A two-stage process for plasma nitriding with an active screen

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Abstract

In the present work a two-stage technique was applied in the ASPN to obtain a better control on the nitriding process and to meet the treatment requirements for a wide range of the steel types, different load size and workpiece geometry. A nitriding temperature range of 400-500 °C for 2 h and 570 °C for 4 h were chosen for the first and the second stage, respectively. Two concepts of a reduction of the nitriding potential of the N_2-H_2 plasma in the second stage were proposed. The first one (concept A) is based on reduction of the N_2 to H_2 gas ratio without change of the bias power applied to the workload, whereas the second concept (B) uses a low level of the bias power without change of the N_2 to H_2 gas ratio, keeping it similar to the first stage. The advantages and applicability of both concepts in the ASPN process were discussed. A response of the nitriding results of different alloyed steels on the variation of the process conditions in each stage was presented and discussed.

Keywords: active screen plasma nitriding, two-stage process, alloyed steels, case depth

Introduction

The use of two-component atmosphere of dissociated or diluted with N_2/H_2 gas ammonia was first proposed by Floe as a two-stage process [1]. Besides the economical reasons, the method allowed producing of nitriding case with required hardness in the first stage and acceptable case depth in the second stage. The morphology and the thickness of the compound layer can be effectively controlled in the Floe process. A few efforts have been made in the past to adopt the two-stage technology to the plasma nitriding process [2]. A nitrogen free gas media were used in the second stage leading to a limitation of growth or even to a disappearance of the compound layer (CL). The active screen plasma nitriding (ASPN) is a novel plasma assisted nitriding technique with a high application potential [3]. The main advantages of the ASPN over a conventional plasma process are caused by the replacement of the glow discharge from the components to a separate metal screen (active screen, AS) surrounding the entire workload. During the nitriding process the active screen plays a twofold role - it generates a mixture of active species required for the nitriding process and it radiates the heat, produced by the plasma discharge, resulting in a uniform temperature distribution over the workload parts. A weak cathodic potential (bias) applied to the treated parts is essential for nitriding in the plasma unit of industrial scale [4, 5, 6].

Experimental

The AS plasma nitriding was performed in the large laboratory plasma unit powered by 15 kW pulsed DC plasma generator (f= 1 kHz and 60% duty cycle). The inner volume of the reactor was about 1 m^3. As an active screen a double wall metal mesh with 800 mm in diameter and 750 mm in height was placed in the middle of the reactor surrounding the workload. A second generator (Magpuls, P= 10 kW) running at f=10 kHz with a 50% duty cycle supplied a negative Bias-voltage to the worktable. The pressure in the furnace and the total gas flow rate in all experiments were set constant at 300 Pa and 80 nl/h, respectively. The process gases were introduced into the discharge chamber through separate mass flow controller (MFC) Two precursor gases were used: N_2 and H_2. The gas mixture was varied by changing the partial gas flows. The temperature was varied from 400 to 500 °C in the 1st Stage and kept at 570°C in the 2nd Stage (see Table 1 for details). Three commercial grade steels (42CrMo4, 31CrMoV9 and X38CrMoV5-1) commonly used for mechanical components and tools were used in this study. All the samples were wet ground down to 1000 grit SiC paper, cleaned and dried with hot air before the treatment. The nitriding results were metallographically analyzed. The microhardness and composition profiles were obtained by means of microhardness test and glow discharge optical emission spectrometry (GDOES), respectively.

Table 1. The ASPN process parameters used in the two-stage treatment.

	1st Stage (2h)			2nd Stage (4h)		
	T, °C	N_2:H_2	Bias, W	T, °C	N_2:H_2	Bias, W
Concept A	400, 450, 500	1:1	750	570	1:8	1300
Concept B					1:1	450

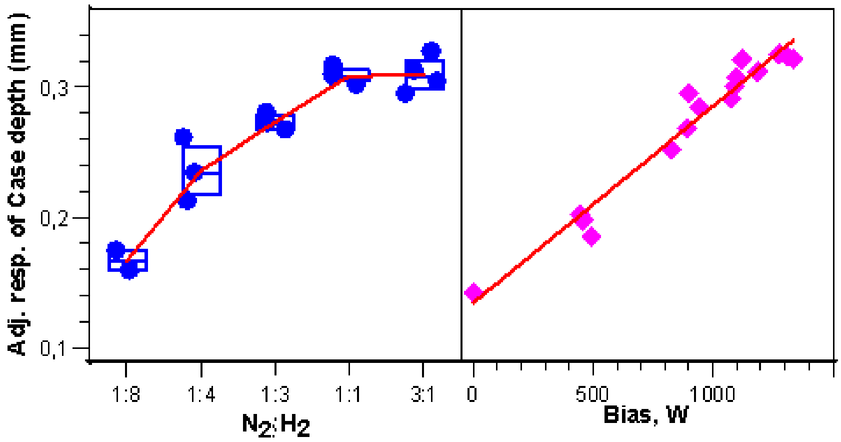

Fig. 1. Adjusted response graph of the case depth measured in the 42CrMo4 steel sample nitrided in the single-stage run (2nd stage conditions) in dependence on two factors: the N_2 to H_2 gas ratio and bias power applied to the probe table.

