A Study on Nitriding Alloy Steel in INTI Plasma Focus Machine

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Abstract. The INTI Plasma Focus machine was used to irradiate AISI 304 stainless steel samples with nitrogen ion beams to improve the hardness of the sample surface. The increase in hardness was plotted as a function of pressure at a constant 12 kV operation. Optimum hardness was found at 1.5 Torr using the Vickers Hardness test. Computation with the Lee codes produces a plot of ion beam energy versus pressure. The plot shows that the optimum ion beam energy of 7.3 Joules occurs at 1.5 Torr. Thus we have correlated the optimum hardness of the treated sample surface to the optimum ion beam energy.

1. Introduction
Nitriding is a process in which nitrogen ions are diffused into the surface of a metal to create surface hardening. The surface hardening is important because it is directly proportional to the stress at the location of an imposed strain to prevent any deformation on the surface. Nitriding can be done via gas nitriding, salt bath nitriding and plasma nitriding [1]. In a research article exploring nitriding on low carbon steel material by using the dense plasma focus machine, the researcher [2] observed that the distance of the sample from the anode has an inverse relationship with the hardness and the further the distance of the sample from the anode, the less was the hardness obtained. Another researcher, Al-Hawat et.al obtained results that showed that the surface hardness increased with increasing shot number and decreased with increasing distance from the anode [3].

In this study, the surface hardening of AISI 304 alloy steel (T300 Series Alloy steel) also known as chromium nickel alloy steel was carried out. This study has two main objectives which are as follows:

- To study the effect of operating the plasma focus in different nitrogen gas pressure on surface hardness of AISI 304 alloy samples treated in the plasma focus
- To study the effect of ion beam and its energy using Lee codes [4] on the surface hardness of AISI 304 alloy

2. Methodology Used
In this research, the following methodology is used. First, the sample is prepared. It is then nitrided by exposing to the plasma focus radiation, and finally, the results are analyzed. The flow chart is as shown in Figure 1.
To prepare the sample, the steel bar (AISI 304 alloy) was cut into the small square sample (25mm × 25mm × T 8 mm) that fitted in the sample holder (placed 40 mm from the anode tip [2]). The samples were then milled to remove the outer layer (remove the oxide layer). The samples were then gradually heated from room temperature to the required temperature (over a period of 3 hours) and then kept constant at 1060°C for 1 hour. The samples were then allowed to cool down for 24 hours [5]. The samples were then polished and grinded to remove any oxide on the surface layer of the steel [6]. The hardness of the steel was measured using a Vickers Hardness test. This process is shown in Figure 2.
Figure 2: The process of preparing the samples

The samples were then placed into the Plasma focus machine. A Franklin vacuum pump was used to evacuate the 6 litre INTI plasma focus chamber to a base pressure of 0.01 Torr. It was then filled with the required nitrogen gas to a higher pressure than required before being evacuated until the desired pressure (0.5, 1, 1.5 and 2 Torr) was reached.

The capacitor bank was then charged up to 12 kV before its energy was discharged into the nitrogen gas. The nitrided steel sample after 4 shots is shown in Figure 3.

Figure 3: The alloy sample after nitriding.

Vickers Hardness test was carried out and the change in hardness was recorded.
At the same time, a Digital Phosphor Oscilloscope [Tektronix-TDS3034C] was used to observe the current derivative waveform obtained using a Rogowski coil. This waveform was numerically integrated to obtain the waveform of the discharge current. The voltage waveform was also monitored. The presence of an intense voltage spike indicates the strong focusing (plasma pinch) action. A sample of the oscilloscope screen is shown in Figure 4.

![Oscilloscope Screen](image)

**Figure 4:** The current derivative (top yellow trace) and voltage waveform from INTI Plasma Focus machine at 1.5 Torr nitrogen gas.

The current derivative data file obtained from the Digital Storage Oscilloscope (DSO) was integrated and the current waveform was obtained. This was then inputting into Lee 6 phase code [7].

To use the Lee code, the bank, tube and operational parameters were input into the codes. The bank parameters consist of the values for the capacitance, inductance and unavoidable stray resistance. The length and radius of the anode, as well as the radius of the cathode, were also input into the code.

For the operational parameters, the voltage of 12 kV, nitrogen gas and pressure of operation (0.5, 1, 1.5 and 2 Torr) respectively were keyed in the input.

