steel during gas quenching ............................................................................................................. 100

CHAPTER 9  Proposed standard II: Measuring the heat transfer coefficient distribution of gas quench furnace ................................................................................................. 110

CHAPTER 10  Conclusions ......................................................................................................... 118

CHAPTER 11  Recommendations for future work .................................................................... 119

CHAPTER 12  Relevant presentations and publications ............................................................. 120
CHAPTER 1 Introduction

1.1 Quenching and gas quenching of steel

Quenching is the rapid cooling process from an elevated temperature [1]. Water; gas; petroleum; vegetable or animal oil; aqueous polymer solution; aqueous(salt) solution; molten salt, fluidized bed and even forced air are selected as quench medium in different circumstances [1] [2]. The effectiveness of the quenching process depends on the heat transfer coefficient of quench medium, the hardenability of the steel and the parts geometry [1]. Martensite and sometimes retained austenite, only formed at relative high cooling rate, are the desired microstructure for as-quenched steel and tempering process afterwards [1] [3]. For specific parts geometry and steel grade, quenching medium with higher heat transfer coefficient (HTC) is preferred, such as water shown in Figure 1-1. However, the cooling rate at the surface and core of the part varies dramatically, which increases the potential for distortion, stress, and cracking [4].

![Heat transfer coefficient for different quenching media](image)

Figure 1-1 Heat transfer coefficient for different quenching media [1]

High hardenability steel, usually with high alloy contents, can form martensite with retained austenite at lower cooling rate compared to low hardenability steel. Therefore, higher hardenability steels and quenching medium with low heat transfer coefficient, such as in nitrogen and helium, are selected to reduce distortion, stress and cracking and keeps the strength of steels [1]. Currently, for the heat treatment of high-speed steels and tool steels, high pressure gas quenching (HPGQ) is the preferred choice and has almost replaced liquid quenching media [1]. Low-alloyed case-hardening steels and medium hardenability steels can be hardened by separate HPGQ quench chambers, or so-called cold chambers [1].

Gas quenching has the following advantages compared to liquid quenching [1]:

- Clean part surfaces, no need to washing
- Environmentally friendly process
• Full flexibility to control quench intensity
• Potential to reduce distortion, stress and cracks
• Possibility to integrate heat treatment into the production line

The disadvantage of HPGQ is that limited quench intensity (i.e. cooling rate) compared to oil or water quenching [1]. It also requires to use high-pressure vessel resulting in high equipment investment and the high pressure and velocity gas causes high noise levels [2]. Figure 1-2 and Figure 1-3 present the ALD ModulTherm gas quenching system.

Figure 1-2 ALD ModulTherm heat treat system with gas quenching chamber [5]
Figure 1-3 ALD gas quench system flow pattern [1]

1.2 Goals and objectives

This thesis is focused on the improvement of fundamental understandings of gas quenching process. The overall project goals are:

- Develop a test to characterize the hardenability of high hardenability steels.
- Develop a test to measure the performance of industrial gas quenching systems.

Specifically, the objectives are:

(1) Compare the cooling performance between liquid quenching and gas quenching
The fundamental difference between liquid quenching and gas quenching is the HTC. For liquid quenching, due to different liquid phase transformation, HTC can vary by several orders of magnitude. For gas quenching, HTC keeps nearly constant. In this thesis, an equivalent HTC for gas and liquid quenching is explored. A model, including heat transfer, phase transformation and hardness estimation, is developed and verified for quenching process.
(2) Develop a standard for characterizing steel hardenability during gas quenching
Hardenability is the key property of steel to determine whether the specific steel is suitable for selected quenching process. The Jominy and Grossmann water quenching steel hardenability tests have been successfully used by industry to define what steel is suitable for liquid quenching process. However, it demonstrated that both these tests cannot be directly used for gas quenching in this thesis. A gas quenching hardenability standard is proposed and verified.

(3) Evaluate the heat transfer coefficient distribution in gas quench system
The gas quenching HTC distribution in the furnace is complex. An easy and quick approach to evaluating the HTC distribution of gas quench system is needed, considering the workload pattern and gas flow condition.

(4) Develop an XRD procedure to accurately measure the c/a ratio in martensite and determine the relationship between the wt% C and c/a ratio.
Martensite crystal structure of steel with different carbon contents remains a debate currently. Modern X-ray machine with high resolution and Rietveld refinement was used to determine the relationship between the wt% C and c/a ratio.

(5) Compare mechanical properties between gas quenching and liquid quenching
After gas quenching process, the hardness of the parts is often the same or similar compared to oil or water quenching. It seems other mechanical properties should also be similar. However, Charpy impact toughness variation after different cooling rates were observed by Christoph Lerchbacher [6]. Martensite and retained austenite crystal structures, morphology and carbon distribution is studied to investigate the mechanism.
Reference:
CHAPTER 2 Literature review

2.1 Heat transfer during quench process

2.1.1 Heat transfer during water and oil quenching

When quenched in water and oil, cooling process can be divided into three distinct stages, film boiling, nucleate boiling and convection stages as shown in Figure 2-1. Many quenching probes were developed to quantify heat transfer coefficient (HTC) during this process, such as CHTE quench probe, Liscic-Nanmac probe, IVF probe, General Motors quenchometer and Grossmann probe [1].

Figure 2-1 Cooling curve and cooling rate curve of typical liquid quenching process [2]

Figure 2-2 HTC of different quench media [3]

Figure 2-2 presents the typical HTCs of different quenching media. HTCs of liquid quenching media show changes at different temperature.
2.1.2 Heat transfer during gas quenching

As shown in Figure 2-2, gas quench HTC at all quenching process is constant, which provides more uniform cooling process and has the potential to reduce distortion. Gas quench type, pressure, velocity, viscosity, compressibility, density, temperature, specific heat and thermal conductivity all have influences on gas quench HTC. It has been well studied and summarized as Equation 2-1 [4].

\[ h = \frac{k}{L} 0.023 \left( \frac{PVL}{\mu ZRT} \right)^{0.8} \left( \frac{\mu CP}{k} \right)^{0.33} \]  

Equation 2-1

h: heat transfer coefficient, W/m²K
P: gas pressure, Pa
V: gas velocity, m/s
L: specify characteristic length (part diameter), m
\( \mu \): dynamic viscosity, kg/ms
Z: gas compressibility and density
R: gas constant, J/Kmol
T: gas temperature, K
\( C_p \): gas specific heat, J/kgK
k: thermal conductivity, W/mK
2.2 Steel hardenability

2.2.1 Hardenability definition

During quenching, HTC is the surface boundary condition, and hardenability is the ability to form martensite [5]. Quenching conditions, size and shape of cross-section would affect the hardness, but only the chemical composition, initial microstructure and grain size determine the hardenability [2]. Many methods exist to measure hardenability, including Grossmann’s method, Jominy end-quench test, SAC rating and P-F test. The most familiar and commonly used procedures are Jominy test and Grossmann’s method [6].

2.2.2 Jominy end-quench test and Grossmann critical diameter test

The Jominy bar end-quench test is the most familiar and commonly used procedure for measuring steel hardenability. This test has been standardized and is described in ASTM A 255, SAE J406, DIN 50191, and ISO 642 [7]. For this test, a 100mm long by 25mm diameter round bar is austenitized to the proper temperature, dropped into a fixture, and one end rapidly quenched with 20-25°C water from a 12.5mm orifice under specified conditions. The austenitizing temperature is selected according to the specific steel alloy. Cooling velocity of the test bar decreases with increasing distance from the quenched end. After quenching, parallel flats are ground on opposite sides of the bar and hardness measurements made along the bar as illustrated in Figure 2-3 Figure 2-4 Figure 2-5 [7].

Grossmann’s method of measuring hardenability uses a number of cylindrical steel bars of different diameters hardened in a given quenching medium. After sectioning each bar at mid-length and examining it metallography, the bar that has 50% martensite at its center is selected, and the diameter of this bar is designated as the critical diameter [8].

Figure 2-3 Jominy end quench test setup1 [2]
Figure 2-4 Jominy end quench test: hardness measurement [7]
2.2.3 Influencing factors on hardenability

The heat treating process has influence on the hardenability. Usually the heat treating process could be divided into austenitizing process and quenching process. The purpose of austenitizing process is to obtain homogeneous austenite with defined grain size. During austenitizing process, there are two important metallurgical phenomena occurring in the austenite. First, the ferrite and pearlite transform to austenite. Second, the carbide dissolves into the austenite. Meanwhile, the austenite grains are growing. Both the chemical composition in austenite and the grain size affect the hardenability of the steel.
Figure 2-7 is AISI4140 TTA (time-temperature-austenitizing) diagram simulated by JmatPro. From the diagram, the homogeneous austenite is formed at 1100°C within 10s, compared to at 900°C within 100s. The homogenous austenite is formed more quickly at higher austenitizing temperature. When austenitizing temperature is selected, homogeneous austenite is more easily formed with low heating rate and longer heating time during heating process. Homogeneous steel has higher hardenability compared to inhomogeneous steel. Figure 2-8 presents the effect of austenitizing temperature on the hardenability of steels. Initial microstructure effect on hardenability is shown in Figure 2-9. Steel with fine initial microstructure would have higher hardenability.

Figure 2-9 Initial microstructure effect on hardenability

Higher austenitizing temperature and longer time increase grain size as shown in Figure 2-10. Holding time’s influence on the grain size is small compared to the austenitizing temperature [11]. For AISI 4140 steel, the grain size is ASTM10 after heat treating at 850°C even after 9 hours. At 1050°C within 9 hours, the grain size increases to ASTM6.5 [12].

Figure 2-10 Holding time effect on grain size

Figure 2-11 AISI4140 TTT diagram based on different grain size

Figure 2-12 4140 CCT diagram based on different grain size
As shown in Figure 2-11, Figure 2-12 and Figure 2-13, larger grain size increases the steel hardenability. However, large grain size is often avoided in industry for strength deduction. Adding alloying elements is used to increase steel hardenability [6].

The variations of chemical elements also have influence on TTT and CCT diagrams. The chemical composition is varied within a small range for specific steel grade. And this small variation has impact on the TTT and CCT diagrams which determines the hardenability of steel. The chemical composition of AISI 4140 alloy steel is presented in Table 2-1. The variation of chemical elements impact on TTT diagram is listed in Figure 2-14. Usually, the TTT diagram moves to right with the increase of the alloying elements, such as C, Cr, Mn, Si, Mo.

![Grain size effect on martensite formation](image1)

![Variation of chemical elements impact on 4140 TTT diagram and hardenability](image2)

<table>
<thead>
<tr>
<th>Element</th>
<th>Content (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Iron, Fe</td>
<td>96.785 - 97.77</td>
</tr>
<tr>
<td>Chromium, Cr</td>
<td>0.80 - 1.10</td>
</tr>
<tr>
<td>Manganese, Mn</td>
<td>0.75 - 1.0</td>
</tr>
<tr>
<td>Carbon, C</td>
<td>0.389 - 0.430</td>
</tr>
<tr>
<td>Silicon, Si</td>
<td>0.15 - 0.30</td>
</tr>
<tr>
<td>Molybdenum, Mo</td>
<td>0.15 - 0.25</td>
</tr>
<tr>
<td>Sulfur, S</td>
<td>0.040</td>
</tr>
<tr>
<td>Phosphorus, P</td>
<td>0.035</td>
</tr>
</tbody>
</table>

Table 2-1 The chemical composition of AISI4140 [13]

In conclusion, the initial microstructure, chemical composition, grain size, austenitizing temperature, heating rate and holding time all have effect on hardenability. In Jominy end quench standard, it mentions that the test piece shall be heated uniformly to the temperature specified in the relevant product standard or fixed by special agreement for at least 20 min and then maintained for 30 min at that temperature. Initial microstructure, chemical composition and grain size should be determined [7]. The above JMAtPro simulation results indicate that above 850°C austenitizing temperature, nearly all the steels have formed homogeneous austenite after 20 min heating process and 30 min holding process.
2.2.4 Characterizing gas quench steel hardenability

2.2.4.1 The Bozidar Liscic device [14]

No standard test method for determining gas quench steel hardenability are well accepted so far. Direct using Jominy end-quench test or Grossmann test were found to be not suitable for gas quenching process, because cooling rate in gas quenching is lower than 2°C/s [15]. Figure 2-15 and Figure 2-16 present Bozidar Liscic’s device for determining gas quench steel hardenability. The concept is based on Jominy end-quench test, by replacing water quenching with gas quenching. Adding ceramic insulation around the test bar is to avoid low-density gas cooling the side of test bar. Due to the gas impinging the end of test bar, the HTC is not easy to be well controlled and calculated.

Figure 2-15 Liscic's end-quench hardenability test device for gas quenched steels [16]  
Figure 2-16 Hardenability comparison by Liscic's device [16]

2.2.4.2 The Solar Atmosphere device [17]

Figure 2-17 and Figure 2-18 present Solar Atmosphere devices for determining gas quench hardenability. The device in open atmosphere cannot apply high pressure gas. The gas flow cannot be well-controlled for the device with chamber, because the gas velocity is too high and chamber is relatively small [17].
2.2.4.3 Other devices [6]

Some hardenability tests were developed for air-hardened steel as shown in Figure 2-19 and Figure 2-20. A large mass is attached at one end the sample to reduce cooling rate.
2.3 Martensite phase transformation during quenching process

2.3.1 Martensite transformation in quenching

During quenching, austenite transforms to martensite, the Phenomenological Theory of Martensitic Crystallography (PTMC) is the most widely accepted theory [18][19]. Figure 2-21 presents the Bain distortion from FCC lattice of $\gamma$ into the BCC lattice of $\alpha'$. There is a compression along the z axis and a uniform expansion along the x and y axes.

Figure 2-21 Bain distortion [18][19]

Figure 2-22 (a) and (b) show the effect of the Bain strain on austenite, which when undeformed is represented as a sphere of diameter $w_x=y_z$ in three dimensions. The strain transforms it to an ellipsoid of revolution. (c) shows the invariant-line strain obtained by combining the Bain strain with a rigid body rotation through an angle $\theta$ [18][19]

Figure 2-22 presents the invariant-line strain (ILS) during Bain distortion. By rigid body rotation, an ILS was found as a necessary condition for martensitic transformation. During the phase transformation, twinning or slip may exist as shown in Figure 2-23. Twinning and slip can explain habit plane and successfully predict a substructure in plates of martensite (either twins or slip steps) as is observed experimentally [20].

The partisans of the PTMC claims that the theory provides the appropriate habit plane and orientation relationship. Despite its success and its broad use, PTMC remains phenomenological [21]. The exact atomic displacements of the iron atoms during the transformation are not in its scope: “The crystallographic theory of martensite is on the hand called phenomenological; the steps into which the transformation is factorized are not unique and do not necessarily describe the actual path by which the atoms move from one lattice to the other. The theory simply provides a definite link between the initial and final states without being certain of the path in between” [22].
The KSN model is another model to describe martensite transformation, combined Kurdjumov-Sachs and Nishiyama-Wassermann models together. The KS and NW ORs are separated by 5°, and both are at 10° far from the Bain OR. This difference between the Bain distortion and experiment made these authors propose a similar model of lattice distortion by shear and dilatation, which is known as the KSN model. However, KSN model predicted habit planes were not identical compared to experimental result such as {259} and {225}. The fact that Nishiyama himself changed his mind and advocated for the PTMC and Bain distortion was probably decisive in the scientific community to make PTMC wins versus KSN [23].

Bogers etc. [24] and Olson etc. [25] developed a model based on the assumption of Shockley partial dislocation. Other approaches based on strain energy considerations and interfacial dislocations models are reported in a review on martensitic transformations by Zhang and Kelly [26]. Venables [27] found HCP phase for stainless steel and proposed two step martensite phase transformation for stainless steel. However, based on reviewer’s knowledge, no HCP phase has been found for carbon steel or low and medium alloy steel so far. No model discussed above can explain all the phenomenon for martensite transformation.

2.3.2 Microstructure characterization in phase transformation

In order to characterize phase transformation, optical microscopy, scanning electron microscopy, transmission electron microscopy, dilatometry, X-ray diffraction, atom force microscopy and atom probe tomography are utilized. Among those, in situ XRD [28] and Rietveld refinement [29] is a promising technique to investigate the mechanism during phase transformation.

The Rietveld refinement uses a least squares approach to refine a theoretical line profile until it matches the measured profile. It is able to deal reliably with strongly overlapping reflections, such as retained austenite and tetragonal martensite peaks [29]. Using this technology, it is possible to deconvolute martensite (101), martensite (110) and austenite (111) peaks as shown in Figure 2-25.
Figure 2-26 presents the typical in situ XRD work by Jeremy Epp [30]. AISI5120 steel were heated to 900°C and quenched with cooling rate higher than 40 °C/s. FRELON (ESRF, Grenoble, France) camera was used with an exposition time of 0.4 seconds for each frame. This ultra-fast detector can record structure transformation during quenching. In Figure 2-26, it clearly shows that when reaching martensite starts temperature, martensite starts to form body center tetragonal structure.

