Plasma nitriding of various substrates using D.C. glow discharges under a triode configuration

J.C. Avelar-Batista1, E. Spain1, J. Housden2, A. Matthews2 and G.G. Fuentes3

1 TECVAC LTD., Buckingam Business Park, Rowles Way, Swavesey, Cambridge, CB4 5UG. United Kingdom
2 Department of Engineering Materials, Sheffield University, Sir Robert Hadfield Building, Mappin Street, Sheffield, S1 3JD. United Kingdom
3 Centre of Advanced Surface Engineering-AlN, San Cosme y San Damian sn, E-31191, Pamplona, Spain

Experimental

Three different substrates (Ti6Al4V alloy, high speed AISI M2 steel and austenitic stainless steel AISI 316 steel) were plasma-nitrided in a TECVAC P10 PAPVD coater using a D.C. triode configuration. A hot tungsten filament heater was used to adjust the current independently of gas pressure and bias voltage, and thus keep the substrate temperature constant.

For the Ti6Al4V alloy (nominal composition wt%) 5.5-6.7 Al, 3.5-4.5 % balance Ti), fast-acies (25.4 mm x 5.0 mm, 350 HRA), were plasma-nitrided at 650°C, 750°C and 800°C for 135 minutes and at 850°C for 135 minutes, in order to evaluate the effect of temperature under triode conditions on the nitrided layer depth and hardness. For plasma-nitriding at 700°C, two bias voltages were used: -500 and -2000 V and -3000 V.

For both AISI M2 steel (nominal composition wt%) 0.85 % C, 0.25 % Si, 15 % Cr, 1.85 % V, 0.05 % Nb, 0.35 % W, balance Fe) and AISI 316 steel (nominal composition wt%) 17.48 % Cr, 10.04 % Ni, 2.06 % Mo, 2.0 % Mn, <1.0 % Si, <0.05 % S, balance Fe), the effect of gas composition on case depth and hardness was investigated under a D.C. triode configuration. Both substrates were nitrided in the same cycle and, therefore, under the same process conditions. Hardened and polished AISI M2 steel (25.4 mm x 5.0 mm, 850 HRA) and polished AISI 316 steel (20.3 mm x 4.0 mm, 350 HRA) were plasma-nitrided at 550°C for 240 minutes using 5 different gas compositions and bias voltages: 40% Ar + 60% N2, -200 V; 60% Ar + 40% N2, -500 V; 70% Ar + 30% N2, -750 V; 70% Ar + 30% N2, -1500 V.

After nitriding, the nitrided layer hardness and depth were evaluated using Vickers microhardness measurements on polished cross-sections and surface under a load of 0.49 N (50 g). The nitrided polished cross-sections were also examined by SEM. X-ray diffraction (XRD) analyses were performed using CuKα (λ=0.154056 nm) radiation to identify the phases which had been formed during the plasma nitriding treatment.

Results

[Graphs and tables]

Fig. 1: Vickers microhardness profile of plasma-nitrided Ti6Al4V alloy.

Fig. 2: Vickers microhardness profile of plasma-nitrided AISI M2 steel.

Fig. 3: Vickers microhardness profile of plasma-nitrided AISI 316 steel.

Fig. 4: XRD diffractograms of untreated and plasma-nitrided Ti6Al4V alloy.

Fig. 5: XRD diffractograms of hardened and plasma-nitrided AISI M2 steel.

Fig. 6: XRD diffractograms of plasma-nitrided AISI M2 steel showing the nitrided layer under triode plasma nitriding using (a) 60% Ar + 40% N2, 480°C, -250 V; (b) 40% Ar + 60% N2, 500°C, -500 V and (c) 30% Ar + 70% N2, 500°C, -250 V. Etched with 10% Nital. Note that a compound layer is present in (a) and (b) only.

Fig. 7: SEM cross-sectional photomicrographs of plasma-nitrided AISI 316 steel showing the nitrided layer after triode plasma nitriding using (a) 60% Ar + 40% N2, 480°C, -250 V; (b) 40% Ar + 60% N2, 500°C, -500 V and (c) 30% Ar + 70% N2, 500°C, -250 V. Etched with Mtilda's reagent:

Conclusions

Plasma nitriding was carried out in Ti6Al4V, AISI M2 and 316 stainless steel substrates using a D.C. triode configuration in a PAPVD coater. The results indicated that:

1. For the Ti6Al4V alloy, hardening seems to be mainly accompanied by incorporation of nitrogen in the α-Ti phase at temperatures up to 750°C, as the formation of the ternary nitride layer on the surface could not be detected by SEM. For these conditions, the maximum surface hardness (550HV0.05) and nitrided layer depth (30-40 μm) were achieved at 700°C. At 800°C, a ternary nitride layer (TiN) was formed on the surface. The highest carburization achieved using a D.C. triode configuration reduced the treatment time and nitriding temperature required to obtain hard and reasonably deep nitrided cases in the Ti6Al4V alloy.

2. For the AISI M2 steel substrate, the surface hardness after plasma nitriding was increased up to 1370-1524 HV0.05 and case depths of 100-150 μm were obtained at 440-500°C for 240 minutes, depending on total pressure, gas composition and substrate bias. The highest surface hardness and deepest case (150 μm) were achieved by using a 40% Ar + 60% N2 gas composition at a bias voltage of -500 V. Compound layer formation seemed to be influenced by gas composition and bias voltage. For the highest nitrogen concentration and lowest bias voltage, compound layer formation was suppressed. The benefits of high nitrogen dilution and isolation in triode systems (PVD coater) in comparison to conventional plasma nitriding allow case depths of 0.10-0.15 mm to be produced in 4 hours only.

3. Nitrided layer thicknesses of 30-50 μm and surface hardnesses of 800-1000 HV0.05 were obtained in AISI 316 stainless steel substrates after plasma nitriding treatments of 480-500°C for 240 minutes. As with the AISI M2 steel, variations in surface hardness and case depth occurred, depending on gas composition and substrate bias. The highest surface hardness (1020 HV0.05) and deepest case (50 μm) were achieved when a gas composition of 40% Ar + 60% N2 and bias voltage of -500 V in order to obtain similar case depths in stainless steel at the same temperature using the Active Screen Plasma Process: a treatment time of 12 hours would be required. Therefore, a triode configuration assisted by thermionic emissions allows a 5-fold reduction in processing time.

Fig. 8: SEM cross-sectional photomicrographs of plasma-nitrided AISI M2 steel showing the nitrided layer after triode plasma nitriding using (a) 40% Ar + 60% N2, 480°C, -250 V; (b) 40% Ar + 60% N2, 500°C, -500 V and (c) 30% Ar + 70% N2, 500°C, -250 V. Etched with 10% Nital. Note that a compound layer is present in (a) and (b) only.