# HARDNESS, TENSILE AND WEAR BEHAVIOUR OF A NONCONVENTIONAL AUSTENITIC STAINLESS STEEL UPON SENSITIZATION

A thesis in partial fulfillment of requirements for the award of the degree

#### **BACHELOR OF TECHNOLOGY**

**Submitted To** 

#### NATIONAL INSTITUTE OF TECHNOLOGY, ROURKELA

BY

**SUMAN KUMAR** 

110MM0359

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110MM0369



### DEPARTMENT OF METALLURGICAL AND MATERIALS ENGINEERING

#### NATIONAL INSTITUTE OF TECHNOLOGY

ROURKELA -769008

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### **CERTIFICATE**

This is to certify that the thesis entitled "Hardness, tensile And Wear Behaviour of A Nonconventional Austenitic Stainless Steel Upon Sensitization" submitted by **Suman Kumar (Roll No-110MM0359) and Ashadeep Pani** (**110MM0369**), Department of Metallurgical And Materials Engineering, Rourkela, as a partial fulfillment of requirements for the award of the Degree of Bachelor Of Technology has been carried out under my supervision.

**Prof. Krishna Dutta** 

Department of Metallurgical and Materials Engineering,

NIT Rourkela.

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#### ABSTRACT

The objective of this dissertation is to study the effect of sensitization on the mechanical properties such as hardness and tensile strength of a non-conventional austenitic stainless steel with special emphasis on wear properties. A set of samples has been solution annealed by soaking the steel at 1050°C followed by water quenching. On the other hand, a total of four sets of samples of the steel have been given sensitization treatment by holding at 750°C temperature for different soaking time periods ranging from 1 to 7 hours followed by water quenching. The microstructures of both the solution annealed as well as sensitized samples have been observed by optical microscope. The mechanical testing such as microhardness and macrohardness as well as tensile testing of each specimen has been performed. The wear behaviour of the non-conventional stainless steel is determined by using the ball on plate wear testing machine, with varying loads and sliding distances. It is observed that the height loss due to wear increases with increase in sensitization time, applied load and sliding distance. The hardness and yield strength of the investigated stainless steel sharply decreases with increase in sensitization time, whereas the tensile strength of this steel decreases marginally with sensitization time. It is also observed that the ductility values of the specimens decrease with increase in sensitization time. The supporting reason of these results have been discussed in terms of the depletion of solid solution strengtheners due to diffusion of these elements from the austenite matrix to the grain boundary, the increase carbide and nitride precipitates at the grain boundary with increasing sensitization time of the investigated steel sample. Marginal reduction in tensile strength may be attributed to possible transformation of austenite to strain induced martensite. The investigation of the worn out surface of the non-conventional stainless steel with field emission scanning electron microscope (FESEM) shows that the wear mechanism can be classified under abrasive wear.

#### Key Words: Non-conventional stainless steel, Sensitization, Microstructure, Hardness, Tensile strength, Wear.



# INTRODUCTION

#### **INTRODUCTION**

Stainless steel, especially, austenitic stainless steel, because of their high corrosion resistance and customizable mechanical properties has become an indispensable part of the regularly evolving modern day technology. Stainless steels of various grades find applications in numerous fields starting from the household to the nuclear reactors; from food and beverage cans to construction of different automobile parts. The formation of impervious oxide layer on the surface makes it suitable for use in adverse environments such as sea water.

The high temperature application of Austenitic Stainless Steel is somewhat limited because at higher temperatures it undergoes a phenomenon called **Sensitization**. According to Ghosh et al. [1], it refers to the precipitation of carbides and nitrides at the grain boundaries. Precipitation of Chromium rich carbides ( $Cr_{23}C_6$ ) and nitrides at the grain boundaries result when the Austenitic stainless steel is heated and held in the temperature range of  $500-850^{\circ}C$  (773K-1123K). This precipitation of carbides taking place at the grain boundary is because of their insolubility at these temperature ranges. This leads to Chromium depreciated regions around the grain boundaries. So the change in microstructure takes place and the regions with low Chromium contents become susceptible to Intergranular Corrosion (IGC) and Intergranular Stress Corrosion Cracking [1, 2]. Along with carbides and nitrides there is formation of *chi phase*. The chi phase, which is a stable intermetallic compound, consists of Fe, Cr, and Mo of type  $M_{18}C$ . Some studies reveal that sensitization may lead to formation of Martensite. In addition to the altered microstructure, mechanical properties of the Austenitic Stainless steel also become affected.

Sensitization of austenitic stainless steels leads to the diminution of mechanical properties which may lead to different problems during operation of the machine or component. The situation becomes critical when there is use of valves, pumps, conveyer belts, bearings etc., because along with sensitization, wear also plays an important role in

degrading the properties of the steel. Wear may be defined as an unintentional deterioration of the metal surface. The material from the surface is removed gradually because of relative motion between two surfaces in contact, usually in presence of load, [3]. In most of the cases, one of the two surfaces is stationary.

It has been established that sensitization has a huge effect on the mechanical properties of austenitic stainless steel. But there are very few works dedicated to the study of effect of sensitization on the wear behavior. So, this paper focuses on study of effect of sensitization on wear properties of austenitic stainless steel. Shankar et al. [5], in his journal, delineated the effect of sensitization on the tensile properties of stainless steel of particular grade 316LN. He reported that, sensitization has a trivial role on the tensile properties but reduces the ductility notably. According to the author, the interaction of the precipitated phases with the dislocations and built up stresses and grain boundary accounted for the reduction in ductility.

The class of ferrous alloy steels having 16-25% Chromium and sufficient amount of Austenite-stablising elements like Ni, Mn, and N, so that the steels are Austenitic at room temperature, are categorized as Austenitic Stainless Steels. Different grades of Austenitic Stainless Steels have been developed for various applications. High Mo stainless steels are used for brackish water cooling heat exchangers/condenser applications for power stations. Different kinds of non-conventional steels have been developed for specific use. One example is **ISO/TR 15510 X12CrMnNiN17-7-5**. This specific kind of non-conventional stainless steel is manufactured by using N in place of Ni and it finds application in the field of construction of automobile parts, construction of structural members. So for these applications, wear can be considered as an important parameter. So emphasis must be given on the behavior of this kind of nonconventional stainless steel in presence of wear. Very less research works have been dedicated to the effect of sensitization on the wear properties of nonconventional stainless steels. So the present research work encompasses the effect of sensitization on the microstructural, wear and mechanical properties of this particular grade of nonconventional stainless steel.

#### **1.1 OBJECTIVES**

The major objectives and relevant work plans of this investigation can be briefly summarized as:

# (i)To impose the sensitization of the selected non-conventional X12CrMnNiN17-7-5 stainless steel:

This part of the investigation comprises following :( a) Solution annealing of the selected steel at a temperature of  $1050^{\circ}$ C for 1 hour, followed by water quenching. (b)Cutting of total 15 samples from the solution-annealed blanks.(c) Sensitization treatment of 12 samples at a temperature of 1023K for soaking periods of 1,3,5 and 7 hours duration followed by water quenching (at least 3 samples for each soaking period).

