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Effects of Temperature Control on Synthesis of Nitride Thin Films of Titanium Alloy using Atmospheric-Pressure Plasma

大気圧プラズマによるチタン合金の窒化薄膜合成における温度制御の効果

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Titanium nitride thin films have been partially synthesized on the titanium surface using pulsed-arc atmospheric-pressure plasma jet in our previous study. In this paper, we introduced an external heater to the plasma-jet-nitiriding system in order to control separately treatment temperature and the supply condition of nitrogen species, and to form uniform and extensive titanium nitride films on the surface. We have succeeded in forming titanium nitride films on the entire surfaces (15×15 mm^2) by uniform heating of the heater.

1. Introduction

Titanium and its alloys are widely used in a variety of medical applications due to desirable biocompatibility, excellent corrosion resistance, and high mechanical strength, for example, for artificial bones and joints, dental implants. At the present time, it is reported that the performance of titanium as a biomaterial was upgraded more by nitriding or TiN coating, e.g. the reduction of bacteria adhesion and the increase of osteoblastic-like cells adhesion [1,2].

In our previous study, we have succeeded in forming titanium nitride layers and the diffusion layer on the titanium surface using pulsed-arc (PA) atmospheric-pressure plasma jet for the first time [3]. However, this nitriding method has some shortcomings due to the treatment by only heating of PA plasma jet. First, changing the gap between the sample surface and the jet-nozzle tip varies not only the treatment temperature but also the supply condition of nitrogen species. Second, it is difficult to nitiride uniformly the entire metal surface due to local heating by spraying with PA plasma jet.

Here, we introduced an external heater to our experimental setup, i.e. the supply condition of nitrogen species is controlled by changing the gap, and treatment temperature is controlled by the external heater. We report the results of titanium nitride synthesis obtained by this new experimental system for PA plasma jet-assisted nitriding.

2. Experimental

Fig. 1 shows the schematic view of the experimental setup including PA plasma jet and an external heater. The detail of PA plasma jet is described in ref. [3,4]. N_2/H_2 mixture gas used as an operating gas of PA plasma jet is introduced from the upper part of the coaxial cylindrical electrode nozzle at the total flow rate of 20 slm, where the H_2 mixture ratio is 1 %. This mixture ratio is the optimal value for steel nitriding found in our previous study [4]. Pulsed-arc discharge is generated by a low-frequency power source (plasmatreat, FG3001). High voltage pulses (5 kV in voltage, 1.2 A in discharge current, and 21 kHz in repetition) are applied to the inner electrode, and the outer electrode is grounded.

Plasma-jet nitriding was performed in the openable tubular furnace (Asahi Rika, ARF-30K) used as an external heater. The gap between the titanium surface and the jet-nozzle tip is fixed at 30 mm. Treatment temperature is controlled by the heater. Treatment time is 2 h including preheating.

We used titanium alloy Ti-6Al-4V (ASTM F136) as a sample. The hardness of the substrate is

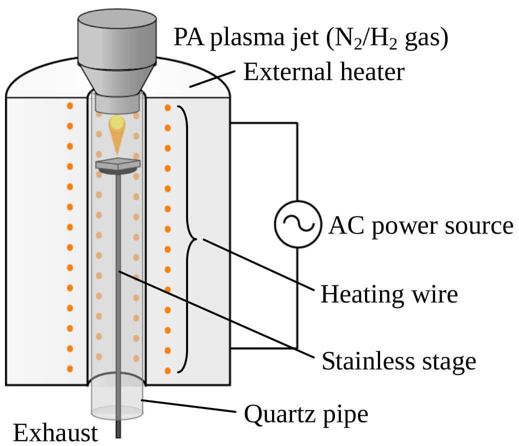

Fig.1 Schematic view of the experimental setup.

ca. 300 $Hv_{0.01}$, and the size is 15×15×4 mm^3. The surfaces are mirror polished and degreased in an ultrasonic acetone bath.

3. Results

The entire surfaces of the sample changed to a golden color corresponding to that of TiN. Fig. 2 shows X-ray diffraction patterns of the sample treated at different treatment temperature. We demonstrated the formation of titanium nitride as shown Fig. 2. Moreover, we found that TiN peaks of sample surface treated under higher temperature condition are higher.

