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HIGH TEMPERATURE GAS NITRIDING TREATMENT OF AISI 430 USING LOW AND HIGH PURITY NITROGEN GAS

Agus Suprihanto
Departemen Teknik Mesin, Fakultas Teknik, Universitas Diponegoro
Jl. Prof. Sudharto, SH Kampus UNDIP Tembalang
E-mail: agusm90@yahoo.com

ABSTRACT

The properties of stainless steels can be improved by high temperature gas nitriding (HTGN) treatment. The improving of their properties are obtained from nitrogen atom which diffuse into stainless steel. Nitrogen gas is the main source of nitrogen atom on the HTGN treatment. Generally, these treatment use high purity of nitrogen gas. The aim of this research is to investigate the effect of nitrogen gas purity on the HTGN treatment for AISI 430. Stainless steel AISI 430 plate 2 mm thick was processed by HTGN treatment. The specimens was exposed at nitrogen gas atmosphere at temperature 1200^{o}C and held for 2 hours prior quenching in water. The treatment used industrial/welding grade (99.5%) as low nitrogen gas purity and ultra high purity (UHP) grade (99.999%) as high nitrogen gas purity. The vickers micro-hardness test was conducted to evaluate the hardness distribution from surface into middle section of the specimens before and after treatment. Light optical microscope was applied to examine the microstructure of specimens after treatment. Metallographic examination shows both treatments using low and high purity gas have the same grain size. However HTGN treatment using low purity of nitrogen gas produces hardness slightly lower than the high purity. This is due the high content of impurity of the low purity gas that reduces the partial pressure of nitrogen gas. Another effect of impurity is the reaction between nitrogen gas and its impurity especially oxygen gas. These reactions reduce the amount of free nitrogen atom which diffuses on the stainless steel.

Keywords: high temperature gas nitriding, nitrogen gas purity

1. Introduction

Ones of materials which widely use in industry is stainless steel due high resistance to corrosion and appropriate mechanical properties. Stainless steel is ferrous alloys which contains at least 11% chromium. Depending on the phase in the microstructure, stainless steel can be grouped as austenitic, ferritic or martensitic.

AISI 430 is the most widely produced ferritic stainless steel. AISI 430 is cheapest and fairly good corrosion resistance in a variety of environments. However in certain uses such as high corrosive environments, the mechanical properties and corrosion resistance are less fulfilling. In order to improve its properties, this stainless steel can be treated by high temperature gas nitriding (HTGN) treatment.

HTGN treatment is able to enhance the properties of stainless steel such as strength, hardness, wear resistance and corrosion resistance [1]. These treatments not only improve the properties, but also it can modify the initial phase [2, 3]. The research of HTGN treatment for AISI 430 has been conducted by many investigators [4, 5]. Depending on the variable process, the micro structure of ferritic stainless steel AISI 430 can be modified into martensitic which hard or austenitic stainless steel which soft and non-magnetic.

The basic process of HTGN is by exposing the stainless steel in high purity nitrogen gas atmosphere at temperature 1050^{o}C ± 50^{o}C within certain time prior quenching [6]. During the process, nitrogen gas become dissociate into two nitrogen atom prior diffuses into stainless steel.

The effects HTGN treatment on the properties of stainless steel depends on the amounts of nitrogen atom which diffuse into stainless steel. The resulting effects depend on the process variable such as chemical composition of stainless steel, temperature process, nitrogen gas pressure and duration of holding time [1, 6, 7].

Generally, the increasing strength obtained after treatment is not higher than increasing of its hardness due to the grain coarsening. Holding time for long duration produce excessive grain size. To overcome grain coarsening, some refinement methods have been developed [8]. The refinement methods promise the prospective application of HTGN treatment in industry.

