The Effects of Rust on the Gas Carburization of AISI 8620 Steel

by

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Abstract

The effects of rust on the carburization behavior of AISI 8620 steel have been experimentally investigated. AISI 8620 steel samples were subjected to a humid environment for time of 1 day to 30 days. After the exposure, a part of the samples was cleaned by acid cleaning. Both cleaned and non-cleaned samples have been carburized, followed by quenching in mineral oil, and then tempered. To determine the effect of rust on gas carburizing, weight gained by the parts and the surface hardness were measured. Surface carbon concentration was also measured using mass spectrometry. Carbon flux and mass transfer coefficient have been calculated. The results show that acid cleaning removes the rust layer effectively. Acid cleaned samples displayed the same response to carburization as clean parts. Rusted parts had a lower carbon uptake as well as lower surface carbon concentration. The surface hardness (Rc) did not show a significant difference between the heavily rusted sample and clean sample. It has been observed that the carbon flux and mass transfer coefficient are smaller due to rust layer for the heavily rusted samples. These results are discussed in terms of the effects of carbon mass transfer on the steel surface and the resulting mass transfer coefficient.

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Introduction

Surface contamination during heat treatment process can greatly affect the quality of the heat treated parts. Although cleaning the post heat treated parts is considered a value added process in heat treatment, cleaning pre heat treated parts is also important and can influence the subsequent process.

The carburizing process can be affected by surface contamination, such as rust. The contaminant on the surface of the part may act as a diffusion barrier layer. AISI 8620 steel is the hardenable chromium, molybdenum, nickel based low alloy steel often used for carburizing to develop a case-hardened part. After carburizing, the steel provides, uniform case depth, hardness and wear properties, and gives the advantage of low distortion.

Literature review has been done for the AISI 8620 steel carburizing, rust formation and cleaning methods which are used to remove rust.

The objective of research is to study the effects of rust on the gas carburizing process and evaluate the efficiency of acid cleaning used to remove the rust. The effect of rust on the carburization behavior of AISI 8620 steel has been experimentally investigated. Hardness after carburizing is used as the parameter to evaluate the heat treatment performance. These results are also discussed in terms of the effects of carbon mass transfer on the steel surface and the resulting mass transfer coefficient. To determine the effect of rust on gas carburizing, weight gained by the parts and the surface hardness were measured. Surface carbon concentration was also measured using mass spectrometry. Carbon flux and mass transfer coefficient have been calculated.

Chapter II　Background

1.0 Heat treatment of AISI 8620 steel

Carburizing is one of the most widely used surface hardening processes. The process involves diffusing carbon into a low carbon steel alloy to form a high carbon steel surface. [1] Carbon transfers from gas atmosphere through the boundary layer, reacts with the steel surface in vapor-solid interface and then diffuses into the bulk of the material. During diffusion, there are several controllable parameters which can be adjusted to meet the customer's tolerances and specifications, including carbon potential atmosphere, temperature and time. The maximum carburization rate can be achieved by controlling the rate of carbon transfer from the atmosphere and the rate of carbon diffusion into the steel. Carburizing process performance strongly depends on the process parameters, as well as furnace types, materials characteristics, atmosphere etc. All of these factors contribute to the mass transfer coefficient (β) which relates the mass transfer rate, mass transfer area, and carbon concentration gradient as driving force. So the mass transfer coefficient and the coefficient of carbon diffusion in steel are the parameters that control the process. [2-4]

The total quantity of the carbon which diffused through the surface can be estimated by integrating the concentration profile over the depth of the carburized layer. Furthermore, differentiation of the total weight gain by the carburizing time yields the following expression for the total flux of carbon atoms through the vapor/solid interface. [5] The flux of carbon atoms diffused in the workpiece through the interface can be presented as shown in equation (1):

$$J = \frac{\partial}{\partial t}\left(\frac{\Delta M}{A}\right) \qquad (1)$$

where J is the carbon flux (g/cm^2*s), ΔM is the total weight gain (g), A is the surface area (cm^2) and t is the carburizing time(s).

The flux in the atmosphere boundary layer is proportional to the difference between the surface carbon concentration in the steel and the atmosphere carburizing potential, the mass transfer coefficient can be presented as follows [6]：

$$\beta = \frac{\frac{\partial}{\partial t}\int_{x_\infty}^{x_0} C(x,t)\,dx}{(C_P - C_S)} = \frac{(\Delta M / A)}{t(C_P - C_S)} \qquad (2)$$

where β is the mass transfer coefficient (cm/s), C_s is the surface carbon concentration in the steel, and C_P is the atmosphere carburizing potential.

AISI 8620 steel is a hardenable chromium, molybdenum, nickel based low alloy steel often used for carburizing to develop a case-hardened part. The well balanced alloy content permits hardening to produce a hard wear resistant case combined with core strength on the order of 862 mPa (125,000 psi). It has excellent machinability and responds well to polishing applications. With the balanced analysis, this steel provides, uniform case depth, hardness and wear properties, and gives the advantage of low distortion. [7] The standard carburization for AISI 8620 is at 900 ^{o}C to 925 ^{o}C (1650 to 1700°F) in an appropriate carburizing medium (C_p = 0.8-1.2 wt% C) and quenched in oil to enhance the surface hardness. Improved carburized case and core properties can be obtained by furnace cooling from carburizing at 900 ^{o}C to 925 ^{o}C (1650 - 1700°F) and then reheating to 860°C (1575°F). Carburizing is accomplished at the same range of 900 ^{o}C to 925 ^{o}C (1650 to 1700°F) in a carburizing environment, followed by oil quench. [8] Fig.1 depicts an schematic illustration showing the locations on the Fe-C phase diagram of the conventional heat treatment in the core of the surface-carburized AISI 8620 steel.[9] During the heat treatment at 900 °C (points a and b), both the carburized surface (0.8% C) and the core of the specimen (0.2% C) remained in the austenitic single-phase region. Oil quenching from 900 °C to room temperature produced a microstructure that nearly all martensite throughout both the core and the case.

The conventional heat treatment of surface carburized steel at ~900 °C

α+γ

γ

α

α+Fe$_3$C

Oil quench

Oil quench

TEMPERATURE (°C)

WEIGHT PERCENTAGE OF CARBON

The core region with ~ 0.2% C content
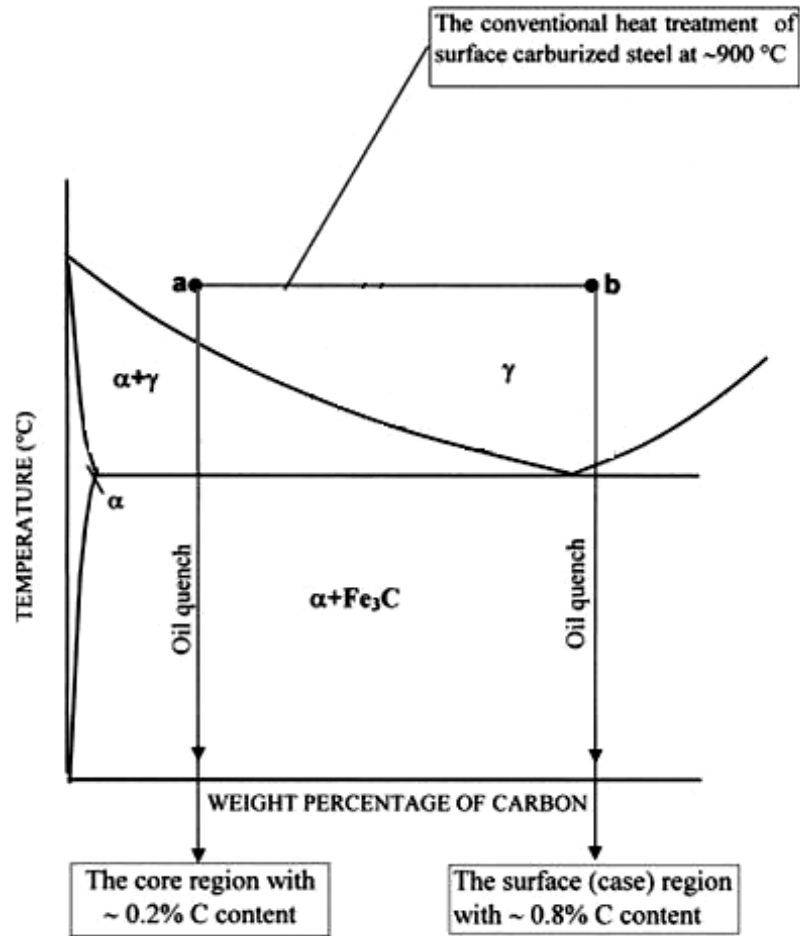
The surface (case) region with ~ 0.8% C content

Figure 1. Schematic illustration of part of the Fe-C phase diagram showing the locations of the heat treatment in the core of surface-carburized AISI 8620 steel. [9]