# (ii) To study the microstructures and to determine related mechanical properties of the selected X12CrMnNiN17-7-5 stainless steel:

This part consists of (a) Microstructural characterization and (b) Determination of hardness and tensile properties.

# (iii) To study the Wear behaviour of the non-conventional X12CrMnNiN17-7-5 sensitized stainless steel:

This part of the investigation encompasses the followings: (a) Determination of wear depth of sensitized stainless steel with varying sliding distances i.e., 2 mm, 4 mm and 6 mm at constant load.(b)Determination of wear depth of sensitized stainless steel with varying load i.e., 10 N and 30 N at constant sliding distance.

# **CHAPTER 2**

# LITERATURE REVIEW

#### **2.1** Stainless Steel

Stainless steels are a large group of special alloys steel containing a minimum of about 10.5% chromium. The chromium forms a protective oxide film, which is the reason why this group of steels has its characteristic 'stainlessness' or corrosion resistance. Other desirable features may include excellent formability, high room-temperature and cryogenic toughness, and good resistance to scaling, oxidation, and creep at elevated temperatures [3]. Chromium is the alloying element which imparts corrosion resistance to stainless steels, but many other elements may be added to stabilize other phases, provided added corrosion resistance, or produce enhanced mechanical properties.

The passive layer formed on the surface of the stainless steel exhibits a unique property. : When the layer gets damaged, it repairs itself by the rapid reaction of chromium from the steel with oxygen and moisture from the environment to reform the oxide layer. The increase in Chromium content beyond 10.5% results in still higher corrosion resistance.. Corrosion resistance as well as other properties of the stainless steel can be improved with addition of 8% or more Nickel. Addition of Molybdenum to the stainless steel increases the corrosion resistance, while the addition of Nitrogen results in higher mechanical strength and resistance to pitting corrosion.

Stainless steels, which exhibit face centered cubic (FCC) lattice structure of austenite over the whole temperature range from room temperature to the melting point are categorized as Austenitic Stainless Steels. Basically, this class of steel is used in reheaters and superheaters. The three grades, 304, 321, and 347, are suitable for high temperature boiler applications while for primary heat transport piping systems of the advanced heavy water reactors, AISI 304LN grade render very useful. Nonconventional austenitic stainless steel of composition ISO/TR 15510 X12CrMnNiN17-7-5 was developed to conserve Nickel and it finds application in automobile parts such as

automotive trim, automobile wheel covers. This can be also used in the construction of structural members and in architecture (Windows, Panels etc.).

#### **2.2** Classification of stainless steel:

The stainless steel can be divided into five groups:

(1) Austenitic stainless steel

(2) Ferritic stainless steel

(3) Martensitic stainless steel

(4) Precipitation-Hardened Stainless steel

(5) Duplex stainless steel

#### (1)Austenitic stainless steel:

This group of stainless steel comprises of at least 16% Chromium and 6% Nickel and expands to the high alloy or "super austenitic" category such as 904L and 6% molybdenum grades. The addition of alloying elements like Molybdenum, Titanium, and Copper enhance some of the properties of this steel and make them useful for high temperature application. Because of the Nickel content in the Austenitic stainless steels, they are suitable to be used in cryogenic applications [4]. By making the steel austenitic by adding nickel avoids the problems of brittleness at low temperatures, which is a problem with other types of steel.

#### (2) Ferritic stainless steel:

This group of stainless steels is straight chromium steels in the 400 Series. These stainless steels are not hardenable by heat treatment and very less hardenable by cold working. These are magnetic in nature. Type 430 is typical of grade in this group of

stainless steel. Their moderate corrosion resistance and poor fabrication properties are improved by addition of more alloying element.

#### (3) Martensitic stainless steel:

Martensitic stainless steels are mainly based on the addition of chromium as the major alloying element but with a higher carbon and generally lower chromium content than the ferritic stainless steel type; Grade 431 of this group has a chromium content of about 16%, but the microstructure is still martensite despite this high chromium content. Because this grade of stainless steel also contains 2% nickel.

#### (4) Precipitation-Hardening stainless steel:

These kinds of stainless steels can develop very high values of tensile strength because of the presence of Nickel and Chromium. The grade "17-4 PH" is the most widely used grade of precipitation-hardening stainless steel. It is also known as grade 630. This sort of stainless steel comprises 17% Chromium, 4% Copper, 4% Nickel, and 0.3% Niobium (Nb). The property that gives this kind of steel edge over other stainless steels is that it can be supplied in the "Solution Annealed" condition. The advantage of this condition is that the steel is machinable in this condition and operations like mechanical forming is easy to carry out. Hardening of these kinds of stainless steels can be brought about by a single considerably low temperature ageing treatment.

#### (5) Duplex stainless steel:

Duplex stainless steels contain ferrite and austenite in their microstructure to combine toughness and weldability of austenite with strengths and corrosion resistance of ferrite. The exact composition is controlled by heat treatment. 2205 and 2507 are the most widely used grade of Duplex stainless steels. They exhibit a good resistance to stress corrosion cracking, but the value is lower than that of the ferritic stainless steels. They exhibit good corrosion resistance but less than that of the austenitic stainless steels. This steel has at least twice the strength of the annealed Austenitic stainless steels. The

corrosion resistance of the he duplex stainless steels is equal or greater than the value of the 304 and 316 stainless steels, but the pitting corrosion resistances are better than that of the 316 stainless steel. Duplex steels have freedom from transgranular stress corrosion cracking, as the ferrite phase is immune to this type of failure. The micro-duplex structure is destroyed in the HAZ, which decreases the strength as well as the corrosion resistance. They don't undergo Intergranular corrosion.

SL.	TYPES	%C	%Mn	%Si	%P	%S	%Cr	%Ni	%Mo	%N
NO										
1	Austenitic	0.03	2	4.5	0.03	0.03	11.25	2.5	0.5	0.08
		-	-		-	-	-	-	-	-
		0.25	19		0.17	0.35	23	38	3	0.4
2	Ferritic	0.08	1	1	0.04	0.03	11.5	0.5	0.6	0.025
		-	-		-	-	-		-	
		0.2	2.5		0.06	0.06	27		2.5	
3	Martensitic	0.15	1-2.5	0.5-	0.04	0.03	11.5	0.75	0.4	0.08
		-		1	-	-0.35	-	-	-	
		1.2			0.06		18	2.5	1	
4	Precipitation	0.05-	0.2-	0.1-	0.01	0.008	11	3.5	0.5	0.01
	Hardenable	0.15	1.25	1	-	-	-	-	-	-
					0.04	0.04	18	27	3.25	0.13
5	Duplex	0.02	1	0.75	0.03	0.01	21	2.5	1	0.01
		-	-	-	-	-	-	-	-	-
		0.04	2	1	0.04	0.03	26	6.5	4	0.13

Below table shows the classification of stainless steel based on the composition.

