Ion-nitriding of Maraging steel (250 Grade) for Aeronautical application

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Abstract. Ion nitriding is one of the surface modification processes to obtain better wear resistance of the component. Maraging steel (250 Grade) is used to manufacture a critical component in the control surface of a combat aircraft. This part requires high strength and good wear resistance. Maraging steels belong to a new class of high strength steels with the combination of strength and toughness that are among the highest attainable in general engineering alloys. Good wear resistance is achieved by ion-nitriding (also called as plasma nitriding or glow discharge nitriding) process of case nitriding. Ion-nitriding is a method of surface hardening using glow discharge technology to introduce nascent (elemental) nitrogen to the surface of a metal part for subsequent diffusion into the material. In the present investigation, ion-nitriding of Maraging steel (250 grade) is carried out at 450 °C and its effect on microstructure and various properties is discussed.

1. Introduction
Surface Engineering means ‘engineering the surface’ of a material or components to impart surface properties, which are different from the bulk of base material [1]. The purpose may be to reduce wear, minimize corrosion, increase fatigue resistance, reduce frictional energy losses, provide a diffusion barrier, provide thermal or electrical insulation, exclude certain wave lengths of radiation, promote radiation, electronic interactions, or simply improve the aesthetic appearance of the surface. Surface engineering processes, which give required properties at surfaces include flame hardening, induction hardening, electron hardening, laser hardening, carburizing, nitriding, cyaniding, plasma nitriding, ion implantation, weld overlay, roll cladding, thermal spraying, plasma spraying, ion plating, CVD, PVD etc. Nitriding is a process for case hardening of alloy steel in an atmosphere consisting of a mixture of ammonia gas and dissociated ammonia [2]. In ion-nitriding (also called as Plasma nitriding or Glow discharge nitriding) method glow discharge technology is used to introduce nascent (elemental) nitrogen to the surface of a metal part for subsequent diffusion into the material [3-5].

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The plasma assisted surface modification techniques offer a great flexibility and are capable of tailoring desirable chemical and structural surface properties independent of the bulk properties [3]. It has other advantages like nil or very thin white layer after nitriding and there is no machining or grinding involved for complex parts after the process. The hardened surface layers become an integral part of the base material without any significant reduction in properties of base material. It is also known to provide the modified surface without dimensional change or distortion of the component. Ion-nitriding provides better control of case chemistry and uniformity [3,6,7]. This method is one of the most effective techniques for increasing wear resistance, fatigue strength, surface hardness and corrosion resistance of industrial components [8]. In this process, vacuum environment and high voltage electrical energy is used to form plasma through which nitrogen ions are accelerated to impinge on work piece. The ion bombardment heats the work piece and cleans the surface as the active nitrogen diffuses through it [3,9].

Maraging steels belong to a new class of high strength steels with the combination of strength and toughness that are among the highest attainable in general engineering alloys [10]. These steels differ from conventional steels in that they are hardened by a metallurgical reaction that does not involve carbon. These steels contain very low carbon (<0.03%) and are strengthened by the precipitation of intermetallic compounds at temperature about 480 °C [11-13]. The term maraging is derived from martensite age hardening and denotes the age hardening of a low carbon, iron – nickel lath martensite matrix [12,14]. Different maraging steels are designed to provide specific levels of yield strength from 1030 to 2420 MPa (150 to 350 ksi). These steels typically have very high nickel, cobalt and molybdenum and very low carbon content. Carbon is treated as an impurity in these steels and is kept as low as commercially feasible (<0.03%) in order to minimize the formation of Titanium carbide (TiC), which can adversely affect strength, ductility and toughness [11,12]. Nominal composition of 250 Grade is Fe-18Ni-8.5Co-5Mo-0.4Ti-0.1Al [10].

Good wear resistance of Maraging steel can be achieved by ion-nitriding process of case nitriding. In conventional gas nitriding process, the nitriding temperature is 500 °C – 550 °C [2,15], which is above the ageing temperature of maraging steel. Hence ion-nitriding of Maraging steel (250 grade) at a temperature lower than the aging temperature has been carried out in the present investigation. Microstructure of the ion-nitrided specimens was examined and properties like tensile, low cycle fatigue, hardness, case depth and corrosion by salt spray test were evaluated. These tests were also carried out on un-nitrided specimens for comparison.