Izciler, and Tabur [10] examined abrasive wear behavior of different case depth gas carburized AISI 8620 gear steel. In their research, 320 min and 660 min at 925 °C in an endothermic atmosphere with constant 0.16% CO_2 presence carburization condition were used. Homogenous matrix at the cross section and composed of pearlite and ferrite microstructure were seen in the core, as shown in Figure 2. Hardness measurements of the specimens were done before and after the heat treatment on a straight line from core to the surface by intervals of 1 mm under the load of 5 kg, the results are shown in Figure 3 and 4 respectively.
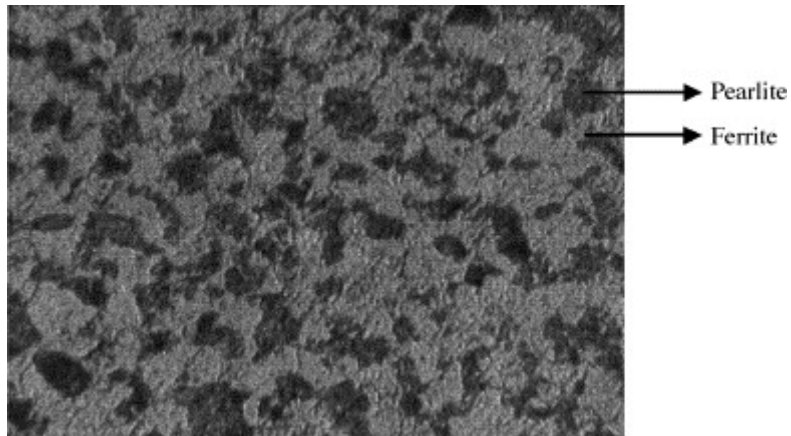
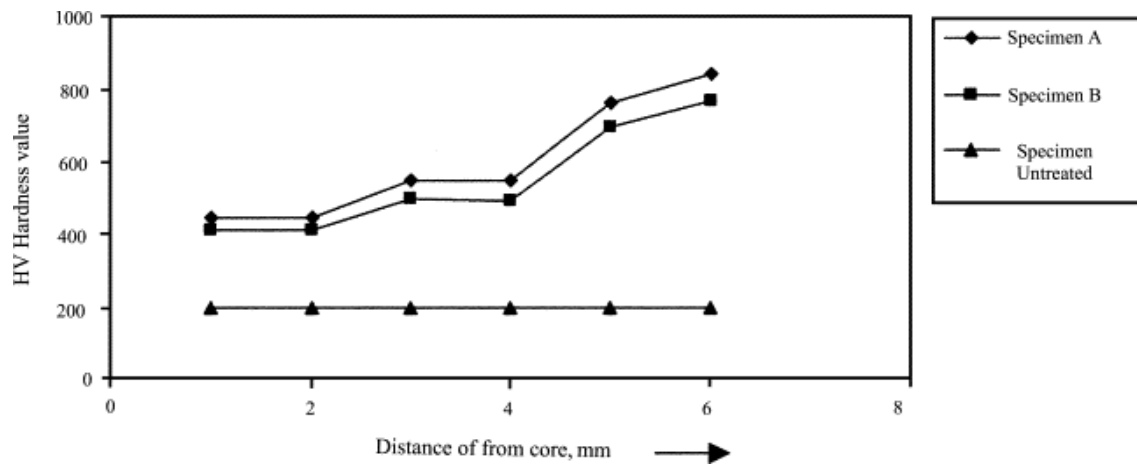Figure. 2. Microstructure of AISI 8620 steel (core).[10]

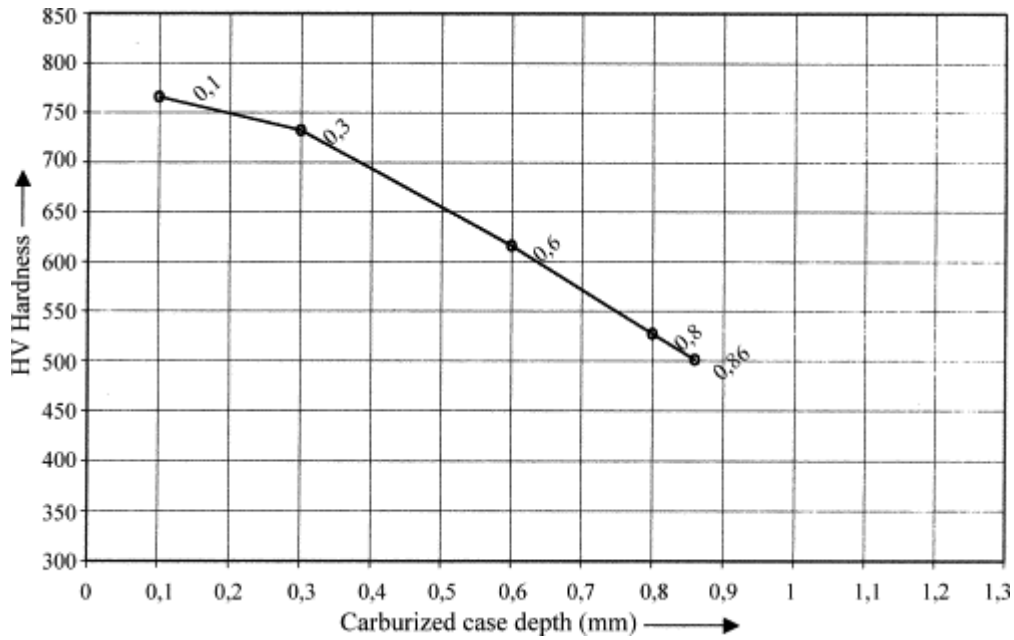Figure 3. Hardness distrubution of the specimens.[10]

Figure 4 Carburized case depth of 320min specimen (from surface to end of case, total case depth 0.86mm).[10]

Erdogan, and Tekeli [9] also investigated carburized AISI 8620 steel. The cross section hardness profile is shown in Figure 5. The carburized surface and the core of the specimen remained in the austenitic single-phase region. Oil quenching from 900°C to room temperature produced a microstructure nearly all martensitic throughout both the core and the case for all the specimens.
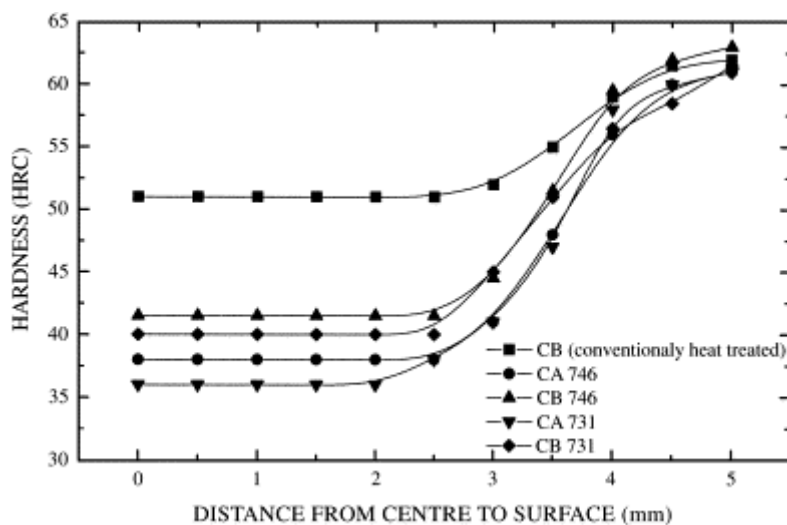

Figure 5. Cross-section hardness profiles of surface-carburized steels after

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conventional heat treatment. [9]

2.0 Rust formation

Rust is a general term for a series of iron oxides formed by the reaction of iron with oxygen in the presence of water. Rust consists of hydrated iron(III) oxides $Fe_2O_3 \cdot nH_2O$, iron(III) oxide-hydroxide (FeO(OH), $Fe(OH)_3$. A tightly adhering oxide coating, a passivation layer, protects the bulk iron from further oxidation. Thus, the conversion of the passivating iron oxide layer to rust results from the combined action of two agents, usually oxygen and water. Other degrading solutions are sulfur dioxide in water and carbon dioxide in water. Under these corrosive conditions, iron(III) species are formed. Unlike iron(II) oxides, iron(III) oxides are not passivating because these materials do not adhere to the bulk metal. As these iron(III) compounds form and flake off from the surface, fresh iron is exposed, and the corrosion process continues until all of the iron(0) is either consumed or all of the oxygen, water, carbon dioxide, or sulfur dioxide in the system are removed or consumed. [11]

The rusting of iron is an electrochemical process that begins with the transfer of electrons from iron to oxygen. The rate of corrosion is affected by water and accelerated by electrolytes. The corrosion of most metals by oxygen is accelerated at low pH. Providing the electrons for the above reaction is the oxidation of iron that may be described as follows:

$$Fe \rightarrow Fe^{2+} + 2\ e^{-} \qquad\qquad (3)$$

Iron dissolved in the water, forms Fe^{2+} ions. In the rich oxygen environment, Fe^{2+} can also react with O_2, forms Fe^{3+}. This reaction is crucial to the formation of rust:

$$2\ Fe^{2+} + 0.5\ O_2 \rightarrow 2\ Fe^{3+} + O^{2-} \qquad\qquad (4)$$

Then, in the following step, iron hydra-oxide with different valence forms.