Despite HTGN treatment use high purity nitrogen gas, there are several grade of purity on the market. The highest purity and the most expensive grade is scientific or research grade which has minimum purity 99.9999%. The other grades are ultra high purity (UHP) grade (99.999%), zero grade (99.998%), specialized zero grade (99.998%), oxygen free grade (99.998%) and industrial/welding grade (99.5%). The grade purity of nitrogen gas depends on the percentage of moisture, oxygen, hydrocarbon, and CO_x.

The most widely available grade is industrial or welding grade. The industrial/welding grade promises in cost production reduction for HTGN treatment. However, the effect of nitrogen gas purity on the effect of HTGN treatment for stainless steel has not yet investigates. This research aimed to investigate the effect of nitrogen gas purity on the hardness of AISI 430 after HTGN treatment.

2. MATERIALS and METHODOLOGY

Specimens were made from 2 mm thick plate of AISI 430. The chemical compositions of AISI 430 are shown in table 1. Specimen was rinsed by ultrasonic cleaner and soaking in acetone before treatment. HTGN treatment was conducted at modified vertical tube furnace as shown in figure 1. Vertical tube furnace equipped with digital pressure gas controller and precision gas regulator.

Table. 1. Chemical composition of AISI 430

C	Cr	Ni	Mn	S	P	Si	Fe
0,03	16,0	0,22	0,604	0,005	0,003	0,22	Bal.

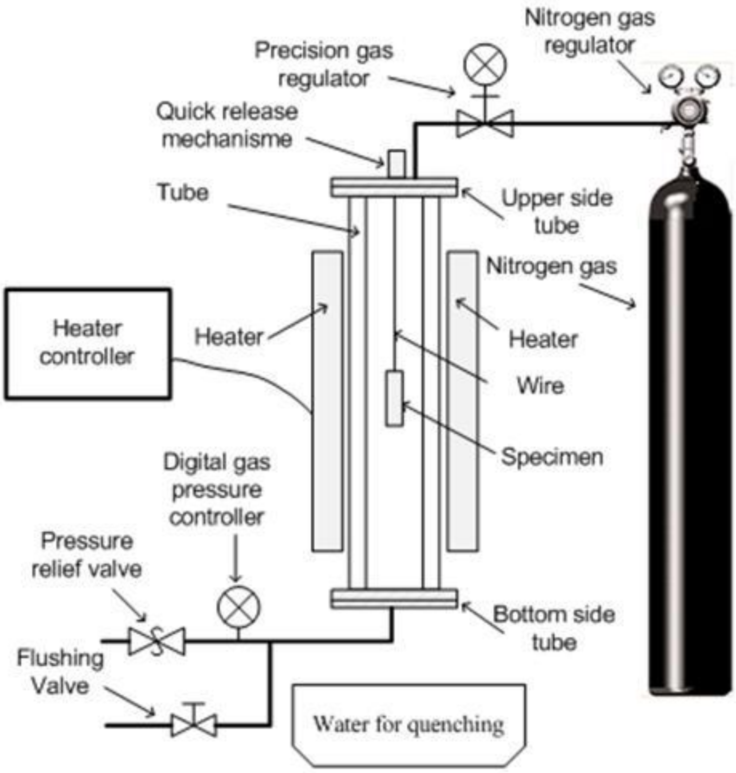


Figure 1. Schematic test HTGN equipment

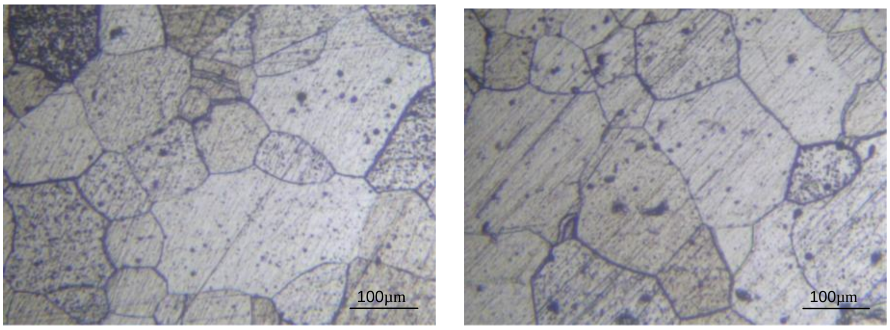
Sample was inserted into tube using wire and the wire inserted into quick-release mechanism in the upper side of the vertical furnace. The upper side and the bottom side of vertical furnace was tightened before vacuum process. The vacuum process was conducted for 20 minutes at the vacuum pressure 5 kPa prior flushing process. During vacuum process, the precision gas regulator was closed. In order to remove the air which still inside the tube, flushing process was conducted.

