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Evaluation of nitrogen diffusion in thermo-mechanically nanostructured and plasma nitrided stainless steel

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ABSTRACT

The effect of grain size on diffusion depth of nitrogen in AISI 321 stainless steel during plasma nitriding was investigated. The repetitive cold rolling and subsequent annealing were conducted to achieve nano/ultrafine grains in AISI 321 stainless steel. The grain size range of 130 nm up to 45 μm was obtained under these conditions. Plasma nitriding was performed at temperatures of 400, 450 and 500 °C for duration of 5 h. Microstructural evolutions were conducted by OM, SEM and TEM. The microstructure and composition of the nitrided layer were characterized by SEM and GDOES. Mechanical properties of the S phase were evaluated by microhardness testing. Results indicated that nitrided layer of the nanostructured steel have uniform appearance with no CrN precipitates, while CrN precipitations were formed in nitrided layer of the micro-grain one. The corrosion resistance of the nitride layer was improved in nanostructured condition because of uniform appearance of the nitride layer. Furthermore, the hardness of the S phase improved by decreasing substrate grain size. Increasing austenite grain size from 130 nm up to 45 μm, caused to increase surface nitrogen concentration in nitride layer. Decreasing austenite grain size led to decreases the S phase thickness, while the nitrogen diffusion mechanism is the same.

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1. Introduction

Plasma nitriding (PN) is one of the thermochemical process used to introduce nitrogen into austenitic stainless steel to improve the surface hardness [1,2]. Since there is a deterioration of corrosion properties when CrN forms, there is a great interest of introducing nitrogen into the austenite matrix without CrN formation [3,4]. Thus, the nitrided steels having high surface hardness and good corrosion properties can be achieved by formation of the nitride layer which is formed at temperatures below 500 °C. Nitriding below 500 °C results in a thin layer that is saturated with nitrogen atoms referred as S phase. In addition, formation of S-phase was observed in samples treated at 20% N_2 atmosphere. The compound layers thickness increases with increasing N_2 content in the atmospheric gas mixture up to approximately 80% N_2 [5]. The S phase was first reported by Zhang and Bell in 1985 [6]. In parallel investigation, Ichii et al. obtain the S phase by low temperature nitriding at 400 °C [7]. The term S phase was invented by Ichii et al. [7] and also referred as expanded or supersaturated austenite by Leyland et al. [9,10], the m phase by Marchev et al. [10,11], and the έ phase [12]. It is also denoted as the Ś phase by Gontijo et al. [13]. Despite of many studies devoted to ion nitriding of austenitic stainless steel, the formation mechanism of expanded austenite is still unknown and is the subject of current investigations.

Pretreatment of austenitic stainless steel like heat treatment can change its properties. Heat treatment influences the grain size, and consequently diffusivity. Manova et al. have reported that slower diffusion rate was observed in larger grains in AISI 304 stainless steel [14]. Tong et al. have indicated that a decrease in grain size from 1000 μm to 13 nm causes a faster diffusion rate by order of 1000 in AISI 304 stainless steel [15]. Additionally, different data sets are reported in the literature concerning nitrogen diffusion in austenitic stainless steel without providing any information about the grain size [16–18]. There is no model explaining these quite diverging results [14]. Moreover, scanty studies have been performed on nitrogen diffusion in connection with microstructure, so the published diffusion values could be misleading. The present work focused on the effect of nanograins on diffusion depth of nitrogen in AISI 321 stainless steel through plasma nitriding. In addition, the effect of nanograins on mechanical properties and corrosion behavior of nitride layer was investigated.

2. Materials and experimental procedures

The substrate material was made of AISI 321 austenitic stainless steel with the composition of: C 0.06 wt.%, Si 0.75 wt.%, Mn 1.22 wt.%, P 0.043 wt.%, S 0.003 wt.%, Cr 17.88 wt.%, Ni 9.28 wt.%, Ti 0.36 wt.% and Fe balance. The mean grain size of the as received material was about

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45 μm. The smallest grain was about 4 μm and the largest grain was about 130 μm. The microstructure evaluations were performed using clemex software. Hot rolled steel strips with 10 mm initial thickness were rolled with 90% thickness reduction at best possible temperature of −20 °C [19]. The cold rolling was followed by annealing treatments in an electrical furnace at 800 °C for 4,6,8,10,15,20,30 min to achieve grain size ranges of 0.13–5 μm. The nitriding process was performed in a pulsed DC plasma nitriding unit. Prior to nitriding the specimens were polished and cleaned ultrasonically in acetone for 15 min. In order to carry out plasma nitriding, the vacuum chamber was pumped down to 0.05 Torr and then the specimens were subjected to sputtering for surface cleaning using an atmosphere composition of Ar 33 vol% —H_2 67 vol% under the pressure of 0.3 Torr for 1 h. Afterwards, plasma nitriding was performed for 5 h for various temperature ranges from 400 to 500 °C by using a gas mixture N_2 20 vol% —H_2 80 vol% under the pressure of 3 Torr. The process was conducted on specimens with average grain size of 0.13–45 μm. At the end of the treatment, the samples were slowly cooled down under vacuum.

