Journal of Materials Science and Engineering A 8 (1-2) (2018) 17-24
doi: 10.17265/2161-6213/2018.1-2.003

Influence of Medium Used during Ferritic Nitro-Carburizing of AISI H-13 Hot Work Tool Steel

Valmik Bhavar, Prakash Kattire, Jagdish Sherkar, Digvijay Sheed and RKP Singh
Kalyani Center for Technology and Innovation (KCTI), Bharat Forge Ltd., Pune 411036, India

Abstract: Ferritic nitro-carburizing is one of the most popular surface hardening methods used to improve lifespan of hot work tool steels. Different types of mediums like gas, liquid, plasma and fluidized bed are generally used during ferritic nitro-carburizing process. In this paper, various ferritic nitro-carburizing methods were compared where gas, salt bath and fluidized are used as mediums. AISI H-13 hot work tool steel specimens were treated by using these different methods of nitro-carburizing and their performance was evaluated by using micro-structural and mechanical analysis. Optical microscopy, micro-hardness testing and X-ray stress analyzer were used for specimen characterization. Moreover, pin on disk dry sliding wear tests were performed to compare wear performance of specimens treated with different nitro-carburizing methods. It is perceived that, medium used during nitro-carburizing has significant influence on the final surface properties that can be achieved by ferritic nitro-carburizing.

Key words: Nitro-carburizing, gas nitrocarburizing, liquid nitriding, fluidized bed nitro-carburizing, hot work tool steels.

1. Introduction

Mechanical behavior of components under service can be significantly improved by changing surface properties. Different types of mechanical and thermo-chemical surface treatments may be used to modify surface properties like hardness, wear resistance, corrosion resistance, contact fatigue etc. [1, 2].

Ferritic nitro-carburizing is one of the most popular thermochemical surface hardening treatments that involves diffusion of nitrogen and carbon into the part surface at sub critical temperatures. The ferritic nitro-carburizing usually takes place at 550-570 °C temperature. Significant improvements in wear, anti-seize, corrosion and fatigue properties with minimum part distortion are the main benefits of this hardening process. Various surface treatments like gas nitro-carburizing, salt bath nitro-carburizing and fluidized bed nitro-carburizing are commercially available based on media used for diffusion of nitrogen and carbon [3].

Ferritic nitro-carburizing was first carried out by using salt bath furnace where liquid salt is used as source of nitrogen and carbon. When steel parts are placed into a preheated liquid salt, there is sufficient energy localized near the surface due to differences in chemical potential that then allows nitrogen and carbon to diffuse from the salt into the steel substrate. There are several commercial salt bath nitro-carburizing treatments available like ARCOR, MILONITE and TUFFTRIDE. Different trade names are used due to differences in the chemistry of the nitriding chemicals and the secondary processing steps. This method is used less frequently because of cyanate bath high toxicity [4].

Gas nitro-carburizing is garnering major interest since it uses improved environment as compared to salt bath [5]. The atmosphere contains ammonia, carbon monoxide and hydrogen as source of nitrogen and carbon. A number of different variants of gas nitro-carburizing are available in the market like nitemper, nitrotec, nitroc etc. Regardless of the relatively extensive use of the gas nitro-carburizing method, this technique may be dangerous, both for the

Corresponding author: Mr. Valmik Bhavar, M.Tech (IIT Bombay), KCTI, Bharat Forge Limited, Pune, research fields: surface treatments, additive manufacturing.

employees and natural environment, due to the produced exhaust gases and explosion risk of flammable atmosphere in the absence of appropriate precautions behavior [6].

Drawbacks of gas nitro-carburizing are significantly reduced in the atmospheric fluidized-bed method, where there is no risk of explosion in the furnace workspace. This method is gaining popularity due to some inherent advantages as compared to conventional techniques. Fluidized bed furnace contains mixture of sand and alumina particles which are forced to behave like a liquid by method of fluidization [7]. This can be achieved by passing stream of gas upward through particle bed. The velocity of gas stream is raised until it overcomes the resistance caused due to mass of particles. This velocity is known as critical velocity at which particles behave like liquid. The turbulent flow of particles in fluidized bed furnace causes higher heat transfer rates and uniform temperature distribution. Owing to their excellent energy efficiency, temperature uniformity, rapid heat transfer, and producing no toxic waste, nitro-carburizing in fluidised bed furnaces have seen widespread use in the heat treating industry since their inception [8].