Results and discussion

The problem of a reduction of the nitriding potential in the second stage of the two-stage process performed by conventional plasma nitriding method was usually solved by strong reduction of the nitrogen concentration in the N_2-H_2 plasma. The ASPN process provides an additional process parameter – a bias activation, which results in significant improvement of the nitriding results. The effect of both factors (the N_2 to H_2 gas ratio and bias power) on the nitriding response in the ASPN was investigated. The results of the regression analysis based on a number of experiments with variation of these two factors are presented in the Figure 1. Both factors showed equally strong impact on the hardness (case depth). It is worth noting the linear dependence of the case depth on the bias power. A slow rate of the case depth growth for higher N_2 to H_2 gas ratios depicts the situation of nitrogen diffusion after the formation of compound layer, when only process temperature and treatment duration have a major impact on development of the case depth.

Table 2. Thickness of the compound layer (CL) and the case depth (CD) measured in the 42CrMo4 steel sample after AS nitriding under variation of the N_2 to H_2 gas ratio and the bias power in the single-stage run (2nd stage conditions). Bias power is given in percentage of the AS power.

N_2:H_2 / Bias, %	1:1		1:4		1:8	
	CL, µm	CD, mm	CL, µm	CD, mm	CL, µm	CD, mm
15	13,9	0,35	4,5	0,29	0	0,22
10	7	0,29	2	0,26	0	0,18
5	0	0,24	0	0,15	-	-

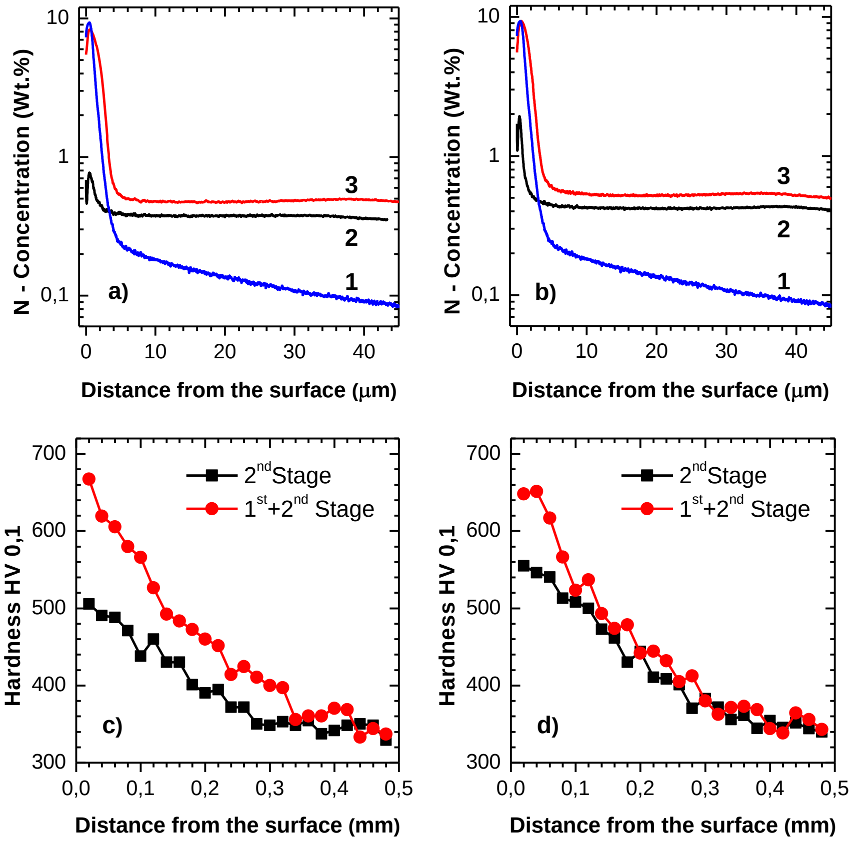

Fig. 2. Comparison of the results obtained in the AS nitrided 42CrMo4 steel sample after the two-stage treatment following two concepts for the 2nd stage: (a, c) und (b, d) for concept A und B, respectively, where (a, b) are the nitrogen concentration profiles measured by GDOES after the 1st (curve 1) or after the 2nd stage (curve 2) - both as single-stage run, or after the two-stage process (curve 3); (c,d) are the hardness depth profiles after the single- and two-stage treatment.

