



### **Science Arts & Métiers (SAM)**

is an open access repository that collects the work of Arts et Métiers Institute of Technology researchers and makes it freely available over the web where possible.

This is an author-deposited version published in: <https://sam.ensam.eu>  
Handle ID: [.http://hdl.handle.net/10985/19679](http://hdl.handle.net/10985/19679)

#### **To cite this version :**

K. RAM MOHAN RAO, A.S. KHANNA, Karanveer S. ANEJA, Kalimi TRINADH, Corinne NOUVEAU - Plasma Nitriding of 90CrMoV8 Tool Steel for the Enhancement of Corrosion Resistance - Materials Today: Proceedings - Vol. 24, p.1006-1010 - 2020

Any correspondence concerning this service should be sent to the repository

Administrator : [scienceouverte@ensam.eu](mailto:scienceouverte@ensam.eu)



IConAMMA 2018

# Plasma Nitriding of 90CrMoV8 Tool Steel for the Enhancement of Corrosion Resistance

K. Ram Mohan Rao<sup>a\*</sup>, Corinne Nouveau<sup>b</sup>, A.S. Khanna<sup>c</sup>, Karanveer S. Aneja<sup>c</sup>, K. Trinadh<sup>d\*</sup>

<sup>a</sup>Department of Chemistry, GIT, Gandhi Institute of Technology & Management (Deemed to be University), Gandhinagar, Visakhapatnam-530045, Andhra Pradesh, INDIA

<sup>b</sup>Laboratoire Bourguignon des Matériaux et Procédés (LaboMaP), Arts et Métiers ParisTech de Cluny, Rue Porte de Paris, F-71250, Cluny, FRANCE.

<sup>c</sup>Department of Metallurgical Engineering and Materials Science, Indian Institute of Technology Bombay, Powai, Mumbai-400076, Maharashtra, INDIA

<sup>d</sup>Department of Chemistry, GIS, Gandhi Institute of Technology & Management (Deemed to be University), Gandhinagr, Visakhapatnam-530045, Andhra Pradesh, INDIA

---

## Abstract

In the present studies, efforts were made to improve corrosion resistance of 90CrMoV8 tool steel by following plasma nitriding. Plasma nitriding of this steel at 500 °C for 6 and 8 h significantly improved the corrosion resistance when compared to the as-received steel. X-ray diffraction reveals  $\gamma'$  (Fe, Cr)<sub>4</sub>N and  $\epsilon$  ((Fe, Cr)<sub>2-3</sub>N) phases formed after nitriding. Potentiodynamic polarization tests in 3.5% NaCl reveal that plasma nitriding significantly improved the corrosion resistance as compared to untreated steel. The improvement in corrosion resistance may be attributed to the N solid solution and the presence of Fe-nitrides formed in the compound layer.

*Keywords: corrosion, low alloy steel, plasma nitriding, potentiodynamic polarization*

---

## 1. Introduction

Plasma nitriding has been accepted as one of the most industrially accepted plasma based processes for the enhancement of both mechanical and corrosion resistance properties of steels due to the presence of iron nitrides in the compound and diffusion layer [1-14]. A host of literature on nitriding elemental nitrogen adsorb on the surface of the metal part and subsequently diffuse into the bulk of the material [22-24]. However, to our knowledge plasma nitriding and corrosion resistance properties of 90CrMoV8 tool steel which is used in machining industries has so far not been studied in detail. The surfaces of these tools encounter high frictional forces and corrosives in the service conditions which synergistically degrades the tool life. Hence, for the wider applications of these steels in normal atmospheric conditions such studies are necessitated.

---

\* Corresponding author. Tel.: +919701841790; fax: +91-891-2795 311.  
E-mail address: rammohanrao.k@gmail.com

Previously, plasma nitriding of 90CrMoV8 steel had shown the improvement in hardness; however the corrosion propensity in wood juice was shown to be increased [25]. Apart from this specific wood juice electrolyte there is no other literature available so far on corrosion studies of this steel in a common electrolyte found in the atmosphere. As it is known that halides accelerate corrosion in Fe based alloys and also these are commonly found atmospheric corrodents, in the present study electrochemical investigation was conducted in NaCl electrolyte.

## 2. Experimental Details

### 2.1. Plasma nitriding process:

Steel samples in as-received condition were cut from a sheet to a dimension of 10 x 10 x 3 mm<sup>3</sup>. The composition of the steel is given as below in the Table 1.