$$Fe^{2+} + 2\ H_2O \rightleftharpoons Fe(OH)_2 + 2\ H^{+} \qquad\qquad (5)$$

$$Fe^{3+} + 3\ H_2O \rightleftharpoons 2\ Fe(OH)_3 + 3\ H^+ \qquad (6)$$
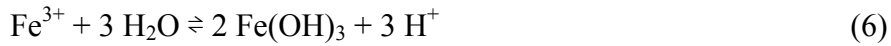
The following dehydration reaction may also take place in rust formation:

$$Fe(OH)_2 \rightleftharpoons FeO + H_2O \qquad (7)$$

$$Fe(OH)_3 \rightleftharpoons FeO(OH) + H_2O \qquad (8)$$

$$2\ FeO(OH) \rightleftharpoons Fe_2O_3 + H_2O \qquad (9)$$

From the above equations, it is also seen that the corrosion products are affected by the water and oxygen. And complex compounds are formed during corrosion. Figure 6 is a schematic diagram showing various stages of reaction for rust formation.
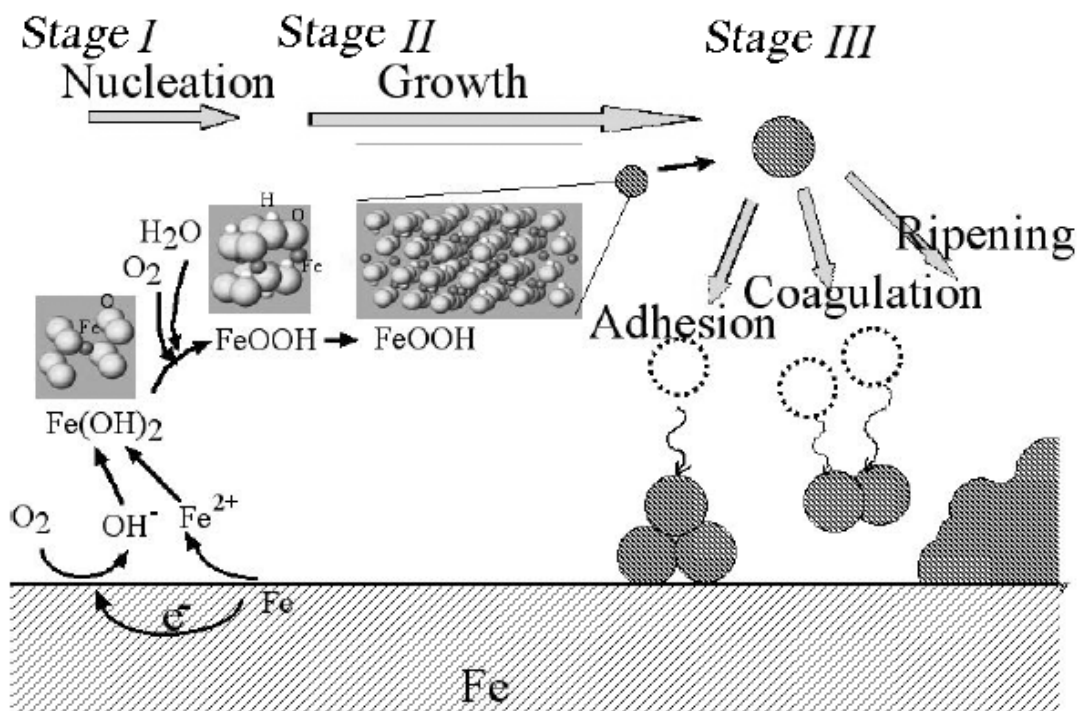

Figure 6 Schematic diagram showing reaction of rust formation. [12]

With limited dissolved oxygen, iron(II)-containing materials are favored, including FeO and black Fe_3O_4. In high oxygen concentrations, $Fe(OH)_{3-x}O_{x/2}$ may

form.[13] The nature of rust changes with time, reflecting the slow rates of the reactions of solids. Furthermore, these complex processes are affected by the presence of other ions, such as Cr^{n+}, which serve as an electrode, and thus accelerate rust formation, or combine with the hydroxides and oxides of iron to precipitate a variety of Cr-Fe-O-OH species.

3.0 Cleaning method

Due to the adhesion mechanism, rust is attached to metal surface, the cleaning method to remove rust often contains metal loss, physically or chemically. For choosing the proper cleaning method for rust, there are several aspects that should be considered. [14]

- Thickness of rust or scale

- Composition of metal

- Allowable metal loss

- Surface finish tolerances

- Shape and size of workpieces

- Production requirements

- Available equipment

- Cost

- Freedom from hydrogen embrittlement

There are various cleaning methods available to remove rust from the part surface [14-15].

Abrasive Blast Cleaning - Abrasive Blast Cleaning is widely used for removing all classes of scale and rust from ferrous mill products, forgings, castings, welding, and

heat-treated parts. Depending on the requirement, abrasive blast cleaning can be the sole process, or combined with pickling, which is applied after to remove the remainder.[14-15,17,20]

Tumbling – Tumbling is the least expensive method. However, the size and shape of parts are the limiting factors for this the process. Tumbling in dry abrasives is often used to clean small workpieces, and the parts with complex shape can not be descaled uniformly. Adding descaling compounds often decrease the required time by 75%.[14-15,18]

Pickling - Pickling in hot, strong solutions of sulphamic, phosphoric, sulfuric, or hydrochloric acid is used for complete removal of scale from mill products and fabricated parts. Pickling is generally used as the second step after abrasive blast cleaning or salt bath descaling. At acid concentrations of about 3% and at temperatures of about 60°C or lower removed the steel [14-16]

Salt Bath Descaling - Salt bath descaling is an effective way to remove scale on alloys and tool steels. Several types of salt baths either reduce or oxidize the scale. It operats at temperature range of 400 to 525 ^{o}C. [14-15,19]

Alkaline Descaling - Alkaline descaling is more costly and slower in its action than acid pickling of ferrous alloys, but no material is lost using this method. The action stops when the rust or scale is removed. Immersion baths are usually operated from room temperature to 71 ^{o}C and can also be used between 93 to 99°C with a concentration of 20% alkali compound.[14-15,19]

Acid Cleaning - Acid cleaning is effective to remove light rust, such as the rust forms on ferrous metal in storage under high humidity. In acid cleaning, detergents, liquid glycol ether, and phosphoric acid are effective in removing the heavy oil compounds from the engine parts, even after it dried. By using a power spray, these acid solutions can clean the parts without manual scrubbing. Phosphoric acid cleaners may cause some discoloration, but it will not etch steel. Acid cleaners are usually used in a

power spray. Some cleaners remove light blushing rust and form a thin film of protection temporarily. They are high in cost, but still often used in large ferrous parts, such as truck cabs. Phosphoric or chromic acid cleaners, with power spray or soak cleaning are used in removing most cutting fluids. These methods are expensive. But in some cases, they are still used because of their ability to remove light rust. [14-15]

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Chapter III Journal Manuscript

The effect of rust on gas carburizing of AISI 8620 steel

The effect of rust on gas carburizing of AISI 8620 steel

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Abstract

The effect of rust on the carburization behavior of AISI 8620 steel has been experimentally investigated. AISI 8620 steel samples were subjected to a humid environment for time of 1 day to 30 days. After the exposure, a set of the samples was cleaned by acid cleaning. Both cleaned and non-cleaned samples have been carburized, followed by quenching in mineral oil, and then tempered. To determine the effect of rust on gas carburizing, weight gained by the parts and the surface hardness were measured. Surface carbon concentration was also measured using optical emission spectrometry. Carbon flux and mass transfer coefficient have been calculated. The results show that acid cleaning removes the rust layer effectively. Acid cleaned samples displayed the same response to carburization as clean parts. Rusted parts had a lower carbon uptake as well as lower surface carbon concentration. The surface hardness (R_c) did not show a significant difference between the heavily rusted sample and clean sample. It has been observed that the carbon flux and mass transfer coefficient are smaller due to rust layer for the heavily rusted samples. These results are discussed in terms of the effects of carbon mass transfer on the steel surface and

the resulting mass transfer coefficient.