The precision gas regulator was open and set at the pressure 1 bar prior flushing process. The flushing process was conducted for 20 minutes using nitrogen gas. During flushing process, the nitrogen gas was flowed to the environment by opening flushing valve. After flushing process, the precision gas regulator was set at 0.3 bar, the flushing valve was closed and the heater was turned on at heating rate 10^{o}C/minutes. During heating process, the pressure inside the tube was maintain at 0.3 bar. If there were over pressure, the pressure relief valve in the gas controller equipment will open automatically and the pressure inside the tube was back at 0.3 bar. The sample was exposed at temperature 1200^{o}C for 2 hours prior quenching in the water by opening the bottom side tube and quick release mechanism.

After quenching, the sample was rinse by water and minor polished using rust removal. The sample was cut into small section prior mounted using resin for hardness test and metallographic examination. Hardness test used Buehler micro hardness tester. The hardness test was conducted from surface into middle section with equal distance. After hardness test, sample was send into metallographic examination. The microstructure was examination using Olympus light optical microscope (LOM). These procedures repeated for HTGN treatment using industrial/welding nitrogen gas grade (99.5%) and ultra high (UHP) purity grade (99.999%).

3. RESULTS and DISCUSSION

Figure 2 show the micro structure of AISI 430 after treatment. Both treatment using industrial grade and UHP grade of nitrogen gas show the same grain coarsening. Generally HTGN treatment produces grain coarsening because the process temperature is above the recrystallization temperature (T_{rec}) of steel. Figure 2 show there is no significant difference in grain size both industrial/welding grade and UHP grade purity of nitrogen gas. These indicates that the purity of nitrogen gas has not influences in grain coarsening of the sample. Grain coarsening only depends on the duration of holding time.



a. Industrial grade b. UHP grade

Figure 2. Micro structure AISI 430 after HTGN

The hardness of AISI 430 before HTGN treatment is 200 VHN. After treatment, the hardness increase significantly. Figure 3 show the hardness distribution of AISI 430 after HTGN treatment. In the surface the hardness increases from 200 VHN up to 560 VHN. The hardness is gradually decreased from surface in to middle section. However, the hardness on the middle section is higher than hardness before HTGN process.

The research on the HTGN process by other investigators show that the HTGN process depend on chemical composition of stainless steel, temperature process, duration of holding time and partial pressure of nitrogen gas. Our results show that the HTGN treatment also depends on the purity of nitrogen gas.