#### **2.3** Effect of alloying elements on stainless steel properties:

Stainless steels are a kind of alloy steels which consist of different alloys in different proportion to meet the desired specifications. Alloying affects the mechanical, physical, electrical, thermal properties of the stainless steel [13]. To improve one property of the stainless steel, a particular alloying element is added but it may or may not affect other properties. Effects of various alloying elements is delineated below .

#### (1) Carbon (C):

Steel is the alloy a ferrous alloy produced by the combination between iron and carbon. Carbon lowers the melting point of iron. As the carbon content in the steel increases, the melting point is lowered. Alloying with Carbon increases the hardness and strength of steel. Induction of hardening or strengthening of Pure iron is not possible by heat treatment whereas addition of carbon enables the manufacturer to produce steels with a wide variety of hardness and strength. Due to carbide formation during welding, low carbide content is desirable in austenitic and ferritic stainless steels. High carbon content in austenitic stainless steels also results in sensitization at high temperatures.

#### (2) Nickel (Ni):

Nickel is one of the important alloys that are added to the 300 grade steels. It is basically added to high Chromium content steels. The formation of an Austenitic structure due to presence of Ni results in increase in strength and toughness even at sub-zero temperatures. It results in no change in ductility. It doesn't affect the stability of passive layer directly but it increases the resistance of metal towards acid attack. It improves the cold formability at room temperature.

#### (3) Chromium :

Chromium is the element responsible for formation of the passive layer on the surface of stainless steel. As the Chromium content of steel exceeds 11%, the layer on top of the surface becomes stable and impervious [17]. It increases the high temperature strength and reduces the oxide formation tendency. It also helps in improving the wear resistance.

#### (4) Manganese (Mn):

Manganese increases the strength of the steel at high temperatures by preventing the chance of formation of FeS. It increases the shock resistance, toughness, hot formability. Manganese is an austenite stabiliser just like Nickel so sometimes it is used as a replacement of Nickel. It exhibits no change in ductility. Handsfield steel which contains about more than 10% Mn is used in railway construction, earth moving equipment.

#### (5) Nitrogen (N):

Nitrogen reduces the tendency to localized pitting attack and corrosion. It enhances the strain ageing process of steel. Thus the yield point becomes visible which has a positive impact on strength while a negative effect on ductility. It also helps in stabilising Austenite. As the presence of Carbon results in reduction of yield strength, addition of Nitrogen nullifies it.

#### (6) Titanium (Ti):

It is a stronger carbide former than Chromium. So it is used in stainless steels to prevent sensitization at high temperatures. When the stainless steel was melted in air, Ti was used to stabilize the carbon level. Now it has been replaced by Argon Oxygen Degasser. It puts a boundary on Austenitic grain size. It helps in prevention of Intergranular corrosion.

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#### (7) Copper (Cu):

The presence of Mo in steel requires addition of Cu to improve the corrosion resistance to acid attacks ( $H_2SO_4$ ). It also provides resistance against corrosion induced by the environment. Precipitation hardening is enhanced by addition of Cu. Despite of its good qualities, it is undesired in spring steels.

#### (8) Molybdenum (Mo):

The primary attributes of Molybdenum include increasing the high temperature properties of steel. The chance of temper embrittlement is significantly reduced by the presence of Molybdenum. It helps to overcome the deleterious effects of Chloride. It is useful in high speed steels.

#### (9) Zirconium (Zr):

Zirconium has stronger affinity to Carbon than Chromium so it helps in reducing the interaction between Chromium and Carbon. It also helps in increasing the corrosion resistance of steel. Below freezing temperatures, the strength can be significantly increased in presence of Zirconium. It helps in resisting fracture and limiting grain size.

#### (10) Niobium (Ni):

Niobium also plays a role similar to Zirconium and Titanium. It is a strong carbide former. It increases resistance towards corrosion. It helps in providing a microstructure with fine grains. It prevents the formation of  $Cr_{23}C_6$  so that the there is adequate amount of Chromium to form the passive layer. Fatigue and creep resistances are improved by the addition of Niobium, especially in ferritic stainless steels.

#### (11) Vanadium(V):

The use of Vanadium in steels increases the strength as well as the high temperature strength. Ductility reduction can be neutralized by promoting a fine grain size. It is used in high speed steels as it is a strong carbide former.

#### (12) Silicon (Si):

Silicon is added in small amounts to increase resistance towards acid attack. Silicon improves strength and increases the size of grains. This further leads to better magnetic properties. It improves the oxidation resistance. High Silicon content in Austenitic stainless steels prevent the formation of carbides at high temperatures

#### (13) **Oxygen(O):**

Presence of Oxygen in steels is completely restricted as it leads to formation of very brittle oxide at grain boundaries. Hence the deoxidation operation is carried out to remove the dissolved Oxygen with Al and Si.

#### (14) **Phosphorus** (**P**):

This is an undesired impurity. Effect of Phosphorus on hot cracking is quite deleterious. Presence of Phosphorus may lead to cold shortness or blue brittleness at low temperature.

#### (15) Hydrogen (H):

Hydrogen diffuses along the grain boundaries as it is a very small element. The formation of molecular hydrogen from atomic hydrogen at the grain boundaries creates pressure. Combined with external load, it may lead to cleavage fracture, which is popularly known as Hydrogen Embrittlement. Presence f  $H_2$  is detrimental.

#### **2.4** Sensitization of stainless steel:

The high corrosion resistance of stainless steels is attributed to the low conductivity and to the formation of a bilayer structure which maintains the alloy in a metastable state of passivity. The inner layer of the bilayer consists of Fe, Chromium Oxides with an enriched amount of Cr while the outer layer mainly comprises of Chromium Hydroxide (Cr(OH)<sub>3</sub>). When the austenitic stainless steels are fallaciously heat treated in the temperature range of  $500^{0}$ C (823K) -  $850^{0}$ C (1123K), chromium in the grains diffuse to the grain boundary and combine with carbon at the grain boundaries to form chromium carbides (typically Cr<sub>23</sub>C<sub>6</sub>) [1, 3, 14, 17]. This causes in the depletion of chromium in the grains. This depletion of Cr content in the grains adversely affects the mechanical properties, corrosion resistance of the metal. This phenomenon is known as sensitization and leads to a decrease in the corrosion resistance of stainless steels, notably resistance to intergranular corrosion as the depleted regions become anodic in presence of electrolyte [6]. Further, this leads to disintegration of parts. So to prevent or nullify the effect of sensitization, many methods of de-sensitization or elimination of Cr<sub>23</sub>C<sub>6</sub> have been proposed. Some of those measures are described below.

- 1. Reduction in Carbon content so that there is insufficient amount of C to form carbide with. Examples of such steels 304L, 316L.
- 2. To dissolve the carbides formed during sensitization, heat the steel in a temperature range of  $950^{\circ}$ C-1100<sup>o</sup>C and cool rapidly.
- The content of carbide formers like Niobium and Titanium should be increased. These elements form carbides without affecting the Chromium content of grains.
- 4. Molybdenum content in steel should be increased so that the sensitization time would be increased.



Fig. 2.1: Precipitation of chromium carbide at grain boundary and chromium depleted region.

