

Effect Of Nitriding Time and Surface Contaminants On Case Depth Of CRDI Nozzle.

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Abstract - The fuel-injector nozzle is a vital component in diesel engine, profoundly influencing engine performance. Enduring extreme conditions like high temperatures and pressures up to 3,000 bar during operation, it demands high strength, wear resistance, and fatigue strength, achievable through careful material selection and precise heat treatment. Despite extensive research on gas nitriding process parameters and their impact on nitriding properties across steel grades, understanding the effects of surface structure, composition, and contaminants on the process remains challenging. In the current study, an attempt was made to understand effect of surface contaminants on effective case depth of injector nozzle subjected to zero-flow gas nitriding process which offers precise process control compared to the double component gas nitriding process. While zero-flow technology has been successfully demonstrated in car applications for diesel engine fuel injector nozzles, its adoption in high-end diesel engine applications like trucks and tippers is still limited. In this study, high-end injector nozzles made of H13 steel was subjected to zero-flow gas nitriding at 500±50°C for varying time intervals. Nozzles with RP oil stains, cutting oil stains, and clean surfaces were subjected to gas nitriding to assess the impact of surface contaminants on nitriding properties. Nitriding properties were assessed using optical microscopy and a microhardness tester. The results show that longer nitriding times increases effective case depth, but surface contaminants reduces it by impeding nitrogen diffusion, resulting in non-uniformity. Optimizing these factors significantly enhances nozzle performance, underscoring the importance of thorough surface preparation in gas nitriding processes.

Key Words: Kn, Effective Case Depth, Surface Hardness, Surface Contaminants, Nitrogen Diffusion.

Abbreviations:

CRDI Common Rail Diesel Injector
EDS Electron Dispersive Spectrum
Kn Nitriding Potential
RP Rust Preventive
SEM Scanning Electron Microscopy
ECD Effective Case Depth
DOE Design of Experiment
GN Gas Nitriding
VHT Vacuum Hardening & Tempering

1.INTRODUCTION

The common rail diesel injection (CRDI) fuel injection system stands as a critical component in diesel engines employed in on-highway applications like trucks and tippers. Its primary function is to inject fuel into the engine cylinders through a singular, shared line known as the common rail. This common rail is interconnected with all fuel injectors. Operating as an accumulator, the common rail plays a pivotal role in ensuring a constant fuel pressure delivery to the fuel injectors up to 3000 bar [1][3]. A fuel injector is an electronically controlled valve supplied with pressurized fuel by the fuel pump, that sprays pressurized fuel as a fine mist for efficient combustion in an engine [2][3]. Hence injector nozzle is a vital component of diesel engine for the performance and emissions of modern diesel engines. With key design parameters such as the injector seat, sac, and nozzle hole size and shape significantly influencing combustion characteristics, emissions stability, performance consistency, and injector durability over time [2][3].

The fuel injector nozzle operates under extreme conditions, enduring high pressure and temperatures during the combustion process in an internal combustion engine which makes them susceptible to accelerated wear and defects. Therefore, the design of fuel-injection nozzles must prioritize adequate strength, impact resistance, fatigue resistance, and abrasion resistance [6]. For high end application such as trucks and tippers wherein injection pressures are beyond 2000 bar, Gas nitriding has emerged as an optimal heat treatment process capable of imparting these essential properties to the nozzle, ensuring durability and performance under extreme conditions. It involves the formation of a robust nitride layer on the surface of the nozzle which significantly enhances the surface resistance to wear and erosion [4]. It imparts good dimensional stability to the component and is considered a low-temperature process, typically conducted at ~ 500°C±50°C. Furthermore, the surface hardness achieved through gas nitriding imparts sufficient fatigue strength to the nozzle. The formation of a white layer during the process enhances the wear resistance and corrosion resistance of the nozzle and serves as an additional protective barrier against the challenging conditions encountered during engine operation.

In the present work, zero flow nitriding technology was used to nitride hot working tool steel H13 fuel injector

nozzles used in truck and tipper applications. A novel case-hardening technology such as vacuum carburizing & vacuum nitriding was introduced by seco in 2014, marking a significant advancement in the heat treatment of diesel engine fuel-injection nozzles crafted from hot-working tool steel, have been employed to enhance surface properties of nozzle, particularly beneficial for complex geometries of nozzles such as blind holes (1 mm deep hole-seat/sac area), as seen in injector nozzles [6][7]. With rigorous testing the technology was deemed ready for industrial deployment, signifying a milestone in enhancing the durability and performance of fuel-injection nozzles in diesel engines [6]. Building upon their earlier innovations, Seco further advanced the field by introducing the zero flow concept—a gas nitriding process that is not only economical but also environmentally friendly. This breakthrough enables a significant reduction in the consumption of industrial gases compared to existing processes, representing a major stride towards sustainability in heat treatment practices for diesel engine fuel-injection nozzles and other components, enabling the formation of layer phase structures with comparable precision to processes utilizing double-component atmospheres [10][11]. This technology is found to have been successfully implemented for car engine parts such as crankshafts, camshafts, piston rings, poppet valve springs and discs, piston pins, or nozzles [8][9]. It became popular because it utilizes a single-component atmosphere, specifically raw ammonia (NH₃), for the nitriding process, and control of the nitriding potential K_n is achieved by adjusting the chemical composition of the nitriding atmosphere, which is solely managed by temporarily halting and then reactivating the feeding of NH₃ into the furnace [10][11]. Some studies have shown that diesel engine nozzles used in trucks and tipper applications can also be vacuum nitrided with the use of a mixture of gases such as NH₃, CO₂ & N₂ for the nitriding process [7]. While zero flow technology has been demonstrated for diesel engine fuel injector nozzles in-car application, its adoption in high-end diesel engine applications such as trucks and tippers remain limited. The current study attempted to apply zero flow nitriding technology to diesel engine fuel injector nozzles used in truck and tipper applications, where high injection pressures up to 2000 bar are common. While doing this, our focus is on comprehending the impact of nitriding process parameters and surface contaminants on the effective case depth to unveil the correlation between these parameters for the H13 injector nozzle.

2. MATERIALS AND METHODS

2.1. Material

In the current work, ingot casted, hot rolled & spheroidized annealed H13 steel was studied as an experimental material. Table 1 shows the standard chemical composition of the steel. The microstructure of H13 steel is composed of a ferrite matrix, with a uniform distribution of

spheroidized carbides, shown in Figure 1. These carbides are composed of molybdenum, vanadium, chromium, and iron.

