International Journal of Science, Technology and Humanities 1 (2014) 93-98
Available online at www.svmcugi.com
International Journal of Science, Technology and Humanities
DOI: 10.26524/ijsth18

Plasma (Ion) Nitriding of Low Alloy Steel (EN19 grade) and Investigation of Its Physico-Mechanical Properties

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Received: 16 September 2014: Received in revised form 16 October 2014; Accepted 25 October 2014

Abstract

The Plasma (Ion) Nitriding technology shows a clear orientation towards future developments. Hence, it ideally satisfies current and future industrial demands for economical and efficient solutions to the treatment of surfaces. It is also an answer to social demands for improved environmental protection. This technology uses Plasma as a gaseous charged particles (electrons, ions etc.,) as well as electrically neutral atoms and molecules the plasma envelopes the work pieces to be treated, which are set up, electrically insulated in a vacuum vessel. The electrically activated plasma and the influence of pressure and temperature now induce a thermo-chemically controlled change on the surface of the work pieces. The pulse plasma technique can be applied to the hardening, coating or etching of surfaces. The process is precisely controlled and easily adapted to many fields of application by using different gases and elements. Precision control is guaranteed by plasma activation in pulse - like intervals. The destructive effects of electric arcs to the treated surfaces are reliably avoided. The distinctive features of this technique are its highly efficient use of energy and gas as well as the omission of chemical baths, substances and waste products. In the present study, we attempt to develop a novel correlation between the micro-structural features of the sample materials with their macro level Physico-Mechanical properties. i.e., we are interested to study the physico mechanical behaviors of EN 19 Steel at Liquid, Gas and Plasma Nitriding process. After Nitriding, the qualitative results are compared and conclude, which is the successive process for EN 19 steel, in timely as well as economically and also we interested to study the physico-mechanical characteristics of this EN19 steel.

Key words: Ion/Plasma Nitriding (PIN); Mechanical Properties of EN19; Metallurgical Aspects of Plasma (Ion) Nitriding; EN19; Nitrided steel.

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1.0 Introduction

Nitriding is mainly used for ferrous components such as valves, camshafts and piston rods in the mechanical engineering and automotive industries. Other applications are cutting tools or large forming dies. Cast iron parts, such as pump and gear houses, can also be nitrided. Nitriding is one of the processes of surface hardening of steel in which Nitrogen is allowed to diffuse into the surface layer by heating the metal in contact with the Nitrogenous medium in the temperature range of 3500^{o}C to 6000^{o}C. This process can effectively give high surface hardness, wear resistance along with anti galling properties, improved fatigue resistance, better creep resistance and enhanced corrosion resistance (except in the case of stainless steels).

Nitriding is carried out at temperature much less than the carburizing and hardening temperatures (below the lower critical temperature) produces negligible distortion in the component (1).

During the process of Nitriding the Nitrogen content reaches a value about 5.5% up to a depth of 16 – 50 µm (Microns) and then gradually decreases with distance from surface this surface layer containing high N_2 content results in the formation of and' – Nitrides in the interior contains a dispersion of '– Nitride in – phase. These two layers are called the compound layer and diffusion layer respectively. The Nitriding process using Liquid Nitriding or gas was developed in the early 20th century in Germany and the United States. The development of ion or plasma Nitriding started in the 1930s but was not commercially used until the 1970s. All three Nitriding methods have advantages and the selection of a particular method depends on the specific application of the nitrided component (2).

Ion Nitriding is special form of Gas Nitriding in which a D.C voltage is supplied between the steel to be case hardened (Cathode) and the furnace chamber as (Anode) The furnace first evacuated and then filled with nitrogen and hydrogen to a pressure of 250 – 600 pa. When voltage is applied the current is small at lower potentials but glow discharge occurs at higher voltages when the whole cathode emits light and the current increases. The power required for speeding up the nitrogen and hydrogen ions to the cathode during this abnormal glow discharge period depends on the load, area, pressure of gas mixture. The bombardment of the Ions on the cathode increases the temperature of the specimen. Due to the heat generated in the steel specimen (Cathode) and due to the bombardment of the ions, it gets heated to 375^{O}C to 550^{O}C and at this temperature; nitrogen diffuses rapidly in steel while hydrogen keeps the surface oxide free. The Plasma Nitriding gives a very fine surface finish, almost free of pores, that lends itself for a high polish. Due to this low porosity plasma nitrided cam- and crank shafts are for instance used for high performance motors. As plasma nitriding allows a large variety of nitride layers, its fields of application are also varied. Examples are the surface treatment of forming dies such as large plastic extrusion dies and auto body blanking dies, tools for stainless steel deep drawing, casting, hot forgoing and extrusion dies. Other specific applications are corrosion resistant engine valves, high speed steel cutting tools and many applications in mechanical engineering (3).

