



## Characteristics Of Surface Roughness And Microhardness Of Nitrided Pure Iron

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### ABSTRACT

The nitriding process on metal is made to improve hardness, tribological properties, machinery components, and parts durability. This nitriding process shall affect surface roughness. The surface roughness determined the quality of components at the time of its use. The surface hardness changes through the surface chemistry, and to increase the surface roughness are important things to observe the surface modification in nitriding components.

The nitriding process of pure iron has been carried out to achieved high surface hardness. The nitriding process was done at the temperature of 1000 °C for 1 hour in the nitrogen atmosphere. This work will discuss the roughness and microhardness characteristic of untreated and treated by nitriding of the pure iron surface to show the correlation of surface roughness to its microhardness. Surface roughness testing had done by mapping the sample area in an array. The surface hardness test is used on the Vickers scale. The surface roughness results showed two critical values: surface roughness (Ra) and the mean surface roughness depth (Rz). The surface roughness value of pure iron increases from 0.0465  $\mu\text{m}$  before treatment to 0.1089  $\mu\text{m}$  after treatment. The change of Rz value before treatment is 0.2333  $\mu\text{m}$  and the highest value after treatment is 1.160  $\mu\text{m}$ . The surface microhardness after nitriding increased significantly. The average increase that occurred was from 122.8 HV become 533.8 HV.

**Keywords:** Nitriding, High Temperature, Surface Roughness, Microhardness

### 1. INTRODUCTION

Improving the surface properties of materials such as hardness, tribological properties and enhanced the endurance services can be overcome with surface nitriding. The nitriding process involves a case hardening. The nitrogen would diffuse to the material surface under the activation condition. Many techniques progressively develop to replace the conventional nitriding of iron and steel [1]. The conventional nitriding has weakness in the long duration of the nitriding process that would occur hot spot on the surface of the material and found a white layer on the surface. Besides, these techniques can be implemented on material with an irregular shape.

The mechanical strength of material iron-based degrades above 600 °C [2]. Iron degradation at high temperature limits the thermal plasma's use by an arc or torch that operated at an atmospheric or above in iron nitriding process. Several researchers have successfully demonstrated arc plasma for nitriding of stainless steel, high carbon steel, low carbon steel and titanium at 1000–1200 °C [3]. The synthesize coating of carbon nitride with the extended nitride graphite method has been done [3]. Nitriding combine with carbon possible occurred. The carbon reaction with nitrogen made its more stable solid in natural carbon is not a stable element in solid.

The nitriding mechanism on the metal surface has been revealed and extensively studied. The new method as plasma nitriding agrees for a significant role of  $N_2^+$ ,  $NH^+$ ,  $NH_2^+$  and fast neutral molecules in the nitrogen transfer on the solid surface [4]. The gas nitriding that using of ammonia would occur dissociation at 500–600 °C [5].

Nitridation and surface diffusion are entirely the responsibility of nitrogen [5]. In contrast to the gas nitriding process at a high temperature around 1000 °C which is rare to find in the literature. The gas nitrogen molecules have triple bonds with 945 kJ/mol, the high temperature necessary to dissociate  $N_2$  [6]. At this temperature, there is a small chance the diffusion of nitrogen atoms on the surface. Meanwhile, at the temperature 1000 °C the nitriding process possible to occurs. A nitride layer could form by the participate flux of ion  $N_2$  that diffuse on the surface material [2]. Solubility of nitrogen in pure iron at standard pressure described with equations [7],

$$\begin{aligned} \gamma - \text{range: } \log[m - \% N] \\ = \frac{441.0}{T} - 1.930 \end{aligned} \quad (1)$$

$$\begin{aligned} \delta - \text{range: } \log[m - \% N] \\ = \frac{1600}{T} - 0.9372 \end{aligned} \quad (2)$$

Where the nitriding temperature at 1000 °C is took in  $\gamma$ -range.

