Radiation shielding, mechanical and tribological properties of treated AISI304L using H₂/N₂ rf plasma

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Abstract. The electro-slag re-melting technique was used to produce locally two standard stainless steel samples, AISI304L and AISI316L. The shielding properties of both neutrons and gamma radiations were determined using He-3 and NaI(Tl) detectors. A mixture of hydrogen and nitrogen gases (1:1) was used to investigate the effect of rf plasma surface engineering on properties of austenite stainless steel AISI304L which is used in many parts of nuclear power plant. The X-ray diffraction technique was used for recognizing the structural phases on the studied austenite stainless steel samples. The rf plasma process was performed for half an hour at stable gas pressure and water cooling rate. As a result of the rf plasma surface engineering process, enormous enhancement was achieved in surface microhardness, tribological properties and remarkable increase in the absorption cross section for slow neutrons. Moreover, the gamma ray attenuation properties for the HN304L sample is to great extent comparable with those of AISI316L and AISI304L austenite stainless steel samples. As a consequence, the obtained results give a further support in favor of the rf plasma engineering in the nuclear domain.

1. Introduction.
The construction of the nuclear power plant unit is based mainly on different grades of stainless steel; such as austenite AISI316L and AISI304L stainless steels [1-2]. Where, they can be used for construction of reactor pressurized vessels, control rods, pipes and valves. The long-term operation of these important parts inside the nuclear power plant may demand specific characteristics for stainless steels comprising them. The rf plasma surface engineering treatment process is considered a good choice for enhancement standard austenite stainless steel [3-6], without any side effects on nuclear properties of the material [7]. The paper points out the role of rf plasma surface engineering by the ratio (1:1) gas mixture of hydrogen and nitrogen gases on physical and nuclear properties of AISI304L standard austenite stainless steel as compared with that of untreated austenite AISI316L and AISI304L stainless steels.
2. Materials and methods
Two austenite stainless steel samples AISI304L and AISI316L were prepared using a 30 Kg pilot plant medium frequency induction furnace (IF) followed by refining in electro-slag refining (ESR) at the steel making pilot plant unit in Central Metallurgical Research and Development Institute (CMRDI), Egypt [1-2]. The produced ingots were hot forged to steel bars with cross section 30x30 mm2. Samples from the forged steels were initially solution annealed at 1050°C for 30 min and followed by water quenching. The elemental concentration of these two standard samples is presented in table 1

Table 1. Elemental concentration for austenite stainless steel samples

| Elemental cont., wt. | AISI304L | AISI316L |
|---------------------|----------|----------|
| C                   | 0.017    | 0.02     |
| Mn                  | 1.49     | 1.22     |
| Si                  | 0.453    | 1.94     |
| Cr                  | 18.48    | 16.41    |
| Ni                  | 8.08     | 9.89     |
| Mo                  | 0.166    | 2.00     |
| V                   | 0.077    | 0.115    |
| Ti                  | 0.004    | 0.246    |
| S                   | 0.004    | 0.010    |
| P                   | 0.035    | 0.010    |
| N                   | 0.020    | 0.030    |
| Fe                  | Balance  | Balance  |

One of these austenite stainless steel samples AISI304L was treated by radio frequency plasma surface engineering process and compared by the other two untreated native austenite AISI304L and AISI316L stainless steel samples.

The radio frequency plasma surface engineering process was performed inside a quartz plasma reactor tube filled with a mixture of hydrogen to nitrogen gases (1:1) at stable gas pressure of 7.5×10⁻² mbar and water cooling rate of 3200 cm³/min. The treatment plasma power was fixed at 500W for half an hour. The rf plasma system includes a thermocouple for measurement of the HN304L sample surface temperature during treatment process [3-7]. All selected samples were polished and cleaned by alcohol using an ultrasonic apparatus for 20 minutes before treatment process.

XRD instrument type X’PERT PRO. PANLYTICAL was used for recognizing the structural phases on the studied AISI316L, AISI304L and treated HN304L austenite stainless steel samples.