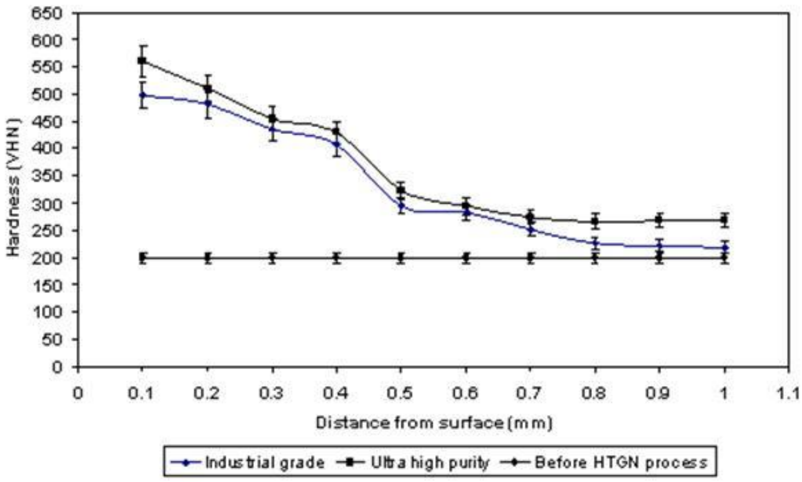


Figure 3. Hardness distribution before and after HTGN process.

Ultra high purity grade more effective than industrial grade. In the surface section, the hardness obtained by HTGN process is 561 VHN and 498 VHN for UHP grade and industrial grade respectively. In the middle section, the hardness obtained by HTGN process is 269 VHN and 219 VHN for UHP grade and industrial grade respectively. The difference of hardness between 2 grade nitrogen gas purity is about 12% and 20%.

The effectiveness of UHP grade can be explained with the effect of gas pressure on the HTGN process. The increasing of the gas pressure resulted in the increasing nitrogen atom which diffused in to stainless steel [4, 9]. Nitrogen gas UHP grade and industrial grade have different purity. Generally the impurity of nitrogen gas is moisture, hydrocarbon, oxygen and carbon-oxide gas. If the HTGN process is only depend on the nitrogen gas, then the amount of nitrogen atoms which diffuse in to stainless steel depends on the partial pressure of nitrogen gas being used. In these experiments, the gas pressure is maintain in 0.3 bar which indicate the total pressure inside the tube. The nitrogen gas pressure or partial nitrogen gas pressure in the tube is lower than the pressure that show in the digital gas pressure gauge. The partial pressure of nitrogen gas UHP grade is 0.299997 bar and the partial gas for industrial grade is 0.284 bar. The higher partial pressure of UHP grade than industrial grade is believed to be causes the higher hardness of the UHP sample.

There is correlation of increasing hardness with increasing of nitrogen contents of stainless steel after HTGN process [5, 9]. The concentration of nitrogen atom in the surface at the same partial pressure is depend on the temperature. In the distance below the surface, the nitrogen concentration not only depend on temperature but also the duration of holding time. The longest holding time produce the highest nitrogen concentration. However the maximum nitrogen concentration is limited by the maximum solubility nitrogen in the stainless steel.

Nitrogen gas which contact with surface of stainless steel is rapidly dissociation into nitrogen atom. The nitrogen atom will rapidly diffuse into the surface. Despite the nitrogen atom can easily diffuse at the surface, but it diffuse slowly into depth section. Our results show the hardness in the surface for UHP grade is slightly higher than industrial grade. These results indicate that the HTGN process is also influenced by impurity in the nitrogen. Moisture, oxygen gas and hydrocarbon as impurity in the nitrogen gas can also dissociate and it reduce diffusion process. The presence of metal which can be act as catalyst and the evaluated temperature caused the reaction between nitrogen gas and its impurity become possible. The possibility reaction is between nitrogen atom and oxygen atom. Nitrogen atom not only diffuses into stainless steel but also produces small amount of NO_x gas. This fact reduces free nitrogen atom which diffused into stainless steel.

4. CONCLUSIONS

The nitrogen gas purity has influences on the HTGN process. The low purity nitrogen gas not only causes to the lowering partial gas pressure but also reduces free nitrogen which diffused into stainless steel. The lowering partial gas and reducing free nitrogen atoms are the factor that reduce the hardness of AISI 430 between HTGN using low purity and UHP purity grade. However, the grain coarsening is only depends on the duration of holding time and it not depends on the purity of nitrogen gas.

The high purity nitrogen gas produce slightly higher hardness than low purity. However the difference of hardness obtained is not significant. Because the price of UHP grade is expensive, the using low grade purity reduce the cost of HTGN process.

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