The quality of industrial components at the service, determined by the important role of surface roughness improvement. The significant application on cutting tools, electric hardware, automotive components, and coated parts [8]. Increasing the properties of surface material such as hardness and roughness could occur by changing the surface chemistry, and of course, there are modifications to the surface. Means of the surface roughness is regarding endurance and the nature of the material. Utilize surface hardening treatment to increase the lifetime of cutting tool components would occur. The nitriding process is the proper and perfect technique to do the thermochemical treatment. This treatment had better precision compared to the other thermochemical treatments such as carburizing and carbonitriding [8]. The nitriding process carried out at 1000 °C needed to fill the gap about interaction iron with nitrogen, that affect to surface characteristics. Furthermore, this work investigates the surface roughness and hardness of untreated and treated pure iron by nitriding and

showed the correlation of surface roughness to its microhardness.

## 2. METHODOLOGY

### 2.1. Sample preparation

The material used is pure iron which has been hot-rolled and annealed. The composition of pure iron is shown in Table 1. The dimensions of samples as follow, the sample width is 20 mm, the length is 50 mm, and the thickness is 2 mm.

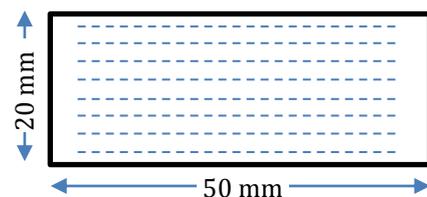
Before treated with nitriding, the surface of the material is grinding with sandpaper with a mesh size of 1000 and polished. The nitriding process's temperature and time are carried out at a temperature of 1000 °C and held for 1 hour, in order to increase the possibility of dissociation and diffusion of nitrogen into iron [3]. The gas used in the nitriding process is  $N_2$ . Untreated and treated pure iron were characterized for surface roughness and microhardness.

**Table 1.** Chemical composition of pure iron (%wt) [9].

C	Al	Mn	N	P	S	Si	Fe
0.0028	0.001	0.16	0.0017	0.011	0.004	0.001	Balance

### 2.2. Surface roughness testing

Pure iron samples were tested for surface roughness using a Surtonic 25 roughness tester. The surface roughness apparatus resolution is 0.01  $\mu\text{m}$ . Before the test, for untreated and treated pure iron samples were cleaned with a fine tissue to remove dust and grease adhering to the surface so that the measured surface is free from debris or additional vibrations. The roughness test is carried out with the schematic, as Figure 1 described. The surface roughness path measurement is illustrated with a dotted line (the results have given the notation 0,1,2,3,4,5,6, and 7). The measurement length is 40 mm, and the distance from one line to the next is about 2 mm.



**Figure 1.** Schematic of surface roughness measurement.

The surface roughness measurement introduces two value calculated by the analytical software that came with the apparatus. The surface roughness ( $R_a$ ) value is derived from calculating the average

length between the peaks and valleys, and the deviation from the mean lines over the entire surface within sampling length. Averaging all peaks and valleys of the surface roughness profile made an outlier, and extreme points effect is eliminated from  $Ra$ 's final results [10]. The mean roughness depth ( $Rz$ ) value calculates the average vertical distance from the highest peak to the lowest valley from five sampling lengths [10,11]. This calculation makes  $Rz$ 's final value highly influenced by the distance between the five samples' peak and valley. The equation for calculating the  $Ra$  and  $Rz$  are shown below [10],

$$R_a = \frac{1}{n} \sum_{i=1}^n |y_i| \quad (3)$$

Where  $y_i$  is the acquired height and  $n$  is number the data taken from the sample.

$$R_z(ISO) = \frac{1}{n} \left( \sum_{i=1}^n p_i - \sum_{i=1}^n v_i \right) \quad (4)$$

$$R_z(DIN) = \frac{1}{2n} \left( \sum_{i=1}^n p_i + \sum_{i=1}^n v_i \right) \quad (5)$$

Where  $p_i$  is for height of peak and  $v_i$  is for deep of valley and  $n$  is number of the data taken from the sample.