Table - 1: Chemical Composition of H13 Steel (mass %)

C	Si	Mn	P	Cr	Mo	V	Fe
0.35-0.45	0.80-1.20	0.20-0.50	0.03 max	4.75-5.50	1.10-1.75	0.80-1.20	Bal

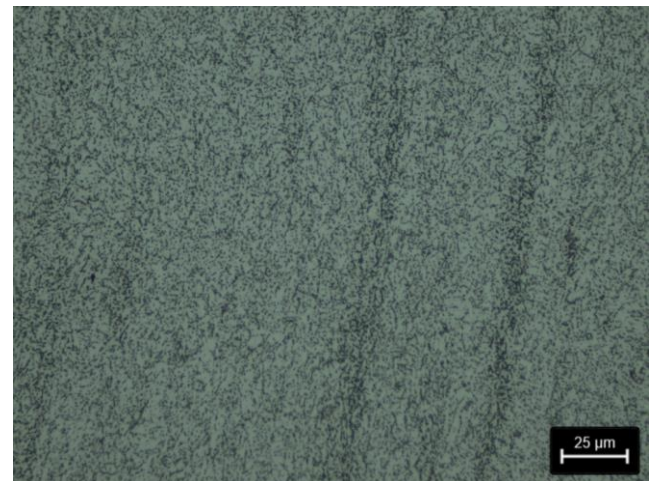


Fig-1: Microstructure of Ingot Casted, Hot Rolled & Spheroidized Annealed H13 Steel at 500X Magnification.

2.2 Manufacturing Method of Injector Nozzle

The processing of the nozzle right from the raw material stage to finish machining is explained in a pictorial view as shown in figure 2.

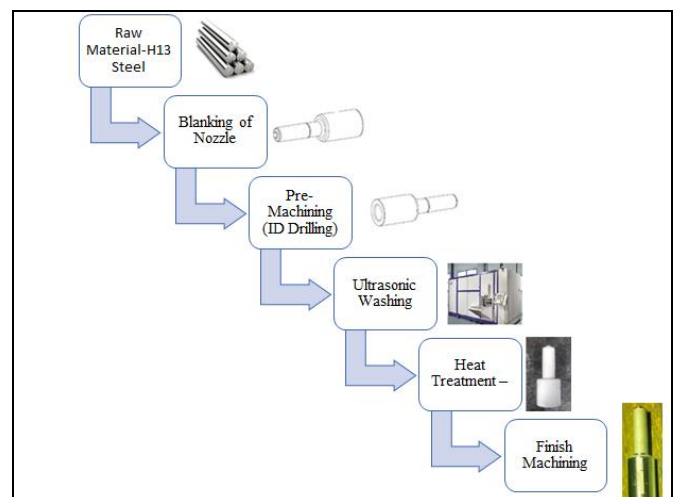


Fig-2: Manufacturing Route of Injector Nozzle.

2.2.1 Heat Treatment Process

Pre-machined (blanking+ id drilling) nozzles were RP oiled to protect them from rust generation. Before heat

treatment, ultrasonic washing of nozzles was carried out to remove RP oil from parts. During ultrasonic washing, it was observed that some parts washed with a 15-minute washing cycle left some traces of RP oil stains in a 1mm deep hole of the nozzle which is the seat/sac area as shown in figure 5 (c). The other areas of the nozzle such as outer surface & id were found getting cleaned with no traces of any RP oil stain.

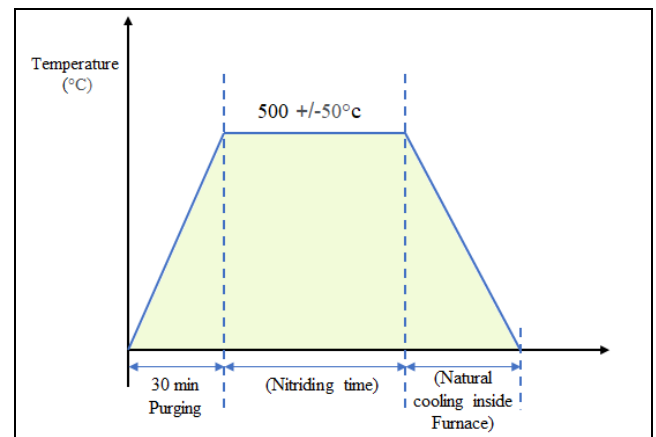
Post washing, parts were loaded into heat treatment fixtures for vacuum hardening & tempering followed by gas nitriding. The heat treatment process was designed to achieve effective case depth controlled within a few hundred microns while targeting surface hardness greater than 800 HK and core hardness as low as 462 HK. To impart sufficient hardness to the nozzle, vacuum hardening was carried out at a temperature of $1030 \pm 50^\circ\text{C}$ for 16 ± 2 hrs and gas quenched at 15 ± 5 bar pressure to produce martensitic structure. To eliminate stresses generated during gas quenching & improve toughness, tempering was carried out at $530 \pm 50^\circ\text{C}$ for 4 ± 4 hrs. Once the nozzles were vacuum hardened & tempered, gas nitriding was carried with controlled nitriding potential at a constant temperature of $\sim 500^\circ\text{C} \pm 50^\circ\text{C}$, for time 18-43 hrs.

The wide range of time was chosen to understand the effect of nitriding time on the effective case depth of the nozzle. To ensure precision about compound layer and diffusion zone thickness, it is important to have good furnace temperature uniformity, typically $\pm 5^\circ\text{C}$ ($\pm 41^\circ\text{F}$) which was ensured in gas nitriding furnace. For both processes i.e. hardening/tempering & nitriding, a seco furnace developed by manufacturer seco (sweden) was employed.

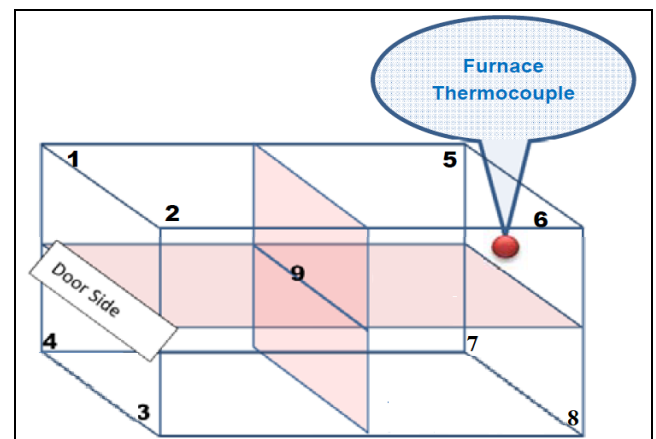
The gas nitriding cycle is shown in Figure 3 (a), The process steps in the nitriding cycle have three major steps:

- 1) Heating to temperature $500^\circ\text{C} \pm 50^\circ\text{C}$
- 2) Holding at temperature for a sufficient time to reach the required nitriding depth and
- 3) Cooling with Inert N_2 Gas.

During VHT & GN, for metallurgical inspection, nozzles were loaded at 9-location of the furnace as shown in Figure 3 (b). This is to capture the overall furnace atmosphere and its effect on metallurgical parameters.