1. Introduction

Carburization is a common process used in surface treatment for low carbon steels and alloys. The carbon diffused into the surface makes the surface harder and more abrasion-resistant. Gas carburizing uses the high carbon concentration gas as atmospheres which normally consist of a mixture as endothermic atmospheres (CO, CO_2, H_2O, H_2).[1] The carbon diffuses into the steel alloys by exposure to the gas at temperatures 880 °C to 980 °C. Subsequently, the material is quenched in mineral oil. Martensite is formed in the high carbon concentration after quenching. Due to the high carbon concentration in the surface and martensite from the quenching, high hardness surface layer is formed. Quenching is followed by tempering process between 300 and 550°C to obtain the final desired properties. AISI 8620 in the carburized condition is used for gears, ring gears, shafts and crankshafts.[2]
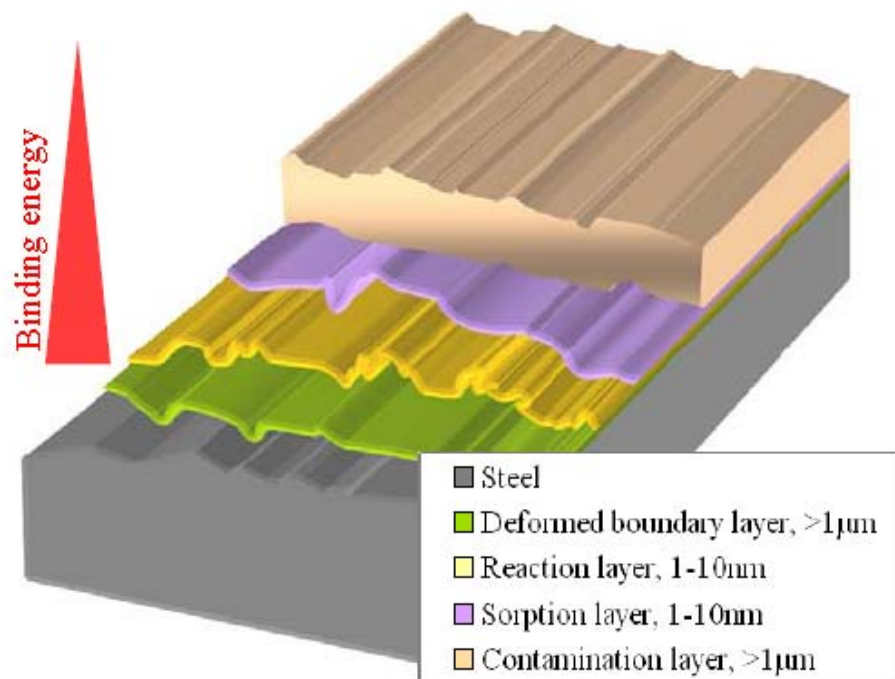

Figure 1. Contamination layers on the surface of workpieces before the heat treatment.

Figure 1 illustrates the various layers of contaminates that may form on a part during manufacturing processing. In terms of different binding energy, the material is covered by four layers, which are the deformed boundary layer, reaction layer, sorption layer and contamination layer and these layers are intergraded to the metal.[3] The deformed boundary layer is formed during the mechanical treatment. The reaction layer which includes metal oxides, sulfides or phospohorous compounds are thin, but have excellent adhesion and can be very dense. The sorption layer which contains the oil and grease is bound by chemisorptions or physisorptions. Final layer is the contamination layer. This layer contains residues of the previous processing steps, e.g. oil, grease, chips, cleaner residues, rust or water ingredients. During the time between previous steps and carburizing, rust can be formed due to the high humidity and temperature in the work shop or it can also be formed during transportation.

Cleaning method

Due to the high binding energy of rust to steel, the cleaning method to remove rust often removes metal. There are several aspects that should be considered in selecting a rust removal method.[4]

- Thickness of rust of scale

- Composition of metal

- Allowable metal loss

- Surface finish tolerances

- Shape and size of workpieces

- Production requirements

- Available equipment

- Cost

– Freedom from hydrogen embrittlement

Abrasive blast cleaning, tumbling, pickling, salt bath descaling, alkaline descaling and acid cleaning are generally used to remove the rust.

Acid cleaner is effective to remove light rust, such as the rust forms on ferrous metal in storage. By using a power spray, these acid solutions can clean the parts without manual scrubbing. Phosphoric acid cleaners may cause some discoloration, but it will not etch steel. Acid cleaners are usually used for spray or immersion. Some cleaners remove light blushing rust and form a thin film of protection from corrosion temporarily. They are high in cost, but still often used in large ferrous parts, such as truck cabs. Phosphoric or chromic acid cleaners, with power spray or soak cleaning are used in removing most cutting fluids. These methods are expensive. But in some cases, they are used because of their ability to remove light rust, such as the rust forms on ferrous metal in storage under high humidity.[5]

Acid cleaning of high strength steels may cause hydrogen embrittlement. To reduce the possibility of hydrogen embrittlement, the time of exposure of the steel to the acid should be minimized. Acid cleaning of soft annealed parts prior to carburization heat treatment is typically not an issue.

The objective of research is to study the effects of rust on the gas carburizing process and evaluate the efficiency of acid cleaning used to remove the rust.

2. Experimental Procedure

2.1 Sample preparation

The AISI 8620 steel is used in this study. It is a hardenable chromium, molybdenum, nickel low alloy steel often used for carburizing to develop the

case-hardened products, such as gears. The composition of the material is presented in Table 1.

Table 1 Composition of the AISI 8620 in wt%

C	Mn	P	S	Si	Ni	Cr	Mo	Fe
0.21	0.83	0.008	0.031	0.25	0.65	0.57	0.16	balance

The cylindrical steel bars were obtained in hot rolled condition. The bars were normalized at 900 ℃ for 4 hours. Disks of 3.125 cm in diameter and 0.8 cm in thickness with a 2mm diameter hole near the edge were machined from the bar. Figure 2 shows the schematic of the sample and its loading configuration for the carburization experiments.
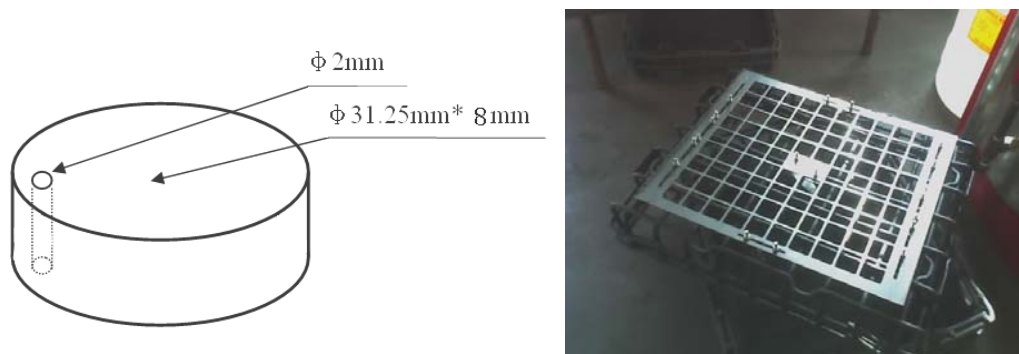
Figure 2 Sample shape and workload used for the carburizing experiments.

The sample surface is ground by using 240 grit SiC paper to create a standard surface finish, and the roughness of the sample is measured by UBM Scanning Laser Microscope with a Keyence LT 8010 confocal laser sensor, 0.4 x 0.4 mm^2 area with a 2 μm sampling interval. The roughness parameter is S_a = 0.3-0.9μm.

The samples were placed in a controlled environment of 25℃ and 80% humidity to form rust. The times which have been chosen are 30 days, 7 days, 3 days and 1 day, as presented in Table 2. The flowchart of the experimental plan is shown in Figure 3. The samples which were polished, divided into three groups: as-polished, rust-not-cleaned and rust-cleaned. The samples were carburized in the same batch to

ensure the same carburization condition; the batch was quenched in mineral oil and tempered. The workload used in carburization experiment is showed in Figure 2, and specimens are randomly attached to the steel fixture with steel wires. Each sample was weighed before and after the carburization using Mettler AB54-S scale to determine the carbon uptake and the total flux into the steel. The surface hardness-HRc and surface carbon concentration were also measured by mass spectrometry. This data was used to calculate the surface mass transfer coefficient for each sample. Three samples for each condition were heat treated and analyzed. By comparing the as-polished and rust-not-cleaned samples, the effect of rust on gas carburization can be studied. By comparing the rust-cleaned and rust-not-cleaned samples, the cleaning method for remove rust can be evaluated.
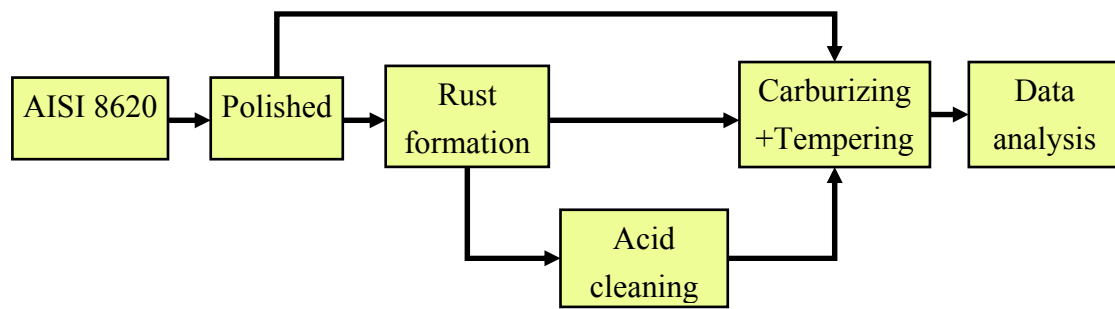

Figure 3 The flowchart for the experiment.