![Figure 2-25 Rietveld refinement of as-quenched AISI52100 steel](image)

Figure 2-25 Rietveld refinement of as-quenched AISI52100 steel

![Figure 2-26 Evolution of lattice parameters of ferrite/martensite as a function of temperature and calculated carbon content in solution [30]](image)

Figure 2-26 Evolution of lattice parameters of ferrite/martensite as a function of temperature and calculated carbon content in solution [30]

![Figure 2-27 Schematic illustrations showing lath martensite structure in (a) low carbon (0-0.4%C) and (b) high carbon (0.6%C) steels [31]](image)

Figure 2-27 Schematic illustrations showing lath martensite structure in (a) low carbon (0-0.4%C) and (b) high carbon (0.6%C) steels [31]

2.3.3 Martensite and retained austenite structure

Martensite is the major as-quenched microstructure in Fe-C alloys with less than 0.6 wt% C. Lath martensite mainly forms in low and medium carbon steel [20]. Figure 2-27 presents the well
accepted lath martensite structure [31]. Carbon content of the steel, cooling rate and prior austenite grain size all have effects on the size of packet, block and lath [32,33]. As shown in Figure 2-28, the packet and block size decrease with the decreasing of prior austenite grain size. The lath width was found to be constant with different austenite grain size and cooling rate, but decreases with the increasing of carbon content as shown in Figure 2-28(c) and Figure 2-29 [33]. In Figure 2-29, IBQ stands for high cooling rate and FC stands for low cooling rate.

Grain boundary must exist at the boundary of packet, block and lath. By TEM and Atom Probe Tomography, it was found that thin austenite film is formed between every two martensite lath [33-35]. Figure 2-30 presents the diffraction pattern of austenite film [35]. The carbon content in austenite film is much higher than nominal carbon content of the steel as shown in Figure 2-32. No references about block and packet boundary was found so far. However, it is not surprising if thin film austenite was found at that place.

Figure 2-28 Link between the prior-austenite grain size and (a) packet and (b) block size and (c) lath width in various steels [32]
2.3.4 Relationship between as-quenched structure and mechanical properties

Block size was found to have relationship with yield strength by Morito [36] as shown in Figure 2-31. It should also be noted that Mn addition effects block size[36]. More recent work by Galindo-Nava [32] extended the model for the tempering process.

Figure 2-31 Hall-Petch type plots of yield strength vs block size [36]
Hardness, tensile strength, Charpy impact toughness, fracture toughness and other mechanical properties at working temperature are important in industry. Thin austenite film thickness, and carbon distribution in austenite film and martensite lath, were found dependent with Charpy impact toughness by Lerchbacher [35] as shown in Figure 2-34 and Figure 2-35.

2.3.5 Cooling rate effect on microstructure and mechanical properties

Critical cooling rate, is defined as the slowest cooling rate that generates 100% martensite. The critical cooling rate is the tangent line with nose of CCT diagram [37]. The hardness and other mechanical properties are often treated similar when the cooling rate is higher than critical cooling rate. Directly replacing water or oil quenching with gas quenching is based on this assumption, which as long as fully martensite microstructure forms, the mechanical properties are the same. However, Morito [33] and C. Lerchbacher [35] found that, even when the cooling rate higher than critical cooling rate, different cooling rates still affect the martensite morphology and carbon distribution in martensite lath and austenite thin film, which further influences Charpy impact toughness.

![Carbon concentration profile in martensite lath and austenite film](image)

(a) $\lambda = 0.3$, Cooling rate = 10K/s  
(b) $\lambda = 3$, Cooling rate 1K/s  
(c) $\lambda = 12$, Cooling rate 0.25K/s

Figure 2-32 Carbon concentration profile in martensite lath and austenite film when quenching hot-work tool steel X38CrMoV5-1 with different cooling rates [35]

Figure 2-27 presents the morphology of martensite. It has been demonstrated that higher cooling rate can generate smaller prior austenite grain size, packet size and block size [36]. Austenite film thickness decreases with the increase of cooling rate [35].
Figure 2-33 Development of the interlath austenite film thickness and the volume fraction of retained austenite in dependence of the quenching parameter [35]

Figure 2-32 and Figure 2-33 present the increasing of austenite film thickness with decreasing of cooling rate. It should be noted that X38CrMoV5-1 has ultra-high hardenability and even when the cooling rate is lower than 0.25K/s, no bainite forms. Figure 2-34 presents the Charpy impact toughness of the hardened samples with different cooling rate. “This toughness increase can be explained by the presence of a higher amount of retained austenite, hence, the martensitic regions are less strained due to the carbon enrichment of the retained austenite” [35]. “However, the carbon content in austenite film is not available for the subsequent tempering induced precipitation of carbides within the martensite laths, which are responsible for the typical high-temperature strength of hot-work tool steels” [35]. Figure 2-35 presents that the tempered Charpy impact toughness decreases with the cooling rate (quenching process).

When considering replacing oil quenching with gas quenching, Charpy impact test, fatigue test and other mechanical properties must be checked.

Figure 2-34 Charpy impact energy of the hardened samples in dependence of the cooling rate [35]

Figure 2-35 Charpy impact toughness and hardness values in dependence of the cooling rate corresponding to tempering treatment [35]
2.3.6 Model and simulation of steel phase transformation

Prediction of microstructure and properties in quenching originate from the early 1970s [6]. In 1980s, empirical equation and constitutive models were widely used to predict microstructure and hardness after quenching process as shown from Equation 2-2 to Equation 2-5.

\[
\dot{T} = -(T_a - 297) \frac{4\eta}{\sqrt{\pi}x^2} \phi^3 \exp(-\phi^2) \quad \text{Equation 2-2}
\]

\[
\phi = \frac{\pi}{2} \left[ \left( \frac{T}{T_a - 297} \right) + 0.4406 \left( \frac{T_a - 297}{T_a - 297} \right)^{3.725} \right] \quad \text{Equation 2-3}
\]

\( \eta \) is the thermal diffusivity at distance x (in cm) along the Jominy bar. \( T \) is the temperature and \( T_a \) is the austenitizing temperature [38].

\[
\text{VPN} = Y_1 - \frac{(Y_1 - Y_2)}{3X_0^2} x^2; \quad x < X_0 \quad \text{Equation 2-4}
\]

\[
\text{VPN} = Y_2 + \frac{2}{3} (Y_1 - Y_2) \frac{X_0}{x}; \quad x > X_0 \quad \text{Equation 2-5}
\]

\( X_0 \) is the Jominy bar depth. VPN is the Vickers Pyramid Number. \( Y_1 \) and \( Y_2 \) are the hardness values of martensite and pearlite in the alloy of interest [38].

Many commercial FEA software are capable of simulating the quenching process, such as DANTE, HEARTS, TRAST, SYSWELD and DEFORM-HT [6]. All the software or related package and sub routine are following the quenching mechanism as shown in Figure 2-36.

![Figure 2-36 Physical fields and coupling interactions involved in the quenching process [6]](image-url)
Thermocalc and Dictra are widely used to model diffusion controlled phase transformation. Dmitrieva observed and modelled alloying elements diffusion between martensite and austenite during partitioning as shown in Figure 2-37 [39]. Goune used Darken’s equation and mass balance to model carbon partitioning at 75°C as shown in Figure 2-38 [40]. Both Dmitrieva and Goune modified the diffusivity of alloying elements. It should be noted that the modified diffusivity is much higher than the diffusivity in traditional database, such as Thermocalc.

Figure 2-37 Alloying elements diffusion during partitioning [39]

Figure 2-38 Carbon partitioning from martensite to austenite at 75°C [40]
Reference:


CHAPTER 3  Paper I: A comparison on heat transfer coefficient in liquid and gas quenching

Yuan Lu, Yiming Rong, Richard D. Sisson, Jr.

To be submitted to: International Journal of Heat and Mass Transfer

Highlights:
1. Develop and verify quenching model during liquid and gas quenching of steel.
2. Prove that gas quenching with constant HTC cannot generate the same cooling curves compared to liquid quenching.
3. Propose equivalent HTC of gas and liquid quenching. Gas quenching with dynamic HTC is proved to produce the same cooling curves compared to oil quenching.
A comparison on heat transfer coefficient in liquid and gas quenching

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Abstract: With the development of quench technology, gas quenching is more popular for less distortion and residual stress compared to liquid quenching, as well as reduced environmental impact. The fundamental difference between the liquid and gas quenching is the heat transfer coefficient, not only the values but also the variation with temperature. A quenching model was developed and verified using DANTE. Hardness database of different phases was modified. Using quenching model and experiment, it has been proven that gas quenching with constant HTC cannot generate the similar cooling curves compared to liquid quenching. Gas quenching with dynamic HTC is proposed to have the same cooling curves compared to oil quenching.

Keywords: Gas quenching, Liquid quenching, Heat transfer coefficient, Equivalency

1. Introduction

Gas quenching is becoming popular to replace water or oil quenching [1] for medium and high hardenability steels, such as AISI4140 and AISI4340. It has many advantages such as less distortion, less stress, safer and more environmental friendly [2]. With gas quenching process, mechanical and physical properties can be significantly improved and obtain near shape of metal components [2]. However, the uniformity of gas quenching process is an issue compared with liquid quenching. Based on the work of Jing Wang [3], Elkatatny [2] Bowang Xiao [4] and Cosentino [5], the gas pressure and velocity changes dramatically in the furnace. Current studies on gas quenching are focused on the gas flow in the furnace.

Considering the complex of gas pressure and velocity, the gas quenching heat transfer coefficient (HTC) is noted, since the HTC has direct influence on cooling curves [5]. In this paper, constant HTC and dynamic HTC of gas quenching is compared with HTC of liquid quenching. After verifying the quench model using DANTE [6] by experiment, the model is used to get equivalent HTC for liquid and gas quenching.
2. Develop and verify quenching models

2.1 Jominy end-quench test for AISI4140 and AISI8620

In order to develop and verify quenching model, AISI4140 and AISI8620 were selected to repeat Jominy end-quench test. The austenitizing temperature of AISI4140 is 843°C and of AISI8620 is 899°C, both maintained for 30min at the austenitizing temperature [7]. All the procedures are strictly followed the ISO 642-1999 Steel – Hardenability test by end quenching (Jominy test) [8]. The test results, shown in Figure 1 and Figure 2, fit the USS reference and it indicates that the Jominy test has been repeated successfully. During hardness test, ISO 6508, Metallic materials – Rockwell hardness test is followed. The alloy element of experiment result was from OES measurement.

![Figure 1 AISI4140 Jominy hardenability](image1)

![Figure 2 AISI8620 Jominy hardenability](image2)

2.2 Heat transfer models

Heat transfer, phase transformation and mechanical properties model should be considered during quenching process. The model of Jominy test has been created using DANTE as shown in Figure 3 and Figure 4. The temperature can be measured by thermocouples and simulated by DANTE. The heat transfer coefficients database is from DANTE. In order to verify the accuracy of the model, the comparison between the experiment and the simulation for the standard Jominy water quench process has been made. As shown in Figure 5, the comparison shows good correlation between experiment and simulation. The experiment data are from Timken [9].
2.3 Phase transformation models

Phase transformations are also simulated during quenching process. In DANTE, thermal/stress process and phase transformation are coupled together. Figure 6 presents AISI4140 phase percentage in Jominy bar. Near 100% martensite forms at the end quench. With the increase of quench end distance, the martensite percentage starts to drop and lower bainite forms. Figure 7 and Figure 8 is the martensite percentage comparison between experiment and simulation of AISI4140 and AISI8620. The steel chemical elements variation, grain size and initial microstructure all have influence on TTT diagram, which later affects martensite percentage in end-quenched bar. Considering the simulation results from DANTE and Jmatpro have the same trends compared to references, the phase transformation simulation is accurate.
2.4 Mechanical properties models

Figure 9 presents the Jominy end-quench hardenability comparison between experiment and simulation. The hardenability profile does not fit the experiment result well. Near the quenched end location, the hardness is underestimated. Far from the quenched end, the hardness is overestimated. As discussed above, heat transfer and phase transformation model are already proved accurate. If the microstructure percentage, including martensite, lower bainite, upper bainite, pearlite and ferrite, is not accurate, the whole hardness profile should be underestimated or overestimated compared to the experiment result, which is not the case. Mechanical properties models, specify hardness database of different microstructure, is doubted.

Equation 1 is the hardness model used in DANTE. It considers that different microstructures follow mixture law for the total hardness. For all the microstructures, the hardness varies with carbon content. For upper and lower bainite, the hardness also varies with formation temperature. Table 1 presents the hardness database in DANTE.

\[
Hardness = \sum \text{phase} \times \text{phase hardness}
\]  

Equation 1
The literature was reviewed on hardness of different microstructures during quenching process [10] [11]. Martensite hardness has been well studied with the varying carbon contents. The average hardness of all the references were selected as the new martensite hardness shown in Table 2. Comparing to original DANTE hardness database, the hardness of martensite is underestimated, which explains the simulation result near the quenched-end.

<table>
<thead>
<tr>
<th>Carbon level %</th>
<th>austenite</th>
<th>ferrite</th>
<th>pearlite</th>
<th>martensite</th>
<th>tempered martensite</th>
<th>upper bainite</th>
<th>lower bainite</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.2</td>
<td>20</td>
<td>20</td>
<td>25</td>
<td>50</td>
<td>45</td>
<td>30-35</td>
<td>38-42</td>
</tr>
<tr>
<td>0.4</td>
<td>22</td>
<td>22</td>
<td>27</td>
<td>52</td>
<td>47</td>
<td>32-37</td>
<td>40-44</td>
</tr>
<tr>
<td>0.6</td>
<td>24</td>
<td>24</td>
<td>29</td>
<td>54</td>
<td>49</td>
<td>34-39</td>
<td>42-46</td>
</tr>
<tr>
<td>0.8</td>
<td>26</td>
<td>26</td>
<td>31</td>
<td>56</td>
<td>51</td>
<td>36-41</td>
<td>44-48</td>
</tr>
</tbody>
</table>

Table 1 DANTE hardness database for different microstructures [6]

The hardness of lower bainite, upper bainite, pearlite, ferrite and austenite are not easy to obtain and verifying. Considering the high accuracy of heat transfer and phase transformation model in DANTE, the least squares regression method was used to get the hardness of each phase. The hardenability curve of more than 30 different steel grades, including AISI8680, AISI4150,
AISI4340, AISI4140, AISI8650, AISI52100, AISI5140, AISI8620, AISI1020, were simulated based on the original DANTE hardness database.

The minimum and maximum experimental hardenability band of specific steel was obtained from reference [9]. The hardenability band, not hardenability curve for specific steel, is due to the varying chemical composition, grain size, and initial microstructure even for the same steel grade [12]. Figure 10 present the experimental reproducibility of hardness in controlled tests can typically be ±6 HRC. Although ASTM specification A255-10 is highly specific, many instances still exist where deviations from the standard practice are not controllable within the most disciplined laboratory conditions.

![Image](image_url)

**Figure 10** Summary of reported Jominy tests by several laboratories on a AISI 4140 steel of approximately the same composition and grain size [12]

If the simulated hardenability curve is within the range, no modification is made to the original hardness database. When the simulated hardenability curve is beyond the range, the hardness of bainite, pearlite and austenite are adjusted, mainly lower bainite and upper bainite, which forms during quenching process. The modified hardness database is still under development. The hardness of lower bainite and upper bainite is only the average hardness formed at different temperature currently. As shown in Table 3, the hardness of lower bainite and upper bainite is lower compared to the original database. As shown in Figure 9, the higher hardness estimation at far end of Jominy bar reveals that the modified lower hardness of lower and upper bainite should be more accurate. As a conclusion, the hardness in original DANTE hardness database underestimates martensite hardness and overestimates bainite (lower and upper bainite) hardness. Figure 11 presents the simulated hardenability curve based on modified hardness database.
<table>
<thead>
<tr>
<th>Carbon wt%</th>
<th>Martensite</th>
<th>Lower bainite</th>
<th>Upper bainite</th>
<th>Pearlite</th>
<th>Austenite</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.1</td>
<td>40</td>
<td>32</td>
<td>26</td>
<td>20</td>
<td>14</td>
</tr>
<tr>
<td>0.2</td>
<td>50</td>
<td>36</td>
<td>27</td>
<td>20</td>
<td>14</td>
</tr>
<tr>
<td>0.4</td>
<td>60</td>
<td>40</td>
<td>30</td>
<td>20</td>
<td>18</td>
</tr>
<tr>
<td>0.5</td>
<td>62</td>
<td>42</td>
<td>31</td>
<td>21</td>
<td>20</td>
</tr>
</tbody>
</table>

Table 3 Modified hardness database

3. Gas Quench Model Verification

The gas quench system is presented in Figure 12. The cylindrical sample with diameter 25mm and length 100mm is used. The gas flow is assumed the same at the free end of the sample and the sample sides, since the slenderness ratio is large. In this condition, the gas pressure and velocity are steady and well controlled during gas quench condition.