(a)



(b)

Fig-3: (a) Gas Nitriding Cycle of Injector Nozzle (b) Loading of Injector Nozzle in Heat Treatment Furnace at 9-Poles.

2.3 Metallographic Inspection

Sample preparation for metallographic inspection was carried out by cross-sectioning 9-pole nozzles in the centreline to check surface hardness, effective case depth & core hardness at the sac (location M) & seat (location U) area which are the most critical areas in the nozzle from fuel injection perspective as shown in figure 4.

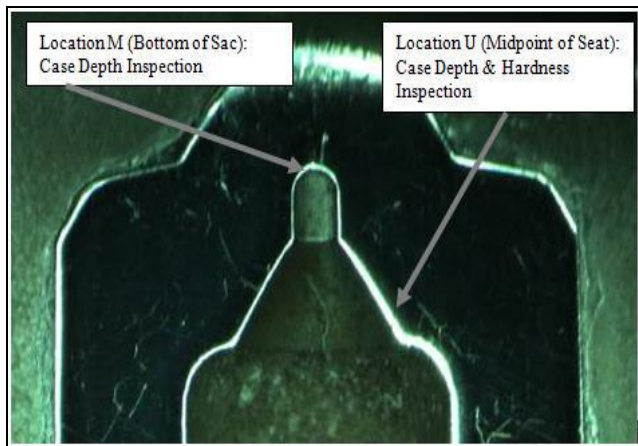


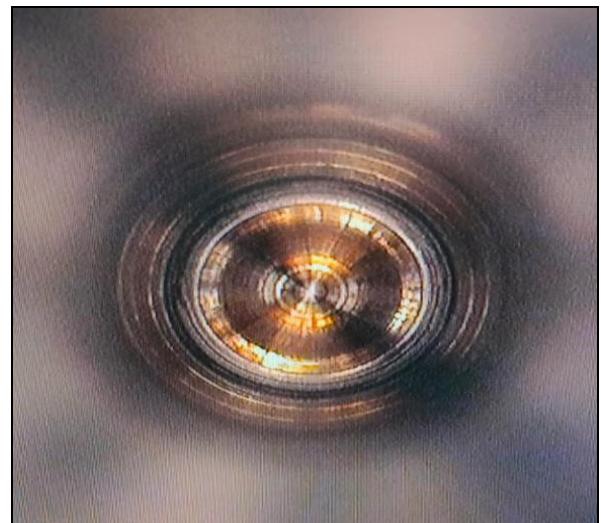
Fig- 4: Cross-sectional view of Seat & Sac Location of 1 mm Deep Hole of Injector Nozzle at 50X.

For optical microscopy, the cross-sections were investigated using Carl Zeiss imaging optical microscope. The samples were mounted, polished with colloidal silica, and etched with nital (2-3 vol.% HNO₃ in ethyl alcohol) for about 10 secs before the examination. Hardness & case depth measurements were carried out for heat-treated samples on the Knoop hardness tester by applying HK0.5 (500 g) load. Surface hardness was measured at a depth of 0.05 mm & core hardness was measured at a depth of 1 mm.

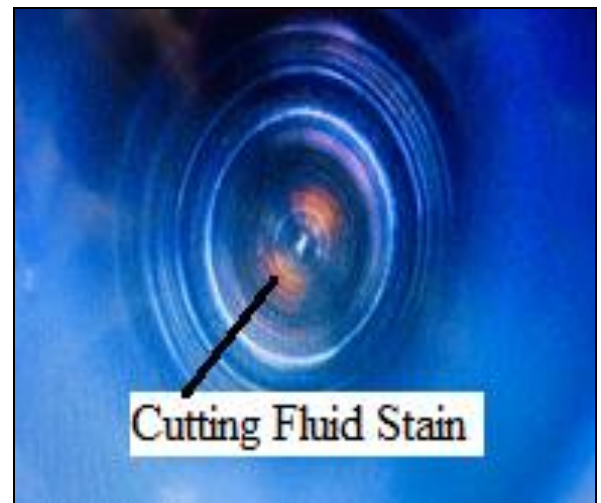
2.4 Design of Experiments (DOE)

The design of the experiments were conducted considering two factors that would impact effective case depth in the gas nitriding process. These two factors were chosen as nitriding time & surface contaminants. To understand the impact of nitriding time, nozzles were nitrided at different nitriding times (18-43 hrs) keeping temperature & kn as constant. To understand the impact of surface contaminants, different sets of samples were prepared and subjected to a heat treatment process. The combination of samples was as follows:

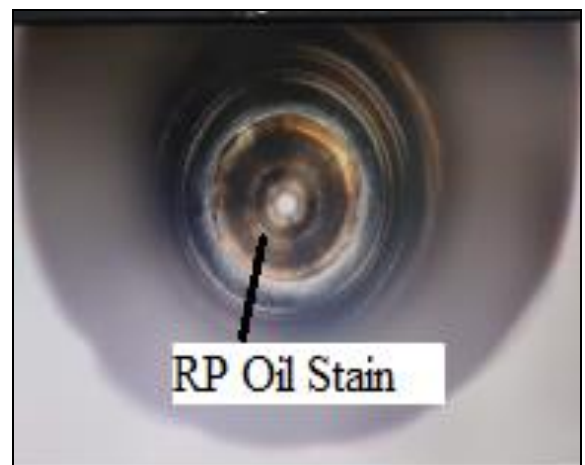
- 1) Nozzle dipped in RP Oil (with no wash)
- 2) Nozzle with cutting oil stain in seat/sac area (with no wash),
- 3) Nozzle with contamination in seat/sac area (contamination could be cutting oil stain or rp oil stain post washing)
- 4) Nozzle with no contamination (with post wash)



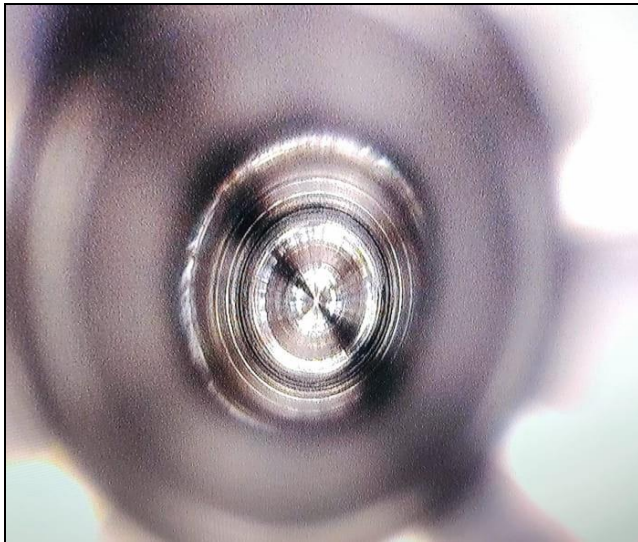
(a)



(b)



(c)



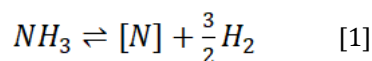
(d)

Fig- 5: Picture of Nozzle Seat/Sac area with (a) RP Oil (b) Cutting Fluid Stain (c) RP Oil Stain (d) No Contamination (No Cutting Fluid Stain/RP Oil Stain) taken with Dinolite Microscope at 20X Magnification.

