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Titanium nitride layer formation by TIG surface melting in a reactive environment

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Abstract

The possibility of forming a hard titanium nitride layer has been studied by melting commercial purity titanium (CP-Ti) surfaces beneath the tungsten inert gas (TIG) torch in a pure nitrogen environment. The surface melting of titanium with TIG torch of energy densities ranging from 46 MJ m^{-2} to 182 MJ m^{-2} produced a melt layer of over 1 mm thickness. The topographies of the molten tracks glazed at energy densities above 68 MJ m^{-2} had rippling marks and they are perpendicular to the glazing direction. However, the tracks glazed at lower energy densities of 46 MJ m^{-2} and 55 MJ m^{-2}, produced cellular type surface structures. Porous edges were present in all the tracks glazed in the nitrogen environment at different energy densities. Those tracks glazed at higher energy densities created surface cracking along the track width, and they propagated down to the melt zone. The track cross sections gave irregular melt profiles; the Maragoni force that produced high nitrogen concentration fluid along the convectional flow lines and the exothermic reaction for the formation of titanium nitride (TiN) are presumed to be responsible for creation of such irregular melt features. The resolidified melt microstructures contained non-uniform distribution of dendrite populations. The XRD analysis at different depths of the melt cross section revealed that the titanium nitride phase dominates the melt pool microstructure. The tracks gave a surface hardness of around 2000 HV. All the tracks glazed at different energy densities produced hardness profiles with gradual decrease in hardness at a higher melt depth. This indicates that the hardness development is directly related to the dendrite population in the melt pool, which was found to decrease at deeper depths because of the lower concentration of nitrogen.

Keywords: TIG torch; Nitriding; Melt depth; Hardness

1. Introduction

Titanium alloys have high strength-to-weight ratio, good mechanical properties as well as excellent corrosion resistance, but these alloys have poor wear resistance and are susceptible to seizure, galling and fretting damages. The surface modification by introduction of nitrogen into the surface melted zone can produce a hard and wear resistant layer of titanium nitride. Laser surface nitriding was initiated by the work of Katayama et al. [1] and since then there have been a number of investigations, e.g., Refs. [2–5]. This treatment is accomplished through laser surface melting of the alloy in a nitrogen environment, which forms titanium nitride. The melted zone consists of dendritic structures of titanium nitride (TiN) which are responsible for the high hardness at the surface. The laser nitrided commercial purity titanium (CP-Ti) and IMI829 alloys are reported to produce surface hardness of about 2000 HV [5]. The laser processing time is very short; however, this type of surface treatment requires high investment for the laser equipment.

The present study has been undertaken to investigate the possibility of producing a hard titanium nitride layer on CP-Ti by surface melting under tungsten inert gas (TIG) torch in a nitrogen environment. The heat energy for surface melting is provided by the electric arc maintained between the tungsten electrode and the specimen. Similar nitriding work performed by Khan and Fowles [6] on Ti–6Al–4V alloy produced nitride structures having a maximum hardness of 1000 HV and a significant improvement in wear behaviour. They observed reduced hardness when nitriding was carried out in a nitrogen and argon mixture. This paper describes the surface topography, microstructural features and hardness of the resolidified

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surface layer formed on CP-Ti using the TIG torch melting process in a reactive nitrogen environment.

2. Experimental

Commercial purity titanium used in this investigation was supplied in the form of rectangular plates of dimensions 100 mm × 25 mm × 4 mm. The specimens were abraded with SiC paper and degreased using acetone prior to surface melting operation. Surface melting of the specimens was accomplished using the Miller TIG torch. A tungsten electrode with a diameter of 2.4 mm was used to create an arc between the tip of the electrode and the specimen surface. The voltage (V) and current (I) to the electrode were maintained at 16 V and 70 A, respectively. The electrode height used was 2 mm. The specimens were held stationary under the moving TIG torch while a shielding gas of either pure argon or pure nitrogen was supplied. Tracks were produced along the width of the specimens at varying electrode-traversing speeds.