Gas quench type, pressure, velocity, viscosity, compressibility, density, temperature, specific heat and thermal conductivity all have influences on gas quench HTC. This has been well studied and is summarized as Equation2 [13].
\[
h = \frac{h}{L} \cdot 0.023 \left( \frac{PVL}{\mu ZRT} \right)^{0.8} \left( \frac{\mu C_P}{k} \right)^{0.33}
\]

Equation 2

h: heat transfer coefficient, \( W / m^2 K \)
P: gas pressure, Pa
V: gas velocity, m/s
L: specify characteristic length (part diameter), m
\( \mu \): dynamic viscosity, kg/ms
Z: gas compressibility and density
R: gas constant, J/Kmol
T: gas temperature, K
\( C_P \): gas specific heat, J/kgK
k: thermal conductivity, W/mK

Figure 12 Gas quench system schematic sketch

The experiments were done with the help of Praxair, Inc. The steel is AISI4140 in the experiment. In Figure 13, the cooling curves under different gas quench condition are measured by thermocouple and simulated by gas quench model. The simulation results match the experimental result and it demonstrates the accuracy of gas quench model. To improve the accuracy, the ambient temperature, transfer time from the heating furnace to the quenching chamber and the time required to build up the pressure and reaching the gas flow speed should be considered.
4. Gas quenching with constant HTC vs. liquid quenching

The HTC is the only difference between liquid quench and gas quench. Chemical reactions with the surface of the steel are ignored in the paper.

Figure 14 presents the HTC of different quench media. Liquid quench exhibits three characteristic quenching processes, film boiling, bubble boiling and convection [12]. For gas quench, the single-phase heat transfer process means that the cooling rate is more uniform [5]. The verified gas quench model is used to simulate the gas quench process and predict the equivalent HTC.
Figure 15 AISI 4140 cooling profile comparison (simulation)

Figure 15 (simulation) is the cooling profile comparison between oil quench and gas quench. The cooling profiles of different gas quench HTCs are simulated to match the cooling profile of oil quench. For gas quench HTC 1000 W/m$^2$C (constant from 20C to 1000C) and HTC 1200 W/m$^2$C (constant from 20C to 1000C), the cooling rates from 850C to 200C is lower than oil quench. In order to increase the cooling rates from 850C to 200C, the gas quench HTC 2000 W/m$^2$C is used. The cooling curves for HTC 2000 W/m$^2$C (constant from 20C to 1000C) matches the oil quench from 850C to 300C. From 300C to 20C, the cooling rates for gas quench 2000 W/m$^2$C is higher than oil quench. No gas quench with constant HTC can be the equivalent HTC compared to oil quench.

5. **Gas quenching with dynamic HTC vs. liquid quenching**

In heat treatment, core microstructures and properties are important, because the core cooling rate is the lowest and may form undesired microstructures such as upper bainite and ferrite. The equivalent HTC between liquid and gas quench is defined as the HTC, which has the same cooling curves at the core of the sample. After two different quench processes, if the cooling curves of the core are the same, these two quench HTCs are considered as the equivalent HTC. One the advantages of gas quench is great process flexibility to vary cooling rates by adjusting gas pressure and velocity. Gas quench with dynamic HTCs are considered to find the equivalent HTC compared to oil quench.

The HTC shown in Figure 16 (simulation) is the equivalent HTC for oil quench. From 1000C to 300C, the HTC is 2000 W/m$^2$C. From 300C to 180C, the HTC is 1200 W/m$^2$C. From 180C to 100C, the HTC is 500 W/m$^2$C. From 100C to 20C, the HTC is 100W/m$^2$C. At each stage, the gas quench HTC is the constant. Figure 17(simulation) are the cooling profiles of oil quench and
equivalent gas quench at the core of the sample. Gas quench with varying HTCs is the equivalent HTC compared to liquid quench.

![4140: Equivalent Gas Quench HTC compared to Oil Quench](image)

**Figure 16** AISI 4140 equivalent gas quench HTC compared to oil quench (simulation)

![4140 Cooling Profile Comparison](image)

**Figure 17** AISI 4140 temperature profile comparison (simulation)

Simulation based on Jominy test was finished to extent the concept of the equivalent HTC. The sketch is shown in Figure 3. The Jominy bar is 25mm diameter and 100mm length. Boundary conditions 2,3 and 4 are air-cooling and boundary condition 1 is oil quench or equivalent gas quench in Figure 18. The temperature profile and the hardenability (along the black line in Figure 19) are compared to verify the equivalency of oil quench and gas quench.
In Figure 18 (simulation), the cooling profiles along the Jominy bar for oil quench and the equivalent gas quench is compared. At 0mm, 10mm, 20mm and 50mm position from the quenched end, the cooling profiles are considered the same for oil quench and the equivalent gas quench. In Figure 19 (simulation), the hardenability of AISI 4140 under oil quench and the equivalent gas quench is simulated. Two hardenability curves match perfectly, which demonstrates the two quench processes generate the same microstructures and properties.

The concept of equivalent HTC should be redefined. After two different quench processes, if the cooling curves, microstructures and properties of all the workpiece are the same, these two quench HTCs are considered as the equivalent HTC.
The AISI 52100 equivalent gas quench process is simulated as well. The equivalent gas quench HTC is the same as AISI 4140’s. The cooling profile comparison and Jominy hardenability for AISI 52100 are in Figure 20 (simulation) and Figure 21 (simulation).

![52100 Jominy test: cooling profile comparison](image)

**Figure 20 AISI 52100 Jominy test: cooling profile comparison (simulation)**

### 6. Conclusions

A robust quenching model has been developed and verified for liquid quenching and gas quenching. The difference between liquid and gas quench is the HTC. After comparing cooling curves after liquid and gas quenching, it has been proven that gas quenching with constant HTC cannot generate the same cooling curves as liquid quenching.

The concept of equivalent HTC is then proposed. After two different quench processes, if the cooling curves of the core are the same, these two quench HTCs are considered as the equivalent HTC. The equivalent HTC prediction was made based on the verified gas quench model. After finding the equivalent gas quench HTC, Jominy test was simulated to compare the cooling curves and hardness for the entire workpiece. Gas quenching with dynamic HTC has the same cooling curves and hardness compared to liquid quenching.
Figure 21 AISI 52100 Jominy test comparison (simulation)

Acknowledgement
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References:


CHAPTER 4  Paper II: The critical heat transfer coefficient method for characterizing hardenability of steel during gas quenching

Yuan Lu, Yiming Rong, Richard D. Sisson, Jr.

To be submitted to: Metallurgical and Materials Transactions A

Highlights:
1. Point out fundamental limitations of Jominy end-quench like method for characterizing gas quench steel hardenability.
2. Propose and test critical heat transfer coefficient method, a Grossmann-like method, for characterizing gas quench steel hardenability. No insulation is needed. The gas flow is steady and well controlled. High, medium and low hardenability steels can be tested in the same system. Sample geometry is simple and fixed.
3. Discuss using HTC to indicate gas quench intensity, instead of gas pressure and velocity. Compare the sensitivity of critical HTC test and Jominy end quench test. Critical HTC test is proved sufficient to distinguish hardenability difference of steel with same grade.
The critical heat transfer coefficient method for characterizing hardenability of steel during gas quenching

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Abstract: Gas quench, with advantages such as reducing distortion and residual stress, is developing rapidly with the intent to replace liquid quench. Medium and high hardenability steels are needed for gas quench, since the quenching power is lower compared to liquid quench. The traditional Jominy end quench test and Grossmann test, designed for liquid quench steel hardenability, does not properly determine the hardenability of high alloyed steels. In order to determine gas quench steel hardenability, a new test is required. In this paper, a critical heat transfer coefficient (HTC) test based on the Grossmann test is proposed. Critical HTC, a concept like critical diameter, was successfully proved to describe the gas quench hardenability of steel. The critical HTC of AISI 4140 steel is 430 W/m²°C and the critical HTC of AISI 52100 steel is 820 W/m²°C, which reveals that the gas quench hardenability of 4140 is better than 52100. The critical HTC test requirements are presented and discussed in detail.

Keywords: Gas quenching, Liquid quenching, Heat transfer coefficient, Equivalency

1. Introduction

1.1 Gas quench and liquid quench

Quenching is the process of rapidly cooling steel from the austenitizing temperature [1]. The purpose of quenching is to obtain steel with higher hardness and other related mechanical properties. With the development of the modern steel quench technology, high pressure and high velocity gas quench has been widely used [2]. The heat transfer coefficient of gas quench could be as high as 2000 W/m²k and is large enough to quench high hardenability steels (AISI4340) and some medium hardenability steels (AISI4140). One of the significant advantages of gas quench is to get the similar mechanical properties compared to water or oil quench, and reduce the distortion and residual stress [3].

1.2 Hardenability

In order to obtain similar mechanical properties such as hardness with lower cooling rate, steel hardenability is one of the most important factors to be considered. Hardenability is the ability of
the Fe-C alloy to be hardened by forming martensite [4]. It is qualitative measure of the rate at which hardness decreases with distance from the surface due to decreased martensite content with reducing cooling rates [1]. Not all the steel can be used in gas quench [5], such as low hardenability steel. In order to select proper steel for gas quench, the gas quench steel hardenability needs to be defined and measured, however, no specific gas quench steel hardenability standard exists.

1.3 Current gas quench steel hardenability test and limitations

Many methods exist to measure hardenability for liquid quench, which including Grossmann’s method, Jominy bar end-quench test, SAC rating and P-F test [4]. The Jominy bar end-quench test is the most familiar and commonly used procedure for measuring steel hardenability. This test has been standardized and is described in ASTM A 255, SAE J406, DIN 50191, and ISO 642. Since water quench Jominy test is widely used in industry, the current gas quench steel hardenability tests are based on the prototype of Jominy test. Figure 1 and Figure 2 are all current Jominy gas quench steel tests.

Solar Atmosphere has designed a Jominy like gas end quench system as shown in Figure 1 (a). The device can generate high velocity gas at room pressure. In Figure 1 (b) test, also designed by Solar Atmosphere, high pressure and high velocity gas can be generated for gas quench, however the gas velocity cannot be controlled and the gas flow is not steady [6]. The Figure 2 (a) test, designed by IWT [7], uses insulation brick around the Jominy bar during gas quench process in order to prevent the side-flow gas from cooling the sample. Figure 2 (b) presents the gas quench hardenability test result from IWT system [7].

Several limitations exist for current gas quench Jominy test:
(1) In Jominy test for liquid quench, only the sample end is quenched. However, since the gas is much lighter compared to liquid, the sample side will be cooled by gas in Jominy-like test for gas quench. The insulation should be added to prevent this phenomenon, which guarantees the Jominy test is one-dimensional heat transfer condition.
(2) When high pressure and high velocity gas impinge on the quenched end, the flow around the rod is complicated. While in liquid quench, the HTC on quenched end can be assumed as uniform.
(3) Traditional Jominy bar, which has 25mm in diameter and 100mm in length, cannot test very high hardenability steel. Even the far-end quenched end of the sample will be fully hardened. The modified Jominy bar, which has a mass on the end of the sample, was proposed to generate cooling rate as low as 0.16 C/s.

Although many improvements were proposed to modify Jominy gas quench test, the fundamental limitations of this method still exit. Low hardenability steel (AISI8620), medium hardenability steel (AISI4140) and high hardenability steel (AISI4340) can be tested by the Jominy water quench test, applying the same water spray (same HTC) quench condition. And using same sample geometry. However, in Jominy gas quench test, one gas quench condition cannot be used for both low hardenability steel and high hardenability steel at the same time.
The simulation results are shown in Figure 3 and Figure 4. From the results, the hardenability of AISI8620 (low hardenability steel) cannot be revealed when low HTC gas quench condition is applied, since even the quenched end cannot form martensite at low cooling rate. Although the hardenability of AISI4340 (high hardenability steel) can be measured under high HTC gas quench condition, AISI4340 still shows high hardenability under low HTC gas quench conditions. For low hardenability steels such as AISI8620, high HTC gas quench condition should be used to ensure that martensite could be formed at the quenched end. For high hardenability steels such as AISI4340, low HTC gas quench condition should be used to reveal its complete ability to be hardened at low cooling rate.
Figure 3 also reveals the limitations of modified Jominy gas quench hardenability test. The same steel grades, 90MnCrV8, has different hardenability curves under different gas quench conditions. If the hardenability of different steel grades need to be compared, using which gas quench HTC would be a debate.

If different steel grades need to be compared, the same gas quench condition should be used to obtain the same quenched end HTC (difficult to control). However, for low hardenability steel, high gas quench HTC condition is needed to obtain martensite near the quenched end. For high hardenability steel, low gas quench HTC condition is needed to reveal its hardenability limitations. This is the fundamental limitations of Jominy test for gas quench that hardenability comparison among steels cannot be conducted especially for high hardenability steel.

2. Critical Heat Transfer Coefficient Test for Gas Quench Steel Hardenability

Modified Jominy gas quench steel hardenability test is demonstrated not to be useful. Therefore, the Grossmann-type test was considered.

Grossmann’s method of measuring hardenability uses a number of cylindrical steel rods of different diameters hardened in the quenching medium. After sectioning each bar at mid-length and examining it metallography, the bar that has 50% Martensite at its center is selected, and the diameter of this bar is designated as the critical diameter [1].

The critical HTC test is proposed based on Grossmann test [4]. In critical HTC test, cylinder samples with the same geometry are used (bar with 25mm in diameter and 100mm in length). The sketch is shown in Figure 6. The gas flow is assumed the same at the sample end and the sample side, since the slenderness ratio of sample is relatively large. Gas flow is fully-developed, turbulent annular flow. In this condition, the gas pressure and velocity are steady during gas quench condition. In the test, different gas quench HTC conditions (gas types and compositions, pressure and velocity) are applied to the sample with the same geometry. After sectioning each bar at mid-
length, the bar that has 50% martensite at its center is selected, and the applied gas quench HTC of this bar with 50% martensite is designated as the critical HTC.

![Figure 5 Grossmann hardenability [4]](image)

Figure 5 Grossmann hardenability [4]

Figure 6 A sketch of the critical HTC test

3. Experimental and analysis

3.1 Praxair gas quench system and sample design

Praxair gas quench system was selected as the prototype for critical HTC gas quench hardenability test. Figure 7 presents the schematic drawing of Praxair gas quench system. The steady gas flow is the advantage of the system. In the system, the heating and cooling curve at the center of the sample, gas pressure, gas mass flow rate and gas temperature can be monitored. Figure 8 is the sample drawing. The screw thread is machined at one end of the sample. Correspondingly, one end of the support rod is machined as well. The 304 stainless steel support rod thread can be used repeatedly.
3.2 Flow analysis in Praxair gas quench system

In critical HTC test, the HTC is considered as steady and uniform for the test bar. However, real outside gas quenching condition is fully developed turbulence in the gas quench chamber. In order to investigate the outside turbulence influence on the cooling rate at the center of the sample and whether constant gas quench HTC can be used to calculate HTC, Fluent were used to model gas quenching process [9]. Fluent is one of the most popular CFD software programs in the world. “The ANSYS Fluent package combines physics and years of simulation development expertise to
solve CFD challenges” [9]. Figure 9 presents the sketch of Praxair gas quench system. The model used in Fluent assumes the constant gas pressure and velocity at chamber inlet. When the gas velocity at the inlet is set as 10m/s, the location with the highest gas velocity is the edge of the test bar and the location with the lowest gas velocity is far end of the test bar.

Figure 9 Gas flow simulation based on ANSYS

Figure 10 Local HTC variation in critical HTC gas quench test

Figure 10 presents the local HTC variation in critical HTC gas quench test. It should be noted that the value of HTC is not accurate, however, large local HTC variation can be found at different locations on the test bar. In critical HTC test, the hardness measured at the location with the lowest cooling rate in the test represents the hardenability of the steel. The concern is whether the center of the test bar always has the lowest cooling rate, when the outside local HTC varies. Figure 11 presents the slowest cooling location movement during critical HTC test. During the whole quenching process, the slowest cooling location is always at the center of the test bar, although it slightly shifts to one end. It demonstrates that in the critical HTC test, the hardness measurement location to determine steel hardenability should be at the center, the location with the slowest cooling rate.
Figure 11 Slowest cooling location movement during critical HTC test

Figure 12 presents the AISI4140 cooling profile comparison between critical HTC test and simulation. The HTC is calculated based on Equation 1 [10]. When assigning boundary condition for simulation, the uniform HTC, not HTC complex distribution was used. The simulation fits the test result well. It demonstrates when mainly considering the core cooling rate and microstructure during gas quenching, uniform HTC can be used.
Based on the simulation, it is predicted that the critical HTC of AISI4140 steel under gas quench condition is 430 $W/m^2K$. A range of gas quench HTCs are selected in the experiment as shown in Table 1. The gas is nitrogen and gas temperature is room temperature. After the gas quench experiments, all the bars were cut to measure the center hardness. The result is shown in Figure 13 and Figure 14. With the increase of gas quench HTC, AISI4140 center hardness increases, since the cooling rate increases and more martensite and lower bainite form. The hardness of 50% martensite is 43 HRC [3]. After drawing a horizontal line which represents for 43 HRC, the horizontal ordinate of the intersecting point is the critical HTC of AISI4140, which is 430 $W/m^2K$. This is the first time that steel gas quench critical HTC was measured by experiment.
Figure 14 presents the AISI4140 critical HTC comparison between test and simulation. The simulated critical HTC is similar compared to the experimental critical HTC. In Figure 15, the measured cooling profiles are compared with AISI4140 CCT diagram. With the decrease of gas quench HTC, martensite decreases and lower bainite increases.