3. RESULTS & DISCUSSION

3.1 Effect of Nitriding Time

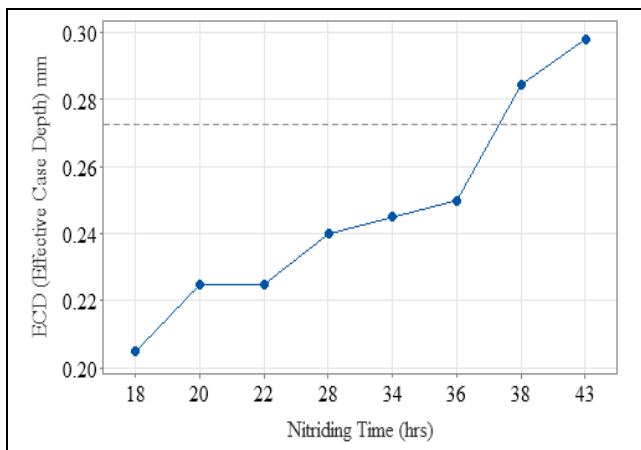
Gas nitriding operates on a theoretical basis involving a heterogeneous reaction between an ammonia (NH₃) gas atmosphere and the surface of steel at temperatures typically ranging between 500-580°C [12]. This process entails chemical reaction, as depicted in Equation 1. Ammonia is transported to the metal surface through molecular diffusion. NH₃ molecules then adsorb onto suitable surface sites and undergo sequential dissociation stages, ultimately producing atomic nitrogen and hydrogen. Nitrogen diffuses into the surface, where it dissolves interstitially and facilitates the development of iron nitrides. Meanwhile, hydrogen either forms molecular H₂ or reacts with oxygen to generate H₂O, and both substances desorb from the surface into the furnace atmosphere [12].



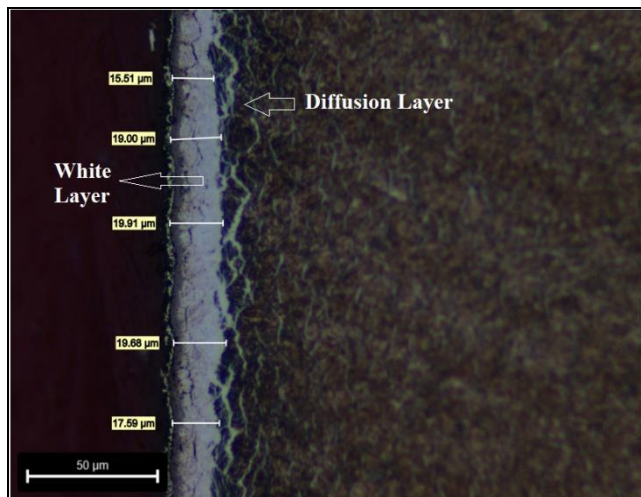
where [N] represents the N atom dissolved in the workpiece of the surface. The above equation presumes that equilibrium has been established. The activated atomic nitrogen dissolves into the iron crystal lattices, facilitating both the nitriding of α -Fe and the formation and growth of compound layers [5]. The growth of the compound (nitride) layer on the surface and the formation of the nitrogen diffusion zone beneath it are controlled by the atomic nitrogen diffusivity [5]. The thickness of these layers is influenced by several factors, including the composition of

the base metal, the duration of the nitriding process, and the temperature employed during the process [14]. As shown in Figure 6b, the compound layer, comprising two iron nitride phases, namely ϵ (Fe₃N) and γ' (Fe₄N), is commonly referred to as the "white layer" due to its persistent white appearance after etching with Nital. Directly beneath the compound layer lies the diffusion zone, characterized by nitrogen in solid solution and stable metal nitrides formed in the shape of needles by alloying elements such as aluminium, molybdenum, chromium, and tungsten. Etching can be employed to reveal these nitrides in the form of needles, making the diffusion zone visible. The thickness of this diffusion zone was measured using a microhardness tester. The effective case depth is a crucial parameter determined from the diffusion zone. It represents the distance inward from the surface of a part to a specific hardness level designated by the design engineer. To ascertain whether the intended hardness has penetrated to the correct depth, measurements are taken with a microhardness tester. This measurement is essential for ensuring that the desired hardness profile has been achieved at the specified depth, validating the integrity and performance of the treated part.

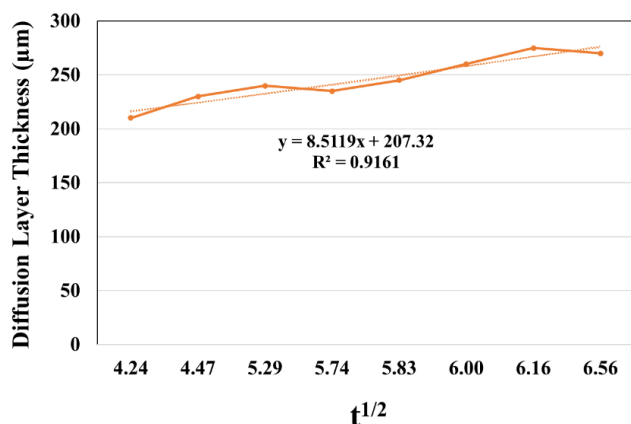
In order to achieve specified ECD (0.20 mm minimum), multiple batches of injector nozzles were produced with different nitriding times (18-43 hrs). The components from each batch were then subjected to various quality tests to assess the impact of nitriding time on the properties of the nitride layer. Data from the DOE trial, including ECD measurements were collected and analyzed statistically. Statistical analysis techniques were employed to identify trends and correlations between nitriding time and ECD. The lowest ECD was reported for nitriding time-18 hrs which was 0.22 mm, on the other hand, the highest ECD was reported for nitriding time-43 hrs which was 0.30 mm. It is observed that, with an increase in nitriding time, ECD also increases. Based on the findings from the DOE trial, recommendations were made regarding the ideal nitriding time i.e., 43 hrs to achieve the desired nitride layer quality. Figure 6 (a). illustrates the effect of nitriding time on the effective case depth of the nozzle. The microstructure of the nozzle nitrided for 43 hrs has developed a very good case & uniform white layer as shown in figure 6 (b).