2. Experimental

Figure 1 shows a simplified schematic and figure 2 shows the actual ion-nitriding installation. The work load is supported on a hearth plate inside a double walled, water cooled vacuum chamber, connected to vacuum pumps and gas supply. The chamber is evacuated to a pressure of about 2.5 X 10−2 mbar, a pressure low enough for the background level of oxygen to be within acceptable limits (less than 50 ppm), then filled with a low pressure mixture of nitrogen and hydrogen. The use of auxiliary A.C. heaters to heat the cathode to 250 °C is desirable to minimize cycle time. It can also help provide better temperature uniformity of the part in ion-nitriding treatment. The discharge is ignited using a D.C. power supply, and pressure and temperature are raised to the desired operating values by controlling gas flow and pressure, applied voltage and current. The discharge can be monitored by meters and viewed through inspection windows. The work is cathode and the vessel is anode. The furnace is electrically grounded, cool to the touch, and quiet in operation. Maraging steel (250 grade) specimens were prepared from solutionised material and then aged at 450 °C for 3 hrs. These aged specimens were ion-nitrided in the nitriding furnace under the vacuum. Plasma is obtained by passing the gas mixture of H₂ and N₂ gases in the ratio 3:1 into the chamber and maintaining the pressure of 5 mbar. Ion-nitriding is carried out at 450 °C for 10 h.
3. Results

3.1 Chemical Composition

The chemical analysis of the Maraging steel (250 grade) was carried out using Optical emission spectroscopy to confirm the material specification. The values obtained are given in Table 1.

| Element | C   | Ni  | Mo  | Co  | Ti  | Al  | S   | P   | Balance |
|---------|-----|-----|-----|-----|-----|-----|-----|-----|---------|
| Composition | 0.01 | 17.29 | 4.89 | 7.90 | 0.41 | 0.14 | 0.005 | 0.006 | Balance |

3.2 Visual Check

Ion-nitrided samples were checked for any visible defects and discoloration and no defects were observed.

3.3 Microstructural studies

The samples for Metallography were prepared from both ion-nitrided and un-nitrided samples. Microstructural studies were carried out using both optical microscope and scanning electron microscope (SEM). Optical micrographs of un-nitrided specimen is shown in figure 3 and that of ion-nitrided specimen is shown in figure 4. Etchant used here is Ferric Chloride. SEM studies were carried out by etching the specimens using Ferric Chloride. The microphotographs obtained from un-nitrided and ion-nitrided samples are shown in figures 5 and 6 respectively.
3.4 Hardness and Case depth measurement

Hardness was measured for both un-nitrided and ion-nitrided samples using Vickers Hardness Tester. The hardness values obtained on un-nitrided specimen is 616 VPN whereas for ion-nitrided specimen it is 900 VPN.

Case depth was determined by hardness measurements. Hardness values are listed in Table 2. Figure 7 shows the variation of hardness with depth from the surface. Case depth here is taken as distance from the surface to which hardness is 100 VPN more than the core hardness. From the graph the case depth estimated is 93 µm.

Table 2. Hardness and corresponding distance from the surface.

| Distance from the edge (µm) | Hardness (VPN) |
|-----------------------------|----------------|
| 50                          | 797            |
| 75                          | 761            |
| 100                         | 702            |
| 125                         | 626            |
| 150                         | 623            |
| 175                         | 623            |
| 200                         | 623            |
| Core                        | 616            |

3.5 Salt spray test

Salt spray test was carried out on both un-nitrided and ion-nitrided specimens for 144 hrs at 5% NaCl solution in a Salt Spray Test chamber. Figure 8 shows the photograph of samples...
before and after the salt spray test. Corrosion started in un-nitrided specimen after 48 h of exposure, whereas no corrosion was noticed in ion-nitrided specimen.