Processing times required and beneficial effects imparted on parts by using all these treatments may vary depending on the selection of treatment and material to be processed. It is therefore beneficial to understand role of different media used for diffusion of nitrogen and carbon during ferritic nitro-carburizing.

2. Experimentation

The commercial grades of AISI H-13 tool steel in harden and tempered condition were selected for this work. Table 1 shows chemical composition of this material which was analyzed by vacuum emission spectrometer. The hardness of this material in as received (H & T) condition was 47-48 HRC.

Small specimens of appropriate dimensions required for micro-structural, mechanical and wear analysis were prepared from this material for planned research. All these prepared specimens were named as AR (as received) specimens. Three batches of specimens from AR group were then separately nitro-carburized in gas, salt bath and fluidized bed furnaces. The gas nitro-carburizing was carried out in closed gas nitro-carburizing SERLIN make furnace. The specimens were first preheated to 400 °C allowing 5 hours for soaking and then exposed to gaseous environment of N_2 (nitrogen), NH_3 (ammonia) and CO_2 (carbon dioxide). The nitro-carburizing process was carried out in two stages at 570 °C and total 12 hours soaking time. The volume percentages of various gases used during nitro-carburizing were varied between the two stages. The specimens treated in this manner were named as gas NC (nitro-carburized) specimens.

Salt bath (liquid) nitro-carburizing was carried out in electrically heated crucible furnace. The second batch of tool steel specimens was first pre heated to 300-340 °C and then submerged into the salt bath lying in charging racks. The salt bath consists of alkaline cyanate and alkaline carbonate. Through oxidation and thermal reaction with the immersed specimen surface, at nitriding temperature the alkaline cyanate releases nitrogen and carbon which diffuse into the surface of the specimens. The nitro-carburizing process was carried out at 570 °C and total 6 hours of soaking time. The treated batch of specimens was named as liquid NC (nitro-carburized) specimens.

Third batch of tool steel specimens was treated by nitro-carburizing in fluidized bed environment. The nitro-carburizing was carried out at dynamic surface

Table 1 Chemical composition of H-13 tool steel (Harden and tempered condition).

Elements	C	Mn	Si	Cr	Ni	Mo	V	S	P	Fe
Weight %	0.385	0.35	1.049	5.11	0.152	1.278	0.993	0.001	0.014	Remaining

technologies, USA by using Dynablue fluidized bed processing. The specimens are immersed in heated fluidized bed with nitrogen, ammonia and carbon producing gases. Temperature maintained at 570 °C for maximum 5 hours of soaking time. All these specimens were referred as FBR NC (fluidized bed nitro-carburizing) specimens.

All specimen treatment conditions used in this study are summarized in Table 2 along with their nomenclature.

3. Characterization

Microstructure, micro hardness, X-ray stress analysis and wear tests of the DIN1.2714 steel were conducted at room temperature in order to compare the effects of un-treated and nitro-carburized specimens at different environments. Micro hardness indentations with a load of 200 g were performed from the treated surface until a depth at which the initial hardness was not modified by the nitro-carburizing treatment. These tests were performed using aon Matsuawa with Celmex make micro-hardness tester according to the ASTME384 standard. For effective case depth measurement, shot peened specimens were cut in transverse direction and mounted with bakelite powder at 175 °C. Cut faces of samples were then polished with 300 and 400 G polish papers followed by cloth grinding with aluminum oxide paste. The hardness was measured at 15 locations which were kept 0.04 mm apart. Limiting case depth was determined by adding 50 HV to core material hardness of same sample.