(a)



(b)



(c)

Fig-6: (a) Effect of Nitriding Time on Effective Case Depth (ECD) of Nozzle (b) Microstructure of well-developed nitriding layer comprising of White layer & Diffusion Layer of Nozzle Nitrided for 43 hrs at 500X (c) Diffusion Layer thickness as a function of the square root of the nitriding time.

Keeping in mind that nitriding is a diffusion-controlled process, the time dependence on the layer thickness (d) in the gas nitriding is described thermodynamically by an equation relating the nitriding thickness to the process parameters (time and temperature in the case of gas nitriding). For gas nitriding, this equation is typically parabolic and can be expressed as follows [13].

$$d^2 = D_n t \quad [2]$$

where d - thickness of the nitrided layer,

t - Nitriding time

D_N - effective diffusion coefficient of nitrogen, which, considering an arrhenius-type behaviour is calculated according to

$$D_N = D_0 \exp(-E_A/RT) \quad [3]$$

where T is the nitriding temperature (in Kelvin),

R - Universal gas constant,

D_0 - Pre-exponential or frequency factor

E_A - Apparent activation energy of the nitriding process

This theoretical approach is applied ahead, and compared with the experimental results obtained in the present work (see Figure. 6 (a)). The evolution of the diffusion layer thickness as a function of the square root of nitriding time is presented in Figure. 6(c) is strong evidence that the gas nitriding is a diffusion-controlled process. The linear relationship observed in Fig. 6c, data was also mathematically treated to estimate how close it is to the fitted regression line. The equations of the fitted straight lines and the coefficient of determination (R^2) for the obtained data can be seen in Equation.(4).

$$y = 8.5119x + 207.32 \quad [4]$$

$$R^2 = 0.9161$$

The fit of the linear relationships was quite good, since $R^2 \approx 1$.

On the other hand, compressive residual stress analysis was performed on samples placed at 9-locations of furnace and showed that sufficient amount of compressive residual stresses are developed in the diffusion zone to sustain better fatigue life during the application. Table.2 depicts residual stress analysis data.

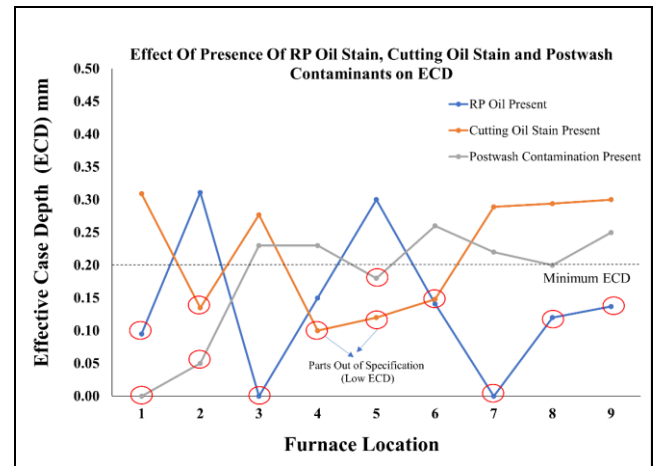
Table -2: Residual compressive stress data of 9 Pole samples subjected to Gas Nitriding.

Sample ID: Nozzle-Bottom Side (Process: VHT + GN)			
Sr. No.	Sample No.	Depth in microns	Normal residual stress in MPa
1	Sample - 1	50 microns	-235 ± 20
2	Sample - 2		-243 ± 9
3	Sample - 3		-193 ± 19
4	Sample - 4		-387 ± 11
5	Sample - 5		-217 ± 12
6	Sample - 6		-230 ± 19
7	Sample - 7		-259 ± 22
8	Sample - 8		-281 ± 22
9	Sample - 9		-262 ± 24

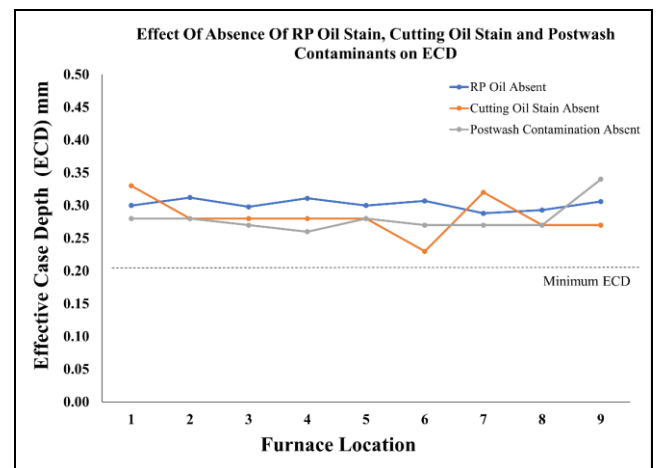
Results confirm that the layer growth is strongly dependent on the nitriding time, being this layer relatively thicker for a longer nitriding time. The experimental data obtained from the nitriding of nozzles, ranging in time from 18 hours to 43 hours, reveals a clear trend: an increase in nitriding time corresponds to an increase in case depth. This observation aligns with the kinetics of the gas nitriding process, where the growth of the nitriding layer thickness follows a parabolic law, as articulated in Equation (2). It is understood that gas nitriding kinetics are characterized by a slow progression, necessitating extended furnace residence times depending upon case depth requirement. Some studies have also shown that for nitriding the gears for wind power plants using the zero flow method (from steels 42CrMo4, 31CrMoV9, 34CrNiMo6) with requirement of case depth layer (0.6 mm-0.8 mm) requires almost 60-80 hrs of nitriding time, whereas plates for casting glass bulbs are made from N135M steel with requirement of case depth layer as 0.20-0.25 mm requires 26 hrs to obtain sufficient case depth [11]. In the realm of gas nitriding, this law postulates that the thickness of the nitriding layer increases proportionally with the square root of the nitriding time in gas nitriding processes' either be it zero flow technology or vacuum technology. This alignment offers valuable insights into the kinetics of gas nitriding, deepening our comprehension of how variations in nitriding time influence the resultant case depth in nozzles.

3.2 Effect of Surface Contaminants on Effective Case Depth:

As shown in Figure 7 (a), parts having the presence of RP Oil, Cutting Oil Stains, Post washing contamination (RP oil stain/cutting oil stain) in the seat/sac area have significantly impacted ECD when subjected to heat treatment, in both locations of nozzle i.e. M (Sac) & U (Seat) location. These parts showed low ECD ranging between 0.00-0.15 mm which is below the required level of specification. On the other hand, Parts with No contamination showed sufficient development of case depth in the range of 0.27 to 0.30 mm as depicted in Figure 7 (b).



(a)



(b)

Fig-7: (a) Effect of Presence of Surface Contaminants on Effective Case Depth of Nozzle at Location U (Seat Location) (b) Effect of Absence of Surface Contaminants on Effective Case Depth of Nozzle at Location U (Seat Location).