Table 2 Test matrix for the effect of rust on the heat treatment performance test

Material	Rust time**	No. of trials (Cleaned or Not)	Carburizing Temp.	Tempering Temp.
AISI 8620 Steel	As-polished	3	925 ℃	177 ℃
	1 day	C*:3	3h	2h
		NC*:3	0.95 wt%C	
	3 days	C*:3		
		NC*:3		

	7 days	C*:3		
		NC*:3		
	30 days	C*:3		
		NC*:3		

*C- cleaned; NC- not cleaned; C: # - cleaned: No. of sample.
**Rust in the environment of 25℃ in temperature and 80% in humidity.

2.2 Acid cleaning

Acid cleaning was selected due to its ability to remove the rust from sample. It is effective to remove light rust, such as the rust forms on ferrous metal in storage under high humidity. Hydrochloric acid cleaner: 50 vol% HCl is used as the cleaner. [9] The samples have been dipped in acid cleaner for 1min in room temperature, rinsed in distilled water twice for 3 min each time, dried and kept in cool and dry environment. Visually, the brown rust layer disappeared after immersion.

2.3 Carburization process

After cleaning, three groups of specimens were carburized at 925 ºC for 3 hours in an endothermic atmosphere enriched with natural gas. The carburizing potential was controlled at 0.95±0.04 wt.% using an oxygen probe. After 3 hours boost, the temperature is lowered to 843 ºC with Cp = 0.80wt% C for 1 hour, then quenched in mineral oil and tempered at 177 ºC for 2 hours. The experiments are performed at a commercial heat treatment plant, following the test matrix shown in Table 2. The carburizing process is shown in Figure 4. The carburizing performance was evaluated in terms of weight gain, microhardness and analysis of selected surface carbon concentration profiles as well as mass transfer coefficient.
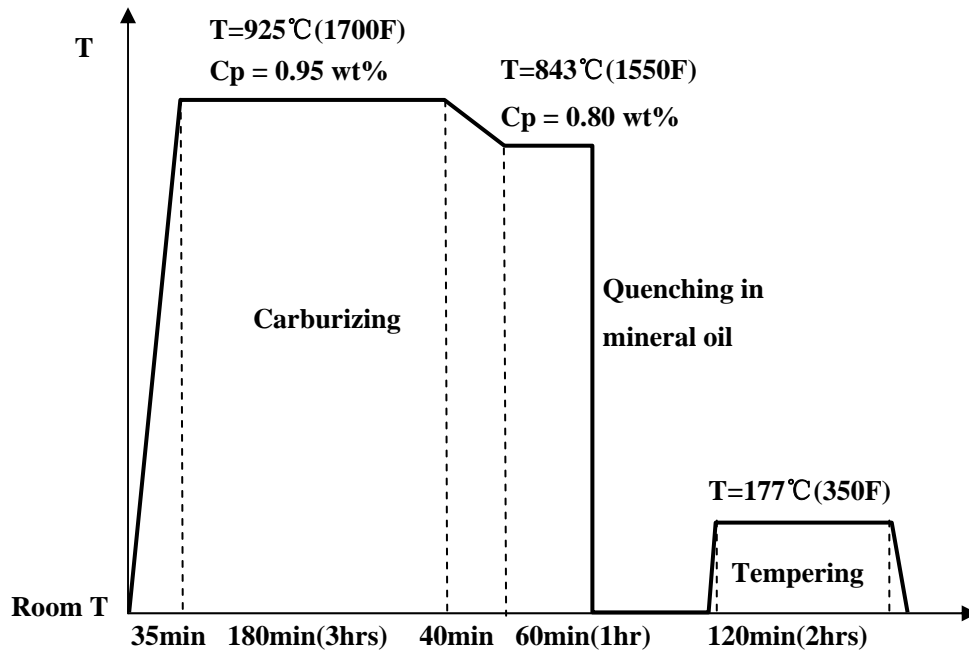
Figure 4 Carburizing process for AISI 8620

Based on the theory of diffusion of carbon in steel, the carbon profile can be estimated for AISI8620 steel carburized in an endo-gas atmosphere. A 1-D carburization simulation software CHTE-CarbTool [6] is used to estimate the carbon concentration vs. depth profile for AISI8620 steel carburized as shown in Figure 5.
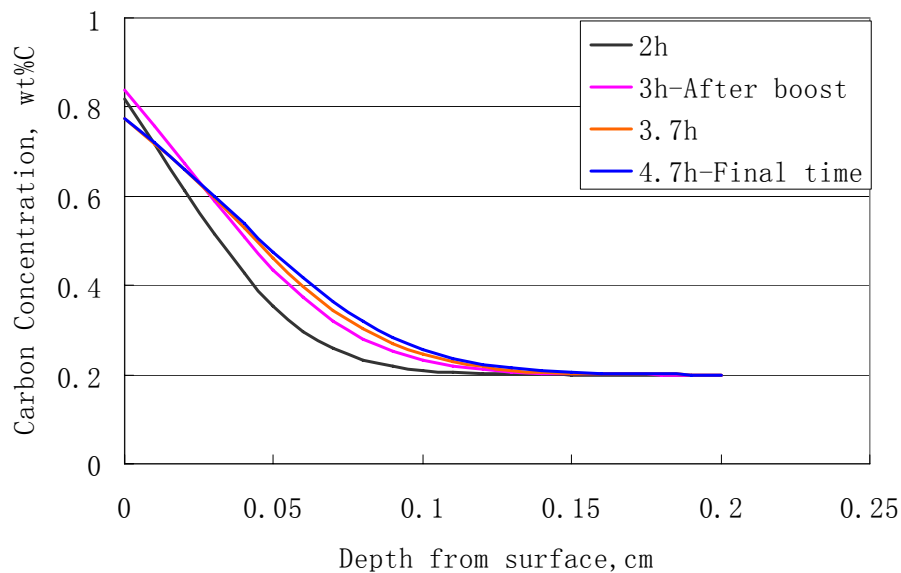

Figure 5. Calculated carbon concentration vs. depth profile by CHTE-CarbTool software[6].

3. Result and discussion

3.1 Weight gain vs. time

The laboratory scale which is sensitive to 0.1mg was used to test the weight gain. The weight of samples was tracked during these experiments. The weight of samples before and after rust process has been recorded and subtracted by original weight to obtain the rust gain during rust forming step. Weight increased when time increased and for the heavy rust sample (30 days rust), shown in Figure 6, around $0.1mg/cm^2$ of rust gained on the surface and formed an almost 1 micron rust layer. The weight after carburizing is also measured.

The weight gain after carburizing was observed to decrease as the rust layer becomes thicker, as shown in Figure 7. Compared to 2.6-$2.8mg/cm^2$ weight gain for other rusted samples, only $2.5mg/cm^2$ of carbon diffused into the 30 days rust sample. Because the rust layer prevents the carbon absorption, less carbon is diffused into sample when the rust layer thickness increased. After cleaning, the weight gain varies in a small range, from 63mg-66mg. The rust layer has removed effectively by acid cleaner.
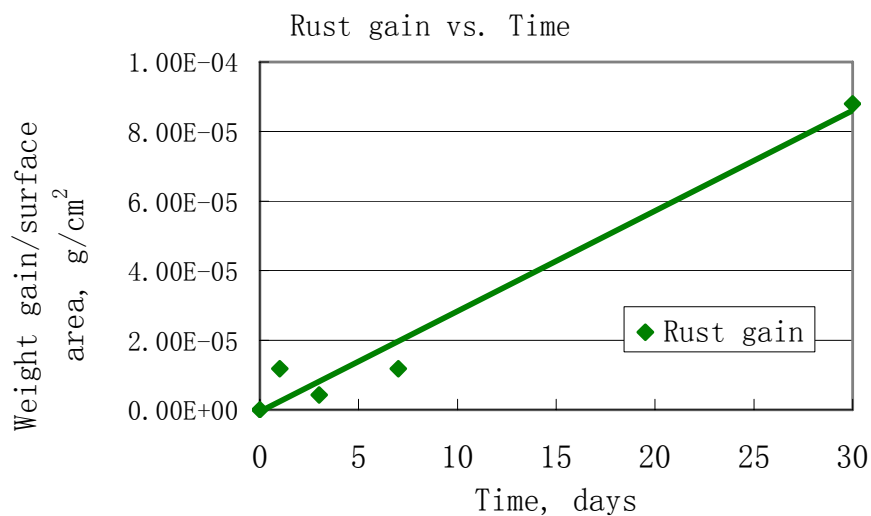

