Surface characterization of Silicon Nitride compound deposited on Plasma Nitrided austentic steel grade 316

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By

Omdutt choudhary

(11309186)

Under the guidance of

Mr. Mukhtiar Singh

(16431)

DEPARTMENT OF MECHANICAL ENGINEERING

LOVELY PROFESSIONAL UNIVERSITY

PUNJAB

CERTIFICATE

I hereby certify that the work being presented in the dissertation entitled "Surface Characteristics of Silicon Nitride Compounds Deposited on Plasma Nitrided Austenitic Stainless Steels 316L in partial fulfillment of the requirement of the award of the Degree of master of technology and submitted to the Department of Mechanical Engineering of Lovely Professional University, Phagwara, is an authentic record of my own work carried out under the supervision of (Name Of Supervisor, Designation) Department of Mechanical Engineering, Lovely Professional University. The matter embodied in this dissertation has not been submitted in part or full to any other University or Institute for the award of any degree.

(Date)

Omdutt Choudhary
(11309186).

This is to certify that the above statement made by the candidate is correct to the best of my knowledge.

Mr.Mukhtiar Singh
(16431)

Mr. Jasvinder singh
HOD (ME)

The external viva-voce examination of the student was held on successfully

Signature of Examiner

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Chapter 1 Introduction

Austenitic stainless steels have good properties in metallurgical angle. These materials also not lose their strength at raised temperatures as fast as ferritic (bcc, body-centered cubic) iron base alloys. Iron and most of its alloy are poor in corrosion resistance when they are into rust environment. To improve this properties in iron and its alloy we add chromium or nickel with it that called stainless steel. Stainless steels are derived by the addition of chromium and nickel with the iron and its alloy.

Stainless steel normally use as a primary raw product in industry.

1.1.1 Stainless steel categories and grades

- Ferritic stainless steel

  The iron and its carbon added alloy material are added with chromium (11.2-19%) and with little amount of nickel.

  The Nickel amount of the ferrite grade makes them higher price stable compare to other because nickel is very expensive material. Sometime molybdenum is added to enhance corrosion resistance as well as niobium and titanium to improve weldability

- Martensitic stainless steel

  In these steel added more carbon content to improve its properties (hardness, strength) in compare to other steel materials. Sometime added nitrogen also added to increase hardness. These grades contain hardly or little amount of nickel but molybdenum added sometime.

  Sometime Sulphur also added to enhance the machinability. This material grades are magnetic and hardenable.

- Duplex stainless steel

  Duplex grades have ferrite-austenitic microstructure. These steel are combination of 50 % austenitic and 50% of ferrite steel with phase balancing. It contain many beneficial properties of ferrite and austenitic. This microstructure also contributes to high strength and high resistance to stress corrosion cracking. Ingredients for duplex are chromium (20.1-25.4%), nickel (1.4-7%). Molybdenum (0.3-4%) and nitrogen added sometimes to enhance corrosion resistance and balance microstructure. Manganese is alloyed in some grades as a replacement of nickel but also rise the capability of dissolving of nitrogen in the material.

- Austenitic stainless steel

  Austenitic stainless steel is largest class of stainless steel. These grades have well to corrosion resistance and good formability, weldability. Their excellent impact strength at low temperature is used in cryogenic application. By cold working with it we increase high strength and hardness.

  These steel are sub divided into (5) groups

  - Cr-Ni grades

    The Cr-Ni grades mostly added with chromium and nickel but no molybdenum. Titanium and niobium are added to enhance or rise the mechanical properties at high temperature. Sulphur is alloyed for enhance machinability and nitrogen for improve strength.

  - Cr-Mn grades

These are defined as "200 series" grades. The nickel content is decrease or replace to manganese and nitrogen to maintain the microstructure.

- Cr-Ni-Mo grades

  In Cr-Ni alloy stainless steel some amount of molybdenum (2-3%) is alloyed to increase hardness. It is also named as 'acid proof' stainless steel material. Some grades are made with nitrogen and sulphur to enhance its strength and machinability.

- High performance austenitics steel

  These steel grades contain higher alloying content. Characteristics for high performance steel are Chromium (17-25 %), nickel (14-26%), molybdenum (3-7%). Copper is added with some steel grades to increase resistance to certain acids.

- High temperature austenitic steel grades

  These steel are produced for use at working temperature above 550C. Characteristics for high temperature austenitic steel grades are chromium (17-25%), and nickel (8-20%) but no molybdenum added. Silicon is added for increasing oxidation resistance.