In the current study, designed experiments can be used to systematically investigate the process variables that affect product quality. The provided data from DOE was analyzed using statistical tools to examine the correlation between RP oil, cutting oil stain, and post washing contaminants with effective case depth. Minitab software was employed for statistical analysis, utilizing the DOE Study tool. The study involved a full factorial analysis considering three factors. The results of the statistical analysis are depicted in pareto chart figure 8. The pareto chart is a useful tool for identifying the magnitude and significance of various effects. On the pareto chart, bars that cross the reference line are statistically significant. In figure 8, bars representing factors A, B, C, AC, and ABC cross the reference line set at 2.120. This indicates that these factors are statistically significant in influencing the ECD. Additionally, their P values

being below 0.05 further confirm their statistical significance, underlining their impact on ECD. On the other hand, the interaction between BC & AB i.e. cutting oil stain with post washing contamination & RP oil with cutting oil stain is non-significant.

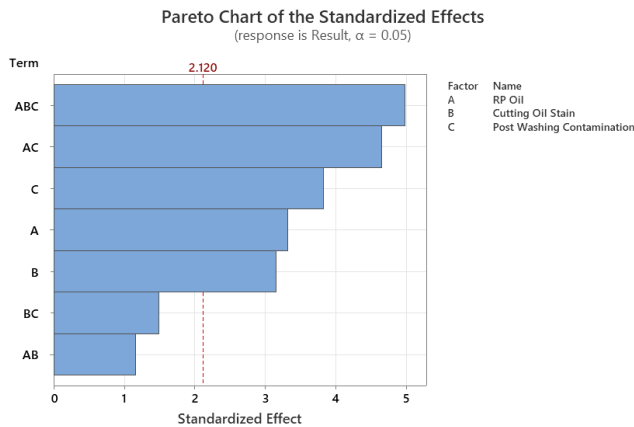


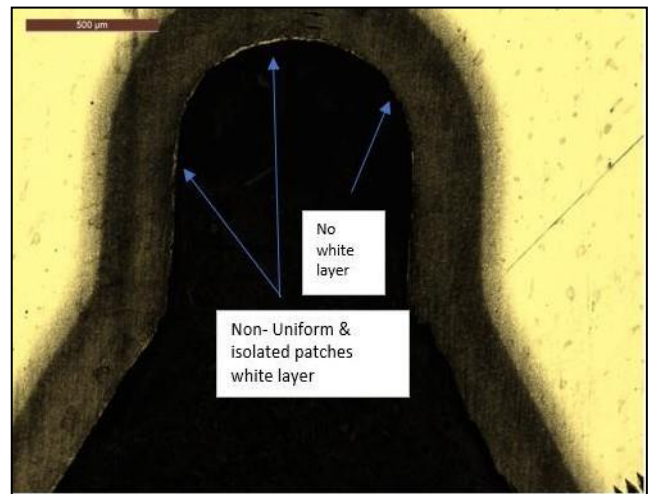
Fig-8: Pareto Chart of the Standardized Effects.

Table -3: P value of Each Factor

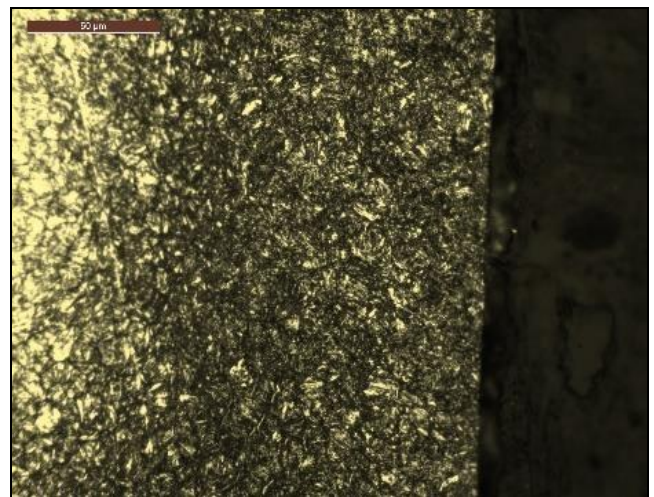
Term	P-Value	VIF
Constant	0.000	
RP Oil	0.004	1.00
Cutting Oil Stain	0.006	1.00
Post Washing Contamination	0.001	1.00
RP Oil*Cutting Oil Stain	0.262	1.00
RP Oil*Post Washing Contamination	0.000	1.00
Cutting Oil Stain*Post Washing Contamination	0.154	1.00
RP Oil*Cutting Oil Stain*Post Washing Contamination	0.000	1.00

The comparative microstructure analysis of nozzles having no stain and stain was carried out. The case depth structure of parts having RP stains with Low ECD (0.15 mm ~Below 0.20 mm) is shown in Figure 9 (a) & (b). Part shows non-uniform & isolated patches of nitriding layer consisting of white layer and diffusion layer in Seat (U) & Sac (M) area. However, The case depth structure of the part having no stain with moderate ECD of 0.27 mm is shown in Figure 9 (c) & (d). Part shows a uniform nitriding layer consisting of a white layer and a diffusion layer.

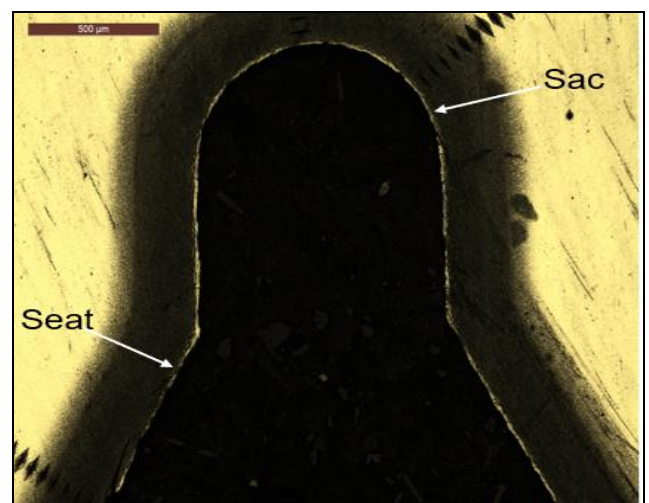
The above observation was also concluded with SEM-EDS analysis. Nozzle having RP Oil stain subjected to heat treatment was measured for effective case depth analysis. Figure 9 (d). depicts a cross-sectional view of this nozzle, having an effective case depth of 0.05 mm at the M location and 0.26 mm at the OD location. EDS traces were taken on the case depth region of the nozzle at the M & OD location. It was observed that at M Location there is a presence of Oxygen (7.20%) with no traces of nitrogen, as shown in Table. 4 (a). However, at OD location, the case is vice versa wherein the presence of nitrogen is detected with no traces of oxygen, as shown in Table.4 (b).