Residual stress profiles generated by different nitro-carburizing treatments were determined by XRD (X-ray diffraction) and incremental layer removal by electro polishing. The XRD technique used in the present study to determine residual macro-stresses was the $\sin^2\psi$ method. Measurements were made by using an "Xstress 3000" device as per ASTM000 standard. A Cr-Kα X-ray source was used employing a wavelength of 0.229 nm and measurements were taken on the (211) diffraction peak of the marten site, which was recorded at a 2θ angle of approximately 156º.

Pin on disk wear testing under dry sliding condition was carried out on pin-on-disk DUCOM make tribometer. The specimen shown in Fig. 1 was manufactured and given planned treatments as already described. Pins with five different types of material conditions (AR, Gas NC, Liquid NC, and FBR NC) were tested. This tribometer works on the Archimedes equation given below by Refs. [9, 10].

$$k = \frac{\Delta V}{F_N L}$$

where, k—wear rate in mm^3/Nm, ΔV—the worn out volume of material (mm^3), F_N—Normal force and L—Sliding distance.

Disk and sample pins were cleaned with acetone to avoid any surface contamination. Fresh disk track was used for every new experiment. Table 3 shows testing parameters which were used during wear testing. The results of wear testing are reported in terms of weight loss of pins, wear rate and coefficient of friction. Weight loss of pins was calculated by taking weight of pin before and after testing whereas wear rate was calculated by using Archimedes equation as mentioned above.

Table 2 Sample treatment and their nomenclature.

Sr. no.	Specimen treatment condition	Nomenclature
1.	Harden and tempered (as received)	AR
2.	Nitro-carburizing in gas furnace	Gas NC
3.	Nitro-carburizing in salt bath	Liquid NC
4.	Nitro-carburizing in fluidized bed reactor	FBR NC

Table 3 Pin on disk wear test parameters.

Test condition	Room temperature dry sliding
Load (N)	150
Sliding velocity (m/s)	0.6
Sliding distance (m)	1,200

Fig. 1 Test specimen used in wear testing and pin on disk tribometer.

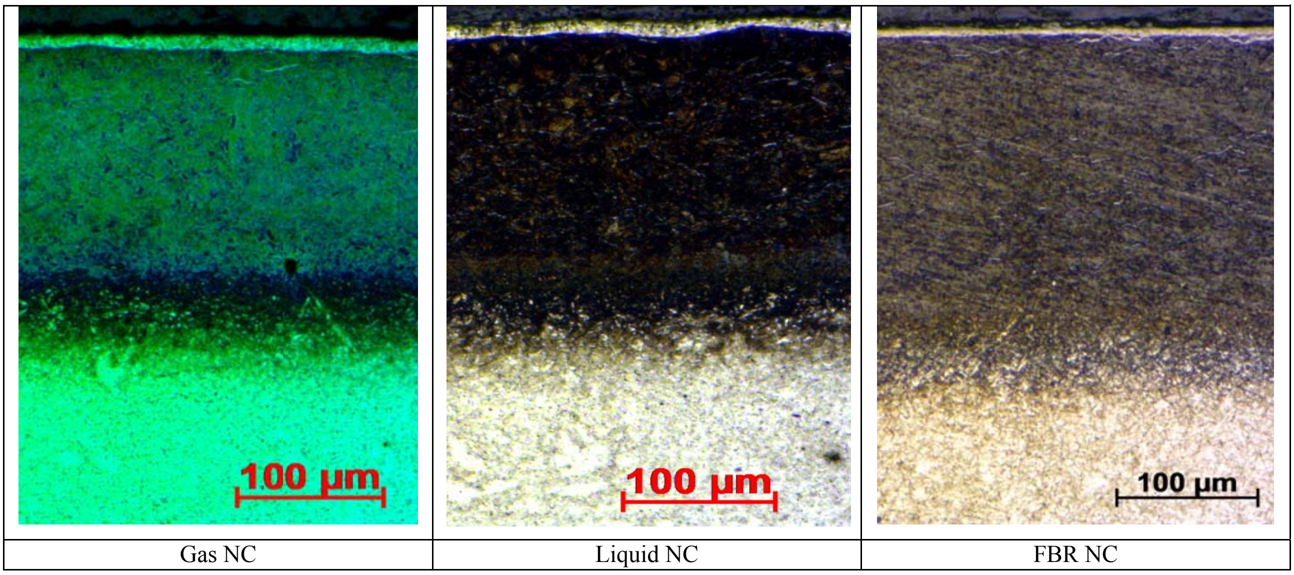

Fig. 2 Surface characteristics of different nitro-carburized surface treatments.