In this research experiment we select austenitic stainless steel 316 for improving its chemical and mechanical properties by some surface treatment technique.

Austenitic stainless steel 316

This steel grade is molybdenum-bearing stainless steel. The high nickel and chromium content enhance the corrosion resistance of this grade

Application

- Food making equipment
- Chemical equipment
- Laboratory apparatus
- value and pump assembly
- Heat exchangers assembly
- Textile industries
- Condensers of industries, evaporators and tanks in industries

Resistance properties

Due to the rise chromium and molybdenum content enhance the corrosion resistance. Good oxidation resilience in discontinuous service to 870C.

Good weldiability by all standard fusion method, both with or without filler metal. Annealing temperature maximum and minimum limits are 1038 to 1149°C.It cannot be hard with heat treatment. Best forging temperature between 2100ºF and 2300°F; do not forge below 1700ºF.

Chapter 2 Objective of Research

To enhance following surface properties of austenitic stainless steel 316 by silicon nitride compound deposition on nitrided stainless steel using PECVD technique.

(1) To enhance wear resistance.
(2) To improve surface hardness and strength
(3) To reduce friction coefficient

The composition and structure of the surface layers were characterized using XRD and SEM testing technique.

Surface hardening means treatment of steel for enhance the hardness of the outer surface while the core remains relatively soft. The combination of a hard surface and a soft interior is more valued in engineering because it unaffected with more stress and fatigue, that property is needed as gears and anti-friction bearings. Surface-hardened steel is also useful for its low cost and superior flexibility in manufacturing because hardened metal is usually more brittle.

Different surface hardening process –

(1) Heat Treatment

– Induction

– Flame

– Laser

– Light

– Electron beam

(2) Case Hardening

– Carburizing

– Carbonitriding

– Nitriding

Induction hardening

In induction hardening Induced eddy current used to heat the outer surface of the steel fast. The heating process is quickly followed by jets of water to quench the component. A hard outer surface layer is produced with a soft core. Slideways on lathe are made by induction hardened. The desired section of the component kept inside an induction coil. A.C. current moving through coil produce magnetic field around the component. Magnetic field produces eddy currents in the component. The eddy currents are predominantly generated near the surface by adjusting the frequency of the A.C. current, depth of heating can be adjusted. Heated part is then quenched in water or oil.

Flame hardening

Gas flames raise the temperature of the outer surface above the upper critical temperature and inside core material will heated by conduction process. Water jets are used for quenching the component. Heat is supplied to the part being hardened. The result is a hard surface layer limit from 0.050" to 0.250"mm deep. Core mechanical properties are unchanged because the composition of steel is not affected. Flame hardening creates results similar to other conventional hardening processes. Flame hardening has the ability to harden flat surfaces. Flat wear plates and knives can be selectively hardened using this process .Oxy acetylene gas is used for heat the material.

Laser hardening

Laser hardening is a surface hardening process. It is used only on ferrous materials suitable for hardening along with, steels and cast iron with a carbon content of higher than 0.2%.

Carburizing

Carbon diffuses into the steel at high temperature. The diffusion is take palace under certain condition that is helpful in process. The Depth of diffused layer of carbon is controlled by “time” and “temperature”. Methane (CH4) is used as carburizing gas. The carbon gas is produced by solid, liquid and gas medium.

Methane is mixed with a supporting gas to escape soot formation components. Desired carbon potential is maintained by adjusting the reaction equilibrium in furnace atmosphere

$$CO + H_2O = CO_2 + H_2$$

CO/CO_2 ratio adjusted to establish required carbon potential. H_2/H_2O ratio adjusted to maintain the atmosphere reducing for steel. Carbon content on the surface raised to 0.8 wt. % after carburizing .carburizing steels contain low carbon, typically 0.15 to 0.25 wt.%.

Nitriding

Nitriding is a process in which diffusion of nitrogen into the surface layers of low carbon steels at high temperature. The evolution of nitrides in the surface layer enhances mechanical properties of material surface.

Steel is exposed to nitrogenous atmosphere.

$$NH_3 = N + 3H \text{ (at 525-550°C)}$$

• Nitrogen reacts with various alloying elements in the steel (e.g. Al, Mo, Cr etc.) to form nitrides. The nitride layer formed on the surface is very hard.