<table>
<thead>
<tr>
<th>HTC (W/m²°C)</th>
<th>Gas Pressure (Bar)</th>
<th>Gas Velocity (m/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>589</td>
<td>12.6</td>
<td>14.81</td>
</tr>
<tr>
<td>298</td>
<td>13.7</td>
<td>5.82</td>
</tr>
<tr>
<td>206</td>
<td>13.9</td>
<td>3.63</td>
</tr>
<tr>
<td>101</td>
<td>14</td>
<td>1.62</td>
</tr>
</tbody>
</table>

Table 1 AISI4140 gas quench HTC

For AISI52100 steel, the CHTE gas quench hardenability test follows the same procedures as AISI4140 steel. Figure 16 is the test result. The critical HTC for 52100 steel is 820 W/m²°C. It should be noted that the austenitizing temperature has influence on the gas quench hardenability, since the carbides are more easily to dissolve into the austenite at higher austenitizing temperature. For AISI52100, the carbides does not all dissolve into the austenite at 850°C until it reaches 1050°C [11]. The grain size increases dramatically when the austenitizing temperature increases. In all gas quench test, the austenitizing temperature should be recorded.

Figure 17 is the AISI52100 CCT diagram. T10, T11 and T12 cooling profiles are measured result. For T12, almost 100% martensite forms. For T11 and T10, the microstructure contains martensite and bainite. The cooling profiles are measured with thermocouple. The CCT diagram is generated by JmatPro [12].
Figure 15 AISI4140 CCT diagram

Figure 16 AISI52100 gas quench critical HTC test result

<table>
<thead>
<tr>
<th>Test number</th>
<th>Gas quench HTC (W/m²K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>T10</td>
<td>579</td>
</tr>
<tr>
<td>T11</td>
<td>788</td>
</tr>
<tr>
<td>T12</td>
<td>983</td>
</tr>
</tbody>
</table>

Table 2 Gas quench HTC of AISI52100 tests

Figure 17 AISI52100 CCT diagram

Figure 18 Optical microscopy of as-quenched sample T10. The microstructure contains of martensite (grey area), retained austenite (bright area) and bainite (dark area).
Figure 18, Figure 19 and Figure 20 present the microstructure of T10, T11 and T12. The grey area is as-quenched martensite. The dark area is bainite. The bright area may be carbides and retained austenite.

<table>
<thead>
<tr>
<th>Test number</th>
<th>Hardness (HRC)</th>
</tr>
</thead>
<tbody>
<tr>
<td>T10</td>
<td>43.6</td>
</tr>
<tr>
<td>T11</td>
<td>54.9</td>
</tr>
<tr>
<td>T12</td>
<td>63.1</td>
</tr>
</tbody>
</table>

Table 3 Hardness result for tests

Figure 19 Optical microscopy of as-quenched sample T11. The microstructure contains martensite (grey area), retained austenite (bright area) and bainite (dark area).

Figure 20 Optical microscopy of as-quenched sample T12. The microstructure contains martensite (grey area), retained austenite (bright area) and bainite (dark area).

When comparing the T10, T11 and T12 microstructures, the bright area increases and dark area decreases, which indicates that the percentage of as-quenched martensite increases. Higher cooling rate generates more martensite. Therefore, the sample with more martensite has higher hardness.

For Pyrowear53, very low gas quench $100 \, W/m^2\degree C$ is selected, because Pyrowear53 is a very high hardenability steel. After the test, the center hardness is measured as 31.5 HRC, which represents nearly 100% martensite [13].

Table 4 is the simulated critical HTC result for various steel grade. For low hardenability steels such as AISI4120 and AISI8620, the critical HTC is higher than $2000 \, W/m^2\degree C$, which beyond the highest HTC that current gas quench furnace could provide [5].
Table 4 Simulated critical HTC for different steels

<table>
<thead>
<tr>
<th>Steel</th>
<th>Critical HTC (W/m²°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>P53</td>
<td>&lt;100</td>
</tr>
<tr>
<td>S8680</td>
<td>130</td>
</tr>
<tr>
<td>S4150</td>
<td>137</td>
</tr>
<tr>
<td>S4340</td>
<td>162</td>
</tr>
<tr>
<td>S4140</td>
<td>430</td>
</tr>
<tr>
<td>S4047</td>
<td>373</td>
</tr>
<tr>
<td>S8650</td>
<td>503</td>
</tr>
<tr>
<td>S52100</td>
<td>820</td>
</tr>
<tr>
<td>S5140</td>
<td>1102</td>
</tr>
<tr>
<td>S4319</td>
<td>1172</td>
</tr>
<tr>
<td>S4120</td>
<td>2826</td>
</tr>
<tr>
<td>S8620</td>
<td>3098</td>
</tr>
</tbody>
</table>

Table 5 HTC and hardness result for gas quenching

Table 5 is the HTC and hardness result for gas quench. For AISI4140, different combination of gas pressure and velocity has the same HTC and the same hardness. Figure 21 is the cooling curves for AISI4140 part2-3 and part2-4. Although the gas pressure and velocity are different for part2-3 and part2-4, the cooling curve are very similar, since the HTC is the same. Figure 22 and Figure 23 present the microstructure for AISI4140 part2-3 and part2-4. The bright area is as-quenched martensite. The dark area appears to be bainite that formed at the prior austenite boundary. From the microstructure, the part2-3 sample and part2-4 sample are very similar. From the above analysis, the cooling curves, microstructure and hardness for 4140 part2-

4. Discussion

4.1 Gas pressure and velocity influence on gas quench hardenability test

Gas pressure and velocity can be adjusted easily in the test apparatus based on the requirements during gas quench process. However, there is no need to use gas pressure and velocity to indicate the gas quench intensity, because the different combination of gas pressure and velocity can have the same HTC. When considering gas quench intensity, the HTC of gas quench condition can be used instead of gas pressure and velocity. In order to demonstrate the same HTC, which is generated by different combination of gas pressure and velocity, would lead to the same cooling profile, microstructure and properties (hardness), AISI4140 and AISI52100 were tested.

Table 5 HTC and hardness result for gas quenching
3 sample and part2-4 sample are the same under the same HTC gas quench condition, although the combination of gas pressure and velocity are different.

![4140 Cooling Curves](image)

Figure 21 AISI4140 cooling curves for gas quench under the same HTC

![Optical microscopy](image)

Figure 22 Optical microscopy of as-quenched sample AISI4140 Part2-3. The microstructure contains of martensite (bright area) and bainite (dark area).

![Optical microscopy](image)

Figure 23 Optical microscopy of as-quenched sample AISI4140 Part2-4. The microstructure contains of martensite (bright area) and bainite (dark area).

Figure 24 is the cooling curve for AISI52100 under the same HTC condition. The cooling curve difference is due to the inaccuracy of being able to maintain constant mass flow for each test piece. In the manual operation, there is slight delay in mass flow over the quench time and speed in opening the valve to quench flow. This is the reason that the hardness for two parts is slightly different. If automated precise mass flow control were applied, the cooling profile would be the same.

Figure 25 and Figure 26 present the microstructure of AISI52100 part2-1 and part2-2R samples. The grey area is as-quenched martensite. The dark area appears to be bainite that formed at the prior austenite boundary. The bright spot may be carbides and retained austenite [11]. When
comparing Figure 25 and Figure 26, the microstructure is very similar. However, the percentage of dark area varies when choosing different filed of view under the microscope.

Figure 24 AISI52100 cooling curves for gas quench

Figure 25 Optical microscopy of as-quenched sample Part2-1. The microstructure contains of martensite (grey area) and bainite (dark area).

Figure 26 Optical microscopy of as-quenched sample Part2-2R. The microstructure contains of martensite (grey area) and bainite (dark area).

After testing AISI4140 and AISI52100 under the same HTC (different combination of gas pressure and velocity), it is obvious that the same type of steel would have the same/very similar cooling profile, microstructure and properties under the same HTC gas quench condition, although the gas pressure and velocity may be different.

4.2 Sample diameter influence on critical HTC test

In the above discussion, bar geometry is 25mm diameter and 100mm length for all the experiments. When changing the bar diameter, the cooling rates at the center of the bar will change and therefore the hardness.
Based on the model, which has been demonstrated to be accurate above, bars with different diameters have been gas quenched to find the critical HTC. Figure 27 is the simulation result. With the increase of the bar diameter, the critical HTC for steel 4140 increases. The critical HTC is 600 W/m²°C when 40mm diameter bar is applied and 270 W/m²°C when 20mm diameter bar is applied. Figure 28 presents the critical HTC for different sample dimensions when applied to different steels. The bar diameter we are using now is 25mm. The result indicates that too large or too small sample diameter is not proper for the critical HTC test.

If the sample diameter is too small, the difference of critical HTC for steels is not significant, which is difficult to determine the difference from medium and high hardenability steels. If the sample diameter (say 100mm) is too large, the critical HTC will be higher than the maximum HTC we can achieve.

**Figure 27** 4140: Bar diameter influence on critical HTC

**Figure 28** Critical HTC for different sample geometry

### 4.3 Comparison between critical HTC test and Jominy end-quench test

Critical HTC tests can be used to distinguish hardenability difference from different steel grades, such as between AISI4140 and AISI52100. Even for the same steel grade, the accurate chemical
composition varies and affects the hardenability. Critical HTC tests were conducted on AISI4140 from different heats to verify whether this test can distinguish not sever hardenability variations. A06379, NF1210 and M49335 were all AISI4140 steels from different heats. Table 6 provides the chemical compositions based on OES test.

<table>
<thead>
<tr>
<th>Steel</th>
<th>Grain Size</th>
<th>C</th>
<th>Mn</th>
<th>Mo</th>
<th>Cr</th>
<th>Si</th>
<th>Ni</th>
<th>Ideal Critical Diameter (inch)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A06379</td>
<td>6</td>
<td>0.407</td>
<td>0.94</td>
<td>0.21</td>
<td>0.967</td>
<td>0.239</td>
<td>0.074</td>
<td>5.79</td>
</tr>
<tr>
<td>NF1210</td>
<td>6</td>
<td>0.409</td>
<td>0.933</td>
<td>0.174</td>
<td>0.998</td>
<td>0.237</td>
<td>0.068</td>
<td>5.49</td>
</tr>
<tr>
<td>M49335</td>
<td>6</td>
<td>0.385</td>
<td>0.845</td>
<td>0.179</td>
<td>1.000</td>
<td>0.233</td>
<td>0.112</td>
<td>5.06</td>
</tr>
</tbody>
</table>

Table 6 Chemical compositions based on OES test

Samples from three heats were gas quenched at the same heat transfer coefficient in Praxair gas quench system to distinguish hardenability variation. Figure 29 presents the cooling curves at the center of each sample. Based on the thermocouple data acquired at the center of the samples, the cooling rates for all three samples are very similar.

Figure 29 Cooling curves comparison among three different heats

<table>
<thead>
<tr>
<th>Hardness (HRC)</th>
<th>M49335-1</th>
<th>NF1210-1</th>
<th>A06379-1</th>
</tr>
</thead>
<tbody>
<tr>
<td>HTC (W/m²°C)</td>
<td>436.5</td>
<td>436.5</td>
<td>436.5</td>
</tr>
</tbody>
</table>

Table 7 Center hardness of different heats

Table 7 presents the average center hardness of steels from different heats. Although all AISI4140 steels were quenched at the same condition, the center hardesses are different. The hardenability rank is **A06379>NF1210>M49335**. Even considering the little difference on cooling rate as shown in Figure 29, A06379 series steel has the comparative lowest cooling rate and highest hardness at the center, which gives more confidence in characterizing its highest hardenability among three
AISI 4140 steel grades. Critical HTC test has the capability to distinguish hardenability variation even for same steel grade from different heats.

In order to compare the Jominy end-quench test and critical HTC test, the same AISI 4140 samples were tested at Westmoreland Mechanical Testing & Research, Inc., followed by Specification A255-10. Figure 30 presents the result.

![Figure 30: Jominy end quench test for AISI 4140 from three different heats](image)

The hardenability rank is $\text{NF1210} > \text{A06379} > \text{M49335}$ based on Jominy end-quench test. However, the rank is $\text{A06379} > \text{NF1210} > \text{M49335}$ based on critical HTC test.

First, M49335 is always the lowest hardenability steel by two tests. During Jominy test process, the test bar is not only cooled by water from the quenched end, but also the air around. This leads to the inaccuracy result if the location is too far from the quenched end. From Figure 31, the cooling rate decreases intensively from the quenched end to 25mm location. At further location than 25mm, the cooling rate decreases slowly. If focused on the beginning of the hardness curve in Figure 30, the hardenability of NF1210 is the same as A06379 and M49335 is still the lowest. The rank should be $\text{A06379} = \text{NF1210} > \text{M49335}$. It demonstrates that compared to Jominy end-quench test, the critical HTC test can give the same/similar rank of steel hardenability. What’s more, critical HTC test have higher resolution to distinguish the small hardenability difference, such as NF1210 and A06379.

4.4 Computer-aided critical HTC test

Figure 32 presents the relationship between 50% martensite hardness and critical HTC. When the sample center hardness is close to 50% martensite hardness, either higher or lower, the relationship between hardness and gas quench HTC can be considered linear. Based on previous discussion, even the same steel grade has chemical composition variations and caused hardenability
difference. Accurate characterizing critical HTC for a selected steel is necessary, but difficult and time consuming. For any given steel, at least three or more tests have to be conducted. Considering the developed model to simulate gas quenching condition in this thesis, the concept of computer-aided critical HTC test is proposed to minimize the test amount. Figure 33 presents the computer-aided critical HTC test, which can reduce the test batch to one or two tests. Based on simulation, the relationship between hardness and gas quench HTC can be drawn as black solid line before experiment. The predicted critical HTC can be found at the points of intersection between HTC-hardness curve and 50% martensite hardness line. The first experiment can be conducted using this predicted critical HTC. The measured hardness is either higher or lower than 50% martensite hardness, not far away. Since the relationship between HTC and hardness is almost linear when the steel contains around 50% martensite, the modified simulation curves can be moved up and down and ensure the measured points lays on, as shown as green dotted line. The modified critical HTC, although it is still a prediction, should be very close or just the experimental critical HTC. The second test can be conducted using modified critical HTC. Only one or two tests are needed for computer-aided critical HTC test. Figure 34 presents the flow chart of computer-aided critical HTC test.

4.5 Advantages and limitations for critical HTC test

Critical HTC test has many advantages and is similar as real gas quench condition compared to Jominy end-quench test:
(1) No insulation is needed.
(2) The gas flow is steady and can be well controlled.
(3) High, medium and low hardenability steels can be tested in the same system.
(4) The sample geometry is simple and fixed.
Since HTC is used to replace the gas type, gas pressure and gas velocity, the test result is more repeatable. Even the same gas with same pressure and velocity, the cooling performance can be
different due to various gas flow patterns. Although the critical HTC concept and critical HTC test have many advantages, it still needs industry acceptance.

![Flow chart of computer-aided critical HTC test](image)

**Figure 34 Flow chart of computer-aided critical HTC test**

### 5. Conclusion

Critical HTC test for gas quench steel hardenability test is proposed based on Grossmann test. The bars, which are 25mm in diameter and 100mm in length, are quenched under different gas quench condition. After sectioning each bar at mid-length, the bar that has 50% martensite at its center is selected, and the applied gas quench HTC of this bar is designated as the critical HTC. 4140 and 52100 were tested based on critical HTC test. The critical HTC for 4140 is 430 W/m²°C, for 52100 is 820 W/m²°C. The result shows that 4140’s gas quench hardenability is better than 52100. The steel, quenched under the same HTC, which is generated by different combination of gas pressure and velocity, is proved to have the same cooling curves, same microstructure and same mechanical properties (hardness). Gas quench HTC can be used to indicate gas quench condition instead of gas pressure and velocity. The sample geometry effect on critical HTC, sensitivity and computer-aided test method are also discussed. Currently, the sample geometry is 25mm in diameter and 100mm in length. The critical HTC test is proved to test gas quench steel hardenability successfully.

**Acknowledgments**

The authors acknowledge members in CHTE, Worcester Polytechnic Institute who give valuable suggestion that help to find the limitation of Jominy test for gas quench. The work was supported from Center of Heat Treating Excellence, Worcester Polytechnic Institute.
References:


CHAPTER 5  Paper III: Evaluation on heat transfer coefficient distribution in gas quench furnace

Yuan Lu, Richard D. Sisson, Jr.

To be submitted to: Materials Science and Engineering: A

Highlights:
1. Propose a method to evaluate heat transfer coefficient distribution in gas quench furnace.
2. Compare HTC distribution in 2bar nitrogen furnace and 20bar helium furnace.
Evaluation on heat transfer coefficient distribution in gas quench furnace

Yuan Lu, Richard D. Sisson, Jr.

Center of Heat Treating Excellence, Worcester Polytechnic Institute

Abstract: Gas quenching is widely used in aerospace industry when quenching medium or high hardenability steels. High pressure and velocity gas flow induce very complex flow pattern in the furnace. The heat transfer coefficient distribution is localized and affect the cooling rate, microstructure and mechanical properties of working parts. In this paper, a method, using bars with proper geometry and steel grades, to evaluate heat transfer coefficient distribution in gas quench furnace, is proposed. The relationship of the measured hardness at the core of the samples and the outside HTC was built by the authors previously. Using this relationship, by simply measuring the core hardness, the HTC distribution in the gas quench furnace can be mapped. It was verified that 0.5” diameter and 4” length AISI 4340 bar can be used to evaluate 2bar nitrogen gas quench furnace. An attempt to measure 20bar helium gas quench furnace was made as well. Finally, a basic test procedure was proposed.