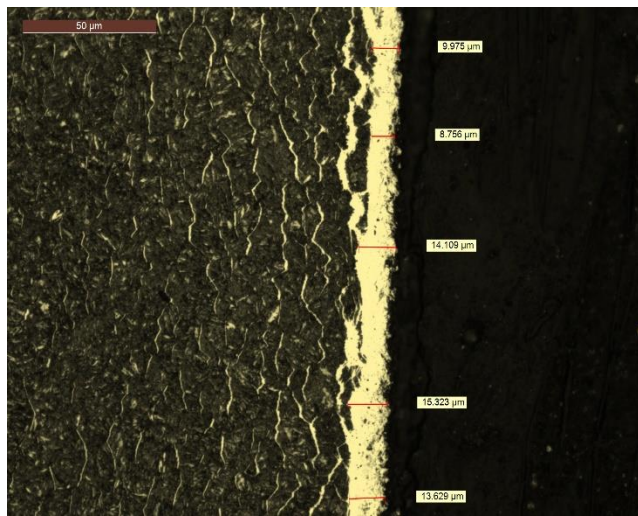
(a)



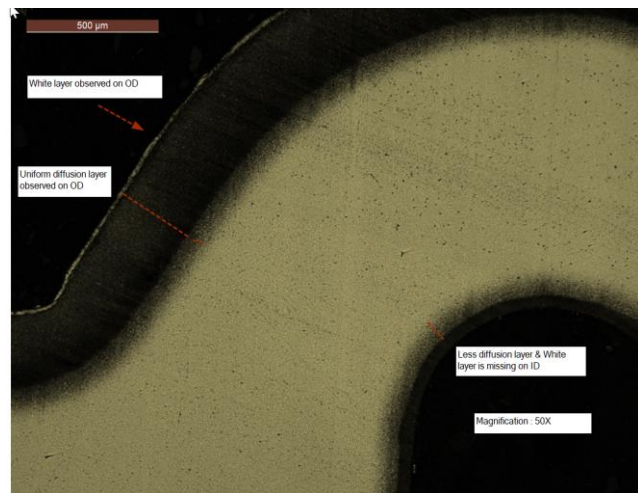
(b)



(c)



(d)

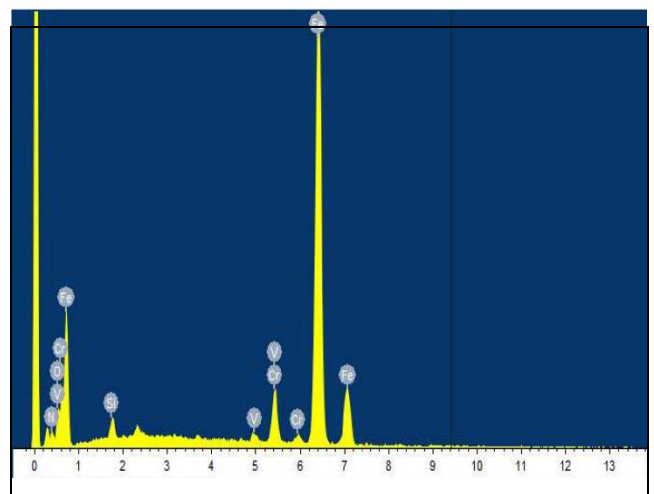


(e)

Fig-9: (a) Cross-sectional view of nozzle having RP Oil/Cutting Fluid stains with low ECD (0.15 mm ~Below 0.20 mm) showing non-uniform & isolated patches of white layer (0-11 micron) in nozzles at 50X magnification (b) Microstructure of nozzle having RP Oil/Cutting fluid stains with Low ECD (0.15 mm ~Below 0.20 mm) showing non-uniform & isolated patches of white layer (0-11 micron) in nozzles at 500X magnification (c) Cross-sectional view of nozzle having no RP Oil/Cutting fluid stains with moderate ECD 0.27 mm at 50X Magnification (d) Microstructure of nozzle having no RP Oil/Cutting Fluid Stains with moderate ECD 0.27 mm showing uniform white layer (10-12 Micron) & diffusion layer at 500X magnification (e) Cross-sectional view of nozzle having effective case depth as 0.05 mm at M location and 0.26 mm at OD location taken for SEM-EDS analysis at 50X magnification.

Table -4: SEM-EDS trace of nozzle (a) At location M showing the presence of oxygen indicating the presence of RP Oil Stain (b) At Location OD showing No Presence of Oxygen.

Elements	Weight%
O	7.20
Si	1.79
V	0.79
Cr	7.09
Fe	83.13
N	6.29
Si	1.24
V	0.98
Cr	6.07
Fe	85.92



(a)

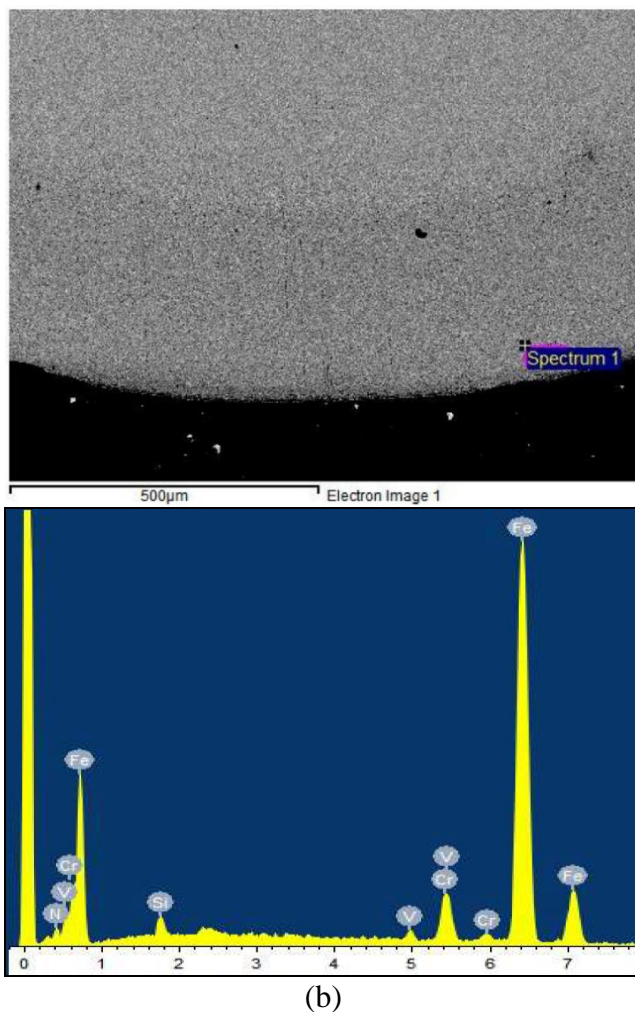


Fig-10: (a) SEM-EDS graph of nozzle at location M (b) SEM-EDS graph of nozzle at location OD (Outer Diameter).