Objective of Nitriding are:

- High Surface Hardness
- Improve Resistant Surface
- Enhance Wear Resistance
- Enhance Tensile Strength
- Enhance Corrosion Resistance

Process methods for Nitriding include:

- Gas Nitriding
- Liquid Nitriding
- Plasma Nitriding

Liquid nitriding

Liquid Nitriding (LN) is a thermo-chemical reaction process whereby nitrogen and carbon are diffused into the surface of iron-based materials. The nitrogen alloyed with the iron to form an iron-nitride mixed layer that gives enhanced surface properties. The nitrogen is produced by a liquid form.

Gas nitriding

Gas Nitriding is a process of surface enhancement of low carbon and low alloy steel materials. In gas nitriding process nitrogen is diffused into the surface layer of material in the form of gas. The nitrogen gas is supplied in the form of gas throughout the process. Basically we use ammonia as medium because of its rich nitrogen nature. When the ammonia comes in contact with the heated surface then it separate into nitrogen and hydrogen. The separated free nitrogen comes in contact with heated layer and diffused on it and creates the nitride layer. While processing we could control on the flow of the gasses medium and control the amount of nitrogen that react with surface that is an advantage of gas nitriding

Chemical vapour deposition

Chemical vapour deposition is a surface coating technique. In this process a solid material is deposited on a surface layer of material. These gases are enclosed into a chamber where gases are reacting with the heated surface and create the coating. In normal CVD process it produced very thin layer of coating.

PECVD (Plasma enhanced chemical vapor deposition)

PECVD reactant gases are passed through active glow discharge plasma.
This is most frequently used because of its ease of construction and good film.
Properties coverage .the voltage division between the plasma and the electrodes depends to first order on their relative sizes, the voltage is largest for the smaller electrode Plasma bombardment can have an important influence on the properties of the growing coatings

Chapter 4 Literature review

C.X Li, T. Bell (2003) Austenitic stainless steel has been plasma nitrided by plasma nitriding technique. The wear properties of untreated austenitic stainless steel 316 are improved by plasma nitriding. Stainless steel 316 having plasma nitriding at 500°C produce a thicker alloyed layer by precipitation of chromium nitride whilst this material at 420°C produce a precipitate free S phase layer . Less temperature plasma nitriding provide improved wear resistance.

Zhao Cheng, C.X Li, T. Bell (2004) plasma nitrocarburising is accomplish at low temperature to enhance surface hardness of austenitic stainless steel 316 without degradation corrosion resistance. It was studied that in previous research that nitrogen and carbon atom simultaneously be dissolved into austenite lattice during the nitrocarburising process. The nitrocarburised layer have high hardness then nitrided layer.

E.L.Dalibon, D.Heim (2015) Stainless steel 316L is used in chemical and other industries due to its excellent corrosion properties. Wear resistance of this material is not good. Plasma assisted process is used for improvement its tribological properties. These coating with high hydrogen content, low C-C bond could improve the 316L austenitic stainless steel tribological behavior.

Ahmad Reza Rastkar, Sephradad Akbari (2014) Silicon nitrided compound deposit on plasma enhanced chemical vapor deposition on plasma nitrided austenitic stainless steel 316L for enhance hardness and wear performance of steels. It is known that silicon nitrided compound have attractive properties (low friction, high hardness, low wear resistance).So the deposition process were accomplish at optimized gas composition and temperature to produce best mechanical properties and tribological attributes.