Keywords: Gas quenching, Heat transfer coefficient, critical HTC test

1 Introduction

Gas quenching is widely used and has great potential to replace water and oil quenching [1] [2]. Its advantages include reducing distortion and stress, environmental friendly and leave dry and clean part after quenching [3]. Although the heat transfer coefficient of gas is a constant during quenching, if ignoring the little temperature variation at gas inlet and outlet [4,5], the HTC (heat transfer coefficient) distribution in furnace varies dramatically at different locations [6] [7] [8]. Several modelling works were focused on using flow modeling to simulate gas quenching HTC distribution [7]. Besides modelling work, experimental measurement on HTC distribution becomes essential to validate the model. Cosentino used thermocouple to measure the cooling rate of samples in the gas quench system [9]. A standard [10] to evaluate furnace uniformity applies the similar method. Using thermocouple is accurate, but has complicated set-up. Based on authors’ previous research on the relationship between hardness at the core of the samples and the outside HTC [11], it is possible to simply measure the hardness and map the HTC distribution in the furnace. As shown in Figure 1, 1” diameter and 4” length AISI 4140 bar was using in the test. When applying different outside HTC, the core hardness varies.

2 Slowest cooling rate modelling

In this paper, it is assumed that although outside HTC distribution is complex, the core of the samples always has the slowest cooling rate, considering the scale of HTC and heat conductivity of steels. HTC simulation was conducted in gas quench furnace using Fluent [12]. As shown in Figure 2 and Figure 3, constant gas quench pressure and velocity were set at the gas inlet.
The gas quench furnace is 2000mm in diameter and 2000mm in height/length. The test bar is 50mm in diameter and 50mm in length. One test bar is located at the center of the gas quench chamber. This simulation is only to determine whether the cooling rate at the center of the test bar is the slowest during the whole gas quenching process. Figure 4 presents that the slowest cooling location is fixed at the center of the sample although the outside HTC is not uniform. This simulation demonstrates the core of samples always has the slowest cooling rate. By only analyze the core hardness, the outside average estimated HTC error could be minimized.
3 Proposed gas quench furnace evaluation procedure

The basic concept is to put test bars at different locations in the gas quench furnace. By correlate the core hardness and outside HTC after quenching, the HTC map can be measured. The following procedure is shown below:
1. **Gas Quench HTC Calculation** - The gas quench HTC is calculated based on equation [14].

\[
h = \frac{k}{l} \cdot 0.023 \left( \frac{P V L}{\mu Z R T} \right)^{0.8} \left( \frac{\mu C_p}{k} \right)^{0.33}
\]

Equation 1

- \(h\): heat transfer coefficient, \(w/m^2K\)
- \(P\): gas pressure, \(Pa\)
- \(V\): gas velocity, \(m/s\)
- \(L\): specify characteristic length (part diameter), \(m\)
- \(\mu\): dynamic viscosity, \(kg/ms\)
- \(Z\): gas compressibility and density
- \(R\): gas constant, \(J/Kmol\)
- \(T\): gas temperature, \(K\)
- \(C_p\): gas specific heat, \(J/kgK\)
- \(k\): thermal conductivity, \(W/mK\)

2. **Select proper sample** – Using Dante to simulate center hardness of AISI4140 and AISI4340 with 0.5”, 1”, 1.5” and 2” under different furnace HTC, from 50-2000W/m²°C. Select proper steel grade and diameter to ensure the center hardness is between 50% (42.2HRC) to 90% (50.0HRC) martensite hardness [3].

3. **Sample preliminary test** - Place the sample in the furnace with the dummy thermal load using desired gas quenching process. After quenching, measure the center hardness of the sample. If the hardness is between 42-51HRC, use this steel grade and diameter. Otherwise, select proper steel grade and diameter.

4. **Normalizing** – After select proper steel grade and geometry for specific gas quench furnace, all the specimen shall be normalized to ensure proper hardening characteristics. The sample shall be held at the temperature listed in Table 1 for 1h and cooled in air. Tempering of the normalized sample to improve machinability is permitted.

5. **Heating** – Place the specimen at desired location in the gas quench furnace at the specified austenitizing temperature (Table 1) and hold at this temperature for certain time. Figure 5 presents the sample locations in one type of gas quench furnace.

6. **Quenching** – Adjust the gas quench furnace to generate different gas quench HTC conditions (different gas pressure and velocity). Record the gas temperature and ensure the specimen is fully cold when removed from the furnace.

7. **Hardness Measurement** – The specimen shall be cut in the middle and Rockwell C hardness measurements should be made at the center hardness measuring point. The thickness of disk cut from the specimen should be no less than 0.5 inch.

8. **HTC modelling** - Based on the database developed with critical HTC test [15] or DANTE [16], determine HTC distribution in the furnace. In Figure 6, the specimen is AISI4140 with 1inch diameter and 4inch length. If this specimen is being used, the measured center hardness 43HRC represents outside HTC is 430 W/m²°C.
Figure 5 Sample locations in one type of gas quench furnace

Figure 6 4140 gas quench critical HTC test

<table>
<thead>
<tr>
<th>Steel Series</th>
<th>Ordered Carbon Content, max, %</th>
<th>Normalizing Temperature, °F (°C)</th>
<th>Austenitizing Temperature, °F (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1000, 1300, 1500, 3100, 4000, 4100, 4200, 4400, 4500, 4600, 4700, 5000, 5100, 6100, 8100, 8600, 8700, 8800, 9400, 9700, 9800</td>
<td>0.25 and under</td>
<td>1700 (925)</td>
<td>1700 (925)</td>
</tr>
<tr>
<td></td>
<td>0.26 to 0.36, incl</td>
<td>1650 (900)</td>
<td>1600 (870)</td>
</tr>
<tr>
<td>2500, 2500, 3500, 4800, 9300</td>
<td>0.37 and over</td>
<td>1600 (870)</td>
<td>1550 (845)</td>
</tr>
<tr>
<td></td>
<td>0.25 and under</td>
<td>1700 (925)</td>
<td>1550 (845)</td>
</tr>
<tr>
<td></td>
<td>0.26 to 0.36, incl</td>
<td>1650 (900)</td>
<td>1500 (815)</td>
</tr>
<tr>
<td></td>
<td>0.37 and over</td>
<td>1600 (870)</td>
<td>1475 (800)</td>
</tr>
<tr>
<td></td>
<td>0.50 and over</td>
<td>1650 (900)</td>
<td>1600 (870)</td>
</tr>
</tbody>
</table>

Table 1 Normalizing and Austenitizing Temperatures [3]

4 Evaluation of 2bar nitrogen gas quench furnace

4.1 Determine gas quench probe design by experiment and simulation

First, 1” diameter and 4” length AISI 4140 bars were tested in 2bar nitrogen gas quench furnace. The thermocouples and sample locations in the furnace were presented in Figure 7 and Figure 8. A hole was drilled from one end to the center longitudinally for temperature measurement, and the other hole was drilled axially at the other end for sample fixing. The steel wires were used to fix the sample during gas quenching process. For AISI 4140 steel, the cooling rate is not high enough to form martensite. All the core hardness of test bars is lower than 34HRC, which also indicates no martensite forms during quenching. As seen from Figure 9, the black line represents cooling curves in experiment. A clear phase transformation happens around 430°C, which is in the bainite transformation temperature range. Different heat transfer boundary conditions were simulated with
DANTE [17], in order to fit cooling curve in experiment. When the HTC is 40 W/m$^2$C, the cooling curve in simulation fits with experiment. It demonstrates the real HTC during experiment is around 40 W/m$^2$C.

To obtain martensite during gas quenching, two approaches may work. One is to reduce the sample geometry, the other is to change to higher hardenability steel. The blue line in Figure 9 presents the cooling curve when the sample geometry is 0.5inch in diameter and 4inch in length. Although higher cooling rate is achieved, no martensite is formed yet. Figure 10 presents the Jominy hardenability for AISI 41XX series steels. AISI 4340 is a good candidate for gas quenching. When the cooling rate is as low as 2$^\circ$C/s, it still contains 80% martensite and the hardness is 50HRC. The cooling rate in gas quench furnace is around 1.5$^\circ$C/s - 2$^\circ$C/s (from 800$^\circ$C to 500$^\circ$C). Based on the simulation AISI 4340 0.5” test bar in gas quench furnace may form 15% martensite at the center.

---

**Figure 7** Sample and thermocouple locations in the furnace

**Figure 8** Sample geometry for experiments (unit: inch)

**Figure 9** AISI 4140 Gas quenching experiment and simulation

**Figure 10** Jominy hardenability for AISI 41XX series steels [18]
4.2 0.5” AISI 4340 gas quench intensity and uniformity experiment

0.5” diameter and 4” length AISI 4340 bars were tested in 2bar nitrogen furnace. The core hardness varies from 44HRC to 49.5HRC, which contains 50% to 90% martensite [3]. From Figure 11, Figure 12 and Figure 13, the front side provides higher gas quench intensity compared to rear side. The weakest quench intensity appeared at lower left corner of rear side and the strongest quench intensity appeared at right side of the front side. Based on the measured hardness, simulation was made by DANTE to get the outside HTC, as shown in Figure 14 and Figure 15. The outside HTC distribution presents that uneven gas flow in the furnace. It should be noted that only using DANTE to simulate outside HTC is not highly accurate, because even for the same steel grade, different chemical composition variation and initial microstructure would strongly affect the phase transformation. In order to get accurate outside HTC, the critical HTC test should be performed with the steel bars from the same heat. The outside HTC is calculated based on lumper HTC method, which not only considers gas type, pressure, velocity and temperature, but also radiation from thermal dummy load. When comparing the gas intensity of different gas quench furnace, gas type, pressure and velocity, as well as weight of the thermal load should be considered. When using same gas pressure and velocity configurations, small load furnace has better cooling performance compared to high volume load furnace. Overall, 0.5” AISI 4340 test bar proved appropriate to quantify quench intensity and uniformity in the 2bar nitrogen gas quench furnace.

![Figure 11 Furnace gas flow direction](image)

![Figure 12 Hardness distribution of front side](image)

![Figure 13 Hardness distribution of rear side](image)
5 Evaluation of 20bar helium gas quench furnace

5.1 1” diameter AISI 4140 gas quench intensity experiment

Besides 2bar nitrogen gas quench furnace, 20bar helium cold chamber gas quench furnace is also widely used in the industry. 1” AISI 4140 test bar were put in 20bar helium gas quench furnace with dummy thermo load to verify whether it can be fully hardened. Figure 16 presents the hardness of AISI 4140 after gas quenching in the furnace. 1” bar was sliced to several pieces for measurement. The average hardness is 54.9HRC, similar to oil-quenched AISI 4140 hardness. The standard deviation is 1.1. It demonstrates that 20bar helium gas quench furnace has the capability to fully hardened 1” AISI 4140 bars. Compared to water quench and oil quench, the error bar of gas quenched AISI 4140 is larger. This may due to unsteady outside HTC during gas quenching.

5.2 1” diameter AISI 4140 experiment gas quench uniformity experiment under different quench intensity

1” diameter AISI 4140 bars were used in 20bar helium gas quench furnace. 13 samples were used in each batch as shown in Figure 17, Figure 18 and Figure 19. The flow pattern is from top to bottom. The heat treatment process is preheating, austenitizing to 843C for 40mins then gas
quenching with maximum gas pressure and 100% (batch1), 75% (batch2) and 50% (batch3) gas velocity. For batch1 with maximum gas pressure and 100% gas velocity, middle layer hardness was analyzed. The average core hardness is 53.9HRC. For batch2 with maximum gas pressure and 75% gas velocity, middle layer hardness was also analyzed. The average core hardness is 52.4HRC. Compared to AISI 4140 oil quenched hardness 54.8HRC, hardness from batch1 and batch2 are similar.

All the core hardness in batch3 were analyzed as shown in Figure 20, Figure 21 and Figure 22. The average core hardness is 53.4HRC. Comparing three layers, top layer hardness is around 2HRC higher than middle and bottom layer. Comparing to 2bar nitrogen furnace, 20bar helium cold chamber furnace provides enough quench intensity for 1” 4140 steels even only applying maximum gas pressure and 50% gas velocity. 20bar furnace also shows good quench uniformity under maximum gas pressure and 75% or 100% gas velocity. Slightly higher hardness at top layer may be due to gas flow direction from top to bottom. Cold helium gas first quenches top layer. Via the parts laid at top layer, the gas temperature increases and causes quench intensity drop. Although gas velocity is as high as 20m/s, it still may cause hardness and other mechanical properties variation at different location in the furnace.

All the hardness is higher than hardness from microstructure containing 90% martensite [3]. Corresponding HTC modeling is only accurate when the hardness from 42.4HRC (50% martensite) to 50HRC (90% martensite). 1.5” or 2” diameter AISI 4140 test bar might be helpful to distinguish quench uniformity in detail for 20bar helium gas quench furnace more accurately.
6 Conclusion

After Fluent analysis, it was proved that the core of the samples has the slowest cooling rate during gas quenching, when the outside HTC distribution is complex. Based on the proposed gas quench furnace evaluation procedure, 2bar nitrogen and 20bar helium gas quench furnaces were evaluated. 0.5” diameter and 4” length AISI 4340 test bar was proved proper to evaluate 2bar nitrogen gas quench furnace. The HTC mapping show the uneven distribution in the furnace. 20bar helium gas quench shows better quench intensity and uniformity compared to 2bar nitrogen gas quench furnace. 1.5” or 2” diameter AISI 4140 is recommended to distinguish quench uniformity for 20bar helium furnace.

Acknowledgments

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References:


CHAPTER 6  Paper IV: Lattice parameter and the tetragonality of as-quenched martensite in steels

Yuan Lu, Haixuan Yu, Richard D. Sisson, Jr.

To be submitted to: Scripta Materialia

Highlights:
1. Using high resolution X-ray diffraction and Rietveld refinement to measure and analyze c/a ratio of as-quenched martensite in steel
2. Modify the classical relationship between c/a and carbon content to: \( c/a = 1 + 0.031 \text{ wt}\% \text{ C} \)
Lattice parameter and the tetragonality of as-quenched martensite in steels

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Abstract: The as-quenched martensite crystal structure is widely accepted to be body-centered tetragonal (BCT). With the increase of carbon content, the c/a ratio increases, following a linear relationship. However, it has been pointed out by Sherby [1] that a steel with less than 0.6 wt% carbon has body-center cubic (BCC) structure and c/a ratio equals to 1. AISI 9310, AISI 4140, AISI 4150, AISI 4161, AISI 1080 and AISI 52100 were austenitized and quenched to form martensite. The lattice parameter was determined by X-ray diffraction with Rietveld refinement. The result shows that for the steel with less than 0.6 wt% carbon, the structure is BCT, however, $c/a=1+0.031\text{wt}\% C$. The slope is smaller compared to traditional c/a model, $c/a=1+0.045\text{wt}\% C$.

Keywords: martensite, X-ray Diffraction, lattice parameter, c/a ratio, Rietveld Refinement, quench

1 Introduction

Martensite is one of the most important microstructures in steels, to provide high strength and wear resistance. However, the structure of martensite, especially for low carbon steel, is still debated. From Adolf Karl Gottfried Martens [2], intensive work has been conducted, focusing on the crystal structures and morphologies of martensite by researchers, including George Krauss [3], Harry Bhadeshia [4] and S. Morito [5]. It is widely accepted that martensite is body center tetragonal. The tetragonality of the martensite, measure by X-ray diffraction, c/a, linearly increases with carbon percentage:

Equation 1 $c/a = 1 + 0.045\ \text{wt}\% \ C$ [4]

However, this relationship is based on data that the carbon content higher than 0.6 wt %. Sherby [1] determined that the c/a ratio relationship was not linear at concentration below 0.6 wt% carbon after extensive review. Based on this determination, Sherby proposed a phase transformation of BCC to HCP to BCT martensite transformation process, different than widely accepted BCC to BCT martensite transformation for carbon steels and low or medium alloy steels [6]. This hypothesis is based on BCC martensite structure when the carbon content is less than 0.6 wt%. The author measured the references by Campbell, Fink and Kurdumoff listed in Sherby’s paper.
Sherby reported that for steels with less than 0.6 wt%, they could not identify the peak split and tetragonal structure [1]. However, compared to X-ray diffractometer in 1930s, the modern X-ray diffractometer has a higher resolution. The PANalytical Empyrean X-ray diffractometer was used in this paper. It has ultra high resolution, with a FWHM of 0.026 degrees 2θ for the first reflection of NIST SRM660a LaB$_6$ [8].

2 Experimental

In order to get better understanding of martensite structure, steels with various carbon contents from 0 wt% to 1.3 wt% were investigated by modern high resolution X-ray diffractometer. AISI 9310, AISI 4140, AISI 4150, AISI 4161, AISI 1080 and AISI 52100 were selected to re-measure the c/a ratio of as-quenched martensite. Each sample was cut into a 25mm diameter and 50mm length disc. Before water quenching, all the steels were normalized. The austenitizing temperature and holding time for these steels are listed in Table 1.