In general any steel can be nitrided but the surface hardness developed is not high in plain Carbon steels in spite of the structural changes during Nitriding. Hence, in general, steels used for Nitriding are medium “Carbon Alloy” steels which acquire high hardness and wear resistance. The high hardness can be attributed to the formation of alloy nitrides. Nitrides of Al, Cr, Mo, Ti and V have increased hardeness. The hardness developed in case of steels containing about 1.0% Al, can be as high as 1200 HV. Such steels are called Nitralloys. But excessive presence of alloying elements may lead to a decrease in case depth and hence a judicious use of alloying is made to obtain optimum case depth and good surface hardness. Some of the steels used for Nitriding are CK 45, EN 19, EN 41B,SAE 4140, AMS 6470, SAE 4340, AISI 420 stainless steel; Hot work Die steels and High speed Tool Steels. Even maraging steels are successfully nitrided.

In the present study, we attempt to develop a novel correlation between the micro-structural features of the sample materials with their macro level Physico-Mechanical properties i.e, we are interested to study the physico mechanical behaviors of low alloy steel i.e. EN 19 Steel at Liquid, Gas and Plasma Nitriding process. After Nitriding, the obtained qualitative results are compared and conclude, which is the Successive process for EN 19 steels, in timely as well as economically. Because, the EN 19 is a special and Important low alloy Steel which is mainly used for Space, Automotives and Railways, components such as Drive Shafts, Crank Shaft, Connecting Rod, High Tension Bolts, Studs, Axles, Propeller Shaft Joints, Rifle Barrels, Breech Mechanism for small Arms, ect.

2.0 Experimental Methodology

Before Nitriding the chemical composition (grade confirmations) of chosen steel material EN 19 was studied by using wet chemistry method (4, 5,6,7,8 & 9) and the percentage of assigning elements are shown in the Table 2.1

Table: 2.1 *Chemistry of EN 19 Grade Steel*

EN19 25mm	*(%) Percentage of Assign. Elements*						
	C	*Mn*	*Si*	*Cr*	*Mo*	*P*	*S*
Spec. min.max	*0.35 0.45*	*0.5 0.8*	*0.10 0.35*	*0.9 1.5*	*0.20 0.40*	*0.05 max.*	*0.05 max.*
Obtained	*0.41*	*0.68*	*0.28*	*0.95*	*0.24*	*0.03*	*0.027*

2.1. Liquid Nitriding Process

Liquid Nitriding is carried out in electrically heated crucible furnaces. After preheating to 350°C, the components are submerged into the saltbath, either hanging in charging racks. The salt bath consists of alkaline cyanate and alkaline carbonate. Through oxidation and thermal reaction with the immersed component surface, at nitriding temperature the alkaline cyanate releases nitrogen and carbon which diffuse into the surface of the component. Pure nitriding is not possible with the salt bath as small amounts of carbon will always diffuse into the surface. The usual process parameters are 90 min at 580°C.

The active nitrogen releasing agent of the salt bath is the alkaline cyanate. Through the reaction of the cyanate ions the amount of alkaline carbonate in the bath increases. By adding an organic polymer the optimal cyanate content of the bath is replenished again. After Liquid Nitriding, quenching in an oxidizing salt bath (380-420°C) produces a black iron oxide (Fe_2O_4) on the surface. It fills the pores of the compound layer and acts as additional corrosion resistant protection. After cooling to room temperature the components can be polished and then re-oxidised depending on the application (10, 11).