### 2.3. Microhardness testing

Hardness on the surface of the untreated and treated samples were measured using a microhardness LECO LM800AT with the Vickers scale. The test load given is 0.5 gf for untreated samples. Meanwhile, for the samples treated with nitriding, the test load was 25 gf, and three repetitions for each sample. The dwelling time for each test is 10 seconds. The indentation of microhardness is carried out in the center position of the sample. The microhardness of material is calculated using equation 6 [12].

$$\text{Hardness (HV)} = \frac{2000 \times P \times \sin(\alpha/2)}{1854.4 \times \frac{d^2}{d^2}} \quad (6)$$

$P$  is the force given during the testing process (gf), and  $d$  is the average of horizontal and vertical indentation diameter ( $d_1$  and  $d_2$  in  $\mu\text{m}$ ). Hardness is the microhardness of material (HV or  $\text{kgf}/\text{mm}^2$ ).

## 3. RESULTS AND DISCUSSION

The surface colour of pure iron changes indicates the element such as nitrogen interacts with the metal. The metal of nitriding changes its colour from silvery to grey.

### 3.1. Surface roughness

The surface profiles of the samples before and after nitriding are shown in Figures 2 and 3. The surface roughness values are shown in Table 2 and the mean roughness depth are shown in Table 3.

**Table 2.** The surface roughness ( $Ra$ ) value in units of  $\mu\text{m}$ .

	Before	After
0	0.0479	0.1044
1	0.0465	0.1036
2	0.0475	0.1031
3	0.0474	0.1089
4	0.0476	0.0969
5	0.0478	0.1062
6	0.0474	0.0973
7	0.0468	0.1031

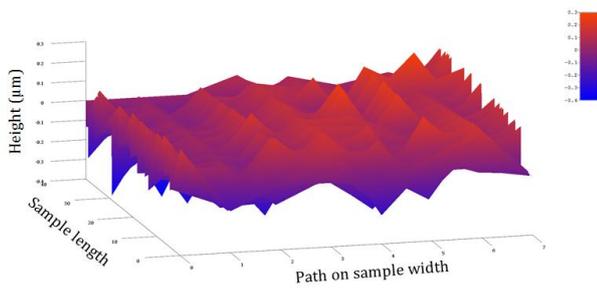
**Table 3.** The mean roughness depth ( $Rz$ ) value in units of  $\mu\text{m}$ .

	Before	After
0	0.2433	0.8267
1	0.2633	0.81
2	0.2867	0.8533
3	0.2933	1.16
4	0.2833	0.9233
5	0.2767	0.9133
6	0.2333	0.78
7	0.28	0.8567

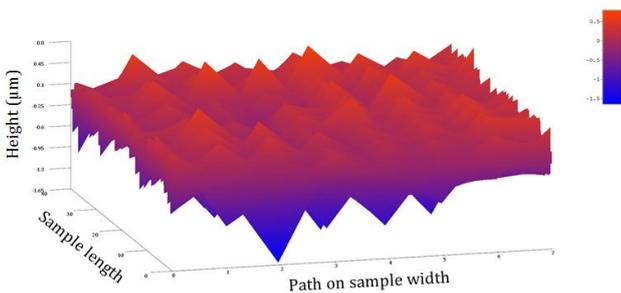
From Figure 2, the surface roughness profile before nitriding is showed. The surface profile before the nitriding process shows an even depth of roughness on the surface. This condition is different from what happened on the surface after the nitriding process, which shows a profile that has a certain depth of roughness with a wider area, as shown in Figure 3. The diffusion of nitrogen on the surface of material formed a nitride layer. This arrangement of the layer on the surface affects the roughness [13].

The surface roughness value ( $Ra$ ) on the pure iron surface before the nitriding process was in the range of  $0.05 \mu\text{m}$  due to the rounding. Meanwhile, after the nitriding process, there was a change in

the order in the 0.10  $\mu\text{m}$  range. Average  $R_a$  value for untreated pure iron is 0.0474  $\mu\text{m}$  and for the pure iron after nitriding is 0.1029  $\mu\text{m}$ . The mean roughness depth on the surface of pure iron also changed significantly.



