Surface & Coatings Technology 306 (2016) 351–357

Contents lists available at ScienceDirect

Surface & Coatings Technology

journal homepage: www.elsevier.com/locate/surfcoat

Nitriding of tool steel using dual DC/RFICP plasma process

Boonchoat Paosawatyanyong [a,b,*], Jiraporn Pongsopa [c], Patama Visuttipitukul [d], Worawan Bhanthumnavin [b,e]

[a] Department of Physics, Faculty of Science, Chulalongkorn University, Bangkok 10330, Thailand
[b] ThEP Center, Commission on Higher Education, Si Ayutthaya Road, Bangkok 10400, Thailand
[c] Nanoscience and Technology Program, Graduate School, Chulalongkorn University, Bangkok 10330, Thailand
[d] Department of Metallurgical Engineering, Faculty of Engineering, Chulalongkorn University, Bangkok 10330, Thailand
[e] Department of Chemistry, Faculty of Science, Chulalongkorn University, Bangkok 10330, Thailand

ARTICLE INFO

Article history:
Received 2 February 2016
Revised 27 July 2016
Accepted in revised form 6 August 2016
Available online 8 August 2016

Keywords:
Plasma nitriding
Dual plasma source
Hard coating

ABSTRACT

The nitriding process of AISI H13 tool steel samples using dual DC/RFICP source is reported. The aim was to explore the advantages of the process in improving the surface hardness of the material. The reactor incorporated a weakly ionized DC plasma source that provides continuous surface cleaning of the sample and a RFICP discharge source that supplies a highly reactive environment to enhance the kinetics of nitriding process. The gas mixture of Ar, H_2 and N_2 was used as plasma medium. The samples were nitrided for times ranging between 1 and 20 h by keeping the DC source at 300 V, RF power at 100 W and substrate temperature at 300 °C. The hardness profiles obtained revealed a significant improvement of the present method over conventional nitriding and some advantage over other plasma-assisted methods, especially in the comparatively lower process temperature. The evolution of the hardness could be related to the formation of CrN and Fe_4N as confirmed by XRD and EPMA analysis.
Abstract code: AO4

1. Introduction

Plasma nitriding process is a well-known technique which can be utilized to improve surface properties of ferrous alloy, and etc. [1–3]. Conventional plasma nitriding processes of alloys have been done with high DC input in the range of several hundred volts. These high voltage, hence high temperature, process can cause further fatigue and degradation in the matrix. On this regards, Radio Frequency Inductively Coupled Plasma (RFICP) sources, which have been of utilized mainly in plasma vapor deposition or etching of advanced materials, can be a suitable alternative plasma source for nitriding process. The reason is not only because their ability to generate uniform high-density plasmas [4–6] but also their flexibility than that of DC plasma source since RFICP system could couple the RF field using an antenna other than the sample itself through an RF transparent window into the reactor generating a sustained discharge, therefore, no electrode is required inside plasma chamber resulting in low contamination from ion sputtering to the electrode [7,8]. Nonetheless, major disadvantage of using sole RFICP in the nitriding process is also related to the reduction of sputtering to the sample. Sputtering is required as to provide continuous cleaning of steel surface from oxide formation. Oxide-free and activated surface easily reacts with the nitrogen ions.

The aim of the present study was to utilize a dual RFICP/DC plasma reactor in nitriding process. The dual reactor was designed and constructed for the purpose of combining the advantages of using two sources of plasma to nitride difficult materials such as aluminum and stainless steel. The reactor incorporates a weakly ionized DC plasma source that provides continuous cleaning of the sample by sputtering and heating by ion bombarding into a RFICP discharge source that supplies a highly reactive environment to enhance the kinetics of nitriding process. In this study, we explored the extent of combine effects in the nitriding of AISI H13 tool steel.

AISI H13 tool steel is one of materials suitable for high temperature applications such as dies and molds for high temperature work [9]. However, due to many severe operation conditions, surface modification of the H13 steel is also needed in order to achieve high surface hardness and good wear resistance. Attempts on formation of nitride by high-temperature plasma nitriding have been reported by several research groups. RF discharge parameters including RF-power, working pressure, dimension of the discharge, the amount of electrical power, treatment temperature, gas mixture, and treatment time affect properties (hardness) and microstructure of the nitride surface [10,11]. The processing time of nitriding for H13 steel is an important factor since further tempering of martensite structure after heat treatment possibly occurs during plasma nitriding with high temperature and long

* Corresponding author at: Department of Physics, Faculty of Science, Chulalongkorn University, Bangkok 10330, Thailand.
E-mail address: paosawat@sc.chula.ac.th (B. Paosawatyanyong).

http://dx.doi.org/10.1016/j.surfcoat.2016.08.016
0257-8972/

processing time. To eliminate the degradation of heat-treated microstructure, this study is, therefore, conducted with relatively lower RF power and DC voltage.