Table 1: Composition of 90CrMoV8 steel

Element	C	Si	Mn	Cr	Mo	V	Fe
Wt. (%)	0.5	1.0	0.5	8.0	1.5	0.5	balance

After metallographic polishing and ultrasonic cleaning in acetone, samples were placed in the vacuum chamber of the nitriding reactor and evacuated to 0.5 Pa. Then nitriding was performed in a glow discharge plasma of H<sub>2</sub> and N<sub>2</sub> (80:20) gas mixture the temperature range of 450-500 °C for 1-8h.

### 2.2 Microstructural characterization:

For the structural characterization the samples after nitriding were exposed to X-ray diffraction (XRD) (XRD — INEL CPS 120 diffractometer —  $\theta/2\theta$  configuration) using Co- $k\alpha$  radiation source. Cross sections of the samples were observed in scanning electron microscope (SEM- model SEM-Jeol JSM-5900) to inspect the nitrided layer. The cross sections after metallographic polishing and etching with Vilella's reagent were exposed to SEM.

### 2.3. Electrochemical characterization:

To assess the corrosion resistance property, steel samples before and after plasma nitriding were subjected to detailed corrosion tests by following potentiodynamic polarization. To carry out these tests Biologic SP300 potentiostat with EC Lab software was used. The three electrode corrosion test cell constituted was consisting of the nitrided steel sample as the working electrode with 0.636 cm<sup>2</sup> exposed to the electrolyte, a saturated calomel electrode (SCE) as reference electrode and a platinum counter electrode. All the tests were performed in 3.5% (wt.%) NaCl electrolyte with pH=5.

## 3. Results and Discussion

### 3.1. X-ray diffraction and phase evolution

From the previous studies on similar steel, it was found that there was no significant improvement in micro hardness for the steel samples nitrided for shorter durations within the range of 1-5 h. However, a significant improvement of hardness was shown after nitriding for 6 and 8 h at all the temperatures (450-520 °C) [25]. It can be seen from the following Fig. 1 that, nitriding at lower temperature of 450 °C improved the hardness to ~ 1150 Hv however, the case depth formed was shallow which was only ~ 40  $\mu$ m thick [25].

With the view to obtain a wider case depth and hence to prolong the tool life, in the present studies, nitriding was performed at the temperature of 500 °C by varying the exposure time for 6 h and 8 h.

Fig.2 shows the XRD patterns of as-received and nitrided steels at 500 °C for different treatment times (6 h and 8 h). In the nitrided samples, mainly the peaks of Fe and  $\gamma'$ -(Fe, Cr) 4 N and  $\epsilon$ -(Fe, Cr)<sub>2-3</sub>N were revealed. Fe and other nitride peaks are much broadened, which is indicative of nitrogen incorporation in the crystal lattice.

Fig.3 represents SEM micrographs of the cross sections of the nitrided steel at 500 °C for 6 and 8 h. It is evident that a very thin and uniform compound layer is formed on top of the surface. After this a diffusion/hard layer was formed. This thin compound layer is known to be beneficial from the corrosion resistance point of view. The bright particles on the surface are formed by agglomeration of iron nitrides particles which depend on the nitrogen concentration [20].

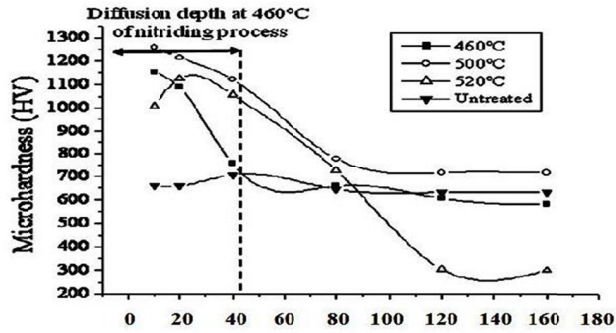


Fig. 1 Microhardness vs. depth profile of plasma nitrided steel for the duration of 10 h at temperatures 460, 500 and 520 °C along with untreated steel [25]

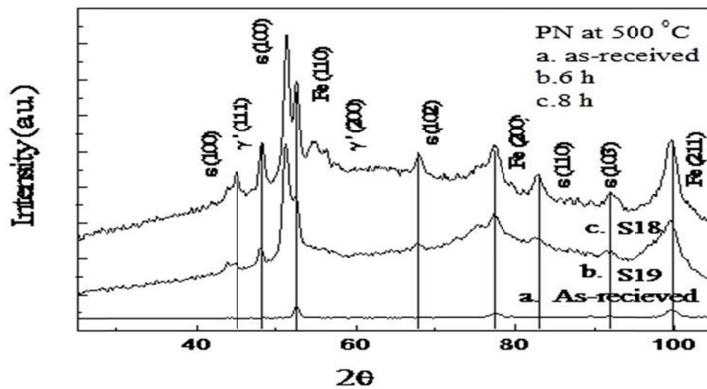


Fig.2 XRD patterns of (a) steel in as-received condition; and after plasma nitriding at 500 °C for the duration of; (b) 6 h (S19) ; and (c) 8 h (S18).

