

## EFFECT OF NITROGEN HYDROGEN RATIO ON PLASMA NITRIDING FOR 38CrMoAl

Lu Song<sup>1)</sup>, Tiantian Peng<sup>1)</sup>, Xiaobin Zhao<sup>1,2)</sup>, Jing Hu<sup>1,2)\*</sup>

<sup>1)</sup> Jiangsu Key Laboratory of Materials Surface Science and Technology, Changzhou University, Changzhou 213164, China

<sup>2)</sup> National Experimental Demonstration Center for Materials Science and Engineering (Changzhou University), Changzhou, 213164, P.R. China

Received: 27.07.2018

Accepted: 01.09.2018

\*Corresponding author: e-mail: jinghoo@126.com Tel.: 86+0519-86330095, complete postal address (Jiangsu Key Laboratory of Materials Surface Science and Technology, Changzhou University, Changzhou 213164, China)

### Abstract

Critical nitrogen hydrogen ratio in plasma nitriding was primarily investigated to get enhanced performance for 38CrMoAl steel. The modified surface layer was characterized by optical microscopy (OM), X-ray diffraction (XRD) and micro-hardness tester. The results showed that the critical nitrogen hydrogen ratio was 1: 5 while plasma nitriding at 540°C for 6 h. Under this condition, no compound layer was formed, and accompanied with high surface hardness, while the compound layer was formed accompanied with lower surface hardness with nitrogen hydrogen ratio higher than the critical value.

**Keywords:** 38CrMoAl steel; plasma nitriding; XRD; compound layer

### 1 Introduction

38CrMoAl steel is a kind of metal material commonly used for producing high-pressure valve due to its excellent wear resistance [1,2]. And a high-pressure valve is generally subjected to severe impact and wear in real applications. Therefore, optimal combination of hardness, wear resistance and toughness are required for 38CrMoAl high-pressure valve. To meet the technical requirements, surface modification of high-pressure valves is necessary [3-5], and plasma nitriding is one of the most widely used surface modification techniques in industry, since a modified layer with high surface hardness and excellent wear resistance can be obtained [6-9].

Unfortunately, during conventional plasma nitriding, a hard and brittle compound layer (also called white layer) is generally produced, which is easy to crack and peel off from the surface during the service life of 38CrMoAl steel high-pressure valve upon subjecting the severe impact and cyclic load [10-12], and thus leading to premature failure of the components. Hence, it is of significant value to control the formation of compound layer during plasma nitriding for high-pressure valve.

It has been reported that compound layer can be avoided through low temperature plasma nitriding [13-15]. However, the combined properties of the nitriding layer obtained by low temperature plasma nitriding can't meet the advanced technical specifications in some real applications, such as surface hardness and wear resistance.

In this study, plasma nitriding at different nitrogen hydrogen ratio was carried out for 38CrMoAl steel to effectively control the formation of compound layer, and it was found that there existed a critical nitrogen hydrogen ratio for avoiding the formation of compound layer.

## 2 Experimentals

38CrMoAl steel was selected as the substrate material with the chemical composition (wt. %) of: 0.37 C, 1.5 Cr, 0.45 Mn, 0.17 Mo, 0.95 Al and balance Fe. The specimens were machined into a size of 10mm×10mm×5mm, followed by quenching at 930 °C and tempering at 600 °C to get a substrate with uniform microstructure. All the surfaces of specimens were treated by silicon carbide emery papers of different granulometry (240, 500, 1500 and 2000 mesh) to achieve a fine finish, then ultrasonically cleaned in anhydrous ethanol prior for 15 min and dried before nitriding.