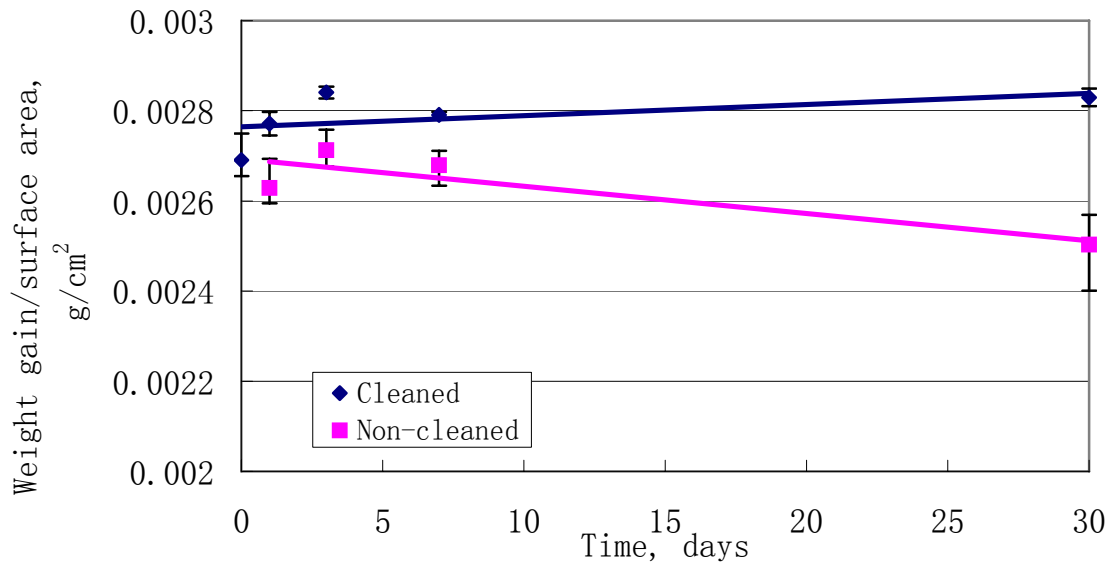Figure 6 Average weight gain of rust sample due to rust vs. time curve.

Figure 7 Average weight gain of rust sample after carburizing vs. time curve.

3.2 Surface hardness vs. corrosion time

Surface hardness was measured with Wilson Rockwell Hardness Tester. The Rockwell scale characterizes the indentation hardness of steel through the depth of penetration of an indenter, loaded on a sample and compared to the penetration in some reference material. The HR_c was measured on each sample for 5 times. Average HR_c was calculated and plotted with time line, which has been shown in Figure 8. The hardness of samples is between 59 and 61, the results is similar to Karabelchtchikova.[7] HRc resulta have been convert to HV data and shown in Figure 8. The non-cleaned sample has lower hardness compare to clean samples.
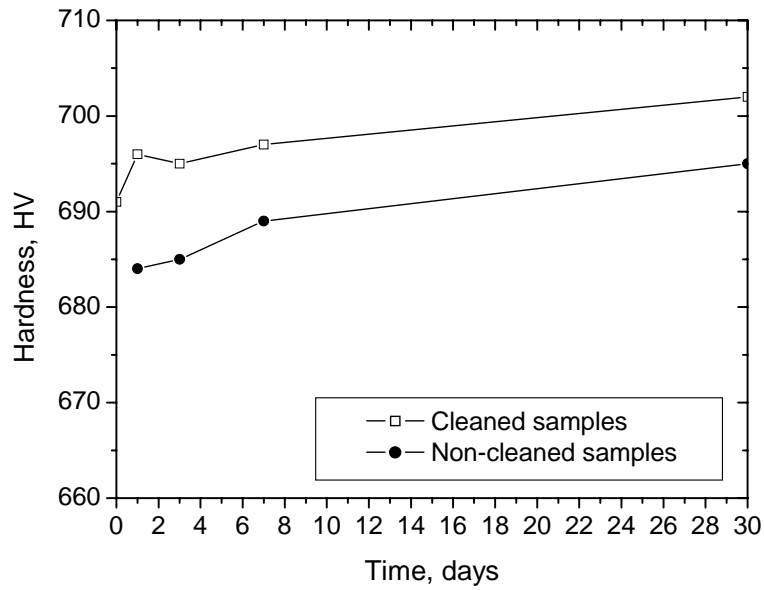
Figure 8 Hardness and surface carbon concentration vs. rust time curve.

3.3 Cross-section hardness vs. corrosion time

The Vickers hardness test is used in testing the cross-section micro hardness. The samples have been cut, polished with 800 grit sand paper and 200gf load and 15s loading time is used in Shimadzu HMV-2000 Micro Hardness Tester. The measurement starts at 100μm depth from the edge, and 50μm per measurement was taken at 100-800μm depth, 100μm per measurement was taken at 800μm-1400μm. Figure 9 shows the diamond shape made by the indenter with square-based pyramid with an angle of 136° between opposite faces.
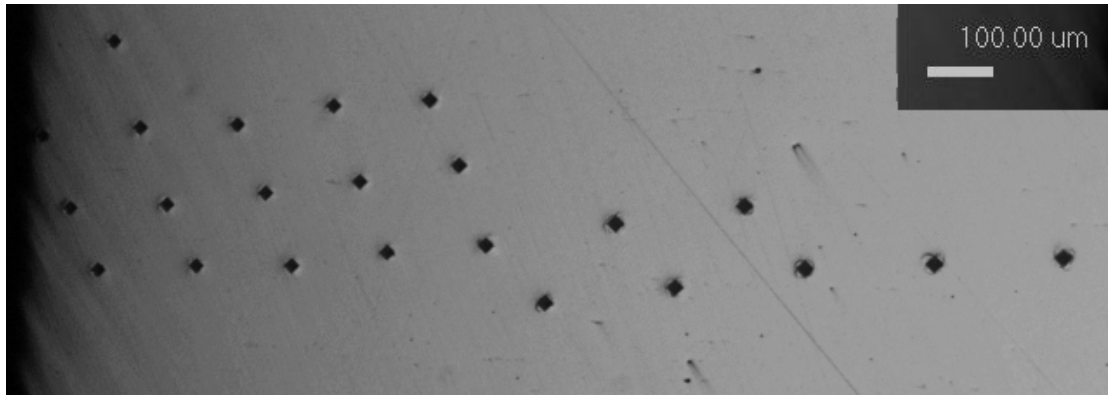
Figure 9 The optical image of the diamond shape holes made by the indenter under 200gf for 15s for Vickers Hardness test.

The results were collected and plotted in Figure 10-11 respectively. As shown in Table 3, the cross-section hardness of non-cleaned sample is slightly lower than cleaned sample. The total and effective case depths data, given in Table 3, were obtained from the micro hardness data (to 600HV, 550HV and 500HV), the data clearly suggests that carburizing the parts of the same steel grade in the same workload, and therefore, the same carburizing process parameters will be affected by the rust layer.
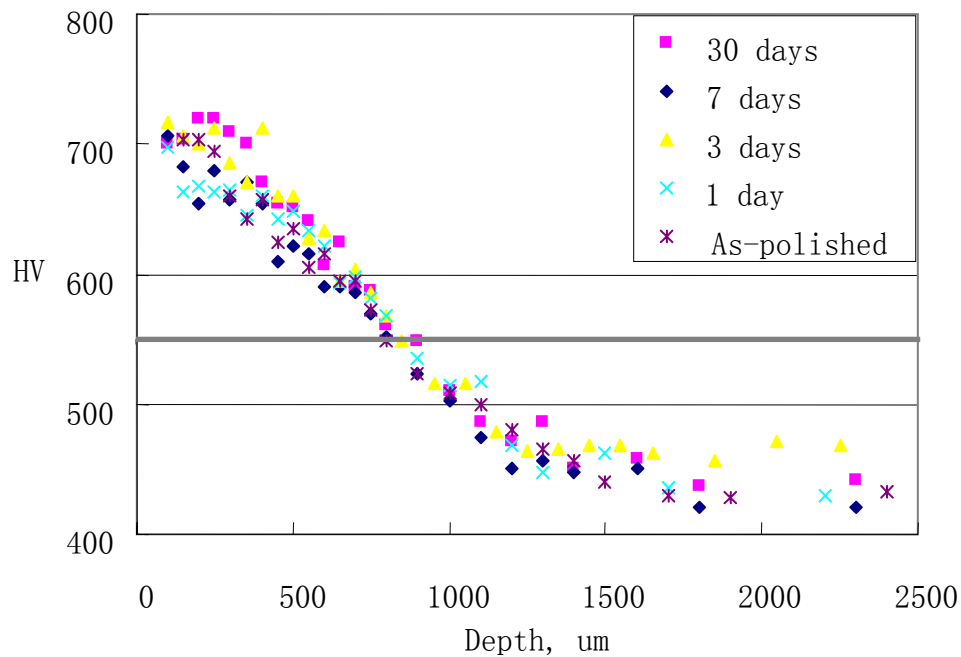