Finally, the model parameters (mass and current factor in both axial and radial phase) were obtained by fitting the computed current waveform to the measured current waveform. The flow chart for this is shown in Figure 5. The current fitting is shown in Figure 6.
Figure 5: The steps in using the Lee Codes.

Figure 6: Computed current waveform of INTI machine at 12 kV, 1.5 Torr nitrogen gas fitted to the measured current waveform.
Once the current waveform had been fitted, the plasma dynamics and properties of the focus for that particular shot were computed within the code and also printed in the code output.

3. Results and Discussion

We measured the average of the hardness value of the sample. The pattern of plasma treatment is represented in Figure 7. The hardness reading at the centre of the pattern was taken. The average was calculated by obtaining the hardness value from the centre, right, left, above and below. The outer rings were not measured since these apparently were caused by shock wave interaction whereas the central region was primarily due to ion beam interaction.

![Surface affected by the shockwave](image)

**Figure 7:** The formation of rings on the test sample

The results obtained from the Vickers Hardness Test is shown in Table 1.

| Sample No. (pressure) | Surface Hardness at the center (HV 0.3 kgf) Average value after nitriding (HV 0.3) | Average value before nitriding (HV 0.3) | Percentage improvement in hardness |
|-----------------------|----------------------------------------------------------------------------------|----------------------------------------|----------------------------------|
|                       | Center | Right | Left | Above | Below          |                                        |                                   |
| 1 (0.5 Torr)          | 260.7  | 244.5 | 250.8| 225.2 | 219.0 | 240.0 | 154.6 | 55.2 |
| 2 (1.0 Torr)          | 309.5  | 303.7 | 294.0| 295.4 | 289.9 | 296.7 | 158.3 | 87.4 |
| 3 (1.5 Torr)          | 351.2  | 434.1 | 306.6| 389.4 | 402.0 | 376.6 | 158.1 | 138.2 |
| 4 (2.0 Torr)          | 298.1  | 337.5 | 274.7| 353.0 | 327   | 318.2 | 156.5 | 103.3 |

Table 1 is plotted in Figure 8, representing the percentage improvement in hardness at different pressures. The best pressure for improving the hardness was at 1.5 Torr which improved the hardness by 138%.
From Lee code, when the computed current waveform was fitted to the measured current waveform, the information on the ion beam energy [8,9] was extracted to find the pressure where the maximum value occurred. This data is shown in Table 2.

| Sample No. (Pressure) | Ion Beam Energy (J) |
|-----------------------|---------------------|
| 1 (0.5 Torr)          | 2.55                |
| 2 (1.0 Torr)          | 4.44                |
| 3 (1.5 Torr)          | 7.25                |
| 4 (2.0 Torr)          | 6.23                |

Figure 8: Improvement in hardness at different pressures

Figure 9 shows a plot of the values of beam energy at different pressures.

Figure 9: Beam energy at different pressure
When the beam energy and average hardness is plotted together as shown in Figure 10, it shows that the hardness and maximum beam energy occurs at the same optimum pressure of 1.5 Torr. There is good correlation between the hardness of the face of the treated sample and the ion beam energy produced by the plasma focus.

Figure 10: Plot of beam energy and hardness after nitriding versus pressure
(Note: Magnitude not using same scale)

Extracting information from Tables 1 and 2, the waveform of the percentage improvement in hardness versus Beam Energy was plotted as shown in Figure 11.

Figure 11: Plot of the percentage improvement in hardness vs beam energy

The figure shows that as the beam energy increases from 2.5 to 7.3 Joules, the hardness improves from 55 % to 138 %. The plot in Figure 11 reveals that for this sample, the hardness relates to the beam energy, by the equation:

\[ \% \text{ improvement in hardness} = 25.6 E^{0.82} \]
4. Conclusion
Nitriding improves the hardness of the steel due to the penetration of nitrogen ions into the surface of the steel. The ions are emitted in a narrow beam along the axis of the device. This narrow beam strikes the central region of the interaction pattern. The high density of energy deposited by the ion beam in this central region causes a high temperature and localized melting. The results of computation show that the beam energy correlates with the hardness of the irradiated sample. This is consistent with the concept of increase of hardness due to the diffusion of nitrogen ions into the surface of the metal. This paper is a preliminary study in investigating the relationship between beam energy and improvement in hardness of the AISI 304 stainless steel alloy. Further studies should be conducted to determine the upper limit of hardness and the beam energy required to achieve the upper limit. The effect of the number of shots required to diffuse the required number of nitrogen ions should also be considered.

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