<table>
<thead>
<tr>
<th>Steel</th>
<th>Austenitizing Temperature [°C]</th>
<th>Holding Time [h]</th>
</tr>
</thead>
<tbody>
<tr>
<td>9310</td>
<td>850/950</td>
<td>1</td>
</tr>
<tr>
<td>4140</td>
<td>850/950</td>
<td>1</td>
</tr>
<tr>
<td>4150</td>
<td>950/1050</td>
<td>1</td>
</tr>
<tr>
<td>4161</td>
<td>950/1050</td>
<td>1</td>
</tr>
<tr>
<td>1080</td>
<td>950/1050</td>
<td>1</td>
</tr>
<tr>
<td>52100</td>
<td>1050/1150</td>
<td>1</td>
</tr>
</tbody>
</table>

Table 1 Heat treating parameters for steels

Two austenitizing temperatures were selected for each steel grade, in order to investigate the effect of austenitizing temperature and grain size on the martensite structure. The samples were placed into the furnace at the austenitizing temperature. After 1h, the samples were taken out of the furnace and quenched in 20°C stagnant water within 5 seconds. To prevent oxidation and decarburization, high-temperature protective coatings were used. Each sample was cut by Mark V CS600 specimen saw. The core of the samples was used for characterization. Chemical compositions were measured by Optical Emissions Spectrometer (OES) on the SPECTROMAXx. Phase identification and lattice parameter measurement were conducted by PANalytical Empyrean 2 X-ray diffractometer (XRD) with Cr-Kα radiation after electropolishing. Rockwell C hardness was measured using equipment from Stanley P. Rockwell Co. The samples were ground, polished and etched with 4% nitric solution. A Nikon optical microscope was used to obtain optical microscopy and JEOL JSM-7000F Scanning Electron Microscope (SEM) was used for higher magnification characterizations.

3 Result: OES and XRD

After heat treating, the OES analysis was conducted at the core of all the samples. Table 2 presents the measured chemical compositions of steels. For the same steel grade, such as AISI 9310, the chemical compositions did not vary with austenitizing temperature. It is clear that accurate
measured carbon content is not the same as the steel grade. For AISI 52100, the specified carbon content is 1 wt%, however, the measured carbon content is 1.24 wt%. When plotting the relationship between carbon content and c/a ratio, the measured carbon content was used. Figure 1 presents the X-ray Diffraction (XRD) pattern of AISI 1080 steel quenched from 950°C and 1050°C. α’ (002) and α’ (200) two peaks from BCT structure can be clearly observed. Although the AISI 1080 was quenched from different austenitizing temperature, the XRD patterns are the same. Similar conditions were observed for all the other steels. This observation can be a sign of all the alloying elements has been totally dissolved into austenite. After the austenite is homogeneous, continuing increasing austenitizing temperature will only increase austenite grain size. The Rockwell C hardness of AISI 4140 from 950°C quenching is lower than from 850°C, which also demonstrates that the increase in austenite grain size causes hardness to decrease.

<table>
<thead>
<tr>
<th>wt%</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Cr</th>
<th>Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td>9310 850C</td>
<td>0.124</td>
<td>0.254</td>
<td>0.63</td>
<td>1.16</td>
<td>3.07</td>
</tr>
<tr>
<td>9310 950C</td>
<td>0.123</td>
<td>0.248</td>
<td>0.63</td>
<td>1.15</td>
<td>3.07</td>
</tr>
<tr>
<td>4140 850C</td>
<td>0.396</td>
<td>0.225</td>
<td>0.92</td>
<td>0.86</td>
<td>0.09</td>
</tr>
<tr>
<td>4140 950C</td>
<td>0.406</td>
<td>0.225</td>
<td>0.93</td>
<td>0.86</td>
<td>0.09</td>
</tr>
<tr>
<td>4150 950C</td>
<td>0.449</td>
<td>0.228</td>
<td>0.92</td>
<td>1.02</td>
<td>0.152</td>
</tr>
<tr>
<td>4150 1050C</td>
<td>0.449</td>
<td>0.229</td>
<td>0.93</td>
<td>1.03</td>
<td>0.153</td>
</tr>
<tr>
<td>4161 950C</td>
<td>0.56</td>
<td>0.169</td>
<td>0.98</td>
<td>0.81</td>
<td>0.191</td>
</tr>
<tr>
<td>4161 1050C</td>
<td>0.57</td>
<td>0.169</td>
<td>0.97</td>
<td>0.81</td>
<td>0.189</td>
</tr>
<tr>
<td>1080 950C</td>
<td>0.76</td>
<td>0.271</td>
<td>0.83</td>
<td>0.183</td>
<td>0.069</td>
</tr>
<tr>
<td>1080 1050C</td>
<td>0.77</td>
<td>0.272</td>
<td>0.82</td>
<td>0.185</td>
<td>0.068</td>
</tr>
<tr>
<td>52100 1050C</td>
<td>1.26</td>
<td>0.243</td>
<td>0.397</td>
<td>1.59</td>
<td>0.052</td>
</tr>
<tr>
<td>52100 1150C</td>
<td>1.24</td>
<td>0.243</td>
<td>0.399</td>
<td>1.59</td>
<td>0.053</td>
</tr>
</tbody>
</table>

Table 2 Chemical composition of the steels used in the study

![XRD Patterns of AISI1080 Quenched from 950°C and 1050°C](image)

Figure 1 XRD pattern of AISI 1080 quenched from 950°C and 1050°C
Auto-tempering at room temperature is another issue when accurately measuring martensite c/a ratio. AISI 52100 samples after water quenching and water quenching + liquid nitrogen cryogenic treatment were measured and compared as shown in Figure 2. The XRD experiments were conducted within 30 minutes after the samples were water quenched or cryogenic treated. From the XRD patterns, after cryogenic treatment, the amount of retained austenite decreases from 20.1% to 13.2%. The c/a ratio of martensite, as seen from $\alpha'$ (002) and $\alpha'$ (200), stays the same. Villa and Somers observed slight increment in c/a of AISI 52100 after cryogenic treatment, due to newly formed martensite [9]. Since the increment is rather small, c/a ratio from as-quenched martensite at room temperature was used in this analysis.

Figure 2 XRD patterns of AISI 52100 steels before and after cryogenic treatment

Figure 3 presents the XRD patterns of the as-quenched steels. As the carbon content increases from AISI 9310 to AISI52100 steels, the martensite tetragonality increases. The $\alpha'$ (002) and $\alpha'$ (200) peaks split when the carbon content increases. $\gamma$ (111) peaks moves to a lower angle and the intensity increases. The increase of austenite peak intensity indicates that the fraction of retained austenite is increasing and the peak shifts indicate carbon content of retained austenite is also increasing. With the increase of carbon content, the $\alpha'$ (101) martensite peaks shifts to a lower angle, indicating larger lattice parameter and tetragonality. Although martensite has BCT structure, the slightly distorted c/a is not easy to measure. From Figure 3, for low carbon steel, the $\alpha'$ (002) and $\alpha'$ (200) peaks are highly overlapped. For high carbon steel, $\alpha'$ (002), $\alpha'$ (200) and $\gamma$ (111), peaks are overlapped, which gives great difficulty to accurately measure and calculate c/a ratio.
4 Results: hardness and microstructure

Rockwell C hardness test as also measured. The relationship between carbon contents and as-quenched hardness is shown in Figure 4. The relationship is linear until the carbon content exceeds 0.6 wt%. The hardness is very similar to hardness of as-quenched microstructure from references [1].
Figure 5 presents the optical micrography for the different steel grades. Low carbon steels, including AISI 9310, AISI 4140, AISI 4150 and AISI 4161, presented lath martensite morphologies. Higher carbon steels, including AISI 1080 and AISI 52100, presented plate and lath martensite morphologies. The morphologies of martensite under high magnification are presented in Figure 6. With the increase of carbon content, the amount of lath martensite decreases and the amount of plate martensite increases.

Figure 5 Optical microscopy of as-quenched microstructure
To accurately measure the c/a ratio, Rietveld refinement is used. "It uses a least squares approach to refine a theoretical line profile until it matches the measured profile" [10]. Using this technology devised by Hugo Rietveld, the most accurate lattice parameters for martensite can be determined. HighScore Plus 4.1 developed by PANalytical was used to conduct Rietveld refinement analysis for martensite crystal structure [11]. Figure 7 presents the peaks of AISI 4150 martensite. In Figure 7, the black dots are the experimental result and red line is the refinement result. If treating...
martensite as BCC structure, the peak shape should be symmetry without left hand tail. The residual error between experiment and refinement is 12% when treated martensite as BCC. When treating martensite as BCT structure, martensite peak (002) gives the same left hand tail as experiment. The residual error drops to 5%. For AISI 4150 and other low carbon steel, although only one peak shape is observed, the left hand tail represents that the structure is BCT, not BCC.

Figure 7 AISI 4150 martensite peaks (002) and (200)

Figure 9 presents the lattice parameter for martensite in as-quenched Fe-C alloys. The measured lattice parameter of a and c axis are smaller than Bain’s measurement (classical model). Figure 8 presents the c/a ratio for martensite in as-quenched Fe-C steels as a function of carbon content. The black solid line represents classic model developed by Honda, Nishiyama and many other researchers. The blue dots were measured c/a results by the author. The blue dotted line is the linear fit. The red dotted line is the reference from atomistic modeling result [12]. Compare to the traditional model, the modified model has also the linear relationship between carbon content and c/a ratio with smaller slope. The equation is:

\[ \frac{c}{a} = 1 + 0.031 \text{ wt}\% \text{ C} \]

Considering the classic model, the slope 0.031 is 31% smaller than the slope 0.045. This difference may be from chemical composition and c/a measurement inaccuracy. With modern OES machine and X-ray diffractometer, 0.031 slope may be considered more accurate. Compare to the Sherby’s model, the c/a ratio is not 1 when the carbon content is less than 0.6 wt%. The reason that Campbell, Fink and Kurdumoff, G. considered c/a ratio as 1, is lack of more accurate X-ray diffractometer and Rietveld refinement with related software to deconvolute highly overlapped peaks from one observed peak.
6 Discussion

In this paper, it was considered that the carbon content in martensite is the same as the carbon content measured by OES. However, even during the quenching process, carbon repartitioning from martensite lath/block to lattice defects and thin austenite film [13]. Becquart, Sherman and Morsdorf’s research on atom probe tomography has shown carbon depletion in martensite lath and carbon enrichment in austenite film [13-15].

The new relationship between martensite c/a ratio and wt% C can be used to estimate carbon content in as-quenched martensite and tempered martensite by determining the c/a ratio. This could also help to develop more accurate hardness and other mechanical properties model based on martensite structure.

Acknowledgement

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References:


CHAPTER 7  Paper V: Microstructure and mechanical properties comparison on AISI4140 and Pyrowear53 after gas and liquid quenching

Yuan Lu, Haixuan Yu, Xiaoqing Cai, Richard D. Sisson, Jr.

To be submitted to: Metallurgical and Materials Transactions A

Highlights:
1. Compare microstructure, hardness and Charpy impact toughness of AISI 4140 and Pyrowear 53 after water, oil and gas quenching. The relationship between Charpy impact toughness and cooling rate was analyzed.
2. For AISI 4140, Charpy impact toughness increases when the cooling rate decreases during quenching. Water quenching and tempering provides the ultimate Charpy impact toughness, compared to oil and gas quenching. Austenite percentage and carbon content in austenite is proposed as the dominated mechanism.
3. For Pyrowear53, Charpy impact toughness decreases when the cooling rate decreases during quenching. Oil quenching and tempering provides the ultimate Charpy impact toughness, compared to water and oil quenching. Carbides is proposed as the dominated mechanism.
Microstructure and mechanical properties comparison on AISI4140 and Pyrowear53 after gas and liquid quenching

Yuan Lu, Haixuan Yu, Xiaoqing Cai, Richard D. Sisson, Jr.

Center of Heat Treating Excellence, Worcester Polytechnic Institute

Abstract: Gas quenching has potential to replace water and oil quenching for medium and high hardenability steels. It can reduce distortion, leave clean and dry parts after quenching and environmental friendly. It was assumed that as long as the hardness are similar after quenching, the microstructure and mechanical properties are similar as well. This is not the case for medium hardenability steel, especially for high hardenability steel, when changing from oil quenching to gas quenching. In this paper, the microstructure and mechanical properties on AISI 4140 and Pyrowear 53 were compared after liquid and gas quenching. For AISI 4140, Charpy impact toughness increases when the cooling rate decreases during quenching. Water quenching and tempering provides the ultimate Charpy impact toughness, compared to oil and gas quenching. Austenite percentage and carbon content in austenite is proposed as the dominated mechanism. For Pyrowear53, Charpy impact toughness decreases when the cooling rate decreases during quenching. Oil quenching and tempering provides the ultimate Charpy impact toughness, compared to water and oil quenching. Carbides is proposed as the dominated mechanism.

Keywords: Gas quenching, Charpy impact toughness, austenite thin film

1 Introduction

Gas quenching is popular, especially in aerospace and automobile industry when using medium and high hardenability steels [1]. Gears, which has high geometry and dimension requirement, used to have distortion using oil quenching [1]. Gas quenching has great potential to reduce distortion and stress if gas parameters, including gas pressure, velocity and flow patterns, and parts layout are carefully adjusted [2-6]. Extensive work has been carried on gas flow pattern [7-10], however, few research on microstructure and mechanical properties, especially Charpy impact toughness has been conducted.

Lerchbacher [11-14] conducted a series work on cooling rate effect on microstructure, hardness and Charpy impact toughness for a hot-work tool steel X38CrMoV5-1. Although the cooling process is not gas quenching, the absolute cooling rate is comparable to gas quenching. He found that significant carbon segregation to dislocations and cluster formation after quenching [11]. The thickness and the carbon concentration of thin austenite film increases with the decreases of cooling rate, which affect Charpy impact toughness, not hardness [11]. Carbon enrichment in thin austenite film is also observed by Sherman [15] and Morsdorf [16].

When replacing oil quenching to gas quenching for medium and high hardenability steels, it is possible that the thickness and carbon concentration of austenite film increases, which cause the decreases of Charpy impact toughness. AISI 4140, a typical medium hardenability steel, and Pyrowear 53, a typical high hardenability steel, were selected in this paper to investigate the microstructure and mechanical properties difference after liquid and gas quenching.
2 Experimental

AISI 4140 and Pyrowear 53 were selected in this paper. AISI 4140 chemical composition is shown in Table 1. Sample geometry is 25mm in diameter and 100mm in length. Samples were preheated, austenitized, quenched and tempered. The detailed heat treat process is shown in Table 2. Quenching in gas use 20bar helium gas quench furnace.

<table>
<thead>
<tr>
<th>Batch</th>
<th>Pre-treat</th>
<th>Austenitizing</th>
<th>Quenching</th>
<th>Tempering</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>300°C, 1h</td>
<td>843°C, 1h</td>
<td>Gas, 18bar, 100% velocity</td>
<td>-</td>
</tr>
<tr>
<td>2</td>
<td>300°C, 1h</td>
<td>843°C, 1h</td>
<td>Gas, 18bar, 75% velocity</td>
<td>-</td>
</tr>
<tr>
<td>3</td>
<td>300°C, 1h</td>
<td>843°C, 1h</td>
<td>Gas, 18bar, 50% velocity</td>
<td>-</td>
</tr>
<tr>
<td>4</td>
<td>-</td>
<td>843°C, 1h</td>
<td>Water</td>
<td>-</td>
</tr>
<tr>
<td>5</td>
<td>-</td>
<td>843°C, 1h</td>
<td>Oil</td>
<td>-</td>
</tr>
<tr>
<td>6</td>
<td>300°C, 1h</td>
<td>843°C, 1h</td>
<td>Gas, 18bar, 100% velocity</td>
<td>250°C, 1h</td>
</tr>
<tr>
<td>7</td>
<td>300°C, 1h</td>
<td>843°C, 1h</td>
<td>Gas, 18bar, 75% velocity</td>
<td>250°C, 1h</td>
</tr>
<tr>
<td>8</td>
<td>300°C, 1h</td>
<td>843°C, 1h</td>
<td>Gas, 18bar, 50% velocity</td>
<td>250°C, 1h</td>
</tr>
<tr>
<td>9</td>
<td>-</td>
<td>843°C, 1h</td>
<td>Water</td>
<td>250°C, 1h</td>
</tr>
<tr>
<td>10</td>
<td>-</td>
<td>843°C, 1h</td>
<td>Oil</td>
<td>250°C, 1h</td>
</tr>
</tbody>
</table>

Table 1 Chemical composition of AISI 4140 (unit wt%)

Chemical compositions were measured by Optical Emissions Spectrometer (OES) on the SPECTROMAXx. Phase identification and lattice parameter measurement were conducted by PANanalytical Empyrean 2 X-ray diffractometer (XRD) with Cr-Kα radiation after electropolishing. Rockwell C hardness was measured using equipment from Stanley P. Rockwell Co. Charpy impact toughness were conducted at Westmoreland Mechanical Testing & Research Inc. The samples were ground, polished and etched with 4% nital solution. JEOL JSM-7000F Scanning Electron Microscope (SEM) was used for higher magnification characterizations.