In the metallography sections, the chromium carbide precipitation is revealed by deep grain boundary attack by certain etching procedure such as the use of electrolyte oxalic acid etching .Generally the carbides are too fine to be resolved by the light microscope, but are indirectly revealed by deep etching of effected grain boundaries. In contrast ,grain boundary in metallographic specimens of austenitic stainless steel without chromium carbide precipitation are  $M_{23}C$  well defined and not deeply etched.

The carbide precipitates in sensitization phenomena have been identified as  $M_{23}C_6$ , where M denotes the metal atom content of the carbide, which may include iron and molybdenum as well as chromium. However, the high concentration of chromium in the  $M_{23}C_6$  particles locally lowers the chromium content of the austenitic to below the 12% required for stainless corrosion behaviour. The analytical transmission electron microscopy study shows that , in austenite adjacent to  $M_{23}C_6$  particles at twin and high angle grain boundaries are preferred sites for precipitation and diffusion because of the relatively high atomic disorder where grains of different orientation meets. Thus  $M_{23}C_6$  particles readily nucleate and grow, severely depleting the adjacent austenite of chromium. Twin boundaries have much better atomic matching than most high angle boundaries [5] and therefore are not as favourable for nucleation and growth of  $M_{23}C_6$  particles.

The catastrophic consequences of intergranular corrosion due to chromium carbide precipitation has led to a number of heat treatment and alloying approaches to minimize or eliminate this problem. One approach is simply to select an extra low carbon modification of austenitic stainless steel .These modifications are designated as types 304L and 316L and have an upper limit of 0.03% carbon. Although chromium carbide formation may not be completely suppressed ,it is greatly reduced , and the low carbon grades are adequate for many application.

The kinetics show "C" curve behaviour with most rapid precipitation occurring between 800 and  $900^{\circ}$ C .Above  $950^{\circ}$ C ,chromium and carbon are dissolved as atoms in

the crystal structure of austenite and there is no thermodynamic driving force for chromium carbide formation. Below  $500^{0}$ C, the diffusion of chromium atoms required for  $M_{23}C_{6}$  formation is too sluggish and carbide formation essentially stops. Based on the  $M_{23}C_{6}$  precipitation kinetics, wrought austenitic stainless steel products are annealed or solution treated at temperature between 1040 and  $1150^{0}$ C and quenched to eliminate sensitization. The solution treatment dissolves the  $M_{23}C_{6}$  carbides , and the rapid cooling prevents the precipitation of such carbides in the critical temperature range around the nose of the C- curve.

Another approach used to eliminate chromium carbide precipitation is to alloy austenitic stainless steel with very strong carbide-forming elements such as titanium, niobium, or tantalum. Such austenitic stainless steels are referred to as stabilized grades. The alloying addition form carbides such as TiC and NbC and reduce the carbon available for  $M_{23}C_6$  precipitation. Stabilizing heat treatments, performed at temperatures between 840 and 900<sup>o</sup>C, are designed to produce the most effective interagranular dispersion of the alloy carbides [13]. Under most conditions stabilized austenitic stainless steels are effective in reducing chromium carbide formation and intergranular attack. However, the very high temperatures adjacent to welds may cause even TiC and NbC carbides to redissolve and make possible the precipitation of  $M_{23}C_6$  if the weldments are held in or slowly cooled through the  $M_{23}C_6$  precipitation temperature range. This may lead to the localized corrosion.

The plot shown below depicts the variation of sensitization time with temperature for various Carbon contents. The effect of Carbon content on sensitization also shown by the figure and it acts as a tool for the operator to avoid sensitization during heat treatment [6]. It is apparent from the figure that for a steel, with 0.062 %C, to avoid sensitization it has to cool below  $595^{\circ}$ C within 5 minutes. But for austenitic grades, containing very low carbon, it could take about 20hrs to cool below  $480^{\circ}$ C without getting sensitized.



George Krauss [17] has mentioned that when the precipitates re analyzed using Transmission electron microscope (TEM) studies, it has been found that sensitization leads to formation of Chromium rich carbides, chi phase, chrome nitrides etc. 'Chi' phase is a stable intermetallic compound. Some studies reveal that there is stress induced transformation of Austenite to Martensite in specific kind of stainless steels.

### 2.5 Wear:

When two surfaces are in contact with each other and they are moving relative to each other under load then it leads to subsequent loss of material from the surface. This phenomenon is known as wear [26]. The relative motion may include:

- (a)Sliding motion
- (b)Rolling motion
- (c)Combination of both

Wear resistance is not an inherent property of a metal which may be considered by itself; rather wear is the resultant of the material itself; of the mating material, the environment, and the operation conditions producing the wear [28]. Wear resistance then is complicated by all these factors and cannot be determined apart from specific the specific condition of service.

Burwell proposed that the four basic types of wear are adhesive wear, abrasive wear, corrosive wear, and surface fatigue.

(a)Adhesive wear: Adhesive wear is best described by the weld theory as proposed by Bowden and Tabor and others. According to this view, friction is due primary to the cold welding of surfaces when they are brought together under load [31].

Subsequent motion results in the tearing of the metal rupture taking place either in the newly formed weld, in the asperity, or in the underlying metal, which ever presents the weakest bond.

(b) Abrasive wear: Abrasive wear results from the ploughing of asperities and the cutting action of either entrapped or free-rolling grit particles between the surfaces. In most conventional engineering applications the design engineer can greatly overcome abrasive wear by the proper choice of materials, lubricants, and surface finish; and by providing for a clean environment. Hardness and smooth finish are the primary means of minimizing abrasive wear [29].

(c) Corrosive wear: Corrosive wear is characterized by interaction of the wear surface with a corrosive environment. It should be remembered that the nature of the oxide must be considered for each material. These products can act as a wear inhibitors in some applications and as primary causes of excessive wear in others. The integrity of the oxide layer is also a factor to be considered, as in the case of tin oxide. When present as an unbroken film, the oxide is protective and wear is low. However, once the oxide layer has been broken, the removed particles become abrasive and promote accelerated wear.

(d) Surface fatigue: Surface fatigue is most often encountered in ball and roller-type bearings, on gear teeth and cams, and other similar types of applications where the surface is subject to a great number of stress reversals at high unit stress [35]. Spalling, flaking, and eventual destruction of the wear surface are the results of surface fatigue.

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#### **Evaluation of Wear:**

Adhesive wear (amount of wear produced) is proportional to distance travelled (except as films may be worn through and thus change the nature of the actual contacting surface [30].

Adhesive wear is proportional to load (below some critical load and as long as the nature of the contacting surface is not changed by the load.)

➤ Abrasive wear is characterized by the ability of a particle to penetrate another, related to the hardness of the metal, and the ability of the particle to remove other particles and present a new wear surface.

Corrosive wear is complex and has not been adequately analyzed .However, it appears to be proportional to load, distance travelled, and to some function of frequency and the corrosivity of the environment [23].

Surface fatigue is a form of wear in which the number of cycles until break down of the surface is inversely proportional to the cube of the load.

➤ The amount of wear certainly depends upon the material in contact, but the rules for relating wear and material are still not firmly established [21].