2. Experimental

2.1. Sample preparation

Samples for the experiments were cut from a cylindrical rod of AISI H13 tool steel of 12 mm in diameter. Prior to the nitriding, the steel was heat treated at 1020 °C for 2 h, quenched in air to room temperature and then tempered at 510 °C for 2 h, followed by forced convection cooling. The obtained steel had a hardness of around 580 HV and basically consisted of martensite microstructure. After that, the steel was cut into a disc shape with the thickness ranging from 2 to 5 mm. The spark emission spectrometer was used to analyze chemical composition of the material. The result was C%:0.35; Si%:1.10; Mn%:0.4; P%:0.02; S:0.02; Cr%:5.10; Mo%:1.14; V%:0.96; Fe:balance [12]. All samples were polished using 400, 600, 800, 1500, 2000, 4000 grade SiC paper, and finally with 1 μm diamond paste. All samples were cleaned in ultrasonic agitator with acetone and kept in temperature and moisture controlled chamber.

2.2. Nitriding procedure

Treatments in this study were performed in a plasma reactor which was adapted from the system in our previous work [13,14]. The main feather of this reactor is that it has two sources utilized in two steps: a cleaning-heating stage done by the weakly ionized DC discharge and the actual nitriding stage with the post-discharge flow of the RFICP plasma. The schematic diagram of the reactor is shown in Fig. 1. The RFICP source consists of an antenna, 13.56 MHz 500 W RF generator (RFPP RF5S) and an impedance matching network. A planar coil antenna was used to couple RF power to plasma gases through the quartz plate. The DC source was basically a cylindrical-shape cathode disc placed between the sample and the irradiative heater underneath. The cathode disc was connected through a vacuum feedthrough to an external DC power supply (Sorensen DCS300). A closed cycle water-cooling circulation line inside the cathode disc was also implemented.

The irradiative heat source was a 300 W halogen bulb. The bulb was installed into a paraboloid-shaped cavity inside a stainless steel block. In order to increase the reflectance of the cavity, the inner surface was highly polished. Using this irradiative heating source together with the cooling water circulation, the substrate could be maintained at a precise temperature between 200 and 500 °C. A thermocouple was embedded in the center of the cathode disc to monitor the sample temperatures by assuming that the thermal equilibrium between the sample and the cathode underneath was reached. Our preliminary studies in several batches of experiments yielded positive nitriding results only when the samples temperatures were around 300 °C or higher. Therefore, during the nitriding process in the present study, the samples temperatures were intentionally controlled at the 300 °C.

Prior to the treatment process, samples were introduced into the chamber. For each treatment condition, three samples were altogether placed on the cathode disc. The chamber was afterward evacuated by a turbomolecular pump until the base pressure was reached. Gases were filled into the chamber until the desired pressure was reached. To demonstrate the merit the DC/RFICP technique developed, the samples were then experimented with three different processes which were: using the DC discharge only; using the RFICP plasma only; and using the combined DC/RFICP process.

In the DC only process, the admixture of argon, hydrogen and nitrogen (Ar 1: H_2 1: N_2 3) was fed in to the chamber via massflow controller. The total pressure was maintained at 0.5 Torr. The DC source was employed and controlled to remain constant at 300 V throughout the process. For RFICP treatment, the same gas admixture and total pressure as stated in the DC process above were used without the employment of the DC supply. The plasma was ignited in the chamber, by applying RF power from the generator through an automatic impedance matching network to a planar-shape coil antenna. The dissipated RF power rate was controlled at 100 W throughout the process. In the DC/RFICP process, argon and hydrogen mixture (Ar 1: H_2 1) was first introduced into the chamber and a voltage of 300 V was applied to generate weakly ionized plasma for 20 min as a pretreatment process. Then the RF

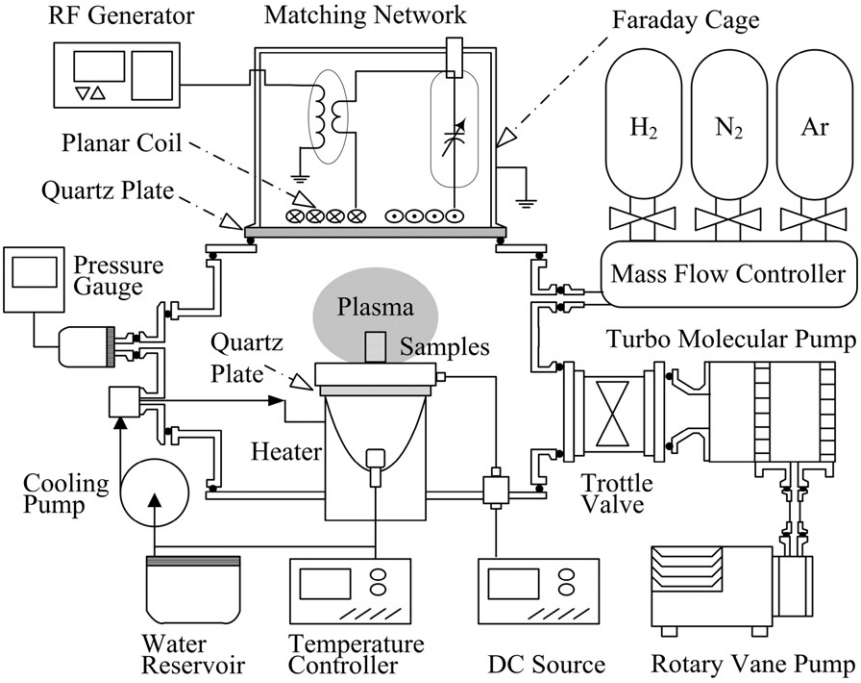

Fig. 1. Schematic diagram of the dual DC/RFICP reactor.