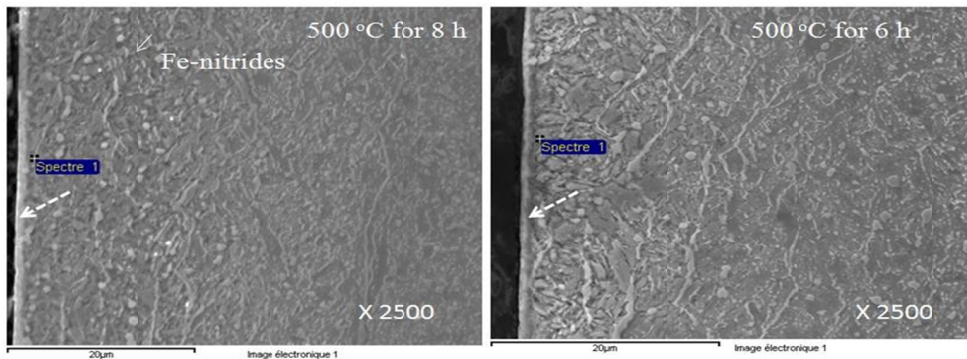


Fig. 3 SEM micrographs of 90CrMoV8 alloy steel after plasma nitriding at 500 °C for the durations of (a) 8 h and (b) 6 h.

### 3.2. Corrosion properties

Fig.4 represents the polarization transients of steel in the as-received condition along with the steel nitrided at different condition of time at the fixed temperature of 500 °C. All the corrosion tests performed at room temperature in an electrolyte containing 3.5% (by weight) NaCl which was freely exposed to the atmosphere. Fig.4 shows potentiodynamic polarization plots of untreated (Bare), S18 (nitrided at 500 °C for 8 h) and S19 (nitrided at 500 °C for 6 h) samples.

Steel sample after nitriding for 6 h (S19) shows more positive corrosion potential than the rest of the other two nitrided steel samples. Not only that a wider passivation range was shown by this steel. It was also revealed by XRD that this steel reveals (Fe, Cr)<sub>2-3</sub>N phase indicating the retention of higher concentration of Cr / (Fe, Cr)<sub>2-3</sub>N even after nitriding.