2.2 Gas Nitriding Process

Gas Nitriding is takes place in a sealed, bell-type Nitriding furnace which provides good gas circulation. The process is mainly controlled by the degree of dissociation of ammonia. The ammonia gas reacts at 500-520°C with the steel surface and decomposes, thereby releasing nascent nitrogen which diffuses into the steel surface. As gas Nitriding uses a lower temperature, process times are 40- 80 hrs. The formation and properties of the compound layer and diffusion zone are similar to those produced by salt bath Nitriding. However, the thickness of the compound zone can be more accurately controlled or even completely suppressed with gas Nitriding (10, 11).

2.3 Plasma Nitriding

Ion or Plasma Nitriding is carried out in nitrogen - hydrogen atmosphere at 400-600°C and a pressure of approx. 50-500 Pa. The plasma is produced in a vacuum chamber with a high voltage whereby the work-piece acts as cathode and the vacuum vessel as anode. Because nitrogen and hydrogen are brought into the vacuum chamber as individual gases, the ratio of nitrogen to hydrogen can be controlled allowing variations of thickness and composition of the compound layer, consequently (10, 11).

2.4 Mechanical & Metallurgical Testing

Surface Hardness of the nitrided Component i.e. EN 19 samples were studied by Rockwell Hardness Tester (HRC-Scale, Make: FIE, Model: RAB 250, Load 150Kg) & Micro Vickers Hardness Tester (HV0.2 Scale, Make: Mitotoyo, Model: MVK E3). Metallographic study is the imaging of topographical or micro structural features on prepared surfaces of materials (12, 13). The structures studied by metallography are indicative of the properties and performance of materials studied. As the evaluation of the Nitriding diffusion is carried out with 100 X magnifications (14) by Matascope make Metallurgical Microscope (Model T1600).

3.0 Results and Discussion

3.1 Results of Liquid Nitrided Sample

From the Mechanical behaviours of liquid nitrided samples (Table 3.1), it is observed that the surface hardness of the sample EN19 sample with hardened & tempered conditions is 620 HV200 and Core hardness is 358HV200 obtained but the surface hardness in without hardened & tempered conditions is 686 HV200 and the Core hardness 264HV200 achieved. From the Microstructure examinations (Figures 3.1 & 3.2), it is noted that the depth of case formation of the sample EN 19 with hardened & tempered conditions is 0.35 mm achieved and also white layer is not formed but case depth in without hardened & tempered conditions is 0.3 mm and obtaining white layer is 5 microns.

3.2 Results of Gas Nitrided Sample

From the Mechanical test of Gas Nitrided samples (Table 3.2), it is observed that the surface hardness of the similar EN19 sample with hardened & tempered conditions is 466HV200 and Core hardness is 337HV200 obtained but the surface hardness in without hardened & tempered conditions is 552HV200 and the Core hardness 286 HV200 achieved and the reference of the Microstructure examinations (Figures 3.3 & 3.4), the case depth of EN19 sample with hardened & tempered conditions is 0.3 mm achieved and also white layer is not formed but case depth in without hardened & tempered conditions is 0.2 mm.

3.3 Results of Plasma (Ion) Nitrided Sample

From the Mechanical test of Plasma Nitrided samples, the surface hardness of EN19 sample with hardened & tempered conditions is 714HV200 and Core hardness is 275HV200 obtained but the surface hardness in without hardened & tempered conditions is 737 HV200 and the Core hardness 289HV200 achieved. Similarly the case depth of EN19 sample with hardened & tempered conditions is 0.3 mm achieved and also white layer is not formed but case depth at without hardened & tempered conditions is 0.15 mm observed and there is no wite layer found (Figures 3.3 & 3.4).