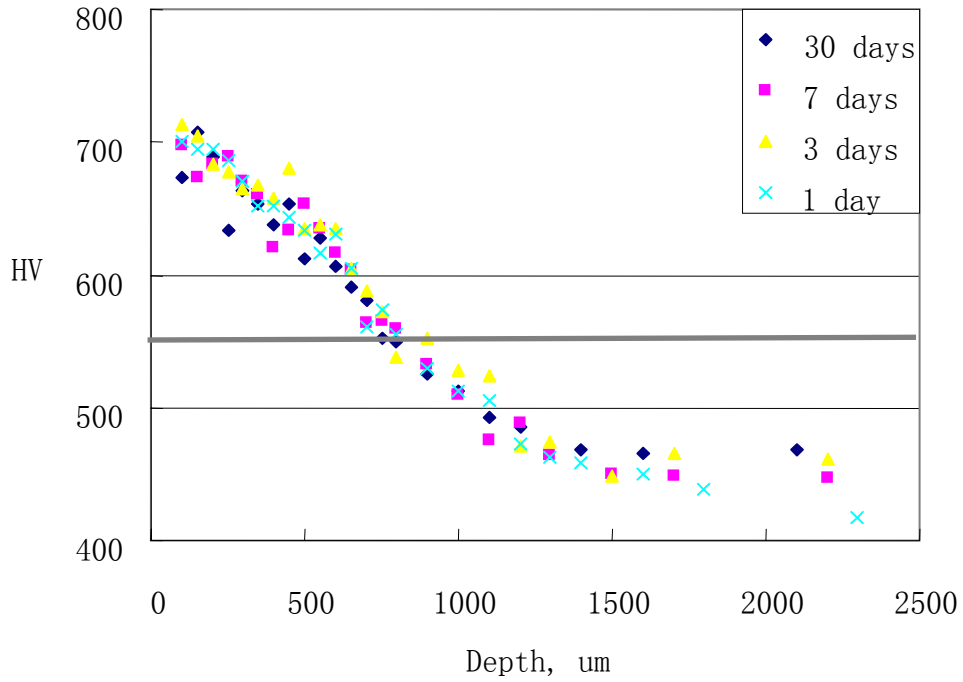Figure 10 The HV profile for rusted then cleaned sample

Figure 11 The HV profile for rusted samples.

The HV with depth curves were fitted using polynomial of degree 6 to determine the case depth for 600HV, 550HV, 500HV hardness. The data is present in Table 3.

Table 3 Case depth based on microhardness measurements.

Case depth for Cleaned sample (μm)				Case depth for Rusted sample (μm)			
Rust t	600HV	550HV	500HV	Rust t	600HV	550HV	500HV
30days	630	850	1070	30days	600	810	1090
7days	600	800	1010	7days	640	830	1050
3days	690	860	1070	3days	690	880	1100
1day	680	880	1090	1day	630	830	1070
As-polished	620	840	1070	--	--	--	--

3.4 Flux of carbon and mass transfer coefficient vs. rust time

The surface carbon concentration for samples for the same carburized condition is in the range of 0.80-0.83wt% C, as reported in Karabelchtchikova's work.[8] Spectro analysis for testing the carburized samples was based on standard ASTM E415 and performed at Caterpillar Inc, in Peoria, IL. Three measurements were taken on one side of each sample and average surface carbon concentrations are calculated and presented in the Table 4.

Table 4 Surface carbon concentration.

Cleaned sample		Non-cleaned sample	
Rust time	C_s (wt%)	Rust time	C_s (wt%)
30days	0.81%（0.79-0.84%）	30days	0.76%（0.74-0.78%）
7days	0.81%（0.79-0.83%）	7days	0.81%（0.78-0.82%）
3days	0.83%（0.82-0.83%）	3days	0.80%（0.79-0.82%）
1day	0.81%（0.79-0.84%）	1day	0.79%（0.78-0.80%）
As-polished	0.80%（0.78-0.81%）	--	--

Mass transfer coefficient in the gas phase is an important parameter. It determines the thickness of the boundary gas layer in front of the gas-solid interface and defines the maximum flux of carbon atoms reaching the steel surface and available for further diffusion towards the bulk of the steel. The total mass of the solid changes per unit surface area should be equaled to the mass accumulation within the solid during carburization, due to the flux balance condition at the steel interface:[9]

$$\int_{x_\infty}^{x_0} C(x,t)dx = \int_{t_0}^{t_f} Jdt = \frac{\Delta m}{A} \qquad (1)$$

where, m is the mass and A is the surface area of the workpiece, C is carbon concentration and J is flux of diffusing species.

The total quantity of the carbon diffusing through the surface is found by integrating the concentration profile over the depth of the carburized layer. Further differentiation of the total weight gain by the steel over the carburizing time yields the Equation 2 for the total flux of carbon atoms through the vapor-solid interface:

$$J^t = \frac{\partial(\Delta m / A)}{\partial t} = \beta(C_P - C_S^t) \qquad (2)$$

The flux in the atmosphere boundary layer is proportional to the difference between the surface carbon concentration in the steel and the atmosphere carburizing potential, the mass transfer coefficient can be presented as follows: [10]

$$\beta = \frac{\frac{\partial}{\partial t}\int_{x_\infty}^{x_0} C(x,t)dx}{(C_P - C_S)} = \frac{(\Delta M / A)}{t(C_P - C_S)} \qquad (3)$$

where, β is the mass transfer coefficient, C_s is the surface carbon concentration in the steel, and C_P is the atmosphere carburizing potential, in this case equals to 0.95wt%. The weight gain is expressed in g/cm^2, time in s, and carbon concentration in g/cm^3, the calculated mass transfer coefficient is expressed in cm/s.

Based on the C_s value and weight gains due to carburization, the mass transfer coefficient β can be easily calculated by using the equation 3and plotted with time line in Figure 12.
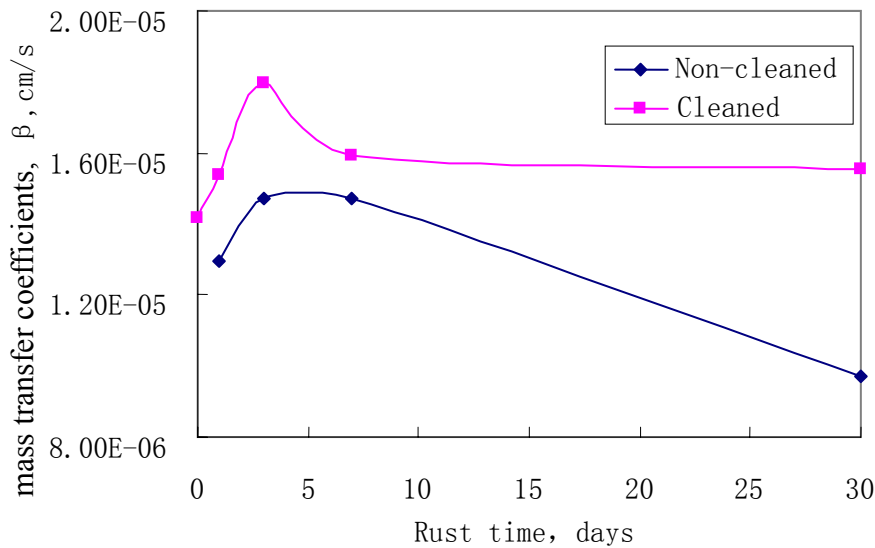

Figure 12 Mass transfer coefficients vs. corrosion times

The flux of carbon atoms diffused in the workpiece through the interface can be presented from the equation of differentiation of the total weight gain by the steel over the surface area by carburizing time. The flux of carbon J was calculated by using the equation 4 and plotted with time line in Figure 13.[11]

$$J = \frac{\partial}{\partial t}\left(\frac{\Delta M}{A}\right) \qquad (4)$$

where J is the carbon flux (g/cm^2*s), ΔM is the total weight gain (g), A is the surface area (cm^2) and t is the carburizing time(s).
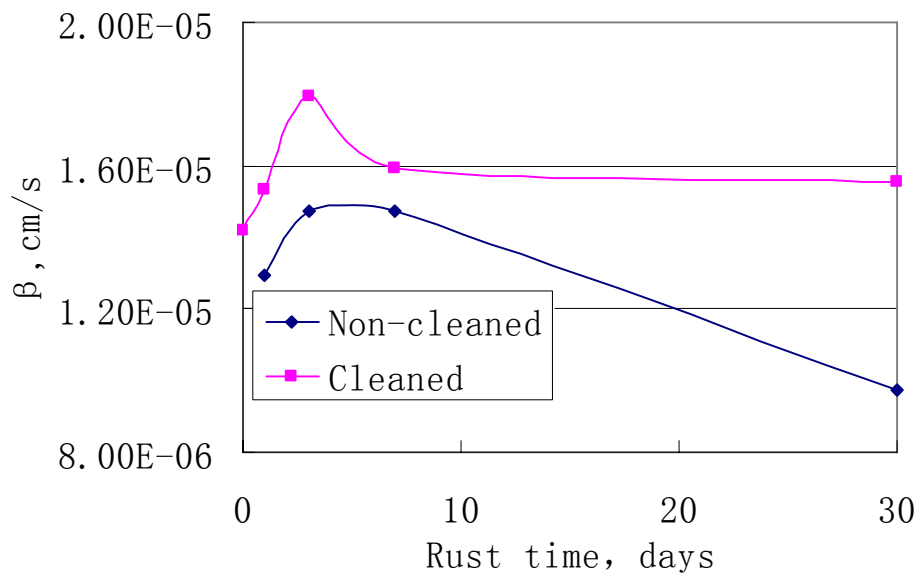