4. Results and Discussion

4.1 Micro-structural Analysis

Fig. 2 shows optical images of tool steel specimens subjected to different nitro-carburized treatments. The images clearly show three distinct zones obtained by nitro-carburizing treatments like compound layer (white layer), diffusion layer and base material.

Gas and liquid nitro-carburized specimen's shows similar compound and diffusion layer thickness. The compound layer thickness around 10-12 µm and diffusion layer thickness of 240 µm is observed in these specimens. Specimens treated in fluidized bed furnace show slightly smaller compound layer and deeper diffusion layer as compared to gas and liquid nitro-carburized specimens. The fatigue performance of nitro-carburized components is greatly affected by compound layer thickness. Higher the compound layer thickness, lower will be fatigue life and vice versa [11]. This indicates that lower compound layer thickness obtained in case of FBR nitro-carburizing may help to improve fatigue properties as compared to other two methods of nitro-carburizing.

4.2 Micro-hardness

The hardness distribution along with the surface layers for specimens treated by different nitro-carburizing treatments (Gas NC, Liquid NC and FBR NC) are shown in Fig. 3. Initial material hardness used in this experiment was 45-47 HRC. Different nitro-carburizing treatments increase this hardness to 61-65 HRC at surface which gradually decreases along depth. Similar hardness and case depths were observed for gas and liquid nitro-carburizing treatments. Gas and liquid nitro-carburized specimen shows maximum surface hardness of 61-62 HRC which then gradually decreases to base material hardness after effective case depth of 0.21 mm. However, nitro-carburizing in fluidized bed results in higher surface hardness of 65 HRC and deeper case depth of 0.24 mm as compared to other treatments. Rapid heat transfer and more uniform temperature distribution in case of fluidized bed may be responsible for this beneficial hardening effect.

4.3 Residual Stresses

The distribution of the measured residual stress in the thickness direction from the surface for the different nitro-carburized treatments is shown in Fig. 4. The values for residual stresses near the surface are similar for gas and liquid nitro-carburized specimens. However, liquid nitro-carburized specimen shows slightly deeper penetration. The maximum residual stresses and penetration depth are achieved in case of fluidized bed nitro-carburized specimens as compared with gas and liquid. Maximum residual stresses up to 415 MPa and penetration depth of 250 μm were observed in FBR nitro-carburized specimens. Alumina particles at fluidization velocity behave like liquid and cause fast heat transfer and uniform temperature all over the furnace. Nevertheless, along

Table 4 Compound and diffusion layer thickness obtained in various nitro-carburizing treatments.

Sr. no.	Nitro-carburizing treatment	Compound layer thickness (μm)	Diffusion layer thickness (μm)
1	Gas nitro-carburizing (Gas NC)	10-12	210
2	Liquid nitro-carburizing (Liquid NC)	8-9	200
3	Fluidized bed nitro-carburizing (FBR NC)	6-7	240