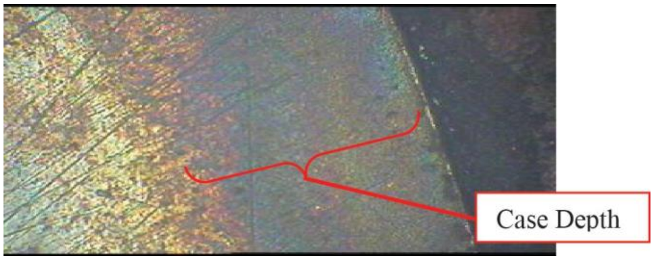

Fig 3.1 *Liquid Nitrided- EN 19 @ 100X with Hardened & Tempered Conditions.*

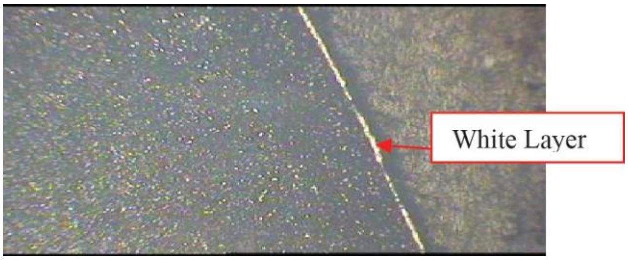

Fig 3.2 *Liquid Nitrided- EN 19 @ 100X without Hardened & Tempered Conditions.*

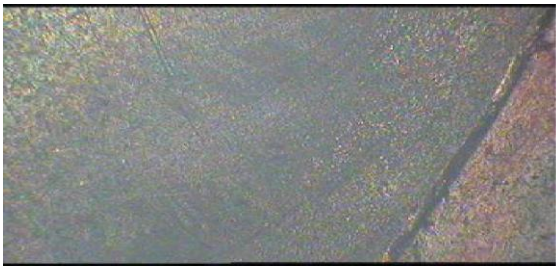

Fig 3.3 *Gas Nitrided - EN 19 @ 100X with Hardened & Tempered Conditions.*

Fig 3.4 *Gas Nitrided - EN 19 @ 100X without Hardened & Tempered Conditions.*

Fig 3.5 *Plasma (Ion) Nitrided - EN 19 @ 100X with Hardened & Tempered Conditions.*

Fig 3.6 *Plasma (Ion) Nitrided - EN 19 @ 100X without Hardened & Tempered Cond.*

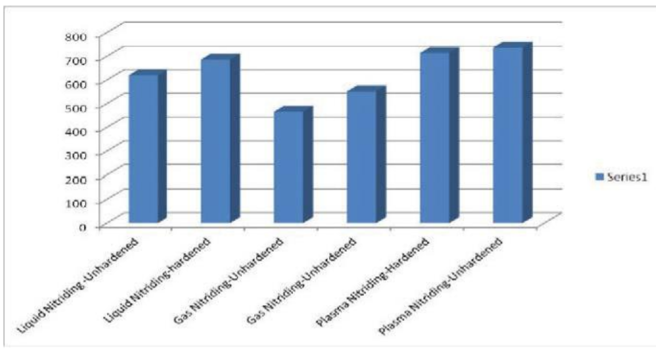

Fig 3.7 *Nitriding Process Vs Obtained Hardness*

Table: 3.1 *Obtained Mechanical Test Results*

Part Name	Nitriding Process	Hardness Value in HV200g		Wight layer (mm)	Case Depth (mm)
		Surfaces	Core		
EN19 With Hardening& Tempering	Liquid Nitriding	620	358	Nil	0.35
EN19 Without Hardened & Tempered	Liquid Nitriding	686	264	5	0.3
EN19 With Hardened & Tempered	Gas Nitriding	466	337	Nil	0.3
EN19 Without Hardening & Tempered	Gas Nitriding	552	286	Nil	0.2
EN19 With Hardening& Tempering	Plasma Nitriding	714	275	Nil	0.3
EN19 Without Hardened & Tempered	Plasma Nitriding	737	289	Nil	0.15

4.0 Sammary & Conclusions

From the overall study, we conclude that the that the Plasma (Ion) Nitrided samples are shortly achieved very good mechanical & metallurgical properties and the observed Physico-mechanical properties are authentically gives, Plasma (Ion) Nitriding is the best and timely, economically as well as pollution free technology in the latest trends of surface diffusion engineering comparatively other Nitriding Process i.e. Gas and Liquid Nitriding for the chosen Sample EN19 grade steel. The obtained surface, Core hardness values and Micro structural features of Plasma Nitrided low alloy steel (grade EN19) is found satisfactory with their macro properties.

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