Figure 13 The total flux of carbon vs. corrosion times

3.5 Microstructure

Figures 14-16 are micrographs of AISI 8620 steel after carburization, followed by quenching and tempering. Cleaned and rusted samples both shows fine grains in the edge due to the carburization treatment. Same grain boundary condition is observed for all samples. The intergranular oxidation is formed during carburization, which is shown in Figure 16.
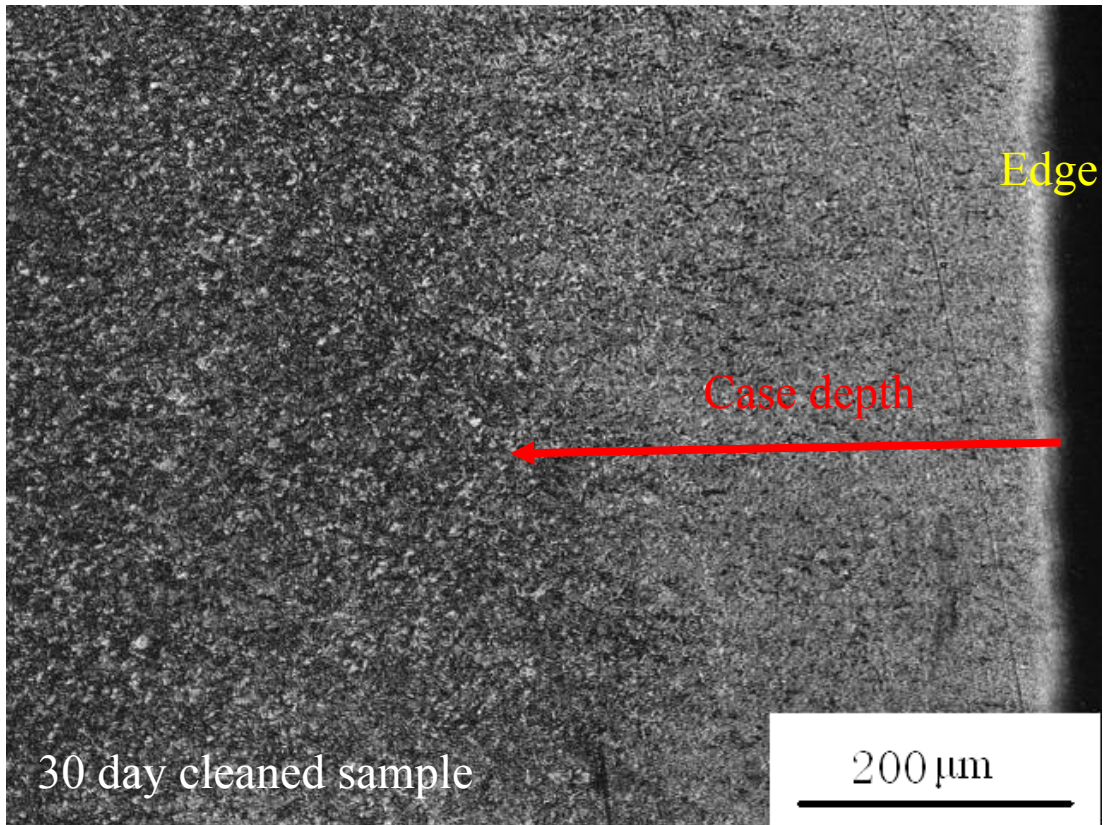
Figure 14 Photomicrograph of the cleaned (30 day rust) AISI 8260 steel's edge, etched with 2% nital etch. Case depth is based on 550HV.
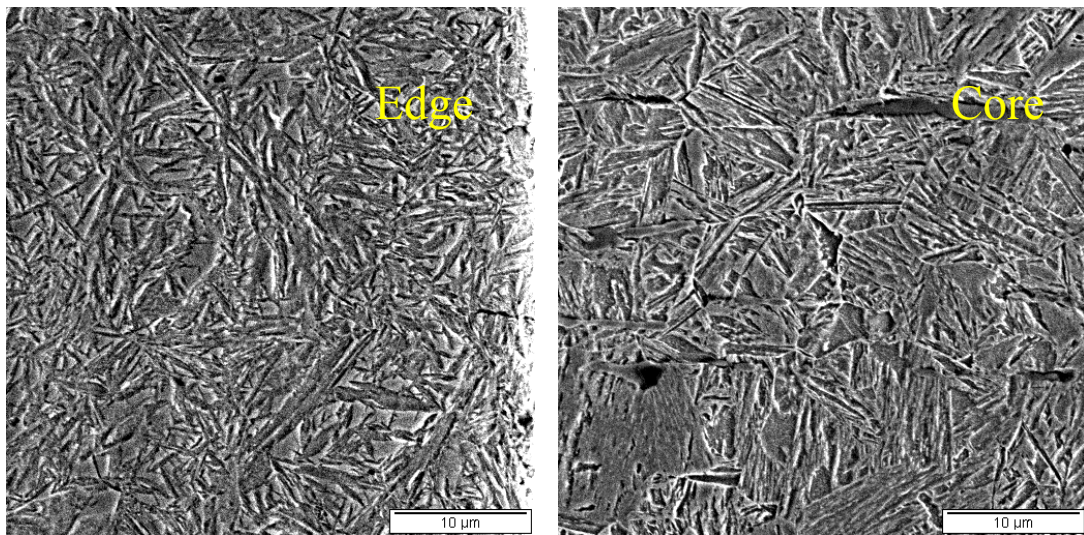

Figure 15 SEM pictures of the AISI 8260 steel's edge and core, etched with 2% nital etch
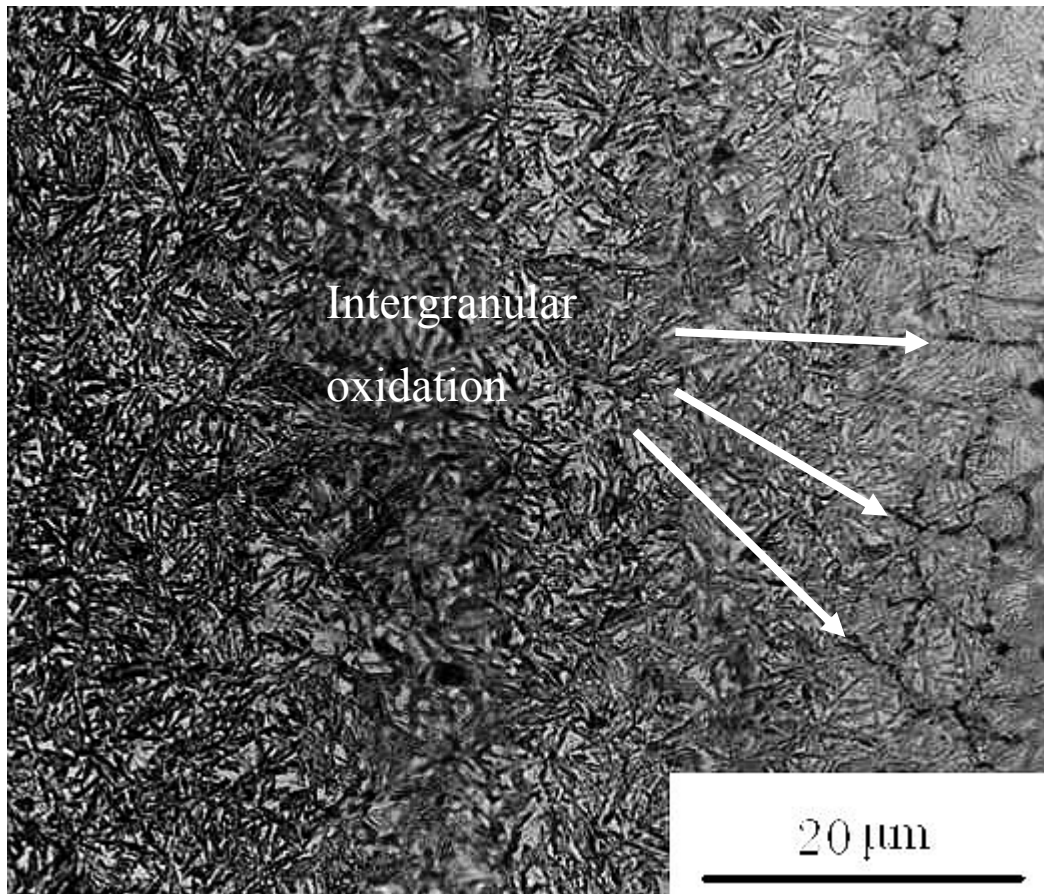
Figure 16 Photomicrograph of the AISI 8260 steel with intergranular oxide at the surface, etched with 2% nital etch.

4. Conclusion

The effects of rust layer on the hardness and mass transfer coefficient were experimentally investigated. The results are summarized as below.

- The surface hardness (R_c) didn't show a significant difference between the heavily rusted sample and clean sample. The hardness of samples carburized at 925°C for 3 hrs is between 59 and 61 HR_c .

- Acid cleaning can remove the rust layer effectively. Hydrochloric acid cleaner: 50 vol % HCl is used. Rinsing completely in distilled water is necessary to remove the cleaner residue.

- Carbon flux and mass transfer coefficient is smaller due to rust layer for the heavily rusted sample.

Acknowledgment

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