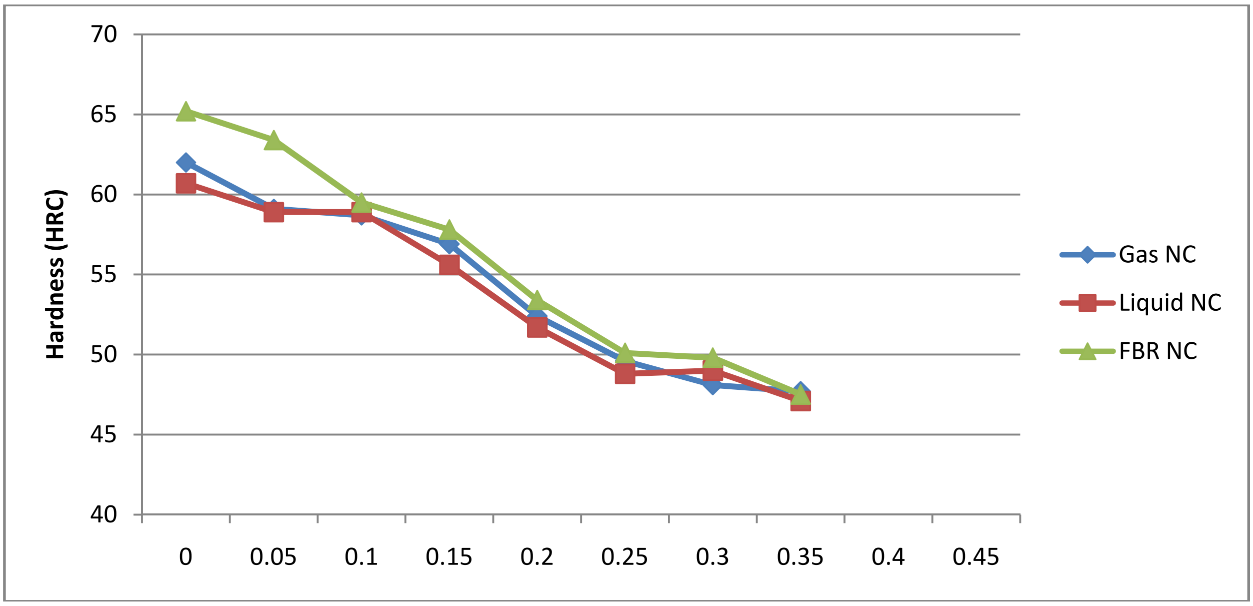

Fig. 3 Hardness distribution along with surface layers for different surface treatments.

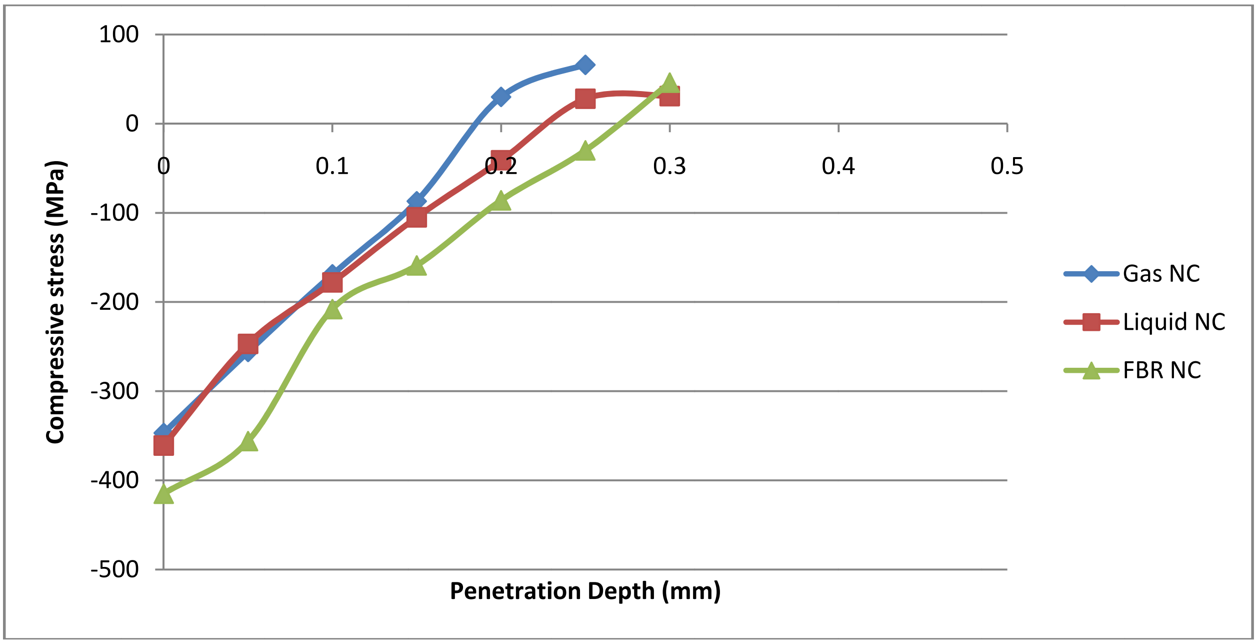

Fig. 4 Residual stress distribution along thickness direction for specimens treated with different surface treatments.