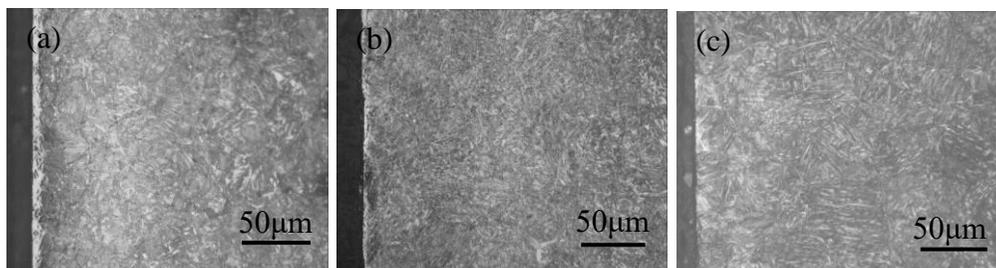
After cleaning with ethanol, the specimens were placed into plasma nitriding equipment (LD-8CL), and evacuated to 18 Pa by a rotary pump. All specimens were sputtered of 30 min by hydrogen with a flow of 500 mL/min at a pressure of 300 Pa. After sputtering process, a mixture gas of N<sub>2</sub> and H<sub>2</sub> with its ratio of 1:3, 1:4 and 1:5 was supplied at a gas pressure of 400 Pa to run plasma nitriding process at the same temperature of 540°C for the same duration of 6 h.

The cross sectional microstructure was observed by optical microcopy. The phase constituents were determined by X-ray diffraction (XRD) with Cu-Kα ( $\lambda=1.54 \text{ \AA}$ ) radiation. Hardness measurements were made in a HXD-1000TMC micro-hardness tester, with the test load of 10 g and holding duration of 15 s. Each hardness value was determined by averaging at least 5 measurements.

## 3 Results and discussions

### 3.1 Microstructure characterization

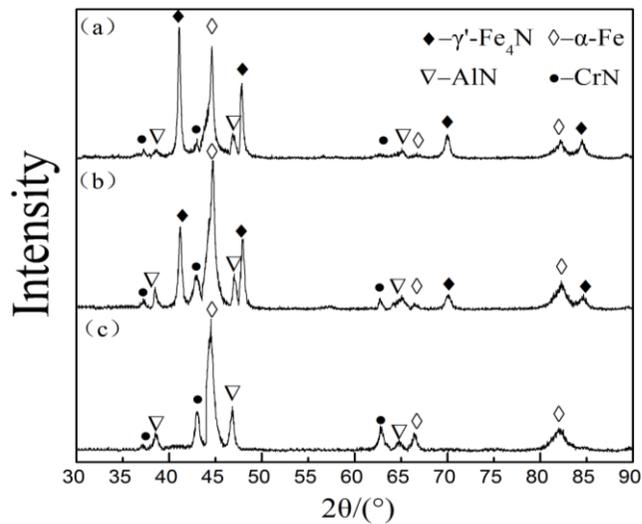
The cross-sectional microstructures of 38CrMoAl steel nitriding with different nitrogen hydrogen ratio are presented in **Fig. 1**. It can be seen that the compound layer thickness decreases with the decrease of nitrogen hydrogen ratio, and no compound layer is formed at the ratio of 1:5 in Fig. 1(c), which implies that the compound layer thickness is dependent on nitrogen hydrogen ratio and there exists a critical ratio to avoid the formation of compound layer. Here in this research, the critical nitrogen hydrogen ratio is 1:5.



**Fig. 1** Cross-sectional microstructure of 38CrMoAl steel nitriding with different N<sub>2</sub>:H<sub>2</sub> ratio (a) N<sub>2</sub>:H<sub>2</sub>=1:3; (b) N<sub>2</sub>:H<sub>2</sub>=1:4; (c) N<sub>2</sub>:H<sub>2</sub>=1:5

### 3.2 XRD analysis

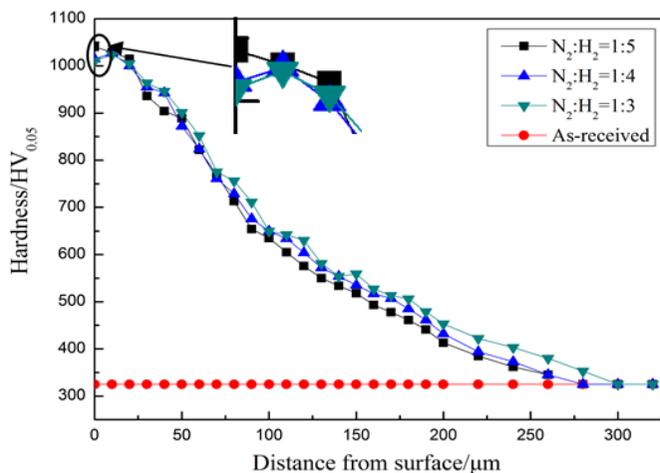
**Fig. 2** shows XRD patterns of 38CrMoAl specimens nitrided with different nitrogen hydrogen ratio. It can be seen that the main phase is  $\alpha$ -Fe along with CrN and AlN for specimen (c) with nitrogen hydrogen ratio of 1:5, while  $\gamma$ '-Fe<sub>4</sub>N is dominant for the other two specimens. Therefore, XRD patterns confirm that no compound layer is formed on the surface of specimens (c), and with compound layer on the other two specimens, which is in good agreement with the results in **Fig.1**.