Table 3 Chemical composition of Pyrowear53
Figure 1 TTT diagram of Pyrowear53

<table>
<thead>
<tr>
<th>Test batch</th>
<th>Austenitizing</th>
<th>Quenching</th>
<th>Tempering</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>913°C, 1h30min</td>
<td>Water</td>
<td>-</td>
</tr>
<tr>
<td>2</td>
<td>913°C, 1h30min</td>
<td>Oil</td>
<td>-</td>
</tr>
<tr>
<td>3</td>
<td>913°C, 1h30min</td>
<td>Air</td>
<td>-</td>
</tr>
<tr>
<td>4</td>
<td>913°C, 1h30min</td>
<td>Gas, 2bar nitrogen</td>
<td>Cold treat+232°C temper</td>
</tr>
<tr>
<td>5</td>
<td>913°C, 1h30min</td>
<td>Gas, 2bar nitrogen</td>
<td></td>
</tr>
</tbody>
</table>

Table 4 P53 heat treat process

3 Results

3.1 Microstructure and mechanical properties comparison between gas quenching and liquid quenching of AISI 4140

3.1.1 Mechanical properties comparison

Mechanical properties of AISI 4140 after quenching is shown in Figure 2 and after quenching and tempering is shown in Figure 3. The cooling rate is calculated when the core temperature drops from 800°C to 500°C. After quenching, the process with different cooling rates provides similar hardness and different Charpy impact toughness. The hardness is 55HRC, which is the typical fully hardened hardness for AISI 4140. When the cooling rate is higher than 20 °C/s, the Charpy impact toughness is the same. With the continuous decreasing of cooling rate, the Charpy impact toughness start to increase, while keeping the hardness are similar. Although the average hardness from slow cooling process is similar compared to fast cooling process, the fluctuation of measured hardness range is larger. After 250°C tempering for 1h, the hardness drops to 52HRC. All the Charpy impact toughness increases, however, batch8 (cooling rate is 14.24 °C/s from 800°C to 500°C) has the lowest Charpy impact toughness. A trend is clearly observed that when process with different cooling rates provides similar hardness, the slower cooling rate would give higher Charpy impact toughness after quenching, but lower Charpy impact toughness after quenching and
tempering. When replacing oil quenching to gas quenching, Charpy impact toughness difference should be carefully inspected.

Figure 2 Mechanical properties of AISI 4140 after quenching

Figure 3 Mechanical properties of AISI 4140 after quenching and tempering

3.1.2 Microstructure comparison

XRD patterns of AISI 4140 under water and gas quenching reveals carbon content in martensite matrix with different cooling rates as shown in Table 5 and Figure 4. With the decreases of cooling rate (water quench > gas quench 100% > gas quench 75% > gas quench 50%), the $\alpha'$ (002) and (200) peaks shift to right, which indicates smaller lattice parameter and lower carbon concentration in martensite matrix. Lerchbacher
observed the same phenomenon for hot-work tool steel X38CrMoV5-1 [11]. The carbon in martensite lath partitions to adjacent thin austenite films. SEM of AISI 4140 is shown in Figure 5 and Figure 6. From Figure 5 (a) and (b), more bainite forms for gas quench 50% with slow cooling rate. Most microstructure is still martensite for as-quenched samples. Carbides morphology difference can be observed from Figure 6 (a) and (b). For water quenching + temper, small carbides were evenly distributed. For gas quenching + temper, carbides size is larger and have clearly parallel distribution, which may be at prior austenite film location. These planar carbides are not available for strengthening and could be the crack initiation during Charpy impact test. Fracture surface of AISI 4140 is observed as shown in Figure 7. Quasi cleavage fracture is shown in Figure 7 (c) and (d). Several dimples with residual carbides are also observed.

<table>
<thead>
<tr>
<th>Quench condition</th>
<th>peak position after quench</th>
<th>peak position after tempering</th>
</tr>
</thead>
<tbody>
<tr>
<td>water quench</td>
<td>68.571</td>
<td>68.697</td>
</tr>
<tr>
<td>gas quench 100%</td>
<td>68.622</td>
<td>68.795</td>
</tr>
<tr>
<td>gas quench 75%</td>
<td>68.688</td>
<td>68.758</td>
</tr>
<tr>
<td>gas quench 50%</td>
<td>68.679</td>
<td>68.827</td>
</tr>
</tbody>
</table>

Table 5 Peak position of AISI 4140 martensite after quenching and tempering

![XRD patterns of water and gas quenching](image)

Figure 4 XRD patterns of AISI 4140 under water and gas quenching
Figure 5 SEM of AISI 4140 - 2500 times

Figure 6 SEM of AISI 4140 - 5000 times
3.2 Microstructure and mechanical properties comparison between gas quench and liquid quenching of Pyrowear53

3.2.1 Mechanical properties comparison

Mechanical properties of P53 is shown in Figure 8 and Figure 9. With the decreases of cooling rate, the hardness slightly decreases. Even for air cooling, the microstructure contains more than 95% martensite. Oil quench (30.57 °C/s) provides the highest Charpy impact toughness after quenching and after quenching and tempering. This trend is different compared to AISI 4140 discussed above. Considering only 0.1 wt% carbon in P53, carbides morphology, size and distribution may be the dominated strengthening mechanism of Charpy impact toughness.
3.2.2 Microstructure comparison

XRD patterns of all the samples after quenching or after quenching and tempering are similar, which indicates that different cooling rate does not have much effect on carbon content in martensite. Considering P53 only contains 0.1 wt% carbon, carbon content in martensite changing from 0.1 wt% to 0.7 wt% could not lead much difference in lattice parameter of martensite and XRD pattern. Figure 10 and Figure 11 present the EBSD patterns of P53 after oil quenching and gas quenching. The matrix is martensite. The red dots represent different carbides, including Fe2C, Fe3C, Fe5C2 and Fe7C3. The accuracy of carbides determination using EBSD is highly depends on the sample microstructure, surface condition and EBSD configuration.

From Figure 12, the average grain size of oil quenching (30.57 °C/s) is 1.5166μm and of gas quenching (0.96 °C/s) is 1.6389μm. Due to slow cooling rate, the grain size of gas quenching increases. Fracture surface of P53 after oil quench and gas quench is presented in Figure 13 and Figure 14. Both fracture surface shows mainly dimples, which indicates microvoid coalescence fracture.
Figure 10 EBSD pattern of P53 after oil quenching

Figure 11 EBSD pattern of P53 after gas quenching

Figure 12 Grain size statistics
4 Discussion

As shown in Figure 2 and Figure 3, the Charpy impact toughness trend of AISI 4140 is the same as shown in Lerchbacher’s research [11]. However, the Charpy impact toughness trend of Pyrowear is the opposite. Considering Pyrowear, with only 0.1 wt% carbon, may mainly segregate to dislocations and clusters even under high cooling rate. No carbon left for further partition to thin austenite film even with slower cooling rate and longer time at elevated temperature. The Charpy impact decreases may be caused by grain growth and carbides uneven distribution for Pyrowear 53 during gas quenching. Figure 15 presents the carbon concentration profile of martensite lath and austenite film conducted by Atom Probe Tomography [11]. It should be noted the highest carbon concentration always appear at the center of the austenite, not at the phase boundary between austenite and martensite. An assumption is that the soft austenite is pressed by the surrounding hard martensite. The diffusion barrier would increase under compressive stress based on first principle calculation by [18]. The decreased carbon potential and diffusivity near the phase boundary would favor to diffuse carbon to the center of the austenite film, where the potential and diffusivity are the highest.

(a) Oil quench  
(b) Gas quench

Figure 13 Fracture surface of P53 - 250 times

(a) Oil quench  
(b) Gas quench

Figure 14 Fracture surface of P53 - 2500 times
5 Conclusion

Microstructure, hardness and Charpy impact toughness of AISI 4140 and Pyrowear 53 after water, oil and gas quenching was compared. The relationship between Charpy impact toughness, hardness and cooling approach/rate were analyzed. For AISI 4140, Charpy impact toughness increases when the cooling rate decreases during quenching. Water quenching and tempering provides the ultimate Charpy impact toughness, compared to oil and gas quenching. Austenite percentage and carbon content in austenite is proposed as the dominated mechanism. For Pyrowear53, Charpy impact toughness decreases when the cooling rate decreases during quenching. Oil quenching and tempering provides the ultimate Charpy impact toughness, compared to water and oil quenching. Carbides is proposed as the dominated mechanism.

Acknowledgment

The authors would like to thank members in Center of Heat Treating Excellence, Worcester Polytechnic Institute, who give fruitful discussion. The work was supported by CHTE, Worcester Polytechnic Institute.

Figure 15 Carbon concentration profile at martensite lath and austenite film [11]
References:


CHAPTER 8  Proposed standard I: method for determining hardenability of steel during gas quenching

Yuan Lu, Richard D. Sisson, Jr.

To be submitted to: ASTM standard proposal

Highlights:
1. Propose standard method for determining hardenability of steel during gas quenching based on critical heat transfer coefficient test.
2. Apparatus, specimen, procedure and examples are illustrated in detail.
Proposed Standard: Method for determining hardenability of steel during gas quenching

1 Scope

1.1 This test method covers the identification and description for determining the hardenability of steels during gas quenching.

1.2 The selection of the test method to be used for determining the hardenability of a given steel during gas quenching shall be agreed upon between the supplier and user. The Certified Material Test Report shall state the method of hardenability determination.

1.3 Based on the original work by M.A. Grossmann [1], it has been found that the hardness is determined mainly by the carbon content, and is raised only very slightly by the presence of alloys. Therefore, the critical hardness is defined as the hardness when the quenched microstructure contains 50% martensite.

1.4 Hardenability is an internal property of the steels. In this standard, critical heat transfer coefficient (HTC) is used to measure the hardenability. The critical HTC is defined as the HTC required to form 50% martensite at the center of a steel alloy rod with a 1 inch (25.4 mm) diameter. Lower critical HTC indicates higher gas quench steel hardenability.

1.5 This standard is to measure critical HTC for steels during gas quenching.

1.6 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.7 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2 Referenced Documents

2.1 ASTM Standards:

E18 Test Methods for Rockwell Hardness of Metallic Materials

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1 This standard is a draft by Center of Heat Treating Excellence, Worcester Polytechnic Institute. The format is followed by ASTM A255-10 standard test methods for determining hardenability of steel.

2 For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards volume information, refer to the standard’s Document Summary page on the ASTM website.
E112 Test Methods for Determining Average Grain Size

3 Description

3.1 This test method covers the procedure for determining the critical HTC of steel. The test consists of gas quenching cylindrical test specimen 1.0 inch in diameter and 4.0 inch long, and measuring the center hardness.

3.2 The configurations of gas quench parameters are listed in Table 1.

<table>
<thead>
<tr>
<th>Designation</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total length of test piece</td>
<td>$(100 \pm 0.5)mm$</td>
</tr>
<tr>
<td>Diameter of test piece</td>
<td>$(25\pm0.5)mm$</td>
</tr>
<tr>
<td>Time during which test piece is maintained at heating temperature</td>
<td>$(30\pm5)min$</td>
</tr>
<tr>
<td>Maximum time lag between removal of test piece from furnace and start of quenching</td>
<td>5s</td>
</tr>
<tr>
<td>Test piece surface roughness</td>
<td>1.6 micrometers</td>
</tr>
<tr>
<td>Precision of gas temperature measurement</td>
<td>$\pm0.1C$</td>
</tr>
<tr>
<td>Precision of gas velocity measurement</td>
<td>$\pm0.01m/s$</td>
</tr>
<tr>
<td>Precision of gas pressure measurement</td>
<td>$\pm0.07bar$</td>
</tr>
</tbody>
</table>

Table 1 Configurations of gas quench parameters

4 Apparatus

4.1 Support for Test Specimen – A fixture for supporting the test specimen horizontally so that the high pressure and high velocity gas flows along the side of the specimen. A satisfactory type of support for the standard specimen is shown in Figure 1.
Figure 1 Test Specimen in Support for Gas Quenching

4.2 Gas Quenching Device – A gas quenching device of suitable capacity to provide different gas quench HTC conditions. Sensors to measure gas temperature, gas pressure and gas velocity should be installed at the gas inlets and outlets. The measurement of gas velocity can be replaced by gas flow rate. A source of sufficient gas to maintain steady gas flow, a gas booster to generate required gas pressure and control valves to adjust gas velocity will be satisfactory.
5 Test Specimens

5.1 *Wrought Specimens* – Critical HTC specimens shall be prepared from rolled or forged stock and shall represent the full cross section of the product. If negotiated between the supplier and the user, the critical HTC specimen may be prepared from a given location in a forged or rolled product or from a continuous cast billet. The test specimen shall be 1.0 inch (25.4 mm) in diameter by 4.0 inch (101.6 mm) in length, with screw for connecting it in a horizontal position for critical HTC test. Dimensions of the specimen are given in Figure 2. The specimen shall be machined from a bar previously normalized in accordance with 6.1 and of such size as to permit the removal of all decarburization in machining to 1.0inch round. The sample side and end shall have a reasonably smooth finish as shown in Table 1. Normalizing may be waived by agreement between the supplier and the user. The previous thermal history of the specimen tested shall always be recorded.

5.2 *Cast Specimens* – A separately cast critical HTC specimen may be used for non-boron steels. Cast specimens are not suitable for boron steel grades due to erratic results. A graphite or metal mold may be used to form an overlength specimen 1.0 inch (25.4 mm) in diameter which shall be cut to the standard specimen size. The mold may also be used to form a 1.25inch (31.8 mm) diameter specimen which shall be machined to the final specimen size. Cast tests need not be normalized.

![Figure 2 Test Specimen](image-url)
6 Procedure

6.1 Normalizing – The wrought product from which the specimen is to be prepared shall be normalized to ensure proper hardening characteristics. The sample shall be held at the temperature listed in Table 2 for 1h and cooled in air. Tempering of the normalized sample to improve machinability is permitted.

6.2 Heating – Place the specimen in a furnace that is at the specified austenitizing temperature (Table 2) and hold at this temperature for 30 min. In production testing slightly longer times up to 35 min may be used without appreciably affecting results.

6.2.1 For a particular fixture and furnace, determine the time required to heat the specimen to the austenitizing temperature by inserting a thermocouple into a hole drilled axially in the top of the specimen. Repeat this procedure periodically, for example once a month, for each combination of fixture and furnace.

6.3 Quenching – Adjust the gas-quenching device to generate different gas quench HTC conditions (different gas pressure and velocity). For each gas quench HTC condition, the time between removal of the specimen from the furnace and the beginning of the quench should not be more than 5s. Direct gas quench, at a temperature of 40 to 85°F (5 to 30°C), flow along the specimen side for not less than 20 min. If the specimen is not cold when removed from the fixture, immediately quench it in water.

<table>
<thead>
<tr>
<th>Steel Series</th>
<th>Ordered Carbon Content, max, %</th>
<th>Normalizing Temperature, °F (°C)</th>
<th>Austenitizing Temperature, °F (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1000, 1300, 1500, 3100, 4000, 4100</td>
<td>0.25 and under</td>
<td>1700 (925)</td>
<td>1700 (925)</td>
</tr>
<tr>
<td>4300, 4400, 4500, 4600, 4700, 5000, 5100, 6100, 8100, 8600, 8700, 8800, 9400, 9700, 9800</td>
<td>0.26 to 0.36, incl</td>
<td>1650 (900)</td>
<td>1600 (870)</td>
</tr>
<tr>
<td>2300, 2500, 3300, 4800, 9300</td>
<td>0.37 and over</td>
<td>1600 (870)</td>
<td>1550 (845)</td>
</tr>
<tr>
<td></td>
<td>0.25 and under</td>
<td>1700 (925)</td>
<td>1550 (845)</td>
</tr>
<tr>
<td></td>
<td>0.26 to 0.36, incl</td>
<td>1650 (900)</td>
<td>1500 (815)</td>
</tr>
<tr>
<td></td>
<td>0.37 and over</td>
<td>1600 (870)</td>
<td>1475 (800)</td>
</tr>
<tr>
<td>9200</td>
<td>0.50 and over</td>
<td>1650 (900)</td>
<td>1600 (870)</td>
</tr>
</tbody>
</table>

A A variation of ±10°F (6°C) from the temperatures in this table is permissible.

Normalizing and austenitizing temperatures are 50°F (30°C) higher for the 6100 series.

Table 2 Normalizing and Austenitizing Temperatures [2]
6.4 *Gas Quench HTC Calculation* – The gas quench HTC is calculated based on equation [3]. The gas velocity can be measured directly or calculated based on the measurement of gas mass flow rate.

\[ h = \frac{k}{L} 0.023 \left( \frac{P V L}{\mu Z R T} \right)^{0.8} \left( \frac{\mu C_p}{k} \right)^{0.33} \]  

Equation 1

- **h**: heat transfer coefficient, w/m²K
- **P**: gas pressure, Pa
- **V**: gas velocity, m/s
- **L**: specify characteristic length (part diameter), m
- **μ**: dynamic viscosity, kg/ms
- **Z**: gas compressibility and density
- **R**: gas constant, J/Kmol
- **T**: gas temperature, K
- **C_p**: gas specific heat, J/kgK
- **k**: thermal conductivity, W/mK

6.5 *Gas Quench HTC Range Selection* – For steels with different carbon content, the gas quench HTC condition to reach critical hardness at the center is different. In order to reduce the amount of the experiments, for low hardenability steels, the recommended HTC test range is from 1000 W/m²°C. For medium hardenability steels, the recommended HTC test range is up to 1000 W/m²°C. For high hardenability steels, the recommended HTC test range is up to 300 W/m²°C. For example, Table 3 is the simulation results of critical HTC for different steels [4].