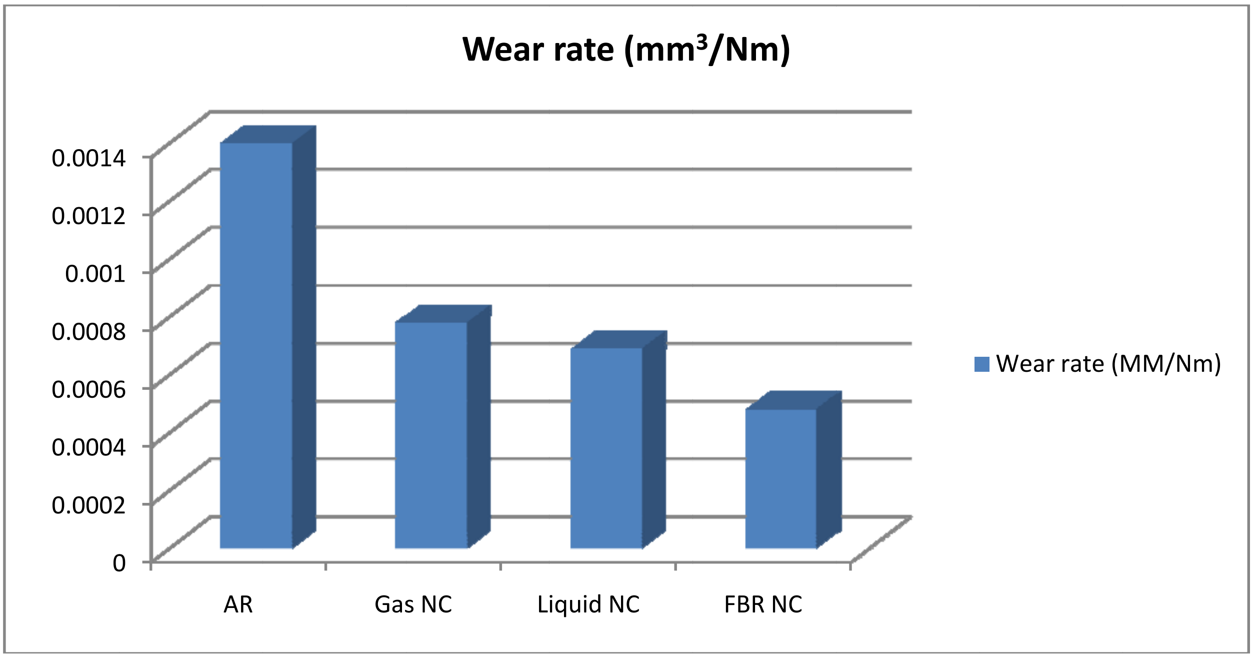

Fig. 5 Wear rate for different specimen treatment conditions.

with fast heat transfer and temperature uniformity, alumina particles may also cause plastic deformation of surface which is responsible for high compressive stresses and their penetration depth.

4.4 Wear Performance

The wear performance of tool steels is very complex phenomenon and it not only depends on the hardness but also on the microstructure, process variables and properties of sliding materials [10]. In the present research work, wear performance in terms of wear rate and coefficient of friction for different specimen conditions are measured. The wear rate for different specimen conditions is given in Fig. 5. The wear rates for nitro-carburized specimens are significantly lower than conventional as received (AR) specimens. It is clearly due to surface hardness enhancement by hardening effect induced by diffusion of nitrogen and carbon during various nitro-carburizing treatments. Wear rate of harden and tempered (AR) specimen is 0.0014 mm/Nm. Nitro-carburizing in gas, liquid and fluidized bed environment reduces this wear by 44%, 51% and 66% respectively. Highest improvement in fluidized bed nitro-carburizing is expected since it has highest hardness.