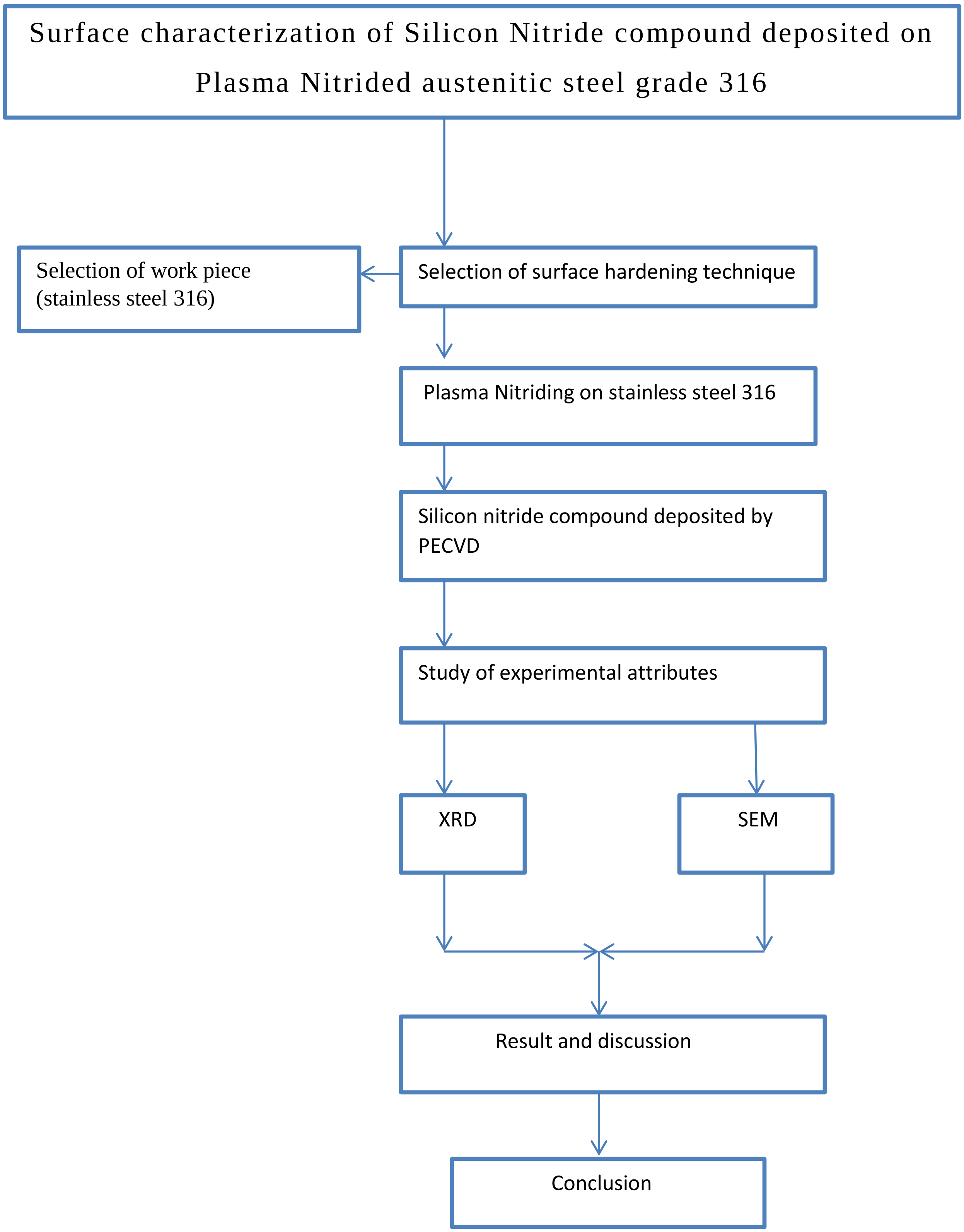
Nitriding enhance surface hardness and wear resistance of steel material. Thermo-chemical diffusion processes like nitriding and carburizing processes alter the surface layer of stainless steel. Plasma technology reduces gas and energy consumption.

Raden Soekrisno, Suyitno, Rini Dharmastiti (2015) The mechanical and chemical properties such Hardness, wear resistance and grain size enhanced with temperature variation in high temperature gas nitriding (HTGN).Corrosion resistance in ringer solution reduced with temperature. HTGN processes at 1000°C for 20 minute are able to enhance the mechanical properties of material and improve the nonmagnetic stability 316LVM.

Hassan R S Mohamoud, Patthi Bin Hussain (2016) there are many way to improve the properties of stainless steel 316L by the mean of thermochemical treatment. The thermochemical process of nitriding/carburizing of AISI 316L at 450°C for 8 and 24 hours demonstrate to produce a layer of expanded austenite phase without precipitation of chromium nitride/carbide. The 24 hour treatment provides higher chromium profile then the 8 hour treatment. The treated layer indicates that the chromium was more reactive in the longer process time.

David M Hoffman, Sri Prakash Rangrajan, Satish D.Athavale (1995) thin films of silicon and germanium nitride can be deposited by chemical vapor deposition. In these thermal processes, silicon nitride films have been produce at high surface temperature (700-900°C) from silane, ammonia or dichlorosilane in the existence of hydrogen at low pressure. Atmospheric pressure CVD of silicon nitride films from tetrakis(dimethylamido)silicon and ammonia produce at 600-750°C but the here the plasma enhanced chemical vapor deposition(PECVD) produce the silicon nitrided layer at 250-400°C.By the comparison of both the processes the PECVD technique would be used at lower temperature.