Ghosh et al. [1] have examined the deterioration of fracture toughness of a 304LN stainless steel due to different periods of sensitization times. These authors have reported that fracture toughness sharply reduces with increased sensitization time. The steel also exhibits martensitic transformation (revealed by transmission electron microscopic studies) during sensitization, as well as the fracture toughness tests. Alvarez et al. [2] suggested that the complex microstructure formed during sensitization can increase the amount of the chrome carbide precipitate as the interface between the alpha and gamma phase acts as a potential site for nucleation. Shankar et al [5] has mentioned that the tensile properties remain unaffected by sensitization while the yield strength reduces significantly. Many of the authors like Hilders et al. [3] and Taveres et al. [4] have studied the effect of sensitization on the fracture toughness of the 304L austenitic

stainless steel. From the studies, they concluded that the fall in fracture toughness during sensitization is due to partial transformation of austenite to martensite.

A critical insight to the investigated reports indicates that effect of sensitization was mainly studied for understanding its effect on various mechanical properties of the respective steel samples. However, as per the knowledge of the current investigators, there is almost no or very few studies of the effect of sensitization on wear behaviour existing in the current literature. Therefore, in this investigation it has been aimed to study the effect of sensitization on the mechanical properties as well as on the wear behaviour of a nonconventional stainless steel.

# **CHAPTER 3**

# EXPERIMENTAL DETAILS

**3.1 Introduction** The objective of this investigation is to study the effect of sensitization on the microstructural variations and variations in hardness, tensile and wear properties of a nonconventional austenitic stainless steel. In order to accomplish these intended objectives, set of various experiments were carried out. These include heat treatment, microstructural characterization, determination of hardness of the virgin and the sensitized samples, determination of tensile properties, wear studies of the samples and scanning electron microscopic studies. A detailed description of various experiment those were carried out are presented in the following sections.

#### **3.2** Material Selection

X12CrMnNiN17-7-5 according to ISO/TR 15510:1997 [11], a special grade of non-conventional austenitic stainless steel was selected for this investigation. It is a commercial pure sample and was available in the form of rod with a diameter of around 14 mm.

#### **3.3** Chemical Composition

The chemical composition of the selected steel was determined at Tata Steel Ltd., Jamshedpur, India using an Optical Emission Spectrometer (Machine model and manufacturer: Thermo Electron Corporation Limited, Switzerland). The details of chemical composition are discussed in section 4.2 of Chapter 4.

#### **3.4** Heat Treatment

The chosen grade of non-conventional stainless steel was procured. As no pre-deformation history was available, so the sample was subjected to heat treatment using solution annealing to dissolve precipitated phases, if any. This treatment was done for 1 hour at  $1050^{\circ}$ C (1323K) followed by water quenching [1, 20]. Then the chosen sample was cut into several pieces so that different amount of sensitization can be introduced into different pieces. To induce sensitization, the samples were heated and soaked at  $750^{\circ}$ C for different time intervals such as 1 hour, 3 hours,5 hours and 7 hours

followed by water quenching. This type of heat treatment schedule was done following Ghosh et al. [1]. The detailed information on the heat treatment is given in the table below.

Serial No.	No. of Samples	Soaking	Soaking Time		
	Used	Temperature (K)	(Hours)		
1	3	1023	1		
2	3	1023	3		
3	3	1023	5		
4	3	1023	7		

Table 3.1 Heat treatments of samples for different soaking periods.

#### **3.5 Metallography**

The next step in the investigation was to determine the microstructure of the heattreated samples. To observe the microstructure of the samples, the samples were roughly polished on both the surfaces using the belt grinder. Water was used to keep the samples cool during the polishing. Then the samples were polished using a series of Emery papers 1/0, 2/0, 3/0, 4/0. The fineness of the papers increases with their grade.

After the polishing with the Emery papers is accomplished, we moved on to fine polishing which was done with the help of a wet rotating wheel covered with a special cloth that is filled with abrasive materials of certain size ranges. This polishing was executed to remove the nicks and inflections from the surface. Then the samples were polished with a diamond polisher up to 0.25um and then etched with freshly prepared aqua regia (3 parts of HCl mixed with 1 part of HNO<sub>3</sub>).

### **3.5.1 Optical Microscopy**

To reveal the microstructures of the prepared samples, an optical microscope (Model- Olympus BX61) was used. Samples were put under the lens of the microscope and it was well focused first by adjusting the position of sample. After that, the microstructures were captured at various magnifications.



**Fig3.1Optical Microscope** 

### 3.5.2 Grain Size Measurement:

To determine the size of the grains of the nonconventional stainless steel samples, liner intercept method was applied. A liner test grid was superposed over the microstructures of the samples obtained from the optical microscope [26].

In the microstructure, the number of grains cut by a line was counted. This was repeated for at least 15 lines on a typical microstructure magnified at  $200\mu m$ . The average grain size was then estimated by using the following formulae:

 $\mathbf{d} = \mathbf{L}_T / \mathbf{N}_L$  , where

 $N_{L}$  = No. of grains cut by a unit true test line length

 $L_T$  = Length of test line (True)

True length of test line means the length of the line at unit magnification

#### **3.6 Mechanical Testing:**

#### **3.6.1 Hardness Determination**

To obtain a detailed knowledge on the effect of Sensitization on the hardness of the samples, two kinds of hardness determination tests were performed.

- (a) Micro Hardness
- (b) Macro Hardness

### (a) Micro Hardness

In order to perform the micro hardness test on the samples, they were first roughly polished using belt grinder. Then they were polished with the Emery papers as described in previous descriptions so as to obtain flat surfaces and to avoid anomalies in the results.

The micro hardness data of the prepared samples were obtained by using a Vickers Micro hardness Tester (Model: Leco LV 700, USA). 5 readings were taken for each sample to calculate the average hardness. An indentation load of 5gf with dwell time of 15secs was used. After calculating the average hardness for each sample, mean, variance and standard deviation (S.D.) were calculated to check the consistency of the data.



Fig 3.2 Leco LV 700 Micro Hardness Tester

 $HV = 1.854P/D^2$ 

Where,

D=Average length of the diagonals.

P=Load Applied

 $D = (d_1 + d_2)/2$ 

 $D_1$  and  $d_2$  are the lengths of two indentation diagonals.

### (b) Macro Hardness

The macro hardness tests of the samples were performed using Macrovickers Hardness Tester at a load of 10kgf with a dwell time of 15 sec [10]. It has a square base diamond pyramid indenter. Five values were taken for each sample to calculate the average macro hardness. The hardness was expressed as hardness number. The vicker hardness number is given by the formula:

DPH=1.854P/L<sup>2</sup>

Where, P=Applied load

L=Average length of diagonals



Fig3.3 Vickers macrohardness tester

### **3.6.2Tensile Testing**

Tensile properties of the non-conventional stainless steel samples were determined by using servo hydraulic INSTRON 1195 testing machine, at a constant cross head speed of 1mm/min. To perform the test, samples were prepared from the initial heat treated rods of dimensions 120mm length and 14mm diameter [11]. The test was performed at room temperature  $(30^{\circ}C)$  on the fabricated samples. The gauge length and the diameter of the fabricated samples were 25mm and 6mm respectively. The specimens were polished to fine polishing in the lathe machine. For each test load, different displacement values are recorded and the data was stored in a computer for further processing.