The microstructures were studied by Optical Microscope (OM, Olympus GX71), Scanning Electron Microscope (SEM, COXEM CX100) and Transmission Electron Microscope (TEM, JEOL JEM-ARM300F). Feritscope device (Fischer FMP30) was employed to measure the austenite volume percentage after heat treatment. The structure and thickness of the S phase was evaluated by SEM. The diffusion depth of nitrogen and surface composition of nitrided steel was measured by means of Glow Discharge Optical Emission Spectroscopy system (GDOES, HORIBA JOBIN YVON). Mechanical properties of S phase was determined using micro-hardness testing (Shimadzu, HMV-2) with a load of 0.1 N. Average results were reported after five trials for micro-hardness testing.

3. Results and discussion

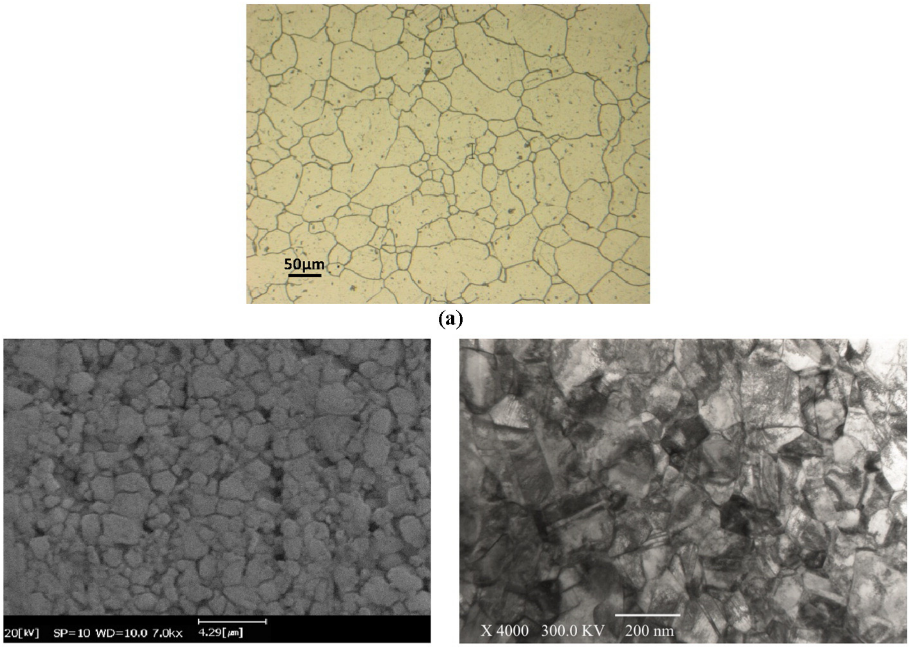
Optical micrograph of as-received material (micro-grain steel) consisting austenite grains has shown in Fig. 1. This indicates that the average grain size prior to cold rolling was approximately 45 μm. The microstructure evaluations were performed using clemex software according to ASTM E112-12 [20]. Fig. 1b shows the higher magnification SEM micrograph after thermo-mechanical process, i.e. cold rolling process and 15 min annealing at 800 °C. Fig. 1c displays the TEM micrograph after cold working and 4 min annealing at 800 °C.

As be seen, average grain sizes achieved to 130 nm and 3 μm (nanostructured and fine grain steels) after 4 and 15 min annealing respectively. Feritoscopic results show that the annealed samples have fully austenitic structure. It revealed that the reversed austenite grains have fully recrystallized and no martensite lathes remain in the matrix.

The SEM micrograph and GDOES profile of the micro-grain steel after plasma nitriding at 500 °C are presented in Fig. 2. As shown in Fig. 2a, the nitride layer thickness for as-received steel is about 24 μm. Besides, grain boundary precipitation of chromium nitride, which causes chromium depletion in the matrix, is observed in the grain boundaries. The chromium nitride precipitations have been seen in other investigations at temperatures above 450 °C [21–24]. In such temperatures, chromium atoms can diffuse rapidly and facilitate the chromium nitride formation. The Chromium depletion could be responsible for the lower corrosion resistance of the layer and unfold the CrN precipitation [21–26]. The GDOES analysis also demonstrates that nitrogen atoms have diffused into the substrate with a depth of about 24 μm (Fig. 2b).

The plateau like appearance of the nitrogen GDOES profile indicates that the nitrogen atoms diffusion don't obey the fickian models. In the literature, plateau shape refers as trapping-detrapping model sign [18, 21,27,28]. In addition, the hardness of the nitride layer was determined to be about 1200 HV. It is due to the supersaturated structure of the nitride layer. Nitrogen atoms in the interstitial sites, reduce the mobility of dislocations and improve the mechanical properties of the layer.

Fig. 3a shows that the nitride layer thickness of fine grain steel is about 13 μm. it is much thinner than that of the micro-grain steel (Fig. 2a). However, the presence of CrN precipitates is also observed in some areas of the grain boundaries. Besides that, the GDOES profile reveals that diffusion depth of nitrogen is also about 14 μm (Fig. 3b). The plateau shape of the nitrogen GDOES profile indicates the trapping-detrapping mechanism of nitrogen atoms. Moreover, the hardness of the nitride layer was about 1250 HV. Further hardness of the fine grain steel nitride layer rather than micro-grain steel is due to



(a)

(b)

(c)

Fig. 1. (a) Optical micrograph of as received AISI 321 stainless steel, (b) SEM micrograph of the specimen annealed at 800 °C for 15 min and (c) TEM micrograph of the specimen annealed at 800 °C for 4 min.

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