S.R Collins, P. Williams and S.V Marx (2014) Low temperature carburization has limited temperature range from 350-550°C at that temperature the process become very slow and less economically feasible. Carburization is performed at high temperature to maximize the solubility and the rate of diffusion. On cooling room temperature a major part of solute atom will participate as carbide phase but at temperature grater then 950°C Participation of carbide can be avoided by high cooling rate. At intense temperatures solutes chromium and nickel diffused more slowly in austenitic stainless steel than carbon.


Surface characterization of Silicon Nitride compound deposited on Plasma Nitrided austenitic steel grade 316
Selection of surface hardening technique
Selection of work piece (stainless steel 316)
Plasma Nitriding on stainless steel 316
Silicon nitride compound deposited by PECVD
Study of experimental attributes
XRD
SEM
Result and discussion
Conclusion

References

[1] Gh. Barati Darband, M. Aliofkhazraei, Plasma electrolytic oxidation of Magnesium and its Alloy material (2005).

[2] J. L. Andu´ jar, E. Bertran, and M. C. Polo, Plasma-enhanced chemical Vapor deposition of boron nitride thin films from B2H6–H2–NH3 and B2H6–N2 gas mixtures (1997).

[3] Ahmad Reza Rastkar, Sepehrdad Akbari Laser and Plasma Research Institute, Shahid Beheshti University, Tehran 1983969411, Iran (2014)

[4] Raden Soekrisno, Suyitno, Rini Dharmastiti and Agus Suprihanto, Mechanical and Industrial Engineering Department of Gadjah Mada University, Indonesia (2015).

[5] Hassan R S Mahmoud, Patthi bin Hussain and Mokhtar C. Ismail, Mechanical Engineering Department, Universiti Teknologi Petronas Bandar
Seri Iskandar, Perak, Malaysia (2016).

[6] David M. Hoffmana and Sri Prakash Rangarajan, Satish D. Athavale and Demetre J. Economoua Department of Chemistry, Jia-Rui Liu, Zongshuang
Zheng, and Wei-KanChu Texas Center for Superconductivity, University of Houston, Houston, Texas (1995).

[7] S.A. Kusmanov, Yu.V. Kusmanova, A.A. Smirnov, P.N. Belkin
Nekrasov Kostroma State University, 1 May, 14, Kostroma 156961,Russia(2016).

[8] S.R. Collins, P.C. Williams, and S.V. Marx, Swagelok Company, A. Heuer, F. Ernst, and H. Kahn, Case Western Reserve University(2014).

[9] E.J. Mittemeijer, Max Planck Institute for Intelligent Systems (formerly Max Planck Institute for Metals Research) and Institute for Materials Science, University of Stuttgart (2013).

[10] L. Martinu, O. Zabeida and J.E. KlembergSapieha,Department of Engineering Physics, ´Ecole Polytechnique de Montr´eal, CP 6079, Statio CentreVille,Montr´eal, Qu´ebec H3C 3A7, Canada.

[11] C.X. Li, T. Bell School of Metallurgy and Materials, The University of Birmingham, Birmingham B15 2TT, UK (2003).

[12] Y. Li, L. Wang , J. Xu, D. Zhang,Department of Materials Science and Engineering, Dalian Maritime University, Dalian 116026, PR China(2006).

[13] S. Thaiwatthana, X. Y. Li, H. Dong, and T. Bell (2002).

[14] Giangiacomo Minak, DIEM Mechanical Engineering Department,Alma mater Studiorum – Università di Bolognz Viale Risorgimento 2, 40136 Bologna, Italia.

[15] Zhao Cheng(a),, C.X. Li(b), H. Dong(b), T. Bell(b), (a)Plasma Engineering Laboratory, Qingdao University of Science and Technology, Qingdao 266042, PR China, (b)Department of Metallurgy and Materials, University of Birmingham, Birmingham B15 2TT, UK.

[16] Dr. Kaustubh Kulkarni, Department of Materials Science and Engineering Indian Institute of Technology Kanpur, U.P. (2015).

[17] P.Elango, PG Student, Department of mechanical Engineering, Bharath University, Chennai –India,(2014).

[18] Dimitar Krastev, Improvement of Corrosion Resistance of Steels by Surface Modification University of Chemical Technology and Metallurgy, Bulgaria.

[19] Austenitic Stainless Steels, www.asminternational.org.

[20] C.X. Li, T. Bell,School of Metallurgy and Materials, The University of Birmingham, Birmingham B15 2TT, UK (2003).