#### Fig3.4 Schematic representation of sample for tensile test



Fig 3.5 Universal Testing Machine (UTM, BISS BANGALORE INDIA)

### 3.6.3 Wear Testing

Computerized ball on plate wear tester (TR-208-M1) employed for the wear testing of the nonconventional stainless steel samples. Cylindrical shaped samples of length around 6-8mm were cut form the initial heat treated rods and the two surfaces of the samples were polished with a belt grinder followed by a series of Emery Papers of grade 1/0, 2/0, 3/0, 4/0 [23].

The test was performed by varying the one of the 2 parameters specified below.

(i) Load (10N, 30N)

(ii) Sliding Distance (628mm, 1256mm, 1884mm)

For different combinations between these parameters, total 6 observations were noted for each sample which was stored in the computer for further processing.



Fig3.6 Ball on plate wear tester (TR-208-M1, DUCOM)

### **3.7 Factography:**

To study the nature of the worn out surfaces of the samples after the wear testing a higher magnifications, Scanning Electron Microscopy (SEM) was employed. The image of a SEM machine is shown in the next page.



Fig 3.7 Field emission scanning electron microscope



#### 

#### **4.1 Introduction**:

The aim of this study is to study the wear behaviour of a non-conventional stainless steel particularly under sensitized conditions. It was also intended to determine wear depth of these steels with varying load and varying sliding distance under different sensitized conditions. To fulfill these goals various experiments have been conducted, which are described in chapter 3. This chapter comprises obtained results of all those experiments conducted during this investigation . This chapter contains following sections: **Section 4.2** deals with chemical composition of the investigated austenitic stainless steel; microstructural analysis of the steel have been discussed in **Section 4.3**; results of conventional mechanical properties of the investigated steel have been given in **Section 4.4** with detail discussion, **Sections 4.5** to **4.6** contains results and discussion of Wear Test of non-conventional stainless steel, and fractographic features of the worn out samples after the wear testing of the specimens.

#### **4.2** Chemical Composition:

The below table 4.1 shows Chemical composition of this investigated special grade non-conventional stainless steel (X12CrMnNiN17-7-5), was investigated by OES. This grade of stainless steel is similar to X5CrNi17-7. This stainless steel has good mechanical properties and good corrosion resistance. The X5CrNi17-7 stainless steels are generally used for automotive parts such as automotive trim, automotive wheel covers and also used in flat conveyor chains, flatware, railroad passenger car bodies, structural members and architectural applications. The austenitic stainless steels possess major alloying elements as Cr and Ni approximately in the range of 16-25% and 8-20% respectively with low carbon content. This non-conventional stainless steel is having 0.14% carbon with common alloying elements of Ni and Cr as 3.66% and 15.6% respectively. This investigated steel also contains 5.49% Mn. Manganese is added to austenitic stainless steel in order to conserve Ni. As the chemical composition is different as compared to that in conventional 300 series austenitic stainless steel, this steel is

referred to as non-conventional austenitic stainless steel. The elements such as Ni, Mn and N are austenite stabilizers and hence this steel is austenitic at room temperature. The crystal structure of this steel is *FCC* throughout the temperature range.

Material	<b>Concentration of Elements (%)</b>				
	Fe	Cr	Mn		
	Balanced	15.6	5.49		
	С	Ni	Мо		
	0.14	3.96	0.2		
ISO/TR 15510 X12CrMnNiN17-7-5	Mn	Р	S		
	5.49	0.042	0.016		
	Cu	Al	N		
	1.05	0.03	0.135		
	Ti	V			
	0.02	0.06			

 Table 4.1 Composition of the investigated nonconventional stainless steel

#### **4.3** Microstructural Analysis:

The microstructure of the non-conventional austenitic stainless steel in the solution-annealed condition is shown in figure 4.1. The microstructure shows nearly equiaxed polygonal grains with annealing twins. Optical microstructures of the non-conventional stainless steels under different sensitization conditions (1 hour, 3 hour, 5hour, and 7 hour of sensitization) were observed at various magnifications. Typical micrographs of the investigated stainless steel under different sensitized conditions are shown in figure 4.2.



The microstructure of the sensitized non-conventional austenitic stainless steel was revealed etching the surface using Aqua regia. The microstructures indicate that the volume fraction of the sensitized grain boundaries increases with an increasing time of sensitization, as it is obvious from this figure 4.2.



Figure 4.2 Typical Microstructures of stainless steel under different sensitized condition (a) 1HR (b) 3HRS (c) 5HRS (d) 7HRS

#### 4.3.1 Grain size measurement:

Grain size measurement was done by linear intercept method without taking into account annealing twins as boundaries according to ASTM standard. Average grain size of the specimen was found to be 34  $\mu$ m. A superimpose liner test grid micrograph of the solution annealed non-conventional stainless steel is shown in the fig4.3.



Fig4.3 A typical superimposed lineal test grid microstructure of solution annealed specimen

#### **4.4** Conventional Mechanical Properties:

The examined conventional mechanical properties of the investigated stainless steel encompass hardness and tensile properties. Further the hardness testing includes:

(a) Microhardness tests and (b) Macrohardness test. The hardness tests of the specimens were carried out using Vickers hardness tester. Tensile testing was done by servo-hydraulic universal testing machine (Model: UTM, BISS).

#### 4.4.1 Hardness Testing:

#### (a) Microhardness Testing:

The microhardness of the non-conventional stainless steel was observed at different positions of the specimens, under both solution annealed and sensitized conditions. At least five readings were taken for each sample to obtain average value of the hardness.

Dwell time for microhardness test is 15 sec, with applied load of 50gf. As per literature, austenitic stainless steels possess hardness  $\approx 200$  VHN. The result indicates that the average hardness of the solution annealed sample is 200.48 HV. From Table 4.2 it is clear that hardness of the investigated stainless steel falls under the hardness range of austenitic stainless steel. The result of all the samples is listed in Table 4.2.

		18									- 82					
	1st reading		2n	id read	ing	3r	d read	ing	4t	h readi	ing	5t	h readi	ng		
Туре	D1, µm	D2, µm	HV	D1, µm	D2, µm	HV	D1, µm	D2, µm	HV	D1, µm	D2, µm	HV	D1, µm	D2, µm	HV	Avg.
A*	21.24	21.64	201	21.28	21.88	294	21.49	21.39	227	21.55	21.41	258	21.39	21.79	198	200
<b>B</b> *	18.11	17.39	294	18.14	17.22	296	18.22	17.29	294	18.02	17.78	289	18.31	17.3 <mark>5</mark>	<mark>2</mark> 91	293
<b>C</b> *	20.19	20.19	227	20.28	20.18	226	20.14	20.08	229	20.51	20.48	220	20.23	20.21	226	226
D*	19.39	18.48	258	19.27	19.23	250	19.21	19.18	251	19.34	19.27	248	19.38	19.32	247	251
E*	18.22	18.37	277	18.48	18.45	271	18.33	18.45	274	18.62	18.56	<mark>268</mark>	18.3	18.37	275	273

Table4.2 Microhardness values of non-conventional stainless steel in both solution annealed and sensitized condition.