5. Conclusions

To summarize, this research discovered the effect of different media used for nitro-carburizing of AISI H-13 tool steel and analyzed mechanisms behind it. The following conclusions can be drawn from the work:

Nitro-carburizing causes major surface characteristic changes like hardening effect and compressive residual stresses generation through diffusion of nitrogen and carbon.

Media used during nitro-carburizing significantly affects the beneficial effects obtained from nitro-carburizing.

Different nitro-carburizing treatments can reduce wear rate of AISI H-13 tool steel by 44-66%.

Best performance of nitro-carburizing in terms of wear rate, surface hardness and compressive residual stresses generation was observed in fluidized bed (FBR NC) nitro-carburizing.

Acknowledgments

The authors gratefully acknowledge the extended support provided to this work by KCTI (Kalyani Centre for Technology and Innovation) for providing financial funding, laboratory and library facilities. The authors also acknowledge the support provided by Bharat Forge Ltd, Pune and DSIR (Department of Scientific and Industrial Research), Govt. of India. Finally, the authors would like to express special thanks and gratitude to review committee and top management of Bharat Forge Ltd for granting the permission to publish/present the research work.

The authors also wish to place their sincere thanks to dynamic surface technology, USA for allowing using their DYNA-BLUE technology for this experimentation work.

References

[1] Ekinci, Ş., Akdemir, A., and Demirci, M. T. 2011. "Effect of Surface Roughness of Salt Bath Nitrided AISI 4140 to the Wear Rate." Presented at 6th International Advanced Technologies Symposium (IATS'11), 16-18 May 2011, Elazığ, Turkey.

[2] Terent'ev, V. F., Michugina, M. S., Kolmakov, A. G., Kvedaras, V., Čiuplys, V., Čiuplys, A., and Vilys, J. 2007. "The Effect of Nitriding on Fatigue Strength of Structural Alloys." *Mechanika* 2 (64), ISSN 1392-1207.

[3] Arslan, K., Selte, A., Ulker, S., and Hatman, A. 2013. "Comparison of Different Thermochemical Treatments on Surface Properties of Impax Supreme Tool Steel." *Journal of Materials Science and Engineering A* 3 (11): 732-7.

[4] Bell, T., Sun, Y., and Suhadi, A. 2000. "Environmental and Technical Aspects of Plasma Nitrocarburizing." *Vacuum* 59: 14-23.

[5] Holm, T., and Sproge, L. "Nitriding and Nitro-Carburizing." *Furnace Atmospheres* 3.

[6] Ciski, A., Babul, T., Lankiewicz, K., Obuchowicz, Z., and Šuchmann, P. 2014. "New Method of Nitrocarburizing of X37CrMoV5-1 Hot Working Tool Steel in Chemically Active Fluidized Bed." *Journal of Achievements in Materials and Manufacturing Engineering* 62 (2).

[7] Renganathan, C., and Raghunath, V. "Fluidised Bed Heat Treatment—A Flexible Heat Treating Technology."

[8] Cicak, I. 2014. "The Study of Metal Nitride Layer Formation by Solid-State Reactions." Master's thesis, Deakin University.

[9] Bhavar, V., Khot, S., Kattire, P., and Singh, P. 2016. "Integrating Deep Cryogenic Treatment with Nitro-Carburizing to Improve Performance of AISI H-13 Tool Steel." Presented at International Federation of Heat Treatment and Surface Engineering 2016 Proceedings of the 23rd IFHTSE Congress, April 18-21, 2016, Savannah, Georgia, USA.

[10] Bhavar, V., Khot, S., Kattire, P., and Mehta, M. 2015. "Effect of Deep Cryogenic Treatment (DCT) on AISI H13 Tool Steel." Presented at the 28th ASM Heat Treating Society Conference, October 20-22, 2015, Detroit, Michigan, USA.

[11] Braza, J., and Pearson, P. 1993. "Tribological and Metallurgical Evaluation of Ferritic Nitrocarburized M-50 and M-50 NiL Steels." *Creative Use of Bearing steels,* ASTM STP 1195, ASTM.