Vickers diamond in position device type Leitz Durimet calibrated at 25°C ± 3°C according to ISO/IEC 17025:2005 [8] was used for surface microhardness measurements by applying load of 100 gmf. A calibrated oscillating ball on disk tribometer was used for investigating abrasive dry wear and sliding friction properties, by 6 mm ball of tungsten carbide moving on selected sample surface at mean velocity 30 mm/s and 3N applied load, humidity 35% - 40% and room temperature of 25°C ± 3°C.

Slow neutrons attenuation properties were recorded experimentally using a 5Ci ²⁴¹Am-Be active neutron source emitting flux of (1.1-1.4) × 10⁷n/sec, which were slowed down by a Perspex sheet and registered by the He-3 neutron detector shown in schematic diagram figure 1, [1-2].
Also, experimental and theoretical studies for gamma ray attenuation properties of the selected AISI316L, AISI304L, [1-2] and treated HN304L austenite steel samples were applied by two active gamma ray sources $^{232}$Th and $^{60}$Co which supply the 238.63, 338.28, 583.19, 911.2, 968.97, 1173.23, 1332.49, and 2614.51 keV gamma ray energy lines. All these lines were detected experimentally by a sodium iodide $1.5\times1.5$" gamma ray detector shown in figure 2, the theoretical study was performed by the Win-X. Com (Version 3.1) [9].

### 3. Results and Discussion

#### 3.1. HN304L microstructure cross section

The high treatment plasma power accelerates active plasma spices inside the plasma reactor tube toward the treated AISI304L sample surface. This leads to an increase in the surface treatment temperature to 788 K, high nitriding rate of 0.67 $\mu m^2/sec$ and a treated thickness of 34.78 $\mu m$. Figure 3 shows cross section for the nitrided surface of HN304L.
50 µm.

Figure 3. Optical cross section micrograph for HN304L treated austenite stainless steel sample.

3.2. X–Ray diffraction analysis

Figure 4 shows XRD for the studied AISI316L, AISI304L and treated HN304L austenite stainless steel samples. The untreated AISI316L and AISI304L samples are basically austenite stainless steel samples composed of γ austenite phase and small α ferrite phase.

From figure 4, one can see that the rf surface plasma treatment process at the previously mentioned condition supports the precipitation of CrN and Fe₄N nitride phases beside the nanocrystalline γ₈ phase. Moreover, the XRD of the treated HN304L austenite stainless steel sample showed a wide γ₈ phase peak which shifts to lower angle from the native γ austenite phase. Based on the XRD results the lattice parameter value for the HN304L was given by 3.80 Å while for both untreated AISI316L and AISI304L were 3.595 Å and 3.593 Å, respectively. This expansion at the lattice parameter value for treated HN304L sample is resulting from the incorporation of plasma spices and creation of crystalline phase γ₈ [3, 10].

3.3. Surface microhardness and tribological properties.
Table 2 shows the surface microhardness and tribological properties of AISI316L, AISI304L and HN304L austenite stainless steel samples.

Table 2. Surface microhardness and tribological properties of AISI316L, AISI304L and HN304L austenite stainless steel samples.

|          | AISI316L | AISI304L | HN304L |
|----------|----------|----------|--------|
| Microhardness (HV0.1) | 180 ± 10 | 196 ± 20 | 1226 ± 28 |
| Wear rate (mm$^3$/m)   | 592.333  | 193.63   | 10.5   |
| Friction coefficient (µm) | 0.48 ± 0.024 | 0.45 ± 0.025 | 0.32 ± 0.020 |

One can see from table 2 that, the surface microhardness of treated HN304L austenite stainless steel sample increased about 5.8 and 5.25 times compared with that of AISI316L and AISI304L samples, respectively. This enhancement is attributed to the existence of newer phases in the HN304L samples resulting from plasma surface treatment process.

Figures 5 and 6 show the optical micrograph for the wear tracks, and track profiles of AISI316L, AISI304L and HN304L austenite stainless steel samples. From these figures it is obvious that, the wear resistance for the HN304L austenite stainless steel sample against abrasive wear is greater than that for AISI316L and AISI304L untreated samples. This result is apparent also from table 2 where the HN304L has recorded the lowest friction coefficient and wear rate.

![Figure 5](image-url)

Figure 5. Optical micrograph of wear tracks of AISI316L, AISI304L and HN304L austenite stainless steel samples.
3.4. Slow neutrons and gamma ray attenuation properties.