**Figure 2.** Profile of surface roughness pure iron before nitriding



**Figure 3.** Profile of pure iron surface roughness after nitriding.

Table 3 showed that mean surface roughness depth ( $R_z$ ) of untreated pure iron around 0.23 to 0.29  $\mu\text{m}$ , for the nitriding of pure iron showed the  $R_z$  around 0.78 to 1.16. The changes  $R_z$  value around three times for a treated surface compare to the untreated surface. Average  $R_z$  value for untreated pure iron is 0.2700  $\mu\text{m}$  and for the nitriding of pure iron is 0.8904  $\mu\text{m}$ .

### 3.2. Microhardness

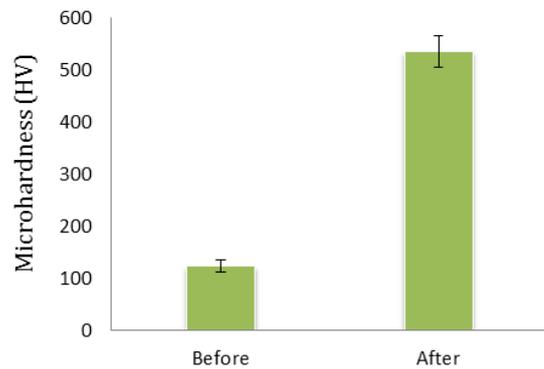
The surface microhardness test results showed an increase in microhardness after nitriding when compared to before. The microhardness after the nitriding process was obtained at 533 HV, from 122 HV previously. Detailed results of surface microhardness as calculated using equation 6, and the measurement results are presented in Table 4, and the comparison before and after nitriding is shown in Figure 4.

A nitride layer is formed on the surface, which increases its hardness [14,15]. The results obtained from this activity were higher than those obtained by Nayak et al., where the hardness was only in the

range of 282 – 380 HV for nitrided grey cast iron [3]. Meanwhile, Wang and Liu showed the hardness value for 501 HV at a depth of 100  $\mu\text{m}$  from the surface of the nitriding of grey cast iron at a temperature of 570  $^{\circ}\text{C}$  [16]. Shen et al. showed similar surface hardness value for nitrided pure iron with simplex nitride technique at 500 HV [17].

**Table 4.** Pure iron surface microhardness before and after the nitriding process.

No.	Before			After		
	d1 ( $\mu\text{m}$ )	d2 ( $\mu\text{m}$ )	HV	d1 ( $\mu\text{m}$ )	d2 ( $\mu\text{m}$ )	HV
1.	8.70	8.70	122.5	9.42	9.06	543.0
2.	8.57	8.79	123.1	9.53	9.72	500.4
3.	8.06	8.06	142.7	9.42	8.81	558.0
	Average		122.8	Average		533.8



**Figure 4.** Surface roughness profile after nitriding.

In the Flury et al. 2012 [18] study about surface roughness's relationship to microhardness for ceramic material. The microhardness results on a rough surface may not show their actual results. The microhardness of material showed a lower value when the  $R_a$  value near to 0.5  $\mu\text{m}$  or higher with the  $R_z$  value near to 2  $\mu\text{m}$  or above [18]. Another result from Sujana and Astana, 2017 about correlation of surface roughness with microhardness on low carbon steel showed after nitriding process the surface roughness increased with lowering its microhardness [19].

The microstructure results showed the nitride layers were brittle, low bonding interface with matrix and non-uniform. Nevertheless, Gamal et al. 2015 [20] showed a positive correlation of the relationship of surface roughness and microhardness for metal material. Although the relationship between surface roughness and surface hardness of nitrided iron cannot be seen clearly from this activity, there is a positive linear relationship between them. Therefore, the nitride

layer formed on the surface iron had an excellent interface and bonding with the matrix. Aside from that, the nitride layer overall showed a uniform and sturdy based on its surface roughness and microhardness after the nitriding process.

#### 4. CONCLUSION

The surface roughness and microhardness after the nitriding process significantly changes. This showed the changed effect on its surface roughness still tolerable for increasing the microhardness. The average surface roughness value changes from 0.0474  $\mu\text{m}$  become 0.1029  $\mu\text{m}$ , with the average surface roughness depth changes from 0.2700  $\mu\text{m}$  become 0.8904  $\mu\text{m}$ . The microhardness was an outstanding increment from 122 HV become 533 HV. We will take further work on how the phase and its microstructure influences the surface roughness.

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