**Fig. 2** X-ray diffraction patterns of 38CrMoAl steel nitrided with different  $N_2:H_2$  ratio (a)  $N_2:H_2=1:3$ ; (b)  $N_2:H_2=1:4$ ; (c)  $N_2:H_2=1:5$

### 3.3 Micro-hardness profile

**Fig. 3** shows the micro-hardness profiles of 38CrMoAl steel nitrided with different nitrogen hydrogen ratio. It clearly indicates that the surface hardness of specimen with nitrogen hydrogen ratio of 1:5 is the highest, though the thickness of effective hardening layer is a little thinner.



**Fig. 3** Micro-hardness profile of 38CrMoAl steel nitrided with different  $N_2:H_2$  ratio

### 3.4 Mechanism discussions

Generally, a nitriding layer formed during plasma nitriding is composed of compound layer and nitrogen diffusion layer. And a compound layer is mainly composed of  $\gamma'$ - $Fe_4N$  or  $\epsilon$ - $Fe_2-3N$ ,

which is much more brittle than the underneath diffusion layer due to the different characteristics, including different crystal lattices, therefore, the compound layer is easy to crack upon subjecting impact loads <sup>[10]</sup>. Moreover, the big difference of elasticity modulus between compound layer and diffusion layer results in uncoordinated deformation, especially upon subjecting cyclic loads <sup>[10,11]</sup>, which makes the compound layer have a tendency to crack and peel off from the surface, and thus result in premature failure. Therefore, though the compound layer can provide high surface hardness and excellent wear resistance, it is of significant value for 38CrMoAl high-pressure valve to get a nitriding layer without compound layer for preventing its premature failure in real applications.

As was reported that there exists a critical nitrogen potential, i.e. the lowest nitrogen potential for controlling the formation of a compound layer, in other words, the compound layer can not be formed when the nitrogen potential is lower than the critical value <sup>[13-15]</sup>. Since in real applications there is no efficient way to evaluate the nitrogen potential of plasma nitriding, nitrogen hydrogen ratio is used to represent nitrogen potential in this study, and it is found that the critical nitrogen hydrogen ratio to control the formation of compound layer is 1:5 for 38CrMoAl steel at 540°C. Therefore, the results can provide valuable guide for designing plasma nitriding process in real applications.

#### 4 Conclusions

In summary, critical nitrogen hydrogen ratio was primarily investigated for 38CrMoAl. The results show that the specimen nitrided at  $N_2:H_2=1:5$  owns the highest surface hardness due to no compound layer formed on the surface, in other words, enhanced combined properties can be obtained for 38CrMoAl steel. Meanwhile, it is found that there exists a critical Nitrogen hydrogen ratio for avoiding the formation of compound layer, and the critical Nitrogen hydrogen ratio for 38CrMoAl steel is 1:5 corresponding to the nitriding temperature of 540°C.