\* A-Solution annealed, B-1 hr sensitization, C-3 hrs sensitization, D-5 hrs sensitization, E-7 hrs sensitization.

The variation of microhardness with sensitization time is shown in the Figure 4.4. The hardness of the non-conventional stainless steel in the solution annealed condition is 200HV. The hardness value of this steel increases up to 273HV.



As per literature review Ghosh et al.<sup>(1)</sup> has suggested that ,the hardness of stainless steel decreases as the time of sensitization increases. The reason behind it is explained as: the diffusion of Cr, C, N, and Ni from the grain interior to the grain boundary. This leads to the depletion of the solid solution strengtheners (C, Cr, N, Ni etc.) from the matrix, which results in softening of the matrix .Thus there is a decrease in the hardness. It can be seen from Figure 4.4 that, the curve does not follow any particular trend. This is due to the fact that microhardness is being taken over very small area, such that a region near the grain boundary can show higher hardness. To avoid ambiguity, macrohardness studies have done and the results are discussed in the following section.

#### (b)Macrohardness Testing:

The macrohardness of the non-conventional stainless steel was observed at different positions of the specimens, under both solutions annealed and sensitized conditions. At least five readings were taken for each sample to obtain average value of the hardness.

The load applied in macrohardness test was of 10Kg. The results for macrohardness values of investigated stainless steel under both solution annealed and sensitized condition are shown in table 4.2. The results indicates the same trend for solution annealed sample as that was in the case of microhardness testing. The mean value of the

solution annealed sample is 206HV, which falls under the hardness range of austenitic stainless steel.

•

Sample	Load(in Kg)	Hardness(HV)	Mean(HV)	Standard
				deviation
Solution	10	206	206	5.45
annealing		213		
		199		
		204		
		209		
1 Hour	10	189	189	4.43
sensitization		182		
		191		
		194		
		190		
3 Hours	10	186	184	4.13
sensitization		181		
		183		
		191		
		180		
5 Hours	10	186	181	3.12
sensitization		179		
		181		
		182		
		178		
7 Hours	10	183	180	4.27
sensitization		186		
		175		
		178		
		180		

Table4.3 Macrohardness values of non-conventional stainless steel in both sensitized and solution annealed conditions

The variation of macrohardness with sensitization time is shown in the Figure 4.5. The hardness of the non-conventional stainless steel in the solution annealed condition is 206HV. The hardness value of this steel decreases up to 180HV with increasing sensitization time.



Here the trend of the macrohardness vs. sensitization time graph is same as it was expected. The hardness of the steel decreases as the sensitization time increases. The obvious reason is the diffusion of Cr, C, N etc. from the grain matrix to the grain boundary. This leads to the depletion of the solid solution strengtheners(C, Cr, N, Ni etc.) from the matrix, which results in softening of the matrix .Thus, there is a decrease in the hardness.

#### **4.4.2** Tensile Testing:

Tensile testing of the non-conventional stainless steel was done in both solutions annealed and sensitized conditions. From the tensile testing, load and elongation data were obtained from the universal tensile testing machine (UTM BISS) for all the specimens. The corresponding engineering stress- strain values were calculated from the load, elongation data. The graphs between the stress and strain were drawn for all specimens. The below figures show, the stress-strain curve for different specimen of the non-conventional stainless steel.



Fig4.6 Engineering stress-strain curve of the investigated stainless steel in solution annealing and sensitization condition;(a) Solution annealing,(b) 1 hour sensitization,(c) 3 hour sensitization.

From the stress-strain curve various tensile parameters such as Yield strength, Ultimate tensile strength, Total elongation, Uniform elongation, Percentage area reduction, Elastic modulus etc. were calculated for each specimen of the nonconventional stainless steel. The various tensile parameters of each specimen are given in the table4.4 below.

Material s	Gage length (mm)	Area (mm <sup>2</sup> )	Yield strength (MPa)	Tensile strength (MPa)	%Are a reduct ion	% Total elongat ion	Elastic modulus (GPa)
Solution annealin g	25	29.228	615	1174	45.579	81.6	198
1 Hour sensitiza tion	25	29.132	596	999	31.271	62.19	210
3 Hour sensitiza tion	25	29.132	587	994	28.765	41.6	371
5 Hour sensitiza tion	25	29.613	413	982	19.213	31.3	229
7 Hour sensitiza tion	25	29.513	375	868	21.372	29.4	287

 Table4.4 Tensile properties of non-conventional stainless steel under solution annealed and sensitization conditions.

### 4.4.2.1

#### Effect of sensitization on tensile properties of the non-conventional stainless steel

The yield strength, tensile strength, and ductility (% total elongation) of the tested specimens were calculated and subsequently plotted with respect to sensitization time as shown in the figure 4.7, figure 4.8, figure 4.9.











#### Fig.4.9 A plot between percentage total elongation and sensitization time.

It can be observed from the figure that there is decrease in yield strength, tensile strength, and in percentage total elongation from solution annealed to sensitized stainless steel. The reason of variation of mechanical properties with increasing sensitization time can be described as:

(1)The precipitation of carbide and nitrides at the grain boundary in sensitization phenomena in austenitic stainless steel results in considerable decrease in solid solution strengtheners such as Cr, C, and N from the grain matrix, which causes to decrease yield strength.

(2)The factors influencing yield strength and tensile strength in this case are same. Apart from this, two additional factors also control the magnitude of tensile strength. The possible factors can be summarized as:

(a) During tensile deformation between yield and uniform strain, there are dislocation multiplications.

(b) There is transformation of austenite to deformation- induced martensite in the austenitic stainless steel .There is enhancement of tensile strength due to these two factors and both factors tend to nullify the effect of the decrease in tensile strength caused by the diffusion of solid solution strengtheners from the austenite matrix to the grain boundary. Thus, there is marginal decrease in tensile strength of non-conventional stainless steel.

There is a significant decrease in ductility of non-conventional stainless steel with increasing time of sensitization. In tensile loading operation, the carbides and the nitrides precipitates act as a potential sites for debonding and void nucleation. With increasing time of sensitization, the increase in the precipitates amount results in an increased void fraction. Thus the ductility decreases.

#### 4.5 Wear Test:

The wear test of non-conventional stainless steel in both solution annealed and sensitized conditions were carried out with varying applied load, and sliding distance. The results were obtained from multiple tests, which were done by keeping one parameter out of two (Load, Sliding distance) constant against wear.