A lot of work has been carried out to understand the effect of gas nitriding process parameters on nitriding properties in various steel grades [16][17]. Indeed, while the nitriding process has been extensively studied, there remain gaps in understanding, particularly regarding the effects of surface structure, composition, and contaminants on nitriding properties. The efficiency of the process is contingent on various factors, including the material and alloy composition, the surface shape and condition, and the pretreatment of the workpiece [12][15]. Surface contaminants, such as remnants from machining, cooling lubricants, and residual protective oils like RP oil used in prior manufacturing steps, can persist on the workpiece. These contaminants pose a significant challenge to the nitriding process at multiple levels. Some of the studies indicated a strong influence of surface contamination on the rate of nitrogen acceptance [12][15]. It is observed that non-volatile contaminants and reaction layers such as phosphates, silicates, and sulfides are stable and hinder gas nitriding during the whole process period; it is therefore necessary to remove these from the surface before Nitriding [12]. The

results also indicated a strong negative influence of machining cutting oil over the nitriding properties, i.e. nitrogen adsorption, leading to a heterogeneous aspect of the parts and a lowering of mechanical properties [12]. If surface contaminants are volatile such as tenside desorb from the surface during gas nitriding [12]. Lightly bonded contamination layers can easily be removed by liquid cleaning processes. Strongly bonded reaction and adsorption layers cannot unfortunately be reduced by conventional cleaning, and so when these are formed, other methods must be found [12]. This theory correlates with the current study wherein parts having the presence of RP Oil or RP Stains (Grade-Daubert 307HF) and cutting fluid stains (Grade-Ecocut HSG 211 HD), which are of non-volatile grades act as a formidable barrier to nitrogen diffusion. Some studies have proved that such a contamination layer can be removed by in situ pre-treatment (surface activation) of the surface before nitriding, enabling it to reach the reference nitriding properties, even for samples that are highly contaminated [15].

As a result, based on microstructure, SEM/EDS, microhardness & statistical analysis it is evident that the diffusion of nitrogen has been affected by the presence of surface contaminants. The presence of surface contaminants acts as a barrier for nitrogen diffusion, impeding the process of forming a robust nitriding layer and, consequently, limiting the attainment of an adequate case depth. Conversely, parts free from any surface contaminants demonstrate a positive outcome, showcasing a well-developed case depth. In summary, the presence of certain non-volatile surface contaminants can significantly impact the gas nitriding process, emphasizing the importance of maintaining a clean surface for optimal nitriding properties and achieving the desired case depth.

4. CONCLUSION

1) The duration for which components are exposed to the nitriding atmosphere is a crucial factor in the nitriding process. Nitriding time directly affects the ECD, representing the depth to which nitrogen atoms have diffused into the material. A longer nitriding time is associated with an increased ECD, leading to enhanced surface hardness and improved wear resistance. This correlation highlights the significance of optimizing nitriding parameters, including time, to achieve the desired material properties and performance outcomes in the treated components.

2) Surface contaminants, such as RP oil stains and cutting fluid stains, especially when they are non-volatile, can create a barrier that interferes with the smooth and uniform diffusion of nitrogen into the material. The consequence is uneven nitriding, potentially leading to variations in the ECD across the component's surface. Therefore, thorough cleaning of the part's surface or ensuring it is free from any contamination is crucial before subjecting it to gas nitriding. This emphasis on cleanliness is essential for achieving

consistent and desired nitriding outcomes, including uniform ECD and optimal material properties.

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REFERENCES

1. Abhishek Balasaheb Shinde, Omkar Anil Umadi, Shivaji V Gawali, Arun Kamble, International Research Journal of Engineering and Technology (IRJET), 7, 3095-3102 (2020)
2. Min-Seop Kim, Ugochukwu Ejike Akpudo, Jang-Wook Hur., Journal of the Korean Society of Manufacturing Process Engineers, 10, 1-16 (2021)
3. Amir H. Shamdani, Amir H. Shamekhi, M. Z. Basharhagh, S. Aghanajafi, Modeling and Simulation of a Diesel Engine Common Rail Injector in Matlab/Simulink, ("4th Annual (Mechanical Engineering Conference, Isfahan University of Technology 2006) p. 1-7.
4. Şeyda Polat, Ş. Hakan Atapek, Fatih Gümüş, GAS NITRIDING OF A HOT WORK TOOL STEEL AND ITS CHARACTERIZATION, (International Iron & Steel Symposium, Karabük, Turkey, 2012) p 257-263.
5. E. J. Mittemeijer, M. A. J. Somers, Journal of Surface Engineering, 13, 483-497 (1997)
6. N. Sylaa, F. Aliaja, B. Dalipi, ACTA PHYSICA POLONICA A, 130, 83-86 (2016)
7. O. Irretier, C. Naber, J. Dong, H. Klumper-Westkamp, B. Haase, K. Bauckhage, Surface Engineering, 12, 331-334 (1996)
8. K. Genel, Mehmet Demirkol, Surface & Coating Technology, 195, 116-120 (2005)
9. B. Guillot, S. Jegou, L. Barrallier, Surface & Coating Technology, 316, 59-70 (2017)
10. Tomasz Przygoński, ZEROFLOW@GAS NITRIDING IS A MODERN TECHNOLOGY THAT MINIMIZES THE CONSUMPTION OF PROCESS MEDIA AND REDUCES EMISSIONS OF POST-PROCESS GASES.
11. S. S. Akhtar, A. F. M. Arif, Bekir Sami Yilbas, International Journal of Advance Manufacturing Technology, 47, 687-698 (2009)
12. Syed Sohail Akhtar, Abul Fazal M. Arif, Bekir Sami Yilbas, International Journal of Advance Manufacturing Technology, 58, 57-70 (2011)
13. Maciej Korecki, Michal Bazel, Michal Sut, SECO/WARWICK S.A.; Piotr Kula, Emilia Wolowiec, LPC and LPN of Tool Steel Fuel-Injection Nozzles, (Lodz University of Technology Poland, 2014), p 39-42.
14. KOWALSKA, J., MAŁDZIŃSKI, L., Combustion Engines, 167, 3-7 (2016)
15. Kowalska, J., & Małdziński, L., ZeroFlow - new, environmentally friendly method of controlled gas nitriding used for selected car parts, (OP Conference Series: Materials Science and Engineering Poland 2016), p 1-10.
16. Qiang, L., & Sugiyama, T. "Heat Treatment Method for Fuel Injection Nozzle of Engine Fuel System", Accessed 11 Feb 2020.
17. Małdziński, L., Tacikowski, J., Thermochemical Surface Engineering of Steels, 459-483 (2016)

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