A suppression of the CL grow without significant shortcomings in the nitriding were realized in the 2nd stage of the ASPN based on two concepts of nitriding potential reduction: i) by a strong decrease of the nitrogen partial pressure in the process gas – the way usually used in conventional plasma nitriding (Concept A), or ii) by a reduction of the bias power. The results given in the Table 2 provided the proper combination of two factors for both concepts, namely 1:8 and 15% for concept A, and 1:1 and 5% for concept B, for the N_2 to H_2 gas ration and percentage of the bias power, respectively. The comparable nitriding results presented in the figure 2 have confirmed the applicability of both concepts for the 2nd stage in the two-stage ASPN process. However, the concept B seems to be more elegant and very attractive for industrial use due to a wide process window for the smooth control of the nitriding potential in the 2nd stage, moreover it follows the main idea of the AS process to minimize a direct plasma impact on the components to be treated.

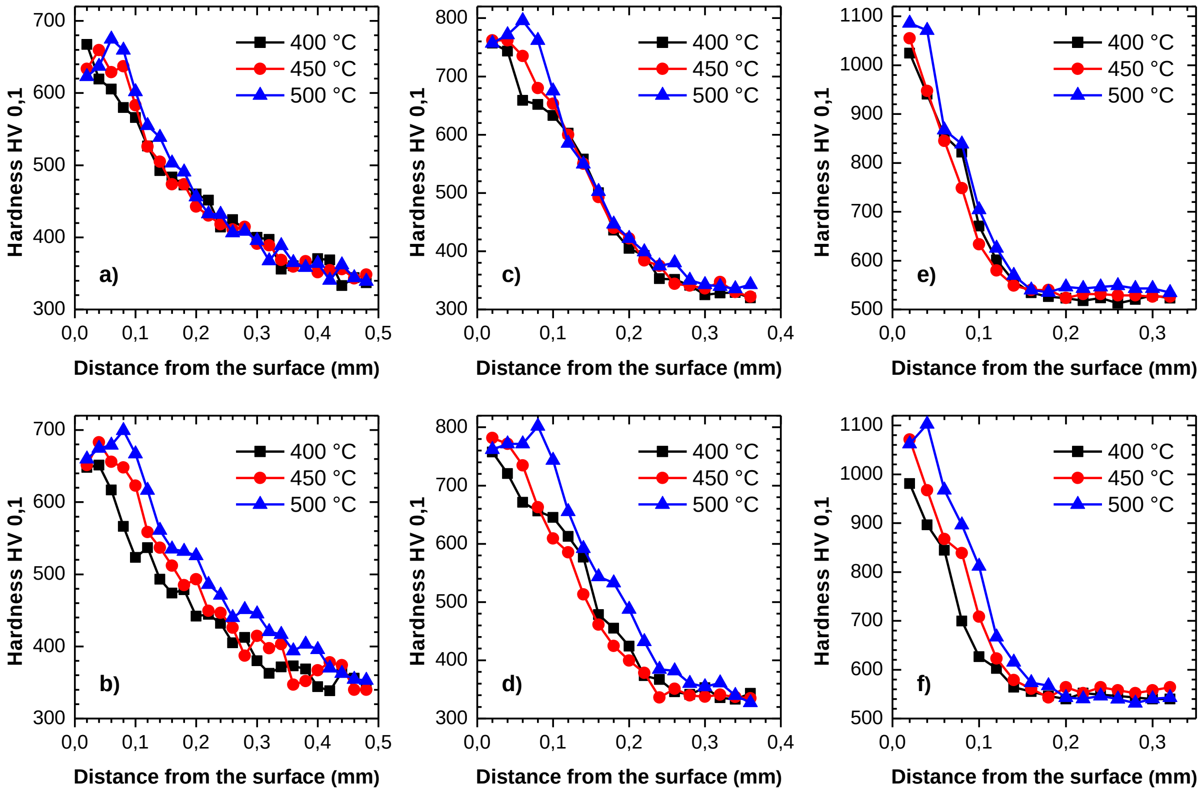

Fig. 3. Effect of process temperatures in the 1st stage on the hardness measured in the cross section of samples of three steels: (a,b) for 42CrMo4, (c,d) for 31CrMoV9 and (e,f) for X38CrMoV5-1, AS nitrided in the two-stage process following two concepts for the 2nd stage: (a, c, e) and (b, d, f) for concept A and B, respectively.

A general requirement for optimization of the process temperature in the 1st stage has a purpose of further improvement of the case hardness near the surface region. Two concepts of the two-stage process provided a similar trend in response of the case hardness on the temperature elevation from 400 to 500 °C in the 1st stage, see Fig. 3 for three alloyed steels. A small weakness of hardness near the surface can be explained by coarsening of precipitated nitrides at elevated temperature. A more pronounced effect on hardness is seen for all materials processed with the concept B, which indicates that the nitriding potential in the 2nd stage of the concept B was slightly higher than that of the concept A.

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