6.6 *Hardness Measurement* – The specimen shall be cut in the middle and Rockwell C hardness measurements made at the center hardness measuring point shown in Figure 1. The thickness of disk cut from the specimen should be no less than 0.5 inch.

<table>
<thead>
<tr>
<th>Steel</th>
<th>Critical HTC (50%martensite) W/m²°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>P53</td>
<td>&lt;100</td>
</tr>
<tr>
<td>58680</td>
<td>130</td>
</tr>
<tr>
<td>54150</td>
<td>137</td>
</tr>
<tr>
<td>54340</td>
<td>162</td>
</tr>
<tr>
<td>54140</td>
<td>430</td>
</tr>
<tr>
<td>54047</td>
<td>373</td>
</tr>
<tr>
<td>58650</td>
<td>503</td>
</tr>
<tr>
<td>552100</td>
<td>820</td>
</tr>
<tr>
<td>55140</td>
<td>1102</td>
</tr>
<tr>
<td>54319</td>
<td>1172</td>
</tr>
<tr>
<td>54120</td>
<td>2826</td>
</tr>
<tr>
<td>58620</td>
<td>3098</td>
</tr>
</tbody>
</table>

Table 3 Simulated Critical HTC for Different Steels
6.6.1 The preparation of the disk must be carried out with considerable care. The two cut surfaces of the disk should be mutually parallel. Very light cuts with water cooling and a coarse, soft-grinding wheel are recommended to avoid heating the specimen. In order to detect tempering due to grinding, the flat may be etched with one of the following etchant solutions:

NOTE 1 - 5% nitric acid (concentrated) and 95% water by volume
NOTE 2 - 50% hydrochloric acid (concentrated) and 50% water by volume.

Wash the disk in hot water. Etch in solution No.1 until black. Wash in hot water. Immerse in solution No.2 for 3s and wash in hot water. Dry in air blast.

6.6.1.1 The presence of lighter or darker areas indicates that hardness and microstructure have been altered in grinding. If such changes caused by grinding are indicated, new surface may be prepared.

6.6.2 When hardness tests are made, the test specimen rests on one of its surface on an anvil firmly attached to the hardness machine. It is important no vertical movement be allowed when the major load is applied.

6.6.2.1 The Rockwell tester should periodically be checked against standard test blocks. It is recommended that a test block be interposed between the specimen and the indenter to check the seating of the indenter and the specimen simultaneously.

For general statements regarding the use of test blocks and surface condition, Test Methods E18 can be referenced.

6.6.3 For reporting purposes, hardness readings should be recorded to the nearest integer, with 0.5 HRC values rounded to the next higher integer.

6.7 Critical Hardness Determination – Based on Grossmann’s work [1], the 50% martensite hardness relationship with carbon content is presented in Figure 3.
Figure 3 Hardness of Quenched Structures Containing 50% Martensite, for Different Carbon Contents

7 Plotting Test Results

7.1 Test results should be plotted on a standard hardenability chart prepared for this purpose, in which the ordinates represent HRC values and the abscissae represent the HTC of gas quench conditions. An example is presented in Figure 4.

Figure 4 4140 gas quench critical HTC test
8 Index of Hardenability

8.1 The hardenability of a steel can be designated by the critical heat transfer coefficient.

9 Report

9.1 Report the following information that may be recorded on the hardenability chart:

9.1.1 Previous thermal history of the specimen tested, including the temperature of normalizing and austenitizing.

9.1.2 Chemical Composition

9.1.3 ASTM grain size as determined by Test Methods E112, unless otherwise indicated.

References:

CHAPTER 9  Proposed standard II: Measuring the heat transfer coefficient distribution of gas quench furnace

Yuan Lu, Richard D. Sisson, Jr.

To be submitted to: ASTM standard proposal

Highlights:
1. Propose standard method for measuring the heat transfer coefficient distribution of gas quench furnace by measuring center hardness of test bar.
2. Apparatus, specimen, procedure and examples are illustrated in detail.
Proposed Standard Practice for
Measuring the heat transfer coefficient distribution of gas quench furnace

1 Scope

1.1 This standard provides general principles for measuring the heat transfer coefficient
distribution of gas quench furnace.

1.2 This practice specifies the materials and the construction requirements for a
standardized test specimen

1.3 This standard is used to measure and describe the response of materials, products, or
assemblies to heat and flame under controlled conditions, but does not by itself
incorporate all factors required for fire hazard or fire risk assessment of the materials,
products, or assemblies under actual fire conditions.

1.4 Fire testing is inherently hazardous. Adequate safe- guards for personnel and property
shall be employed in conducting these tests.

1.5 This standard does not purport to address all of the safety concerns, if any, associated
with its use. It is the responsibility of the user of this standard to establish appropriate
safety and health practices and determine the applicability of regulatory limitations
prior to use.

2 Referenced Documents

2.1 ASTM Standards:

E2749 -15a, Standard practice for measuring the uniformity of furnace exposure on test
specimens.

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1 This standard is a draft by Center of Heat Treating Excellence, Worcester Polytechnic
Institute. The format is followed by ASTM E2749 -15 a, standard practice for measuring the
uniformity of furnace exposure on test specimens

2 For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact
ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards
volume information, refer to the standard’s Document Summary page on the ASTM
website.
3 Apparatus

3.1 Gas Quenching Device - A gas quenching furnace is being tested. Sensors to measure gas temperature, gas pressure and gas velocity should be checked and working.

4 Test Specimens

4.1 Specimens shall be prepared from the same steel grade and same heat. The test specimen shall be 0.5inch, 1.0 inch, 1.5inch or 2.0inch in diameter by 4.0 inch in length. Two holes should be machined as shown in Figure 1. The upper hole is to fix the sample with the fixture. The lower hole is to install thermocouple if necessary. The specimen shall be machined from a bar previously normalized and of such size as to permit the removal of all decarburization in machining. The sample side and end shall have a reasonably smooth. Normalizing may be waived by agreement between the supplier and the user. The previous thermal history of the specimen tested shall always be recorded.

![Test Specimen](image.png)

Figure 1 Test Specimen

5 Procedure

5.1 Gas Quench HTC Calculation - The gas quench HTC is calculated based on equation1 [1].

\[ h = \frac{k}{l} 0.023 \left( \frac{PVL}{\mu ZRT} \right)^{0.8} \left( \frac{\mu C}{k} \right)^{0.33} \]

Equation1
h: heat transfer coefficient, w/m²K
P: gas pressure, Pa
V: gas velocity, m/s
L: specify characteristic length (part diameter), m
μ: dynamic viscosity, kg/ms
Z: gas compressibility and density
R: gas constant, J/Kmol
T: gas temperature, K
\( C_p \): gas specific heat, J/kgK
k: thermal conductivity, W/mK

5.2 Select proper sample – Using Dante to simulate center hardness of AISI4140 and AISI4340 with 0.5”, 1”, 1.5” and 2” under different furnace HTC, from 50-2000W/m²K. Select proper steel grade and diameter to ensure the center hardness is between 50% (42.2HRC) to 90% (50.0HRC) martensite hardness [2].

5.3 Sample preliminary test - Place the sample in the furnace with the dummy thermal load using desired gas quenching process. After quenching, measure the center hardness of the sample. If the hardness is between 42-51HRC, use this steel grade and diameter. Otherwise, select proper steel grade and diameter.

5.4 Normalizing – After select proper steel grade and geometry for specific gas quench furnace, all the specimen shall be normalized to ensure proper hardening characteristics. The sample shall be held at the temperature listed in Table 1 for 1h and cooled in air. Tempering of the normalized sample to improve machinability is permitted.

5.5 Heating – Place the specimen at desired location in the gas quench furnace at the specified austenitizing temperature (Table 1) and hold at this temperature for certain time. Figure 2 presents the sample locations in one type of gas quench furnace.

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**Figure 2 Sample locations in one type of gas quench furnace**
5.6 **Quenching** – Adjust the gas quench furnace to generate different gas quench HTC conditions (different gas pressure and velocity). Record the gas temperature and ensure the specimen is fully cold when removed from the furnace.

<table>
<thead>
<tr>
<th>Steel Series</th>
<th>Ordered Carbon Content, max, %</th>
<th>Normalizing Temperature, °F (°C)</th>
<th>Austenitizing Temperature, °F (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1000, 1300, 1500, 3100, 4000, 4100</td>
<td>0.25 and under</td>
<td>1700 (925)</td>
<td>1700 (925)</td>
</tr>
<tr>
<td>4300, 4400, 4500, 4600, 4700, 5000, 5100, 6100, 8100, 8600, 8700, 8800, 9400, 9700, 9800</td>
<td>0.26 to 0.36, incl</td>
<td>1650 (900)</td>
<td>1600 (870)</td>
</tr>
<tr>
<td>2300, 2500, 3300, 4800, 9300</td>
<td>0.37 and over</td>
<td>1600 (870)</td>
<td>1550 (845)</td>
</tr>
<tr>
<td>9200</td>
<td>0.50 and over</td>
<td>1650 (900)</td>
<td>1600 (870)</td>
</tr>
</tbody>
</table>

* A variation of ±10°F (6°C) from the temperatures in this table is permissible.

5.7 **Hardness Measurement** – The specimen shall be cut in the middle and Rockwell C hardness measurements made at the center hardness measuring point. The thickness of disk cut from the specimen should be no less than 0.5 inch.

5.7.1 The preparation of the disk must be carried out with considerable care. The two cut surfaces of the disk should be mutually parallel. Very light cuts with water cooling and a coarse, soft-grinding wheel are recommended to avoid heating the specimen. In order to detect tempering due to grinding, the flat may be etched with one of the following etchant solutions:

*NOTE 1 - 5% nitric acid (concentrated) and 95% water by volume*

*NOTE 2 - 50% hydrochloric acid (concentrated) and 50% water by volume.*

Wash the disk in hot water. Etch in solution No.1 until black. Wash in hot water. Immerse in solution No.2 for 3s and wash in hot water. Dry in air blast.

Table 1 Normalizing and Austenitizing Temperatures  

<table>
<thead>
<tr>
<th>Steel Series</th>
<th>Normalizing Temperature, °F (°C)</th>
<th>Austenitizing Temperature, °F (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1000, 1300, 1500, 3100, 4000, 4100</td>
<td>1700 (925)</td>
<td>1700 (925)</td>
</tr>
<tr>
<td>4300, 4400, 4500, 4600, 4700, 5000, 5100, 6100, 8100, 8600, 8700, 8800, 9400, 9700, 9800</td>
<td>1650 (900)</td>
<td>1600 (870)</td>
</tr>
<tr>
<td>2300, 2500, 3300, 4800, 9300</td>
<td>1600 (870)</td>
<td>1550 (845)</td>
</tr>
<tr>
<td>9200</td>
<td>1650 (900)</td>
<td>1600 (870)</td>
</tr>
</tbody>
</table>
5.7.1.1 The presence of lighter or darker areas indicates that hardness and microstructure have been altered in grinding. If such changes caused by grinding are indicated, new surface may be prepared.

5.7.2 When hardness tests are made, the test specimen rests on one of its surface on an anvil firmly attached to the hardness machine. It is important no vertical movement be allowed when the major load is applied.

5.7.2.1 The Rockwell tester should periodically be checked against standard test blocks. It is recommended that a test block be interposed between the specimen and the indenter to check the seating of the indenter and the specimen simultaneously. For general statements regarding the use of test blocks and surface condition, Test Methods E18 can be referenced.

5.7.3 For reporting purposes, hardness readings should be recorded to the nearest integer, with 0.5 HRC values rounded to the next higher integer.

5.8 HTC modelling - Based on the database developed with critical HTC test [3] or DANTE [4], determine HTC distribution in the furnace. In Figure 3, the specimen is AISI4140 with 1inch diameter and 4inch length. If this specimen is being used, the measured center hardness 43HRC represents outside HTC is 430 $W/m^2C$.

![Figure 3 4140 gas quench critical HTC test](image-url)
6 Plotting Test Results

6.1 Furnace gas flow direction, hardness distribution and HTC distribution should be presented. An example is shown in

![Furnace gas flow direction](image)

Figure 4 Furnace gas flow direction

![Hardness distribution of front side](image)

Figure 5 Hardness distribution of front side

![Hardness distribution of rear side](image)

Figure 6 Hardness distribution of rear side

![HTC distribution of front side](image)

Figure 7 HTC distribution of front side

![HTC distribution of rear side](image)

Figure 8 HTC distribution of rear side
7 Report

7.1 Report the following information that may be recorded on the hardness and HTC distribution map:

7.1.1 Previous thermal history of the specimen tested, including the temperature of normalizing and austenitizing.

7.1.2 Chemical Composition

7.1.3 ASTM grain size as determined by Test Methods E112, unless otherwise indicated.

References:


CHAPTER 10 Conclusions

In this thesis, heat transfer, hardenability and steel phase transformations during gas quenching were investigated. The fundamental difference between the liquid and gas quenching is the heat transfer coefficient, not only the values but also the variation with temperature. A quenching model was developed and verified using DANTE. Hardness database of different phases was modified. Using quenching experiment and model, it has been proven that gas quenching with constant HTC cannot generate the similar cooling curves compared to liquid quenching. Gas quenching with dynamic HTC, not commonly used in industry, is proved to have the same cooling curves compared to oil quenching. Current gas quench steel hardenability tests were reviewed. Several limitations were found, such as unsteady gas flow and not proper to characterize high hardenability steel. Critical HTC test was proposed and verified. Critical HTC, a concept like critical diameter, was successfully proved to describe the gas quench hardenability of steel. The critical HTC of AISI 4140 steel is 430 W/m²°C and the critical HTC of AISI 52100 steel is 820 W/m²°C, which reveals that the gas quench hardenability of 4140 is better than 52100. HTC was proved to be a better indicator for gas quench intensity, compared to gas pressure and velocity. Computer-aided critical HTC test was discussed to reduce experiment time. A standard, “Method for determining hardenability of steel during gas quenching” was proposed.

An attempt to use critical HTC test bar and measure the HTC distribution of gas quench furnace was made. The result indicated that for different gas quench furnace, different sample geometry and steel grade should be selected. Based on modeling and experiment, 0.5” diameter and 4” length AISI 4340 bar can be used to evaluate 2bar nitrogen gas quench furnace. The 2bar nitrogen gas quench furnace has obvious uneven HTC distribution, which may cause microstructure and mechanical properties variations during gas quenching. The experiment, using 1” diameter AISI 4140, to evaluate 20bar helium gas quench furnace was conducted as well. 20bar helium gas quench furnace demonstrate much better quench intensity and uniformity, compared to 2bar nitrogen furnace.

When replacing liquid quenching to gas quenching, microstructure and mechanical properties should be addressed. Gas quenching, usually with slow cooling rate, may reduce hardness and Charpy impact toughness, compared to water and oil quenching. Lattice parameter and c/a ratio of as-quenched martensite in steel was measured using high resolution X-ray diffraction and Rietveld refinement. The modified equation can be used to estimate carbon content in martensite after liquid and gas quenching, which is essential to model mechanical properties afterwards. For AISI 4140, Charpy impact toughness increases when the cooling rate decreases during quenching. Austenite percentage and carbon content in austenite is proposed as the dominated mechanism. For Pyrowear53, Charpy impact toughness decreases when the cooling rate decreases during quenching. Carbides is proposed as the dominated mechanism.
CHAPTER 11  Recommendations for future work

In this thesis, only cooling rate at the core of the samples was considered, because hardenability and Charpy impact toughness were mainly investigated. However, real HTC distribution in furnace is very complex. Flow analysis should be considered if cooling rate at surface or near surface is needed. Constant HTC should be replaced with various HTC. Fluent and Abaqus are recommended. Currently, 1” diameter bar was used in critical HTC test. If ultra high hardenability steel grade needs to be characterized, such as Pyrowear53, 1.5” or 2” diameter bar should be considered. The size of gas quenching chamber should be redesigned to fit the larger samples.

When using the proposed standard to evaluate heat transfer coefficient distribution in gas quench furnace, the initial samples should be selected carefully. A large amount of samples from the same steel grade and the same heat should be prepared, considering the initial microstructure and chemical variation would affect hardenability. In order to improve the accuracy of HTC distribution, the critical HTC test of selected samples needs to be conducted.

TEM and ATP analysis on thin austenite film was recommended. It is helpful to determine the carbon content in austenite film increases with slower cooling rate. Higher carbon concentration in austenite would increase Charpy impact toughness after quenching, but decrease Charpy impact toughness after quenching and tempering. Ion milling and EBSD were recommended to investigate carbides in Pyrowear after quenching and tempering. The Kikuchi pattern of martensite/ferrite and carbides are similar and should be carefully distinguished.
CHAPTER 12  Relevant presentations and publications

12.1 Publications


[4].  Q Sa, JA Heelan, **Y Lu**, D Apelian, Y Wang, Copper Impurity Effects on LiNi1/3Mn1/3Co1/3O2 Cathode Material. ACS applied materials & interfaces, 2015.


12.2 Presentations and posters

Presentation


120


Posters

[1]. Y Lu, J Mocsari, RD Sisson, Y Rong, Critical Heat Transfer Coefficient Test Method for Gas Quenching Steel Hardenability, Student Poster Competition, HTS2015 (Second Prize)