The attenuation properties for slow neutrons of AISI316L, AISI304L and H304L austenite stainless steel samples are shown in table 3 where it is apparently shown that the macroscopic cross section for HN304L treated austenite stainless steel sample is greater than that recorded for the untreated standard AISI316L and AISI304L austenite stainless steel samples. This increase is mainly due to the radio frequency

Figure 6. Track profiles for AISI316L, AISI304L and H304L austenite stainless steel samples.
plasma surface treatment process which leads to denser nitrogen concentration on treated the HN304L sample surface.

Table 3. Neutrons macroscopic cross section for AISI316L, AISI304L and HN304L austenite stainless steel samples.

| Sample   | ΣT cm⁻¹ |
|----------|---------|
| AISI316L | 0.161 ± 0.0196 |
| AISI304L | 0.163 ± 0.024   |
| HN304L   | 0.183 ± 0.0268  |

Gamma ray attenuation properties at the energy range (238.63 – 2614.51) keV for the studied AISI316L, AISI304L and HN304L austenite stainless steel samples are shown in tables 4 and 5.

Table 4. Linear attenuation coefficient for AISI316L, AISI304L and HN304L austenite stainless steel samples.

| Gama Energy (keV) | AISI316L μm (Exp.) | AISI304L μm (Exp.) | HN304L μm (Exp.) |
|------------------|---------------------|---------------------|------------------|
| 238.63           | 0.927±0.010         | 0.874±0.005         | 0.910±0.009      |
| 338.28           | 0.795±0.022         | 0.731±0.011         | 0.720±0.006      |
| 583.19           | 0.565±0.005         | 0.591±0.025         | 0.592±0.001      |
| 911.20           | 0.455±0.011         | 0.47±0.017          | 0.476±0.001      |
| 1173.23          | 0.415±0.015         | 0.41±0.010          | 0.414±0.005      |
| 1332.49          | 0.401±0.001         | 0.390±0.021         | 0.391±0.002      |
| 2614.51          | 0.28±0.003          | 0.296±0.001         | 0.302±0.002      |

Table 5. Half value layer for AISI316L, AISI304L and HN304L austenite stainless steel samples.

| Gama Energy (keV) | AISI316L HVL (cm) | AISI304L HVL (cm) | HN304L HVL (cm) |
|------------------|-------------------|-------------------|-----------------|
| 238.63           | 0.872             | 0.793             | 0.761           |
| 338.28           | 0.748             | 0.948             | 0.962           |
| 583.19           | 1.523             | 1.172             | 1.173           |
| 911.20           | 1.227             | 1.474             | 1.454           |
| 1173.23          | 1.729             | 1.690             | 1.670           |
| 1332.49          | 1.670             | 1.777             | 1.771           |
| 2614.51          | 2.476             | 2.341             | 2.272           |

Figures 7-A and B present the experimental and theoretical mass attenuation coefficients of the AISI316L, AISI304L. While figure 7-C presents the same comparison between treated HN304L and AISI304L austenite stainless steel samples as function of gamma ray energies in the energy range (238.63 – 2614.51) keV.
Figure 7. Experimental and theoretical mass attenuation coefficients for AISI316L, AISI304L and HN304L austenite stainless steel samples
From the obtained attenuation parameters for gamma rays at the energy range (238.63 – 2614.51) keV it is obvious that all studied untreated and treated austenite stainless steel samples have comparable gamma ray attenuation properties.

4. Conclusion
The ESR technique was used to produce locally AISI304L and AISI316L standard austenite stainless steel grades in the Egyptian CMRDL. To study the effect of rf surface plasma treatment on these materials, only AISI304L was chosen for that purpose. As a consequence, it is found that there is precipitation of the CrN and Fe,N nitride phases beside the nano-crystalline $\gamma_N$ phase. Furthermore, enhanced improvements in the surface micro-hardness and the tribological properties were clearly observed. Also, the nuclear attenuation characteristics of the rf plasma treated HN304L showed better neutron absorption and comparable gamma ray attenuation properties as compared with AISI304L and AISI316L standard austenite steel grades. Finally, the obtained results strongly indicate that the plasma nitriding surface treatment is a promising method for developing austenite stainless steel grades to meet the increased demands for the nuclear materials progress.

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