![Figure 8](image)

**Figure 8.** (a). Photograph of the samples (a) before the salt spray test and (b) after the salt spray test.

### 3.6 Tensile Properties
Tensile properties – Ultimate tensile strength (UTS), 0.2 % Proof stress (0.2 % PS), % Elongation (% El.) and % Reduction in Area (% RA) were measured using TIRA Test 2820S Universal Testing Machine (UTM). The results obtained are presented in Table 3.

| Property       | UTS (MPa) | 0.2% PS (MPa) | % El. | % RA |
|----------------|-----------|---------------|-------|------|
| Un nitrided    |           |               |       |      |
| Sample 1       | 1694      | 1606          | 9.32  | 60   |
| Sample 2       | 1696      | 1619          | 8.84  | 56   |
| Sample 3       | 1702      | 1622          | 9.23  | 55   |
| Ion nitrided   |           |               |       |      |
| Sample 1       | 1847      | 1757          | 5.66  | 25   |
| Sample 2       | 1864      | 1810          | 6.24  | 28   |
| Sample 3       | 1877      | 1813          | 6.70  | 30   |

### 3.7 Impact Strength
Impact strength was measured on Charpy ‘U’ notch specimens using FIE make Charpy Impact Testing machine. The values obtained are given in Table 4.

| Sample       | Sample 1 | Sample 2 | Sample 3 |
|--------------|----------|----------|----------|
| Un nitrided  | 21       | 22       | 22       |
| Ion nitrided | 17       | 19       | 19       |

### 3.8 Low Cycle Fatigue
Low cycle fatigue testing was carried out on smooth specimens in Zwick Roell UTM with applied stress of 1172 MPa and with stress ratio (R) of -1. The values obtained are presented in Table 5.

| Property       | Sample 1 | Sample 2 | Sample 3 |
|----------------|----------|----------|----------|
| Un nitrided    | 3755     | 3287     | 5220     |
| Ion nitrided   | 5500     | 14400    | 11200    |
4. Discussion
Chemical composition analysed conforms to the Maraging steel 250 grade.

Both Optical microscopy and SEM examination confirm that no change in core microstructure of the material after ion-nitriding occurs.

The ion-nitrided specimens exhibit higher surface hardness and good case depth than the un-nitrided specimens. The introduction of nitrogen to the surface of Maraging steel by ion nitriding process improves the wear properties by increasing the hardness.

Salt spray test results show that there is an improvement in corrosion resistance after nitriding. This is due to the formation of an anodic layer after nitriding.

The ion-nitrided samples show higher UTS and 0.2 % PS values than the un-nitrided ones, whereas % El and % RA are less for ion-nitrided samples compared to the un nitrided samples. This can be attributed to the surface layer getting hardened because of ion-nitriding. Impact strength of ion-nitrided samples is lower compared to un-nitrided samples. This is again due to the surface layer getting hardened because of ion nitriding.

The number of cycles to failure in low cycle fatigue test also increase on ion-nitriding. The introduction of nitrogen to surface layers increases the fatigue properties of the maraging steel by introducing the residual compressive stresses.

5. Conclusions
From this ion nitriding study of Maraging steel (250 grade), the following conclusions can be drawn.

Case depth obtained is sufficient for the design requirement.

Ion-nitriding improves the surface hardness of Maraging steel 250 grade.

An improvement in UTS, 0.2 % PS and LCF properties is observed on ion-nitriding. The % El, % RA and impact strength decrease but they satisfy the design requirements.

No change in microstructure observed after ion-nitriding.

Ion-nitriding of 250 Grade appears to be suitable for parts which are subjected to constant wear.

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References
[1] Kumar B, Upadhyay N C 2005 *IM Metal News* 8 5
[2] Sidney Avner H 1997 *Introduction to Physical Metallurgy* 2nd Edition 383
[3] Spalvins T *Ion Nitriding* Conference proceedings ASM International 1
[4] Moller W, Parascandola S, Telbizova T, Gunzel R and Richter E 2001 *Surface & Coatings Technology* 136 73
[5] ASM Handbook 1996 Vol. 4 411
[6] Bernd Edenhofer 1976 *Metal Progress* 181
[7] EPRI Centre for Materials Fabrication Ohio 1994 *Ion nitriding* 2
[8] Ahangarani Sh., Mahboubi F and Sabour A. R 2006 *Vacuum – Surface Engineering, Surface Instrumentation & Vacuum Technology* 1032
[9] Pavel Novak, Dalibor Vojtech and Jan Serak 2006 *Surface & Coatings Technology* 200 5229
[10] Gupta B, 1996 *Aerospace Materials* II 695
[11] ASM Handbook 1991 Vol. 4 219
[12] ASM Handbook 1990 Vol. I 793
[13] INCO Databook, 1976 351
[14] Morito S, X-Huang, Furuhara T, Maki T and Hansen N 2006 *Acta Materialia* 54 5323
[15] David Pye 2005 *Practical Nitriding and Ferritic Nitrocarburizing* ASM International 71