Fig. 3 shows the micro-Vickers hardness profiles of the sample cross-section. The hardness profiles are shown with gray-scale, and the 0 mm point of horizontal axis (radial position) is the jet-spraying center. Note that the entire surfaces uniformly are hardened as shown Fig. 3. In particular, the hard layer more than 1000 $Hv_{0.01}$, corresponding to nitiride layers, is formed down to about 10 μm in Fig. 3(a). In addition, we found that the diffusion layer having the hardness of the range from 400 to 500 $Hv_{0.01}$ is also formed beneath nitride layers.

4. Conclusions

We have succeeded in synthesizing uniform and extensive titanium nitride films by introducing an external heater to our PA plasma-jet-nitriding system. This investigation will lead to understanding the mechanism of plasma-jet-assisted formation of titanium nitride. In the conference, we would like to report the results of the other analysis and the relationship between treatment time and the thickness of titanium nitride layers.

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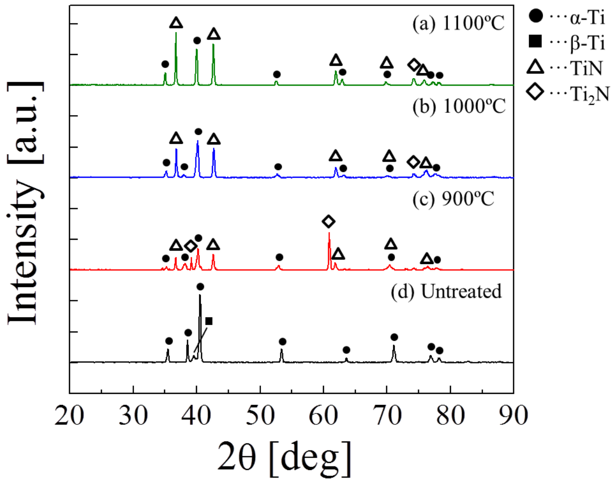

Fig. 2 X-ray diffraction patterns of the sample surface treated at different treatment temperature.

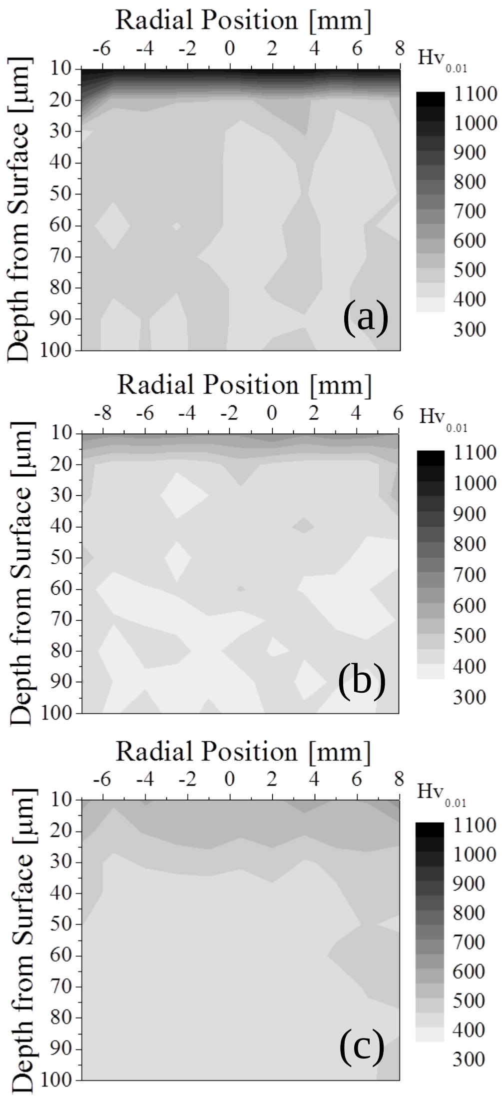

Fig. 3 Hardness profiles of the sample cross-section. Treatment temperature is (a) 1100ºC, (b) 1000ºC, and (c) 900ºC.