#### References

- [1] G.B. Li, X.Z. Li, J.J. Wu: Study of the thermal fatigue crack initial life of H13 and H21 steels, *Journal of Materials Processing Technology*, Vol. 74, 1998, p.23-26, doi: 10.1016/S0924-0136(97)00244-6
- [2] D. Papageorgiou, C. Medrea, N. Kyriakou: Failure analysis of H13 working die used in plastic injection moulding, *Engineering Failure Analysis*, Vol. 5, 2013, p. 355-359, doi: 10.1016/j.engfailanal.2013.02.028
- [3] L.L.G.D. Silva, M. Ueda, R.Z. Nakazato: Enhanced corrosion resistance of AISI H13 steel treated by nitrogen plasma immersion ion implantation, *Surface & Coatings Technology*, Vol. 201, 2007, p. 8291-8294, doi: 10.1016/j.surfcoat.2006.03.063
- [4] M.V. Leite, C.A. Figueroa, S.C. Gallo: Wear mechanisms and microstructure of pulsed plasma nitrided AISI H13 tool steel, *Wear*, Vol. 269, 2010, p. 466-472, doi: 10.1016/j.wear.2010.04.037
- [5] J.Q. Wu, H. Liu, J.Hu: Enhancement of corrosion resistance for plasma nitrided AISI 4140 steel by plain air plasma post-oxidizing, *Journal of Alloys and Compounds*, Vol. 632, 2015, p. 397-401, doi: 10.1016/j.jallcom.2015.01.221
- [6] X.M. Ye, J.Q. Wu, Y.L. Zhu, J. Hu: A Study of the Effect of Propane Addition on Plasma Nitrocarburizing for AISI 1045 Steel, *Vacuum*, Vol. 110, 2014, p.74-77, doi: 10.1016/j.jallcom.2015.01.221

- [7] J.C. Li, X.M. Yang, S.K. Wang, J. Hu: A rapid D.C. plasma nitriding technology catalyzed by pre-oxidation for AISI4140 steel, *Materials Letters*, Vol. 116, 2014, p.199-202, doi: 10.1016/j.matlet.2013.11.033
- [8] R. Bidulsky, M. Actis Grande, A. Zago, Z. Brytan, J. Bidulska: Wear resistance of various type sintered steels, *Archives of Metallurgy and Materials*, Vol. 55, 2010, p. 623-629
- [9] T. Steiner, S.R. Meka, H. Göhring, E.J. Mittemeijer: Alloying element nitride stability in iron-based alloys; denitriding of nitrided Fe–V alloys, *Journal of Materials Science & Technology*, Vol. 33, 2017, p. 23-32, doi: 10.1080/02670836.2016.1155254
- [10] M.F. Yan, X.A. Wang, R.L. Lin, Y.X. Zhang, Y. Yang: Kinetics and wear behaviour of M50NiL steel plasma nitrided at low temperature, *Journal of Materials Science & Technology*, Vol. 33, 2017, p.370-376, doi: 10.1080/02670836.2016.1213027
- [11] R. Mohammadzadeh, A. Akbari, M. Drouet: Microstructure and wear properties of AISI M2 tool steel on RF plasma nitriding at different N<sub>2</sub>–H<sub>2</sub> gas compositions, *Surface & Coatings Technology*, Vol. 258, 2014, p. 566-573, doi: 10.1016/j.surfcoat.2014.08.036
- [12] H. Liu, J. Li, Y. Chai, J. Hu: Kinetics and enhancement mechanism of plasma oxynitriding for AISI 1045 steel, *Surface & Coatings Technology*, Vol. 302, 2016, p. 22-26, doi: 10.1016/j.surfcoat.2016.05.021
- [13] H.Zhong, L.Dai, Y. Yue, B.Wang, X.Zhang, C. Tan, M. Ma, R. Liu: Tribological properties of plasma-nitrided AISI 4340 steel in vacuum, *Journal of Materials Science & Technology*, Vol. 32, 2016, p. 275-281, doi: 10.1080/02670836.2015.1121341
- [14] C.X. Li, T. Bell: A comparative study of low temperature plasma nitriding, carburising and nitrocarburising of AISI 410 martensitic stainless steel, *Journal of Materials Science & Technology*, Vol. 23, 2007, p. 355-361, doi: 10.1179/174328407X161204
- [15] B. Paosawatyanong, J. Pongsopac, P. Visuttipitukul: Nitriding of tool steel using dual DC/RFICP plasma process, *Surface & Coatings Technology*, Vol. 306, 2016, p. 351-357, doi: 10.1016/j.surfcoat.2016.08.016

### **Acknowledgements**

*Authors are grateful for the support by National Natural Science Foundation of China (51774052) and Top-notch Academic Programs Project of Jiangsu Higher Education Institutions (TAPP).*