The arc energy density, E, could be calculated using the formula

$$E = \frac{IV}{r_B s}$$

where r_B is the arc radius and s is the electrode-traversing speed. High speed photography using a Nikon camera at a shutter speed of 1/8000 of a second was used to determine the radius of the arc. Details of this technique of determining arc radius have been described elsewhere [7]. In determining the arc radius, the electrode was kept stationary at a height of 2 mm from the specimen surface while the voltage, current and nitrogen gas flow rate were maintained at 16 V, 70 A and 10 l min^{-1}, respectively. The computed average arc radius was found to be 3.3 mm. The calculated values of the arc energy density, E, corresponding to the various electrode-traversing speeds are given in Table 1.

The topography of each track was examined using the Meiji optical microscope and Joel JSM-5310LV scanning electron microscope. A Shimadzu 6000 X-ray diffractometer was used to determine the phases formed in the resolidified melt pool. X-ray diffraction test was carried out at three different depths measured from the surface of the melt zone, namely 0.2 mm, 0.35 mm and 0.7 mm, to investigate the change in the nitride stoichiometry. Standard metallographic techniques were used to prepare the transverse sections of the resolidified tracks for microscopic examination. The microstructure of the melt zone was revealed by etching the polished samples in a solution of 10 cm^3 of hydrofluoric acid, 30 cm^3 of nitric acid and 50 cm^3 of water for a period of 10 s. Microstructural examination was done using the Meiji optical microscope and the JOEL JSM-5310LV scanning electron microscope. Hardness measurement on the transverse sections of the samples was carried out using the Matsuzawa microhardness tester. A 500 gf load was used to create indentations along the melt depth of the nitrided samples, starting from the surface of the melt zone to the base material.

Table 1
Calculated values of the arc energy density at different electrode-traversing speeds

Electrode-traversing speed (mm s^{-1})	Gas supplied	Gas flow rate (l min^{-1})	Arc energy density, E (MJ m^{-2})
2.5	Ar	10	–
1.9	N_2	10	182
2.5	N_2	10	137
3.8	N_2	10	91
5.0	N_2	10	68
6.3	N_2	10	55
7.5	N_2	10	46

3. Results and discussion

3.1. Surface topography

3.1.1. Surface rippling

All the nitrided track surfaces were gold in colour due to the formation of titanium nitride in the melt zone and a continuous TiN layer at the melt surface. The surface topography of samples nitrided at E = 68–182 MJ m^{-2} revealed rippling marks (Fig. 1), which were not seen on the surfaces of the samples nitrided at E = 46–55 MJ m^{-2}. The ripples were observed to form in two different directions, namely radially along the electrode-traversing direction and perpendicular to the radial direction. At lower arc energy densities, a cellular type of structure was noticed on the surfaces of the tracks nitrided at E = 46 MJ m^{-2} and 55 MJ m^{-2} (Fig. 2).

It was essential for the liquid–vapour interface to be tipped from the horizontal in order for rippling to take place. One of the reasons for the tipping of the liquid–vapour interface could be the surface tension driven (Maragonian-type) convection [5]. As the freezing rate of the melt pool was relatively rapid, waves produced in the melt liquid would be frozen and formed ripples on the surface of the resolidified track.

The cellular surface structures observed for the samples nitrided at E = 46 MJ m^{-2} and 55 MJ m^{-2} were absent for tracks nitrided at higher arc energy densities. The cellular structures were associated with low heat input and rapid solidification of the melt. As the temperature of the melt might just be above its melting point, the melt would be very vis-

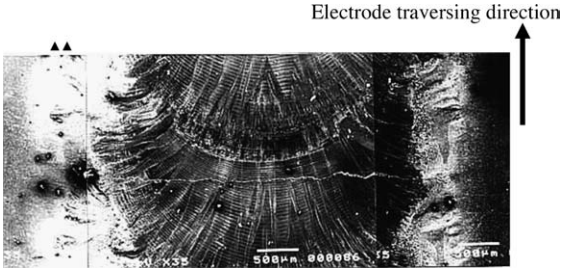

Fig. 1. SEM micrograph showing ripples on the surfaces of the track nitrided at E = 91 MJ m^{-2}.