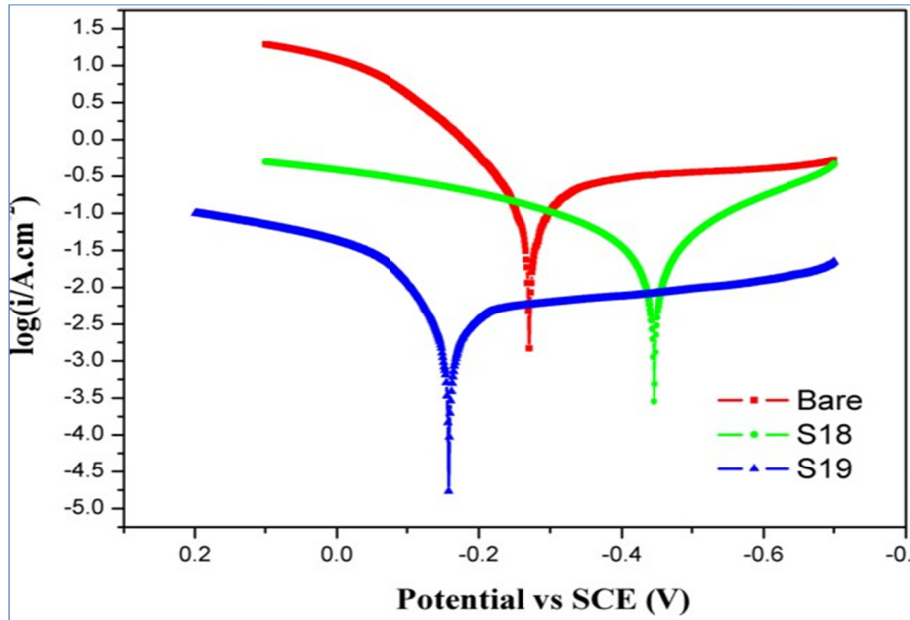


Fig. 4 Potentiodynamic Polarization of Bare, S18 and S19 samples

### 3 Conclusions

$\gamma'$ -(Fe, Cr)<sub>4</sub>N and  $\epsilon$ -(Fe, Cr)<sub>2-3</sub>N are the nitrides formed after plasma nitriding of 90CrMoV8 tool steel which can improve the hardness of steel. Nitrided steels have shown significantly higher corrosion resistance than as-received steel. Presence of nitrogen in solid solution and Fe- nitrides in the nitrided layer could be responsible for the enhancement of corrosion resistance property. To meet the requirements of corrosion resistance properties of 90CrMoV8 tool steel, nitriding at 500°C treatment would a good choice.

#### Acknowledgements

The authors thankfully acknowledge GITAM (Deemed to be University) for kindly giving us the opportunity to carry out this research and granting the permission for the publication of the work. The technical support extended by the team members of corrosion centre of the Department of Metallurgical and Materials Engineering, IIT Bombay, Powai, India and Ecole Nationale Supérieur of Art et Metiers, France is gratefully acknowledged.

#### References

- [1] A. Maniee, F. Mahboubi, and R. Soleimani, *Mater. Des.* 60 (2014) . 599-604
- [2] E. Menthe, K.T. Rie, J. W. Schultze, *Surf. Coat. Technol.* 74 (1995). 412–416.
- [3] M. Berg, C.V. Budtz-Jorgensen, H. Reitz, K.O. Schweitz, J. Chevallier, P. Kringhoj, *Surf. Coat. Technol.* 124 (2000) 25–31.
- [4] T. Nobuteru, M. Yoshiaki, C. Akiyoshi, H. Yuji, *Surf. Coat. Technol.* 196 (2005). 271–274.
- [5] W. Liang., *Appl. Surf. Sci.* 211(2003) 308–314.
- [6] T. Jun, O. Yuusuke, M. Hiroshi, K. Hideyuki, K. Shiomi, T. Imao, *J. Mater. Sci.* 21(1986) 2493–2496.
- [7] D. Pye, 2003 Practical nitriding and ferritic nitrocarburizing ed ASM International (Materials Park: Ohio) chapter 4 pp 31– 35.
- [8] M. Nikolussi, A. Leinweber, *J. Mater. Res.* 98 (2007) 1086-92.
- [9] Z. Pokorný, V. Hruby and Z. Studeny, *Metall. Mater.* 54 (2016) 119-124.
- [10] J.M. O'brien, D. Goodman, 1991, *ASM Handbook*, 4 pp. 420–424.
- [11] M.K. Lei, Z.L. Zhang, *Surf. Coat. Technol.* 91(1997 ) 25–31.
- [12] L. Chekour, C. Nouveau, A. Chala, M.A. Djouadi., *Wear*, 255 (2003) 1438.
- [13] Kishora Shetty, Subodh Kumar and Raghothama Rao P., IOP Publishing, 2008, *Journal of Physics: Conference Series* 100 062013
- [14] Sh. Ahangarani, F. Mahboubi and A. Sabour, *R2006 Vacuum 80 – Surface Engineering, Surface Instrumentation & Vacuum Technology* 1032
- [15] X. Li C, T. Bell, *Corr. Sc.* 48 (2006) 2036-2049
- [16] Magdalena Łepicka, Małgorzata Gradzka-Dahlke, *acta mechanica et automatica*, 7 (2013) 7155-159

- [17] A. Maniee, F. Mahboubi, R. Soleimani, *Mater. Des.* 60 (2014) 599–604
- [18] A. Mashreghi, S. Soleimani, S. Saberifar, *Mater. Des.* 46 (2012).532–538
- [19] A. Allenstein, C. Lepienski, A. Buschinelli, S. Brunatto, *Appl. Surf. Sci.*, 277 (2013). 15–24
- [20] H. Forati Rad, A. Amadeh, H. Moradi, *Mater. Des.*32 (2011) 2635–43
- [21] D.C. Wen, *Surf. Coat. Technol.* 204 (2009) 511-19
- [22] T. Spalvins, *Ion Nitriding Conference proceedings ASM International* 1986.
- [23] W. Möller, S. Parascandola, T. Telbizova., R. Gunzel. and E. Richter, *Surface & Coatings Technology* 136 (2001) 73
- [24] *ASM Handbook* 5 (1996) 411
- [25] Corinne Nouveau, Philippe Steyer, K. Ram Mohan Rao, Denis Lagadrillere, *Surf. Coat. Technol.* 205 (2011) 4514–4520.