- (1) Load vs. Wear
- (2) Sliding distance vs. Wear

The results data from the above tests were noted down and various plots such as wear depth vs. sliding distance at constant loads, wear depth vs. loads at constant sliding distances for each tested samples of the non-conventional stainless steel were made. Apart from that various histograms of wear depth were also made with varying sliding distances at constant loads for each specimen of non-conventional stainless steel. These graphs, histograms, table are given below. The results of wear tests of all the samples of non-conventional stainless steel in both solution annealed and sensitized condition with varying loads (10N, 30N) and with varying sliding distance (628mm, 1256mm, and 1884mm) are given in table 4.5.The results show that height loss due to sensitization increases with increasing sensitization time in all condition of loads and sliding distance. The results also show that with increase of loads the height loss due to wear increases for all samples at a constant sliding distance. Further, the results say with increase in sliding distance for all samples at a constant load. There are various graphs have been drawn in the support of these results which are given below.

Material	Load (N)	Sliding D	istance (mi	n)	Wear	Depth (µ	um)
		SD1	SD2	SD3	WD1	WD2	WD3
	10	628	1256	1884	5.45	16.2	45.81
Solution	30	628	1256	1884	7.1	22.01	78.07
Annealed							
	10	628	1256	1884	11.31	20.69	60.98
1HR	30	628	1256	1884	23.02	29.44	79.13
Sensitized							
	10	628	1256	1884	16.22	33	81.21
3HRS	30	628	1256	1884	26.22	46.74	106.64
Sensitized							
	10	628	1256	1884	35.11	56.69	106.06
5HRS	30	628	1256	1884	64.11	<b>79.84</b>	120.39
Sensitized							
	10	628	1256	1884	61.81	79.84	119.61
7HRS	30	628	1256	1884	77.25	138.52	152.19
Sensitized							

 Table 4.5 Wear at different test parameters for all the samples



Fig4.10 Plot between wear depth vs. sliding distance at constant loads : (a) Load 10N, (b)Load 30N.

This figure 4.10 shows that as sliding distance increases the wear depth increases as the time of residence of load increases. This plot also says that as the sensitization time increases the wear depth increases as the curves are shifting upwards.







This figure 4.11 shows that as load increases ,wear depth also increases for all samples, this plot also suggest that as sensitization time increases then wear depth increases as the curves are shifting upwards.





Fig4.12 Histogram showing values of wear depth for each specimen with varying sliding distances (a) 628mm,(b) 1256mm,(c) 1884mm at load

The above histogram shows that as the sliding distance increases then wear depth increases at constant load 10N, it also show that as the sensitization time increases the wear depth increases.





Fig4.13: Histogram showing values of wear depth of each specimens with varying sliding distances (a) 628mm,(b) 1256 mm,(c) 1884mm at load 30N.

The histogram shows similar results as the sliding distance increases the wear depth increases at a constant load 30N.It also shows that as the sensitization time increases the wear depth increases.

#### 4.6 Analyses of worn out surface:

The worn out surfaces of samples in solution annealed as well as sensitized condition of non-conventional stainless steel were studied by field emission scanning

electron microscope. The various profiles of worn out surfaces were graved in the secondary electron mode of field emission scanning electron microscope. Typical illustrations of the micrographs at high and low magnification are given in the below figures.

The worn surfaces of the specimens were observed under FESEM to examine the wear mechanism. The low magnification FESEM micrographs show fine scoring marks as it is shown in figure 4.14 for solution annealing and sensitized samples. The scoring depth in case of 7 hour sensitized condition is more as compared to solution annealed condition. It indicates that wear is highest in 7 hour sensitized condition and lowest in case of solution annealed condition. The possible reason for presence of scoring marks may be due to abrasion by entrapped debris, hard asperities on the hardened steel counter face or due to work hardening.

The high magnification FESEM micrographs of the specimens are shown in the figure 4.15. The micrographs show the evidence of substantial plastic deformation and cracks. It can be seen from the figure that, there is some wear debris so the reason of removing materials from the place can be given as follows: it may happen that the hard precipitates or fractured pieces get mechanically dislodged during wear. During these process pinholes get formed which act as a potential site for nucleation and growth of cracks. These cracks grow and get interconnected, which causes a layer of metal removal from the surface.

# Worn surfaces at low magnification:



Fig 4.14The above figure shows image of FESEM of (a)7 hour sensitized samples with sliding distance 628 mm (b) 7 hour sensitized sample with sliding distance 1256 mm,(c) 7 hour sensitization with sliding distance 1884 mm, (d) solution annealing sample with sliding distance 628 mm,(e) solution annealing sample with sliding distance 1256 mm,(f) solution annealing sample with sliding distance 1884 mm at a magnification of 1000X.





Fig 4.15The above figure shows the image of FESEM of (a) 7 hour sensitized sample with sliding distance 628 mm,(b) 7 hour sensitized sample with sliding distance 1256 mm,(c) solution annealing sample with sliding distance 1884 mm,(d) solution annealing sample with sliding distance 628mm,(e) solution annealing sample with sliding distance 1884 mm , at magnification of 10,000X.

# CHAPTER 5

# CONCLUSION AND SCOPE FOR FUTURE RESEARCH

#### **5.** CONCLUSIONS AND SCOPE FOR FUTURE RESEARCH.

#### **5.1** Conclusions:

From the obtained results and the analyses related to wear behaviour and mechanical testing of ISO/TR15510 X12CrMnNiN17-7-5 stainless steel and its associated metallography following conclusions can be drawn:

(1) The microstructure of the non-conventional austenitic stainless in solution annealed condition contains equiaxed polygonal grains with annealing twins. On the other hand, the microstructures of sensitized non-conventional stainless steel showed increase in volume fraction of the sensitized grain boundaries with increase in sensitization time.

(2) The hardness value of the investigated steel decreases with increasing sensitization time.

(3) The yield strength of the investigated stainless steel decreases with increasing sensitization time. There is little decrease in tensile strength of the investigated stainless steel with increasing time of sensitization. The possible reason for decreasing ductility is precipitation of carbides and nitrides at the grain boundary during sensitization phenomena. The precipitation act as a site for nucleation of voids and due to increase in void fraction ,the ductility decreases, whereas, there is marginal decrease in yield strength and hardness due to the diffusion of solid solution strengtheners from the austenite matrix to the grain boundary.

(4) The ductility of the 7 hour sensitized non-conventional stainless steel decreases by approximately 29% from that of its solution annealed state.

(5) The height loss due to wear increases with increase in sensitization time at a constant load and sliding distance. The height loss due to wear increases with increase in load at a constant sliding distance. The height loss due to wear increases with increase in sliding distance at a constant load. (6) The investigation of the worn out surface of the non-conventional stainless steel after wear test by FESEM shows that the wear mechanism was abrasive wear.

#### 5.2 Scope for future research:

(1) In the present study, effect of sensitization on mechanical properties and wear behaviour of non-conventional austenitic stainless steel has been observed only up to the sensitization period of 7 hours. It is therefore recommended to carry out sensitization studies for more duration of time.

(2) Degree of sensitization is another important parameter, which can be obtained by double loop-electropotential reactivation test (DL-EPR Test), using corrosion setup. So, it could be taken up for potential direction of research.

(3)During deformation this kind of steel transforms to strain- induced martensite. Some TEM studies can reveal the extent of transformation.

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